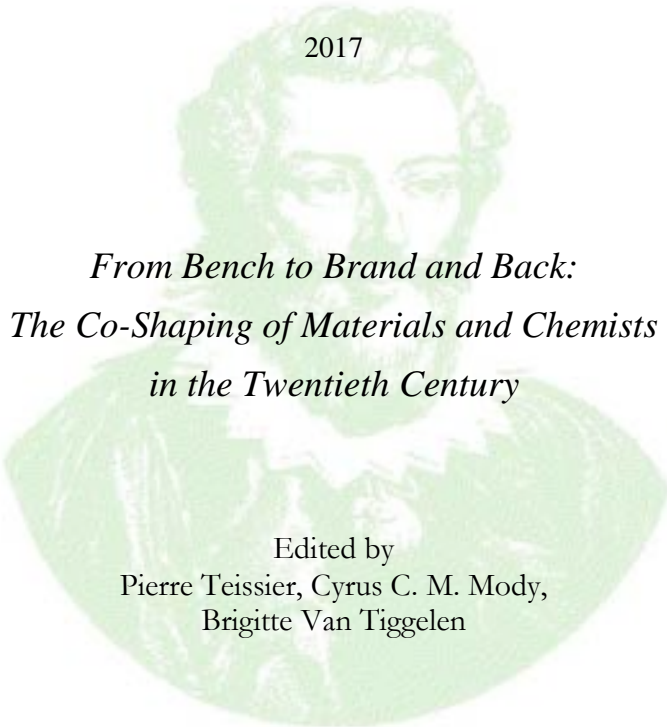


# CAHIERS FRANÇOIS VIÈTE

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*From Bench to Brand and Back:  
The Co-Shaping of Materials and Chemists  
in the Twentieth Century*

Edited by  
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Brigitte Van Tiggelen

Centre François Viète  
Épistémologie, histoire des sciences et des techniques  
Université de Nantes - Université de Bretagne Occidentale

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## Introduction

### Material Things, Scales and Trans-Operations

*Pierre Teissier, Cyrus C. M. Mody  
Brigitte Van Tiggelen*

#### Short Story of the Collective Project

Increasingly since the 19th century, chemists' dual role in society has been to enhance natural knowledge by making new forms of matter and to improve the human condition by making useful substances or materials. Chemists have thus become architects of both matter and society. At the same time, materials have shaped chemists and their science by stimulating the founding or reorganizing of disciplinary fields, epistemic communities, instrumental toolkits, cognitive representations and experimental practices. We can therefore speak of a co-construction of the subject and the object of chemistry. New materials, and their chemist-advocates, help initiate new behaviors in society, such as the past century-plus reconfiguration of consumption habits around the ever-growing number of synthetic materials used in commercial brands. In addition, new materials and social configurations orient chemists to pursue some research questions and neglect others.

We had these ideas in mind in Spring 2012 when we planned the organization of an international meeting on this theme. Entitled "Materials and Chemistry from Bench to Brand and Back", the symposium took place the 26th of July 2013 during the 24th International Congress of the History of Science, Technology and Medicine (ICHSTM) in Manchester. It was organized by Brigitte Van Tiggelen and Pierre Teissier, under the auspices of the Commission on the History of Modern Chemistry. It was partitioned in four sessions with eight speakers, including Cyrus Mody, and four commentators and gathered an average audience of thirty scholars per session for an entire day. A second symposium on the same theme took place one month later at Uppsala. Entitled "Materials in the 20th and 21st Century", it was part of the 9th International Congress for the History of Chemistry, on 24th of August 2013, and featured four speakers and two commentators.

The first symposium raised the interest of the London based publisher Pickering & Chatto for a collective book for the "History and Philosophy of Technoscience" series edited by Alfred Nordmann. The theme of

Nordmann's series appealed to enough of the contributors to the two symposia that we started to work on a collective book dealing with the co-construction of chemists and materials in the 20th century. Unfortunately, the acquisition of Pickering & Chatto by Routledge (Taylor and Francis Group) in March 2015 significantly slowed down our editorial process, leading us to switch from a private to a public press, the *Cahiers François Viète*, an academic publisher from the (public) University of Nantes. This option had the advantages of being reliable, free and open access while keeping high academic standards through a review process including two referees for each chapter. Along the way, these circumstances and reorganizations co-shaped the volume and its object, as much as the rearrangements in the list of contributors.

The collective book gathers eight case studies related to the long 20th century and to the interaction between materials and people. The contributors work in six different countries (Belgium, France, Germany, The Netherlands, Switzerland, and United Kingdom). The cases are grounded in a variety of regions (France, Germany, United Kingdom, United States, Western world) and methodological perspectives (chemistry, history, literature, museum studies, philosophy). In addition to the more traditional sources of historians, including institutional archives and scientific articles, other kinds of documents have also been used: ads and illustrations (§1), artifacts (§2), oral archives (§5, 7), popular literature (§6). The contributions furthermore cover a wide spectrum of materials: inorganic, organic, biologic, arts materials.

### **Historiographic Position in the “Thing Turn”**

The collective book instantiates the recent focus on material culture in academic research in general and in the history and philosophy of science in particular. In the last decades of the 20th century, Science and Technology Studies (STS) emphasized the co-construction of science and society. Since the turn of the century, though, a new trend has developed which focuses on the role of instruments, materials, and objects (Rheinberger, 1997; Baird, 2004; Daston, 2004). Chemistry and materials science represent fruitful ground for both the earlier and the newer directions of investigation – and for reflection on how the co-construction and materiality perspectives relate to each other. On the one hand, chemistry and materials science allow one to trace the changing relationships among bench scientists, production engineers, inventors, and markets. On the other hand, chemistry and materials science are inherently techno-scientific disciplines situated between knowing and making. Thus, these disciplines offer an

original perspective from which to explore the material culture of the “thing turn”. Our volume brings the synthetic sciences – fields that both make *and* understand stuff – to the fore in both history of science and technology. The focus on materials allows our contributors to investigate the intermingling of facts and artifacts, knowledge and know-how, cognition and application. It also, following recent contributions (Bensaude Vincent *et al.*, 2017), further erodes the still-sharp distinctions between history of science and history of technology.

To address these topics, we have chosen to focus on the long 20th century. This has to be justified. The first reason is institutional and pertains to the history of science, since the two 2013 symposiums were organized under the auspices of the Commission on the History of Modern Chemistry, which fosters a particular emphasis on 20th and 21st century chemistry. The second reason is historiographical and more related to the history of technology. Our chapters examine the period bridging the “second” and “third industrial revolutions” (Caron, 1997). The “second industrial revolution”, running from the 1870s to the 1920s, is commonly associated with the industrialization of electricity and chemistry in Europe and America based on the formalization of research and development (R&D), the building of electrical networks, and the invention of means for “scaling-up” chemical reactions. The “third industrial revolution” (Dosi & Galambos, 2013) is a fuzzier concept, but roughly it refers to late 20th century developments linked to the progressive integration of African, Asian and Oceanian actors into post-1980 neo-liberal globalization. With respect to the history of science and technology, the period between the second and third industrial revolutions was characterized by the presence of the “welfare state” and the “cold war”. The perspective of “*temps long*” (long term) history, unfolding over around a century and a half, allows us to stress the continuity of phenomena and to soften the importance of ruptures. Indeed, most of our case studies overlap at least one of the two revolutions mentioned above without reifying ruptures between them. On the contrary, the long 20th century exhibits coherent features that weave in and out of most of the case studies: the consumer society; the developmental state; ideological confrontation between East and West; economic and military confrontation between North and South; the instrumentation revolution in chemistry; the capillarity of economic discourse spreading to all corners of society, including science; etc.

In spite of our strongly empirical perspective on history of science and technology, we would like to contribute to two STS debates. The first one deals with the changing organization of science and technology in society, related to the concept of “regimes of production of knowledge” (Pe-

stre, 2003a). This debate centers on whether the entanglement of science and technology is a recent (post-1980) phenomenon or has roots going back at least to the “second industrial revolution”. A simple and much-cited framework adopted by Michael Gibbons *et al.* (1994) roughly discriminates so-called “mode 1”, or traditional disciplinary sciences, from “mode 2”, or modern trans-disciplinary ones. A number of strong critiques of this framework have been made, however, which offer more thorough interpretations of developments over the long term. For example, Dominique Peestre (2003b) argued for a long-lasting evolution since the 15th century in Europe. However, like Gibbons *et al.* (1994), he agreed that the 1970s mark a neo-liberal rupture in twentieth century science and technology. Other models have also appeared, such as the “triple helix of university-industry-government relations” (Etzkowitz & Leydesdorff, 1996) or the post-1980 “epochal break” (Nordmann *et al.*, 2011; also Forman, 2007).

We did not want to choose among the existing models but we acknowledge the fact that each highlights a certain facet of the problem. None of them, however, is able to capture the complex entirety of the co-shaping of chemists and materials. Some of our case studies might provide empirical data to facilitate the refinement of sociological models that explain late 20th-century transformations in science and technology. Instead of endorsing a model, we adopt the transversal conception of science offered by Terry Shinn and Pascal Ragouet (2005), which stresses that the research process is shaped not only by scientists but also by social and cultural features, including material and instrumental opportunities and constraints (Mody, 2011). Indeed, even though each of our cases examines a very localized and finite object of investigation (a material), all of the contributions do this in a historically sensitive way, bringing in the context of time and space, both local and global, and expanding the theoretical framework through comparisons.

The second debate is that concerning objectivity. Daston and Galison’s (2007) groundbreaking work on *Objectivity* showed that scientific identity is co-produced with communally shared norms for robust knowledge production. Yet their equally influential claim that the making of technoscientific objects represents a new form of objectivity is more questionable. In contrast, our chapters demonstrate that in chemistry and materials science technoscientific objects have underwritten objectivity for well over a century. We follow here the literature on “techno-sciences”, after Gilbert Hottois (1984), which emphasizes the close connection between science and technology since, at least, the “second industrial revolution”.



## From Bench to Brand and Back: Scaling and Trans-Operating

This collective book sketches the mirror dynamics between chemists and materials across a wide spectrum of interconnected fields and activities ranging from bench research through engineering processes and brand consumers to human cultures and the natural environment. It mainly focuses on the circulation and interaction of people, things, and words. The endless back and forth between bench substances and brand products exhibits two transversal concepts that permeate most of our case studies.

First, the importance of *scaling* in grasping the interaction between chemists and materials. By scaling, we mean movement both up and down along both natural and cultural scales, as well as the dynamic interactions between those scales. Chemists, more than most scientists, are often looking to scale up, to amplify what they do in the laboratory in order to build the factory and influence the mass-market. It is striking, when reading the eight following chapters, to realize the great diversity of the institutions involved in chemistry and materials science in terms of their sizes, organizational models, and goals: start-up companies, laboratories, universities, communities, trade unions, multinational firms, states, international markets, global networks, etc. Yet chemists are also just as often employed to scale down by grasping a bit of the world to isolate it and study it out of its normal context or to manipulate it and combine it in the mixed entities known as materials. They thus build an astonishing variety of heterogeneities and combinations, at scales ranging from the (sub)atomic to the macroscopic. The circular dynamic of scaling up and down becomes even more complex and stimulating when new materials enter the natural environment, posing unexpected challenges for regulation, clean-up, and recycling. We thus consider scaling as a process and scales as contingent and evolving things rather than essential and static objects.

The second transversal feature of our collective volume is situated at the conjunction of the transgressive character of chemistry and the operative dimension of techno-science – a conjunction we label *trans-operating*. Chemistry is transgressive in that it blurs traditional dichotomies between natural and artificial, making and knowing, realism and positivism (Bensaude Vincent, 2005; Llored, 2013). Like other techno-sciences, it is also able to operate on its surroundings. Chemists' hemi-synthesis of molecules from natural products, for example, is one of the characteristic practices of the artificialization of nature that we wish to highlight. A *trans-operating process* or *trans-operation* can thus be defined as a performative interaction between two entities usually considered to belong to separate spheres (nature versus culture, science versus technology, infrastructure versus superstructure, etc.). The circulation of materials from bench to brand and back

in the eight chapters makes apparent three types of trans-operation: between things and people (part 1); between knowing and making (part 2); and between things and words (part 3). Our concept of “trans-operation” thus provides a theoretical frame to organize the different empirical cases.

## **Editorial Organization of the Volume**

The first part of the volume “The Plasticity of Things and People” is composed of two chapters which tackle the relation between science and design. In chapter 1, “Paint as a Material: The Transformation of Paint Chemistry and Technology in America (ca. 1880-1920)”, Augustin Cerveaux recounts the emergence of modern paint chemistry and technology in the United States at the turn of the 20th century. He shows how legislative regulations and chemists’ professional struggle for jurisdictions (Abbott, 1988) turned paint chemistry from a decorative art and craft to a techno-scientific field based on performance, while paint coats evolved from mere mixtures to brand materials. Chapter 2, “Quality Matters for Historical Plastics: The Past-Making of Cellulose Nitrates for Future Preservation” by Anita Quye, takes the practical problem of material degradation of cellulose plastics in contemporary museums as an opportunity to explore the plasticity of values according to places, times and communities. Thus, one material can lose its aesthetic value for heritage while acquiring both a bench value for conservation scientists in the future and an historical value for historians of science trying to understand the past.

The second part, entitled “Knowing by Making and Making by Knowing” shows how the interaction between material and conceptual aspects of materials fosters a feedback between the creation of materials and the creation of economic value in the market, or the creation of knowledge and techniques. In chapter 3, “Twentieth Century Fertilizers in France from Natural Mixing to Artificial Making (1890-1970)”, Philippe Martin analyzes how the interplay of chemical and agronomic knowledge and know-how and consumption practices drove the gradual transformation of the French fertilizer industry over the course of eight decades. Martin investigates the trans-operations between the structure and composition of materials and the conceptions of rationality and modernity offered by industrialists and administrators who wanted to build faith in artificial materials. Jumping from industrial problems to academic communities, in chapter 4, Apostolos Gerontas considers “Chromatographs as Epistemic Things: Communities around the Extraction of Material Knowledge” during the 1960s and 1970s. By examining the production and dissemination of automated apparatus, Gerontas highlights the consequences that chromatographic technology

had for knowledge production in chemistry. New instruments turned the “separation” of molecules into a menial job, forcing a reorganization of analytic chemistry’s division of labor. Similarly, chapter 5, “The Exotic Glasses of Rennes (France): Local Knowledge-Making in Global Telecommunication”, by Pierre Teissier, shows how postwar research on materials was organized by a transatlantic division of labor, with new materials coming out of Europe and new physical phenomena manifested in those materials discovered in the United States. In Teissier’s case study, the accidental production of “exotic glasses” in Rennes was shaped both by the bench culture of solid-state chemistry and by the telecommunications industry’s support for international R&D.

The third, and last, part of the volume, entitled “Innovating and Recycling: Telling the Stories of Materials,” exhibits the interplay between new stories and old materials, or between old stories and new materials. In chapter 6, “Making Sense of Chemistry: Synthetic Rubber in German Popular Scientific Literature (1929-2009)”, Jens Soentgen analyzes a large set of German popular books to link changing representations of natural and synthetic rubber to changing political contexts. Rubber chemists were alternatively the heroes of industry, autarky, the working class, and the “apolitical” market from the Weimar Republic to the 21st century Federal Republic of Germany (BRD). With chapter 7, “Point and Line to Plan: The Ontography of Carbon Nanomaterials”, Sacha Loeve draws a parallel between the modes of existence of three emblematic nano-materials (fullerenes, nanotubes, and graphene) and the three geometrical figures conceptualized by Vassily Kandinsky (point, line, plan). He shows how, from bench to brand, these materials are continually born anew in the space of indefinite technological possibilities saturated by promises of radical novelty: the “nanoworld”. Finally, chapter 8, “The Diverse Ecology of Electronic Materials”, by Cyrus Mody, investigates alternative histories of microelectronics by following two material alternatives to silicon that did not migrate from bench to brand nor from brand to bench: superconducting materials and fullerenes. This allows a better understanding of the evolving organization of the semiconductor (silicon) industry and, more generally, of changes in the relationship between industry and academia.

## Concluding Remarks

Such an editorial project inevitably yields unexpected features which emerge from the collective efforts of the authors. We have identified at least four themes and questions which recur in stimulating if unanticipated ways across a number of contributions. The first is related to the study of

materials themselves and the making of materials researchers during the long 20th century. Most of the chapters develop the idea that materials are characterized by multiple features beyond their mere physical and chemical properties. Their forms are specifically investigated by chemists for applications (§1) and packaging (§3), in relation to their transformations over time, for worse (§2) or better (§7), their accidental morphology which can be selected (§5) and amplified, or even their systemic integration as devices (§8). All these forms are then brought within the one true dogma of materials scientists since the 1960s: the relationship between composition or structure and performance (§1, 2, 5). This dogma is not new, of course: metallurgists and chemists have formalized it for, among other things, the steels used for building railroads in the “second industrial revolution” (Misa, 1995; Chezeau, 2004). Yet as our chapters show, this dogma has been progressively formalized and expanded over the long twentieth century.

The second recurring theme of this volume stresses the importance of contingency in the historical process. Many of our chapters show that “it could have been otherwise” (§1, 3), “it was otherwise” (§5) or “it was told otherwise” (§6, 7, 8). However, in spite of a deep consciousness of alternative paths, several of our chapters also present linear narratives which convey the impression of a gradual determined evolution: for example, the drift toward a global neo-liberal order in the final third of the 20th century (§5, 8). Such a tension between determinism and contingency poses complications for sociological and economic models, which tend to favor the mechanical dynamics of social groups and markets. Yet the same tension also undermines the consensus in science and technology studies, which declares its faith in contingency and non-linear narratives.

Thus, tensions constitute the third recurring theme of the volume. Such dichotomies can be identified with respect to practices, such as the opposition between wet and dry syntheses (§8), as well as for moral discourses such as the good/evil dualism (§6). One crucial tension operates at a symbolic level between what is usual and what is new. Indeed, in any given chapter (§1, 3, 6), both the novelty of leading-edge research and the repetition of customs can play a role. More deeply, this tension underlies a second tension between tradition and modernity that runs all through the long 20th century. It would be worth studying the evolutions of the meaning of each end of these oppositions over time.

The fourth and last recurring theme deals with the generation of identity among chemists and their many stakeholders. Our authors treat identity as the upshot of a process involving both self and others, in which materiality and technology are implicated. This leads to the main theme of the volume: the shaping of beings confers identities upon things, and the

shaping of things confers identities upon beings (§1, 3, 4, 5, 6 are especially clear in this regard). This permanent, ongoing, mutual shaping of material substances and human societies also travels across all types of discourses on materials and people: commercial ads (§1), collective memory and myths (§2, 5, 7), political economy (§3), discipline-building (§4, 5), literature and propaganda (§6), and historical narratives (§6, 7, 8). Here again, mechanisms are complex. Disciplinary organization (Stichweh, 1994), boundary work (Gieryn, 1999), and commemorative practices (Abir-Am & Elliot, 1999) are central to the shaping of scientific identities. But many other dynamics are involved too (Teissier, 2014): things (devices, materials, brands), bench practices (concepts, instruments, know-how), professional organizations (companies, disciplines, networks, trade unions), collective memory and myths (monuments, narratives, testimonies). Chemists' identities are also built on a series of hierarchical differentiations whether between chemists and others (§1, 3, 5, 8) or among chemists themselves (§2, 4, 6). The latter often, again, reinforces binary dualism: dirty/pure (§1), wet/dry (§8), descriptive/predictive (§1, 3, 5), dull/exciting (§5, 7), self/other (§4), and so on.

Taken together, these four recurring themes offer a concise summary of our point. Materials, in both their form and function, are co-emergent with institutions, communities, organizations, networks, discourses, cultural hierarchies, and all the other ingredients of modern societies. Materials are also co-emergent with the individuals who populate those societies. In other words, the foundational 20th-century sociological debate over the primacy of structure or agency was always missing at least one other active pole: the materials which constrain and enable both social structure and individual agency. Crucially, the powers of materials are neither deterministic nor entirely contingent. Rather, materials mediate the entanglement of social structure and individual agency not just locally in any single interaction, but also through their never-ending circulation from bench to brand and back.

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## **Part I**

*The Plasticity of Things and People*



## Paint as a Material: The Transformation of Paint Chemistry and Technology in America (ca. 1880-1920)

Augustin Cerveaux\*

### Abstract

*This chapter recounts and analyzes the emergence of modern paint chemistry and technology in the United States. Until late in the 19th century, painting was above all a decorative art and craft, and chemists' role in the paint trade was largely circumscribed to the development of new pigments. At the turn of the 20th century, however, the protective dimension of paints rose in prominence and the standing and influence of chemists within the trade and industry rose tremendously. Charles Dudley, a chemist at the Pennsylvania Railroad Company, initiated this movement. A new field quickly coalesced around the American Society for Testing Materials, the Paint Manufacturers Association, and later the American Chemical Society. In the process, the paint coat became firmly established as a material in itself, rather than a mere mixture of heterogeneous ingredients. The erstwhile conflation of "pure" paint with "good" paint became suddenly obsolete.*

*Keywords: paint chemistry and industry, purity and modernity, Charles B. Dudley (1842-1909), second industrial revolution, American science.*

### Résumé

*Ce chapitre retrace et analyse l'émergence de la chimie et de la technologie moderne des peintures aux États-Unis. La peinture, jusque vers la fin du XIX<sup>e</sup> siècle, consistait en un artisanat dont la vocation était essentiellement décorative, et le rôle des chimistes consistait principalement à découvrir et exploiter de nouveaux pigments. Cependant, au tournant du XX<sup>e</sup> siècle, la dimension protectrice des peintures devient prépondérante, et les chimistes et ingénieurs acquièrent une importante position et influence dans le commerce et l'industrie des peintures. Un chimiste de la compagnie ferroviaire Pennsylvania Railroad, Charles Dudley, a initié ce mouvement, qui s'est ensuite développé au sein de la Société Américaine des Tests de Matériaux, de l'Association des Fabricants de Peinture, et plus tard de la Société Américaine de Chimie. Au cours de cette transformation, le revêtement de peinture devient appréhendé comme un matériau en soi, plutôt que comme une simple mixture d'ingrédients hétérogènes. La tradition artisanale identifiant la « pureté » des peintures avec leur performance est brusquement remise en cause et dépassée.*

*Mots-clés : chimie et industrie des peintures, pureté et modernité, Charles B. Dudley (1842-1909), seconde révolution industrielle, science américaine.*

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THE DOMAIN of paints offers a fascinating research avenue to explore and analyze the co-shaping of chemists and materials. Cars, planes, trains, buildings, bridges, and many household appliances such as ovens, refrigerators and lamps are coated by paints. Each one of them has been formulated by paint chemists, tested and gauged in-doors with a variety of laboratory apparatus, and tested outdoors on exposure panels and in field service. In turn, as I show in this chapter, paints have compelled chemists to leave their benches and venture outside to design and implement outdoor testing methods. What makes a paint durable? Why does a formulation perform well on wood, and terribly on metal? What does it even mean for a paint to “dry”? And above all, how should tests and procedures leading to reliable and reproducible experimental data about paints be designed? These were among the most vexing questions that chemists had in mind when they started to erect exposure panel tests throughout America at the turn of the 20th century. By exploring these questions, these chemists contributed to shape the field of materials science and technology in the 20th century.

In this chapter, I recount and analyze the emergence of modern paint chemistry and technology in America, between ca. 1880 and 1920, when chemists and engineers, rather than painters, explored this set of questions. In the process, paint came to be viewed as a material in itself – a coating that could and should be engineered to fit a wide variety of specific purposes. Pre-industrial painting was mostly understood as a decorative art and craft. Yet the chemists and engineers who would shape modern paint technology were above all concerned with their protective properties. Paints’ function shifted from aesthetics to protection and durability. New forms of paints disseminated, the more conspicuous being the commercial availability of “ready-mixed” paints, effectively transforming paints into a commodity. Underpinning these changes lay a radical shift in representation, a disruption in the perceived relationship between materials and function. Traditional knowledge about paints drew a clear-cut line between color, brought about by pigments, and durability, resting on the quality of the oil which binds pigments together. “Oil is the life of the paint”, a saying among painters went. The new representation held instead that both pigments and the binding medium, interacting together to form a material, are responsible for color and durability.

Section 1 briefly touches on pre-industrial painting in Europe, to give a broader insight into the changes taking place at the turn of the 20th century. I show how painting was historically dedicated to beauty and ornament. Section 2 addresses the introduction of ready-mixed paints in America during the 1880s, and how it affected painters and favored the in-

roduction of chemists into the paint trade. Section 3 focuses on the American chemist Charles Dudley, employee of a railroad company, whose research program shattered traditional knowledge about paints. The ensuing organization of paint chemistry and technology – upheld by two entities in particular, the American Society for Testing Materials and the Paint Manufacturer Association – leant on and expanded Dudley’s program, and is the object of section 4. The field organized alongside a nationwide and bitter controversy over paint regulation and labeling enforcement, largely resulting from the dissemination of ready-mixed paints. In the last section I describe how the controversy accelerated the demise of the old, pre-industrial representation of paints.

### **A Long-Lasting Cultural Tradition of Painting**

Unlike other commodities or technologies that emerged entirely out of the industrial revolution – like, say, the railroads or electricity – painting was bounded by a longer tradition. In medieval Europe, guilds of professional painters were established as early as the 12th century. Since the advent of oil painting in the 14th century, linseed oil and lead-base pigments (lead white, lead red and litharge mostly) were the most important materials for painting. Virtually all preparations included lead. Lead white served as a base, and the desired tint was obtained by adding a small quantity of other “colors”, as pigments were called until late in the 19th century. Lead oxides were also added to the preparation, to increase the siccative power of the oil (reduce its drying time). As a professional guild regulated by the state, painters were frequently at odd with plasterers and shipbuilders: painters claimed a monopoly over the practice of oil painting, which plasterers often used for themselves in finishing their works. For instance, a dispute during the 1610s in London was settled by excluding the crucial lead whites from the materials plasterers were permitted to use (Englefield, 1923, p. 74-75).

Painters were primarily engaged for decorative works of various kinds – interior objects like cups and cans, as well as carriages and houses. It is telling that, in the mid-17th century, when lead pigments and linseed oil, praised by master painters, were used in shipbuilding to water-proof the hull, no painter affiliated to a guild was permitted to perform these jobs, which were reserved for carpenters and workers on shipyards (Englefield, 1923, p. 134-136; Armitage, 1954, p. 57-60). Yet most shipyards typically hosted painters for finishing works. Their trade was understood as distinct in nature from carpentry, masonry, and plastering. Painting was mostly a decorative art and craft, aligned with its etymological root – from the latin verb *pingere*, which means to impart color.

The identification of painting with color-bringing is reflected in the textual production of painters, chemists, and philosophers who wrote about the trade. There is no entry for “paint” in either Ephraim Chalmers’ *Cyclopedia*, nor in Jean D’Alembert and Denis Diderot’s *Encyclopédie*, although the entries for “painting” are quite substantial in both. “Paint”, as a noun, surfaces scantily in the early 19th century, as a synonym for pigment or “color”. Until the mid-19th century, “paint” and “painting” referred to a practice rather than a material thing. Aiming primarily at decoration and ornament, it was above all a cultural practice. This is how D’Alembert and Diderot (1765, p. 246) introduced “painting” in their *Encyclopédie*:

To impart colors on a flat surface, so as to represent any figure. Also designates the beautification of diverse ornaments in a bedroom, an office, a gallery. [...] To paint also refers – though improperly – to sizable works on buildings. One has to *paint* a panel, a cradle, or an iron balustrade to prevent their rusting. But, in that case, to *daub* would be more correct.

It’s not that protection was altogether absent from the motives of painters. Rather, “paint” and “painting” were not immediately associated with protection, unlike “varnish”. The function assigned to each of the two major classes of materials in the formulation of paints – vegetable oils and mineral pigments – was clear-cut: the pigments bring the color, and the oil the stability and durability of the whole.<sup>1</sup> Failure of paint-coats to retain their color or to stand the deleterious effects of weather was blamed on “adulterated” ingredients: the substitution of cheaper oil for linseed oil, or cheaper minerals like clay for lead white. A good paint was a “pure” paint,

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<sup>1</sup> John Smith’s *The Art of Painting* (1676), among the oldest painting manuals recorded, gives some indication of how to adjust formulas for outdoor works, exposed to intense weathering. Compared with indoor formulas, he recommends adding stronger solvents and more oil (chap. XVII). The close association of varnish with protection, and painting with ornament is particularly eloquent in *A Treatise of Japanning and Varnishing*, published in London in 1688. In the preface, the authors, John Stalker and George Parker, state that “Painting only is able to keep us in our Youth and perfection. That Magick Art, more powerful than Medæ’s charms, not only renews old age, but happily prevents grey hairs and wrinkles. [...] Well then, as Painting has made honourable provision for our Bodies, so Japanning has taught us a method, no way inferior to it, for the splendor and preservation of our Furniture and Houses. These Buildings, like our Bodies, continually tending to ruin and dissolution, are still in want of fresh supplies and reparations: on the one hand they are assaulted with unexpected mischances, on the other with the injuries of time and weather; but the Art of Japanning has made them almost impregnable against both: no damp air, no mouldring worm, or corroding time, can possibly deface it.”

free of materials coming from other professions, particularly the plasterers – lime, chalk, clay, barytes, gypsum...

Chemists interacted with the paint trade in various ways, the most known being the discovery of new pigments, or new synthetic routes for pigment compounding (Ball, 2003). From the late 18th century on, pushed by a burgeoning paint and varnish industry, chemists prepared general educational textbooks intended for painters and manufacturers. Textbooks usually presented a classification of pigments and associated production processes by color, and a classification of oils, gums and resins (Malepeyre, 1874). Professional chemists probably comprised some manufacturers' staff, to assess the purity of raw materials and more generally to rationalize the relationship between the pigments' production processes and the color obtained, so as to improve the yields and tints. Yet the penetration of chemistry into the paint trade from the late 18th century on was never as extensive as in the textile trade, where chemists' knowledge and practices became essential for the production and innovation of dyestuff (Nieto-Galan & Fox, 1999).

### **The Reconfiguration of Paint Practices by “Ready-Mix” Brand Materials**

- *The Increasing Demand of the Second Industrial Revolution*

In America, the advent of “ready-mixed” paints, also designated as “prepared” paints, was probably the most important contribution of the post-Civil War industrializing trend to the paint trade. Until then, color merchants and druggists sold oils and pigments separately, and the painter mixed them together, on site, to a desired consistency and color according to the type of work to be done and the personal taste of the painter's client. Ready-mixed paints, sold ready for use directly in a can, completely changed this trade regime and condensed the diverse materials and techniques required to prepare the paint into a single product controlled by manufacturers. Ready-mixed paints were bought and sold in retail, and thus opened the way to the commodification of paints and the standardization of their color. Painters, or the railway, building, and carriage-making companies that employed them, became “consumers” of paints, as well as anyone willing to paint a barn, house, or carriage himself.<sup>2</sup>

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<sup>2</sup> Paints thus played a key role in the advent of a “consumer” society at the turn of the 20th century. Charles F. McGovern (2006) analyzes the progressive inclusion of consumption within American values during this period.

The steadily increasing demand for paints certainly pushed for this substitution, as ready-mixed paints offered promising opportunities for value-adding and profits, efficiency, standardization, and labor saving in plants and paint shops, and thus curtailed costs. A rapidly industrializing America needed more and more paint to protect and beautify its houses, barns, ships, carriages, buildings, and railroads. In 1838, a 350 ton vessel required about nine short tons of paint and varnish, while a Navy ship upon entering service in World War I had on its flanks more than one hundred times as much (not including maintenance). In between, the annual domestic production of ships of any kind increased, in tonnage, about three hundred times to reach 3.3 million tons in the late 1910s.<sup>3</sup> One and a half million horse-drawn vehicles were produced in 1900, each requiring between six and thirteen paint coats (Kinney, 2004, p. 34). A similar trend could be highlighted for houses and railroad equipment, the latter demanding paints for rails, freight, and passenger cars. Paint and varnish production grew accordingly, from \$27 million in 1869 to 125 in 1909.<sup>4</sup> In 1890, ready-mixed paints accounted for about twenty-two percent of all the production of paint materials and products, in value, and were largely circumscribed to house-painting. By 1919 its share in the paint trade had reached forty-seven percent, and had penetrated the building, ship construction, automobile, and railroad markets.<sup>5</sup> The master painters, as a body, felt threatened by the introduction of ready-mixed paints. The departure from their traditional paint mixing practices represented, after all, a transfer of techniques and skills from the painter to the manufacturer, and more ominously threatened the very existence of the painting profession. Sometimes master painters went so far as to organize collective boycotts of the manufacturers that sold ready-mixed paints.<sup>6</sup>

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<sup>3</sup> The figure of 1838 comes from (Green, 1965, p. 35), that of 1916 from (Gardner, s.d., vol. 2) and the increase in ship tonnage from *Statistical Abstracts of the United States*, Washington: U.S. G.P.O., vol. 1 (1878) table 137 and vol. 43 (1920) table 267.

<sup>4</sup> Constant 1909 dollar. Source: U.S. Bureau of the Census, Thirteen Census of the United States (1909), Washington: U.S. G.P.O., 1913, Vol. X: Manufactures, p. 595 table 2.

<sup>5</sup> Figures from U.S. Bureau of the Census, Eleventh Census Report, 1890, vol. I Manufactures, Part III, p. 292 table 2 and Fourteenth Census Report.

<sup>6</sup> For instance in 1885-1886 in Philadelphia: *House Painting and Decorating*, vol. 1, n°4 (Jan. 1886), p. 121.

- “Ready-Mix” Paint as Deceptively Impure Commodity

The Harrison Brothers Company of Philadelphia, among the earliest manufacturers in America that ventured into ready-mixed paints, launched a trade journal in 1885 to smooth its relations with master painters. The journal, a monthly entitled *House Painting and Decorating*, featured ads of ready-mixed paints – mostly Harrison brands – and became the official organ of the Pennsylvania Master Painters Association. To ease the switch to prepared paints, Harrison Brothers launched an aggressive marketing campaign persuading painters that their products were as pure as the preparations they could make on their own. They invited any party to send a sample of a suspect preparation for analysis in their labs, and colorfully exposed the cases of adulteration in the journal. For example, in March 1886, a sample sent by a certain “S. & McL”. is decried in the following terms: “The result of a careful analysis shows that your coach black contains forty-five per cent of barytes. Can you hope to do a durable job with such trash? Give it up!”<sup>7</sup> Numerous cartoons were drawn and published highlighting the threat of adulteration, and its “subduing” by chemistry (figure 1).

A glance at other trade journals shows that purity was the most important advertising leitmotiv in the trade. In the late 1880s, as painters organized to deter adulteration, they also contracted with independent chemists and confronted the results with manufacturers’ claims.<sup>8</sup>

Chemists, then, interacted with the paint trade in various ways, but did not dispute the painters’ common knowledge drawing a sharp boundary between respectable and suspect materials. Rather, their expertise in quantitative analysis lent the detection and exposure of adulteration cases more authority. Thus, this new role for chemists in the paint trade espoused a clear division of labor and qualifications: the painter or paint manufacturer expected the chemist to sort out the nature and proportion of the ingredients entering his products, or his peers’ or competitors’, while the assessment of the overall quality of the paint remained his jurisdiction. In the context of the paint trade, the relationship between painters or paint manufacturers on the one hand, and chemists on the others hand, could then be interpreted as “consultant and testing slaves”, as proposed by historian James Donnelly (1994) in his study of the alkali industry.<sup>9</sup>

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<sup>7</sup> *House Painting and Decorating*, vol. 1, n°6 (March 1886), p. 187.

<sup>8</sup> *House Painting and Decorating*, vol. 5, n°6 (March 1890), p. 275-276.

<sup>9</sup> For example, a master painter praised the chemists’ analytical skill but dismissed his contribution beyond that: “The analyses of chrome yellow indicated clearly the importance of employing a chemist who has not a little experience in the manufacture of paints or, at least, knows something on the manufacture of



Figure 1 - “The demons of adulteration subdued by chemistry”, *House Painting and Decorating*, vol. 1, n°6 (March 1886). (Source: Photo taken by the author)

The comparison with another key material or range of materials of the “second industrial revolution”, concretes, and the associated trade and body of occupations and expertise, is particularly helpful to shed light on the historical development of the paint trade. Both concretes and paints underwent tremendous growth in production and consumption in the late 19th century. Unlike the paint trade however, chemists and engineers, not manufacturers or masons, were at the core of the body of expertise setting technical standards over concrete, assessing their overall quality and how it should be laid or applied (Slaton, 2001). Although both materials shared common substances and input, the difference in representation is striking: gypsum, for instance, was considered an essential and valuable ingredient in the concrete trade, whereas in the paint trade it was vilified as an “adulter-

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pigments in general. Unless he has such knowledge he is not competent to draw proper deductions from his analysis such as we think should be submitted to master painters.” (*House Painting and Decorating*, vol. 5, n°6 (March 1890), p. 276).



rant". In the former case, gypsum is a necessary component of an unavoidable material in modern building technology and civil engineering: concrete. In the later, gypsum is a cheap substitute debasing the purity, and therefore the quality, of a decorative preparation. The idea that gypsum is good for concretes but bad for paints testifies to a long tradition of painting which drew a sharp hierarchy between ingredients. In painting, just a handful of pigments were considered respectable materials; for concrete, anything could go, as long as performance followed.

The next section is devoted to Charles B. Dudley (1842-1909), a chemist at the Pennsylvania Railroad Company, who was the first to consistently and persistently challenge this representation in the paint trade, which posited an inherent hierarchy in painting materials. He was invited in 1890 and later in 1892 by the Pennsylvania Master Painters Association to lecture the painters on the composition and durability of various pigments. His underlying thesis – that adulterants were not necessarily detrimental to paints – would be bitterly resisted and the subject of nationwide legal battles before being fully accepted.<sup>10</sup>

### **A Functionalized Material: Charles B. Dudley and the Pennsylvania Railroad Co.**

During the early 1870s the railroad industry aimed at standardizing its mechanical parts and tests assessing the quality and durability of various procured materials, including iron and steel rails. The major companies thus fostered systematic mechanical investigation and testing facilities. The Pennsylvania Railroad Company, one of the largest American railroad companies, implemented a department of physical and chemical tests in 1875, on the premises of the blacksmith and mechanical shops located at Altoona, in central Pennsylvania. Chemical analyses were sometimes performed on lubricants, steel, and other materials by contracting chemists, and the department would internalize the analyses. Yet the management had no clear idea of the department's organization and outcomes, besides the assumption that in-house physical and chemical testing facilities might

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<sup>10</sup> Dudley is a minor figure in the historiography of science and technology, portrayed mostly as one the first leaders of industrial research. His impact on paint chemistry and technology, as well as on the historical development of materials science and technology, has been overlooked. At any rate, he deserves a more prominent place in the historiography. He was in his time a chemist of very high standing, with tremendous influence in both industrial and academic circles. He presided over the American Chemical Society in 1896 and 1897.

benefit the company. Dudley, a Ph.D. in chemistry freshly graduated from the Sheffield Scientific School at Yale, was hired to run the chemical part of the department. With no pre-established specific missions and duties, he was granted, as a managerial experiment, considerable latitude in the choice of his investigations and the organization of the laboratory (Usselman, 2002, p. 195-208; Ely, n.d., p. 51).

Why did some burning oils, used by coach drivers as signals and therefore essential for traffic safety, fail entirely in service? When paint on coaches was found badly damaged after cleaning service, who or what was to blame: the paint, the soap, or the cleaners? These were the kinds of issues Dudley initially tackled, which led him to detect “adulterated” burning oils and soaps and to devise tests preventing the purchase of adulterated goods. Interestingly, these early forays into adulterated goods did not condition his approach to the paint issue a few years later, since he came to reject the very notion of an adulterated paint. Rather, Dudley framed his investigations into paints, from the late 1880s on, on the basis of his findings and achievements on steel rails during the 1880s.

Steel rails, made commercially available after the invention of the Bessemer process in 1856, had replaced most iron rails by the late 1870s, on the basis of a better performance in service. However, there was no reliable physical or chemical test of steel from which to infer its actual performance and durability over the span of years or decades. Steel rails’ performance varied importantly from one manufacturer to the next, or even from one batch to the next (Chezeau, 2004). The procurement of steel was thus a source of major conflicts between railroad companies and steel manufacturers. Systematically correlating the observed performance and durability of various samples of rails with the chemical analysis of their constituent steel, Dudley found that the proportion of four elements in the composition of steel – phosphorous, silicon, carbon and manganese – could reliably predict the performance of the rail made thereof. On this basis, he promoted radical changes in procurement practices and specifications which, as one might expect, were met with considerable controversy and triggered heated debates, not least because steel manufacturers were reluctant to be told by steel consumers how to process their steel. Yet eventually Dudley’s philosophy of specifications took hold. By the late 1880s, the role of the laboratory was to a large extent defined by the design and enforcement of specifications (Usselman, 2002, p. 204-209; p. 217-223).

Around 1887 Dudley tackled what he called the “paint problem”.<sup>11</sup> Despite the vast quantities of paints consumed by the railroad industry, there was no reliable guideline securing the purchase of the best paint formulation for any specific application. The economic incentive to devote a large share of Dudley’s laboratory’s resources to paints, in a context of unreliable technological knowledge, was thus enormous. There was not even, in contrast to the steel rail problem, a clear and shared understanding of what “paint” referred to. Dudley felt compelled, at the outset of his studies, to state that paint “may be said to be any liquid or semi-liquid substance applied with a brush to protect or give color, gloss, or all three, to surfaces”. He added that “in this sense, both whitewash and varnish can be regarded as paints” (“Paints”, p. 414). While aligned with the modern definition of coating, this understanding departed radically from the historical conflation of paint with pigment, and reflected the consumer viewpoint of the “problem”. Rather than highlighting the process – the mixing of pigments with a liquid binder – the definition emphasized the function of paints. Dudley, as a railroad man, cared more about the durability of the paint-coat than about the proper color of the pigment used, or whether the substance applied was a paint or a varnish.

As a chemist, Dudley felt all the more puzzled since the relationship between composition and performance seemed even foggier than in the steel-rail case. Immersed in a large railroad network covering Eastern and Midwestern parts of the U.S. territory, Dudley had access to firsthand data about the service performance of numerous paints under a variety of climate and exposure conditions. He also appropriated and developed an experimental apparatus and technique at the core of the painters’ and manufacturers’ practices: the panel test. Painters usually applied their preparations on a wooden board to check the working and drying qualities of any specific preparation. The exposure panel was also a commercial artifact, shown to customers. Dudley had different expectations for the dozens of panels he erected in the vicinity of the laboratory. He had the latitude to devise and conduct experiments aimed at a systematic and general approach to the composition-performance conundrum. Assuming that water was the most significant factor in the degradation of paint-coats, he assessed the

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<sup>11</sup> The following presentation of Dudley’s researches on paints is based on his series of articles published in *The Railroad and Engineering Journal*, with his assistant F. N. Pease: “Paints”, vol. 64, n°9 (Sept. 1890), p. 414-417; “The Working Qualities of Paints”, n°10 (Oct. 1890), p. 452-455; “The Drying of Paint”, n°12 (Dec. 1890), p. 545-548; “The Covering Power of Pigments”, vol. 65, n°2 (Feb. 1891), p. 78-82; “How to Design a Paint”, n°4 (Apr. 1891), p. 174-177; “Paint Specification”, n°5 (May 1891), p. 162-167.

relative absorption of water by dried coats of various formulations – different proportions of “pure” pigments, adulterants such as barytes, and linseed oil. His conclusion was in direct opposition to the then-prevailing theory of pure paints and oil-induced durability of paints: that pigments, or supposedly detrimental mineral “adulterants”, mattered a lot to the durability of the paint coat.<sup>12</sup>

Equally important were his reflections and insights into the physical microstructure of paints. Although there is no evidence that Dudley engaged in microscopic studies of paint films, he identified core issues regarding the relationship between paint properties and physical microstructure – the fineness of pigments’ particles and distribution within the oil medium – on which paint technology would concentrate throughout the 20th century. Dudley was probably the first to expound the modern explanation of the opacity of paints, and highlighted the importance of the pigments’ particle size and refractive index in this concern (“The Covering Power of Pigments”, p. 80-81). His experimental studies and conceptual developments set the stage for the definition of the concepts of hiding power, tinting strength, and the importance of the physical structure of the pigments’ particles. As such, he can be regarded as one of the most important figures in the historical development of modern industrial painting.

### **Reforming the Paint Trade: The American Society for Testing Materials and the Paint Manufacturers Association**

Dudley’s most important legacy, though, is not his forays into paint technology, but the founding of the American Society for Testing Materials (ASTM). The success of Dudley’s approach to the conflicts between railroad companies and steel manufacturers over the durability of rails – bringing together consumers and manufacturers to agree on a set of specifications and tests that steel bars should meet – led to a generalization in the design and enforcement of specifications for other industries and materials.

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<sup>12</sup> “We have very little hesitation in saying, and we think all experiments honestly made under proper conditions will prove this point, namely, that it is essential for a good paint that the amount of pigment per square inch or square foot of surface be large. This may look like making the durability of the paint depend on the pigment, whereas the common idea is that the oil is the life of the paint. We are quite free to confess that in our experience we have not been able to confirm the common belief among paint manufacturers and, indeed, among many of the users, that the oil is the life of the paint. The pigment is the life of the paint according to our experience.” (“How to Design a Paint”, p. 175).

Dudley was the driving force behind the formal institution of ASTM in 1898, and pushed for the creation of a committee specifically dedicated to paints in 1902: the committee on “protective coatings for iron and steel”, shortened to committee E. It was chaired by an engineer from the federal government, and equally composed of chemists or engineers from railroad and construction companies, on the one hand, and manufacturers on the other. The committee quickly realized that the kind of specifications regulating the purchase of steel rails – like tensile strength tests and impurity levels – would be grossly inappropriate for paints. Instead of focusing single-handedly on the search for adequate specifications, the committee focused on a few seemingly simple questions or issues that vexed manufacturers and consumers of paints alike, and tried to standardize testing methods throughout its membership to gain robust and reproducible knowledge on these issues. Is a fast-drying paint good or bad for durability? Should metallic surfaces be carefully cleaned and sand-blasted before painting? How should the tests on exposure panels be prepared and conducted to yield reliable and reproducible data about a given paint formulation? These kinds of questions, if at all explored, were previously circumscribed within the occupational sphere of master painters. Chemists’ new inroads into the technological realm of painters entailed a radically enlarged scope of investigation: from an auxiliary analytical aide to an overwhelming agent of materials’ performance.

Above all, in the spirit of ASTM as envisioned by Dudley, the committee strove to regulate the paint trade so as to ensure a fair competition between manufacturers. The committee’s most important sub-committee was dedicated to “field tests”, meaning the assessment of paint performances in actual service. The sub-committee established restrictive guidelines over who would conduct the field tests and how the tests would be conducted. Worth mentioning is the fact that independent chemical analysis was mandatory – any manufacturer could not at the same time submit a sample for testing and provide the analysis stating its composition. Besides, the committee kept a sample of each tested formulation for future proofs. The kind of chemical analyses performed by Harrison Brothers as a marketing scheme of self-promotion was precisely what was being resisted. Gustave W. Thompson (1865-1942), chief chemist at the National Lead company and the sub-committee’s chair, summed it up this way: “The purpose is not to give any manufacturer any commercial preeminence. It may result, in inspection, in the discovery that certain paints have stood well in their respective treatment”.<sup>13</sup> The promotion of economic fairness and techno-

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<sup>13</sup> ASTM Proceedings, vol. VI (1906), p. 64.

logical efficiency through science and expertise was certainly a hallmark of the Progressive era. ASTM as a body, and most chemists and engineers trying to reform the paint trade, embodied what historian Samuel P. Hayes (1959) depicted as the “gospel of efficiency”. It is not surprising that Thompson later joined the Progressive party (Ingalls, 1930, p. 396), led by Theodore Roosevelt (1858-1919), one of the most influential figures of the Progressive movement.

Among the members of the committee was George B. Heckel (1858-1941), an influential member of the Paint Manufacturers Association, then a recently established national association for ready-mixed paints manufacturers headquartered in Philadelphia. One of the major forces driving the founding of the Paint Manufacturers Association in 1898 was the threat of seemingly imminent government intervention in the regulation of the paint trade. Painters and non-professional consumers protested against “adulterated” paints and several bills circulated to legally enforce, at the state level, paint labeling – the labeling of ingredients, both in composition and proportion. The prepared paints manufacturers felt threatened by such bills, as they were reluctant to disclose what they considered trade secrets, and anticipated the damaging consequences for sales that the listing of “adulterants” on paint labels would entail. Heckel (1931, p. 319-323) monitored the advancement of the bills and for a few years successfully prevented their enactment.<sup>14</sup>

In 1907 Heckel, together with Robert S. Perry, vice-president of Harrison Brothers, instituted a “Scientific Section” formally dependent upon the Paint Manufacturers Association and endowed with laboratory facilities on the premises of Harrison Brothers’ laboratory. The Scientific Section was staffed with about a dozen chemists and assistants (cf. table 1), and basically imported the methodology developed by ASTM for paint testing. The section focused initially on wood-painting – that is, tackled the issue of house-painting which was beyond ASTM’s scope. Exposure tests on wood panels were performed in Atlantic City, Pittsburgh, and Fargo (North Dakota), monitored by ASTM and local associations of master painters. Atlantic City was a favorite choice for early panel tests due to the harsh climatic conditions it offered, the proximity to Philadelphia, and because early ASTM gatherings took place in Atlantic City. Pittsburgh was selected because of the existing connection with the Carnegie Technical

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<sup>14</sup> The regulation of the economic and industrial “jungle”, as it was called by popular muckrakers, was certainly a prominent feature of the Progressive Era. Less known is the legacy of the Progressive Era for paint legislation – that, quite strikingly, either dismissed or altogether ignored the health hazards of lead-paints (Warren, 1999, p. 705-736).

School and the different climate it offered. The choice behind the selection of the Fargo site, detailed in the next section, is more sinuous and yet essential in understanding the rationale behind the panel tests campaign organized by the Paint Manufacturers Association. The campaign basically intended to smash the idea that “pure” paints performed better. Henry A. Gardner (1882-?), the director of the Scientific Section, released the results in bulletin formats in 1909, and published a synthesis in 1911 that concluded unambiguously: “Mixtures of white lead and zinc oxide properly blended with moderate percentages of reinforcing pigments, such as asbestine, barytes, silica and calcium carbonate have proved satisfactory from every standpoint and are superior to mixtures of prime white pigments not reinforced with inert pigments” (Gardner, 1911, p. 190).

Besides exposure panels, Gardner introduced in his 1911 manual a variety of new apparatuses and tests construing the physical and mechanical properties of paint films. He completely overlooked the analytical techniques that aimed to reveal the proportion and stoichiometric formulas of pigments, which until then composed the bulk of the scientific treatises on paints. “The writer’s desire”, as he put it, “being to treat the subject from the standpoint of the physical properties of painting materials” (Gardner, 1911, p. 70). Following the approach favored by ASTM, the Scientific Section departed from the chemical examination of materials to explore their physical aspects. Chemical formulas were deemed unreliable to predict the performance of paints in “field service”. How could paints of similar composition display such wide discrepancies in service performance? The alternative to composition as an explanatory and predictive factor of performance lay in the exploration of paints’ microstructure. Concomitant to the physical and mechanical study of paint films, the Scientific Section systematically examined dried and wet paint films with microscopes. Gardner’s manual is probably the first to introduce microphotographs of pigments dispersed in binding medium, together with a quantitative measure of their size and morphology. The microscope provided a new method for pigment identification beyond the traditional analytical techniques. It was on this basis that the mystery of “reinforcing-through-adulteration” was subsequently explained: Gardner noticed that the thickness of the coat, and therefore, one may somehow infer, its durability, depended on the coarse materials that composed the pigments. The early photomicrographs and particle size-measurements tended to show that asbestos and silica particles were, on average, coarser than the lead and zinc pigments. Not surprisingly then, Gardner (1911, p. 86-95) elaborated a classification of pigments not according to their elementary composition, but to the size of minute particles.

### **The Labeling Issue and the Demise of the Old Representation**

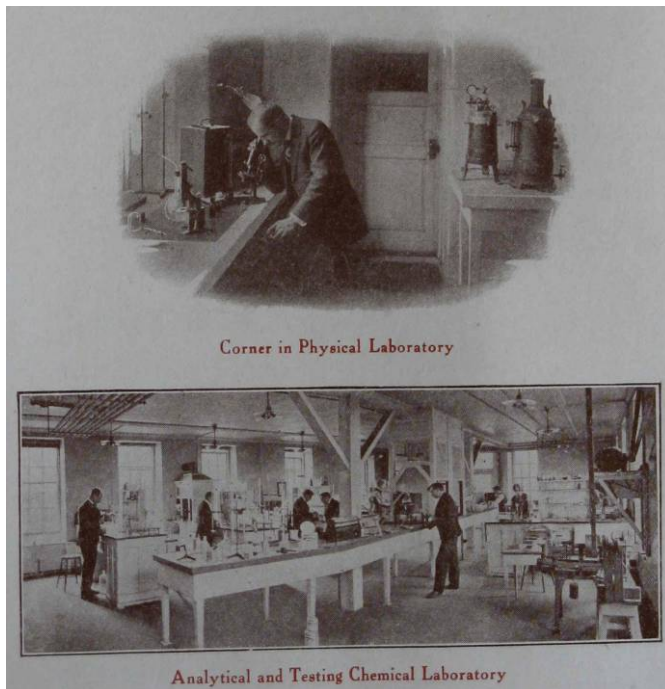
As mentioned above, Heckel and Perry successfully lobbied states' legislatures to prevent paint labeling enforcement. That is, until they came to grips with the North Dakota state legislature, where powerful state chemist Edwin Ladd (1859-1925) had drafted a paint bill in March 1905 which entered into effect in January 1906. The decision to launch the Scientific Section and the panel tests campaign was reached by Heckel after failing to convince Ladd to abandon his bill: "the passage of the North Dakota paint law sharply emphasized the need of marshaling, systematizing and correlating the technical facts scattered through the industry" (Heckel, 1931, p. 81). However, Heckel succeeded in convincing Ladd to host exposure panel tests in Fargo, on the premises of the North Dakota Agricultural Experiment Station run by Ladd. Several chemists of the Experiment Station later joined the staff of the Scientific Section.

Together with the famous chemist Harvey Wiley (1844-1930), Ladd was instrumental in the enactment of the federal Pure Food and Drug Act in 1906, a landmark victory of the progressive movement under the Roosevelt administration (Young, 1989, p. 181-183). During the bitter legislative and political battle over the Act, Ladd acquired an irreversible distrust of manufacturers, and understood the paint adulteration issue just like food adulteration: a conflict of interest between consumer protection and unscrupulous manufacturers. He had little patience for the arguments from industry representatives like Heckel expounding the value of "adulterants" for paint performance. To him, the paint trade was above all ridden by a pervasive hypocrisy, standing on a general claim of purity that, if confronted with impartial chemical analysis, amounted to a massive lie to consumers. To fight adulteration in the paint trade, he distinguished between what he called "statutory pigments" – lead white and zinc oxide – and "substitutes" – the rest of the mineral matter usually introduced in paint formulation, including the most reviled barytes. Labeling paints that were composed of anything besides statutory pigments and linseed oil was mandatory under the state legislation of North Dakota (Holley & Ladd, 1908). In the few years after, Nevada, Texas, Nebraska, Kansas, and Minnesota passed similar laws.

The Paint Manufacturers Association, and Heckel and Perry in particular, sensed that sea changes were under way, and that their networks of informants and lobbyists would no longer prevent the enactment of legislative requirements that would hurt the industry's interests. Yet the industry's prospects were bright: years of continuous growth seemed to lie ahead, and more and more consumers were shifting to ready-mixed paints despite wi-



despread suspicion over “adulterated” products. Re-assessing its interests in the new context of consumer protection, the Association promoted a new marketing and advertising discourse which amounted to a radical change in the industry’s self-portrayal. Rather than parroting the lead manufacturers’ discourse of old masters-sanctioned, pure-white-lead products, the Association attempted to turn a major liability – its dependence upon inferior materials like aluminosilicates – into an asset. After all, didn’t “science” – in the form of ASTM-sanctioned testing methods – prove that adulterated paints could actually perform better than pure paints? Harrison Brothers was among the first companies to embrace this strategy. In the early 1910s the company edited several brochures intended for their dealers and retailers. “The Truth About Paint”, and “Cause & Effect”, two brochures that have survived, explain why a diversity of pigments is good for durability and include photographs of the company’s laboratory facilities, including the recently acquired microscopes (figure 2).



*Figure 2 - Extract of the Harrison Brothers advertising pamphlet “Cause & Effect – a Preachment on Paints”. (Source: Courtesy of the Hagley Museum and Library)*

The modality of exposure tests, under the supervision of “disinterested” parties, enabled Harrison Brothers to portray the claims presented in the pamphlets not as commercial arguments, but as scientific facts.<sup>15</sup> Others manufacturers followed, such as Toch Brothers Co., whose key product, branded R.I.W. – for “Remember, It’s Waterproof”, was claimed to be scientifically prepared in the laboratory of the company. “Pure paint” advertising did not disappear overnight, of course, but gradually faded away during the 1910s and 1920s.

ASTM and the Paint Manufacturers Association issued circulars recommending the adoption of a new nomenclature for paint materials: formerly despised “adulterants” were christened “reinforcing pigments”, “extenders” or “inert fillers”. The new terminology thus conveyed a neutral or positive overtone depicting the variety of minerals, besides lead and zinc, that entered paints’ composition. Guidelines for branding also departed from the obsession with purity: “*Commercially pure* – The use of this term should be avoided if possible” (Gardner, 1915, p. 64). More importantly, ASTM redefined the meaning of “adulteration” and “adulterant”: “a substance substituted partially for another without acknowledgment”, putting aside the issue of performance.

In early 1910, Senator Weldon Heyburn (1852-1912) from Idaho introduced a paint-labeling bill in Congress, modeled on Ladd’s North Dakota bill. Heyburn had previously and successfully introduced the bill which would become the Pure Food and Drug Act. The paint bill however failed to pass Congress. One of the decisive arguments put forth by witnesses to prevent the bill’s enactment was that mandatory labeling would unduly stigmatize valuable materials. Manufacturers’ representatives could rely on a body of data demonstrating their value, and also plead their willingness to reform the paint trade to get rid of the “evil practices” of the past.<sup>16</sup> In Sep-

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<sup>15</sup> Letter, T. J. Armstrong to John Doe, May 15, 1913, Hagley Museum and Library, Charles Demirjian Collection, Box 1, Harrison Brothers advertisements; “The truth about paint”, and “Cause & effect”, *Ibid.* A section of the pamphlet read : “We wish we could have space and your indulgence to allow of a description of the many pigments that go into our products and why they are used. That, however, is impossible here. Suffice it to say that the scientific and progressive manufacturer has been forced to the conclusion after long and careful experiment that the all-perfect pigment has yet to be found. No one pigment which we know now can, used alone, produce a paint capable of withstanding the wide variations of climate and extremes of temperature of this country”.

<sup>16</sup> Congress, House, Interstate and Foreign Commerce Committee, Hearings on H.R. 21901, Manufacture, Sales, etc., of Adulterated or Mislabeled White Lead and Mixed Paint, 61st Cong., 2d sess., 31 May 1910.

tember of that same year Anderson Polk, chief chemist at a major paint company and a long-time member of ASTM, addressed the Master Car and Locomotive Painters Association in St-Louis. His lecture was entitled “Inert Pigments – Their Use and Abuse”, and Polk enjoined the painters and manufacturers to welcome rather than decry inert pigments, and to consider paints as a material that can be designed to fit a specific application:

A great deal of talk has been made concerning the purity of paint; this is an anomaly. We may talk of pure gold or pure linseed oil, or pure turpentine, but one cannot talk about pure shoes, or pure carpets, or pure furniture; there are some ingredients in paint, such as carbonate of lead, oxide of zinc, that are supposed to be pure when as a matter of fact they cannot be absolutely pure under the methods by which they are manufactured. Paint is a mixture of solids and liquids; ingredients that are put into it are for the purpose of making it accomplish something to be desired. That something is to protect and beautify it. Therefore, it is apparent that it does not matter what goes into the paint so long as the consumer is not deceived, and so long as the paint accomplishes its desired purpose, e.g. some paints are designed for painting buildings, some for barns, some for cars, some for bridges, some for signal blades, some for interior decoration, such as painting walls, floors, woodwork and furniture; therefore it is necessary first of all to design the paint for the particular purpose for which it is to be used. (Polk, 1911, p. 27-28)

Thus, in 1910 the paint labeling controversy brought an issue before the federal courts that reform chemists had confronted for several years. In the process, an inherent ambiguity that propelled the pro-paint labeling movement was settled: for what did Ladd really condemn, the discrepancy between the grandiloquent ads and the actual composition of stuff, or the very presence of – supposedly detrimental – “substitutes” into the composition of paints? ASTM and the Paint Manufacturers Association had marshalled sufficient evidence to prove, including in court, the importance of “adulterants” for paints’ material performance. Among his colleagues in the chemical profession, Ladd became isolated in his stance on paint labeling.

## Conclusion

Chemists’ standing and authority within the paint trade and industry changed dramatically at the turn of the 20th century: from “consultants and testing slaves”, per the phrase of historian James Donnelly, to central fi-

gures in the promotion of innovation, economic development, and regulation. This generation of chemists rejected what they pejoratively called the “doctrine of purity” in the paint trade (Hugues, 1911). They gathered around ASTM and the Paint Manufacturers Association, and pushed for a full-fledged recognition and integration in academia. The paint and varnish division of the American Chemical Society was established in 1923, and a community of paint chemists equally represented in academia and industry solidified. The relationship between paints’ microstructure and physical properties, as raised by Dudley and Gardner, became a major research avenue for this community. For this, chemists relied heavily on colloid chemistry and physics, as testified, for instance, by the research program launched by DuPont in the mid-1920s (Cerveaux, 2013, p. 262-288). While color and decoration absorbed these chemists, protection rose in prominence as a function for painting, and became a major objective of their research programs.

The process of industrialization thus triggered changes that stood at odds with the idea that painting was mostly an ornamental and decorative trade, different in nature from the mechanical arts and crafts. Unlike the painters of earlier times, chemists and engineers in the 20th century treated paint no differently than civil engineers and masons would treat concrete: as a reliable material able to fulfill definite functions – namely, the protection and decoration of a variety of surfaces and materials. During its eighteenth annual meeting in 1915, ASTM redefined paint as “a mixture of pigments with vehicle, intended to be spread in thin coats for decoration or protection, or both” (Gardner, 1915, p. 66). A few decades before, painters or chemists would have found this definition jarring. The distinction between paints and varnishes faded: paint, redefined as a coating, came to encompass both terms. This shift in representation was followed by an organizational shift in which Gardner and Heckel played no small a role: the Paint Manufacturers Association merged with the National Association of Varnish Makers in 1933, to be renamed the National Paint, Varnish, and Lacquer Association.

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*Table 1 - Biographical elements of chemists who shaped modern paint chemistry and technology in America (in alphabetical order of name followed by the date of birth)*

<i>Éducation</i>	<i>Carier</i>
<b>Abbott, George Alonzo (1874)</b> 1895: B.S. chemistry, Depauw U. 1896: A.M 1908: Ph.D., MIT	1896-1908: High school teacher 1908-10: Asst prof., North Dakota Col. 1910-: Prof.
<b>Barker, Louis H. (?)</b> ?	1898: Pennsylvania Railroad Co. 1905: ASTM committee E member
<b>Cushman, Allerton S. (1867)</b> 1888: B.S. Worcester, 1889-1890: Freiburg, Heidelberg 1897: A.M. Harvard 1898: Ph.D. Harvard	1892-96: Instructor chem., Saint Louis, Washington 1898: Asst prof. Harvard 1899-00: Asst prof. Bryn Mawr 1901-10: Asst. dir., div. of tests, office of pub. records, USDA 1910-: Director, Inst. of Ind. Research, Washington, D.C.

<b>Dunlop, Frederick Levey (1870)</b> 1892: B.S. Michigan 1895: Sc. D. Harvard 1896: Yale	1896-00: Instr. Industrial chem., Worchester 1900-01: Instr. Inorg. Chem. (Michigan) 1901-07: Instr. anal. chem. Michigan 1907-12: Assoc. chemist, USDA bur. Chem., board of food&drug inspection 1912-16: Consulting chemist, Victor chem works
<b>Dudley, Charles B. (1842)</b> 1875: PhD, Sheffield Scientific School, Yale	1875- : Chemist, Pennsylvania Railroad Co. 1898: ASTM founding member 1902: ASTM president, committee E member and secretary
<b>Gardner, Henry Alfred (1882)</b> 1902: Brown 1903: U. of Penn.	Around 1905: Scientific section, P.M.A., Harrison Bro. Co. 1910: Dir., scientific section, educ, bureau, paint manufacturers assoc. of the US Institute of Paint and Varnish Research, Washington D.C.
<b>Gregg, Norris B. (1856)</b> Washington University, Chemistry	1877-81: Chemist, Southern White Lead Works, Saint Louis 1882-: Chemist, Mount City Paint & Color Co. ?-: President, Mount City Paint & Color Co. ?-: President, P.M.A.
<b>Havens, Franke S. (?)</b> 1896: PhD Chemistry, Yale	?-: Chemist, Harrison Brothers, R.S. Perry assistant, technical matters
<b>Holley, Clifford Dyer (?)</b> 1900: B.S., Maine 1902: M.S. 1904: Ph.D., Michigan	1901-04: Chemist, Maine Experiment Station 1904-07: Prof. ind. chem., North Dakota Agri. Coll. 1908-...: Chief chemist, Acme white Lead & Colors (Detroit)
<b>Holton, E.C. (?)</b> ?: MIT	1898-1930: Chief chemist, Sherwin Williams Co., Cleveland 1902- : ASTM committee E member
<b>Hooker, Albert Huntington (1865)</b> Hon. M.S. Rochester 1920	1889-90: Chemist, Dighton Color Works 1892-93: Opaque Shade Cloth Co. 1894-06: Chief chemist, Heath&Milligan Manuf. Co. 1906-11: Works manager, Hooker Electrochemical Co. (Buffalo, NY) 1911-: Technical director ? - : ASTM committee E member
<b>Job, Robert (1866)</b> 1890: A.B., Harvard	1892-06: Chemist, P. and R.R.R. 1905- : ASTM committee E member 1906-10: Chemist, Booth, Garrett & Blain 1910-: Vice-pres. Milton Hersey Co.
<b>Ladd, Edwin F. Ladd (1859)</b> 1884: B.S., Chemistry, U. of Maine	1884-90: Chief chemist, New York Experiment Station 1890- : Chemist, North Dakota Agricultural college and experiment station 1916: President, Agricultural college and school of chemistry and pharmacy 1921: US Senator, North Dakota
<b>McNaughton, Malcolm (?)</b> ?	?: Dixon Crucible Co., paint and lubricating department superintendent 1902: ASTM member committee E

<b>Nemzek, Leo P. ( ? )</b> ?: B.S., North Dakota U.	?: North Dakota Experimental Station Around 1910: Chief Chemist, John Lucas & Co., Philly Around 1910: Oil and flax seed analysis, P.M.A. Around 1919: Technical director, Paint dpt., Du Pont Co.
<b>Perry, Robert S. ( ? )</b> ?: Lehigh University-Chemistry ?: Royal School of Mines, Freiburg, Germany	1898: Vice-president, Harrison Bro. & Co. 1905: Exposure panel tests, P.M.A.; Scientific section, P.M.A.
<b>Polk, Anderson W. ( ? )</b> ?	1898: Chemist, Lowe Brothers Co. 1902: ASTM committee E member
<b>Sabin, Alvah Horton (1851)</b> 1876: B.S., Bowdoin 1879: M.S.	1876-80: Prof. chimie et physique, Ripon 1882-86: State chemist, Vermont 1897-....: Lecturer, paint and varnish, N.Y. U. 1910-38: Consulting chemist, National Lead
<b>Schaeffer, John A. (1886)</b> 1904: A.B. (U. of Penn.) 1905: A.M. 1908: Ph.D. (chem.)	1908-11: Prof. Carnegie Inst. Of Tech. 1911-20: Research dir., Eagle-Picher Lead Co. 1920-....: Vice-pres., Eagle-Picher Lead Co.
<b>Thompson, Gustave Whyte (1865)</b> - no college education 1927: Hon. PhD, Armour Institute	1902: ASTM committee E member and secretary 1892-19: Chief chemist, National Lead Co. 1919-20: Vice-prs William Harvey Corp. 1920: dir., Titanium Pigment Co.
<b>Toch, Maximilian (1864)</b> 1884-1886: NY U. chemistry (under prof. John W. Draper) + Law School of NY U. LL.B. 1887-1890: Columbia U. (bacteriology & micro-chemistry) 1887 étude avec Ostwald et Witt en Allemagne	1887: Toch Brothers Co. 1904: ASTM committe E member 1905-06: Lecturer organic chemistry, Columbia 1909: Municipal lecturer on paint, Col. City NY 1925-35: Prof. chem. artistic painting, National Academy of Design, NYC 1917-19: In charge of camouflage, USA
<b>Walker, Percy Halgrave (1867)</b> 1885-1887: Virginia 1895: M.S., Iowa 1896-1897: Heidelberg & Berlin	...-1904: Prof. assistant multiple colleges 1904-06: Assayer, USDA Bureau of Chemistry 1906-16: Chief contract Lab USDA Around 1913: ASTM committee E secretary 1914-37: Bureau of Standards

Source: biographical information gathered from Heckel, *Paint Industry, op. cit.*; Ernest T. Trigg, *Fifty-five colorful years*, The Pequot Press, 1954; James Cattell and Dean R. Brimhall, *American Men of Science: A biographical directory*, 3rd edition, The Science Press, 1921; *ASTM Proceedings*, 1903-1906.



## Quality Matters for Historical Plastics: The Past-Making of Cellulose Nitrates for Future Preservation

Anita Quye\*

### Abstract

*The material degradation of an historical artifact through chemical breakdown may place the object at the end of its useful heritage “life” in terms of aesthetic value and appearance. But all is not lost in the ephemeral world of historical synthetic plastics. The chemical analyses of degraded cellulose nitrate artifacts have unlocked material clues that not only help explain stability variations to guide collection care and preservation, but also bring insight into past manufacturing materials, methods and quality control during production. Translating the industrial materials of a degrading artifact by understanding its past to inform its future can revive it with a new cultural significance, and engages heritage scientists, historians and conservators in an innovative community of “complementary science” as defined by Hasok Chang (2004).*

*Keywords: conservation science, modern plastic materials, cultural value, analytical chemistry, industrial heritage, history of science, cellulose nitrate, degradation, modern history.*

### Résumé

*La dégradation matérielle d'un artefact historique par décomposition chimique peut amener l'objet à la fin de sa “vie” patrimoniale utile en termes de valeur esthétique et d'apparence. Néanmoins, tout n'est pas perdu dans le monde éphémère des plastiques synthétiques historiques. Les recherches en chimie analytique sur les artefacts en nitrate de cellulose dégradés ont révélé des indices matériels qui non seulement aident à expliquer les variations de stabilité pour améliorer la conservation mais engendrent aussi une connaissance accrue dans la fabrication des matériaux, les méthodes et contrôles de qualité lors de la production initiale. Traduire les matériaux industriels d'un artefact dégradé en comprenant son passé pour informer son futur peut le relancer dans une nouvelle signification culturelle et rassembler les chercheurs en patrimoine, les historiens et les restaurateurs en une communauté novatrice de “sciences complémentaires” selon la définition d'Hasok Chang (2004).*

*Mots-clés : science de la conservation, matériaux plastiques modernes, valeur culturelle, analyse chimique, patrimoine industriel, histoire des sciences, nitrate de cellulose, dégradation, histoire contemporaine.*

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The creative science of taking a familiar material and chemically changing it into new forms is epitomized by the revolution in the industrial semi-synthesis of plastics from the mid-19th century onwards. The earliest embodiment was cellulose nitrate, known to many by its most familiar name Celluloid. Cellulose nitrate was made by chemical modification of the natural plant polymer, cellulose, and entered commercial production in the 1860s when synthetic organic chemistry and manufacturing technology were opening up new worlds of scientific possibilities for industry.

Historical examples of cellulose nitrate that have survived intact and in pristine condition to the present day are testimonies to the successful balance between raw materials, chemical processing and manufacturing skills. But what can be said of cellulose nitrate when the historical material degrades, as is happening to a small but nonetheless significant number of objects in heritage collections worldwide and exemplified by figure 1? Why are some cellulose nitrate artifacts succumbing to the effects of long-term aging more readily than others? As importantly, does chemical degradation mark the end-point of cultural heritage usefulness for such artifacts? These questions are important for conservation scientists and conservators to answer for the preservation of this landmark historical plastic.



*Figure 1 - Two pictures of the same cellulose nitrate spectacle frames, in 2002 in visibly good condition (left) and in 2014 in a degraded state (right). (Source: Photographs courtesy of Yvonne Shashoua, National Museum of Denmark)*

Degrading cellulose nitrate not only causes its own auto-catalytic destruction, but also releases corrosive volatile chemicals that can induce the breakdown of objects in its vicinity. Conservation research on cellulose nitrate degradation has focused mostly on analytical studies of chemical changes to the modified polymer, loss of its additive plasticizers, and the accelerating effects of increased temperature and relative humidity on its

breakdown (Reilly, 1991; Derrick *et al.*, 1993; Feller, 1994). This understanding has been invaluable for informed management of storage and display environments for historical cellulose nitrate plastic collections (The British Standard Institute, 2012, p. 21), but does not satisfactorily explain what triggers the seemingly random breakdown of the plastic in the first place.

In this essay, I will discuss how reconnecting the history of cellulose nitrate manufacture and the chemistry of past production with present-day material characteristics and chemical composition unveil a fuller picture that helps to explain the preservation behavior of this aged plastic. Within the books and journal articles published between the mid-19th century and mid-20th century about the making of cellulose nitrate, the technical details and chemistry of the process are well-described alongside practical issues that had to be overcome to produce a good-quality material. In these accounts we rediscover the importance of controlling the residual levels of a chemical in a fundamental stage of synthesis for the plastic. We find that the issue persisted from the earliest days of cellulose nitrate production until its industrial decline in the 1960s, and that quality affected the stability of the end-product even within its commercial lifetime. We also see that manufacturers used certain colorants and additives to stabilize the plastic by counteracting the effect of detrimental residues. Revisiting this information allows us to appreciate the material complexities of cellulose nitrate plastic which proved too unpredictable for manufacturers to manage – despite decades of dedicated research – when faced with competition from new petrochemical plastics.

By connecting the historical technical literature for the industrial production of cellulose nitrate plastic with its chemical composition in historical artifacts, we are able to generate a materially-focused body of primary evidence relating a product's quality to its long-term stability. Doing this enables us to re-contextualize the significance of a plastic artifact's materiality as it changes from an un-degraded to degraded state, in terms of its chemical value rather than its aesthetics or function. Thus the cultural value of the degraded heritage artifact takes on new meaning as a consequence of changes in its material composition. Instead of physical degradation marking the end-point in an artifact's usefulness to historical understanding, it becomes a new insight into less tangible aspects of industrial manufacture, such as the undocumented reasons and decisions made by the manufacturers about acceptable quality using technical and scientific know-how that we no longer know or appreciate. This raises two important ethical questions for de-acquisition of degraded historical materials. One is that we undervalue material change as an indicator of past manufacturing practice; if

the object is disposed of, the material evidence goes too. The other is that if the trigger for degradation is inherent in manufacture and in the material itself, and we do not appreciate that the trigger cannot be controlled or removed, then time, effort and resources are expended in a losing battle of preservation. Thus, more interdisciplinary dialogue is required among historians, conservation scientists, and conservators about the significance and value of such materials in a research context. Studying and evaluating our modern industrial material culture through this new perspective opens up a new community in history of science with many possibilities.

In this chapter, I discuss how the chemical challenges faced by cellulose nitrate plastic manufacturers in the past to assure quality control for their brand product has resulted in inherent properties affecting the preservation chances of the material as heritage artifacts. Addressing first the apparently random behavior of the aged plastic and its consequences for heritage collections, I show how documented manufacturing issues involving residual acids, additives, and the limited control of production variables that could not be overcome despite advances in chemical understanding, are linked. They reveal little change in material quality throughout a century of commercial manufacture. These are taken as material reference points to explain the chemical differences between degraded and un-degraded historical plastic examined at the bench of today's conservators and conservation scientists in a quest for strategies to preserve the material. The conclusion is that even if the material cannot be saved it acquires important new value and significance.

### **The Loss of Plasticity: From the Aging of Brand Materials to their Bench Analysis**

- *Historical Context of an Interdisciplinary Project*

As a conservation scientist in a national museum who was surveying plastic artifacts across collections of decorative arts and technical and social history in the early 1990s (Quye, 1993), I, like my peers, was perplexed by the sudden unexpected breakdown of aged cellulose nitrate plastic. Indeed, most curators and conservators were unused to regarding plastics as stable materials and of relatively little research value. In the 1980s, historical interest in the 19th century and early 20th century started to grow, and this is when people were surprised to find that 'everlasting' plastics could fall apart. Analysis of the degraded examples revealed cellulose nitrate to be a vulnerable plastic, along with cellulose acetate, poly(vinyl chloride), poly(urethanes) and synthetic rubber. When curators, conservators and heri-

tage scientists recognized the problem and became more observant, it was realized that certain plastics could degrade within six months even in good museum conditions (Keneghan, 2005). Surveys during the early 1990s of plastic artifacts in the Victoria and Albert Museum and British Museum revealed that 1% were a “high conservation” priority because they were actively degrading (Shashoua, 2009, p. 8-9). This number, although small, has a big impact because the vapors released from degrading cellulose nitrate affect not only the materials of the artifact itself but also other materials nearby. Cellulose nitrate was widely used to imitate relatively stable natural materials like ivory, mother of pearl and tortoiseshell, so it often goes unnoticed until a disguised artifact starts to behave unexpectedly by breaking down.

Most of these historical objects had entered the museum with an unknown user life behind them, yet despite a stable and controllable museum environment, something was causing a few to randomly fall apart even within sets of related objects manufactured at the same time by the same maker. In the late 1980s the degradation problems of historical cellulose nitrate plastic had just been recognized (Green & Bradley, 1988). Some conferences were organized on the subject, like “Saving the Twentieth Century: The Conservation of Modern Materials” held in 1991 in Ottawa (Grattan, 1993). Analytical studies by conservation scientists worldwide started reaching similar conclusions – the material was losing its flexibility-inducing plasticizers and the chemically-modified nitrocellulose polymer was breaking down (Shashoua & Ward, 1995). Yet these chemical changes could not explain satisfactorily the hit-or-miss behavior of the material. Amongst the many chemical complexities of this aged and aging historical plastic, might we be overlooking a basic inherent common factor linking the stability of today’s artifacts to past manufacturing processes?

With awareness rising amongst conservators and curators of unstable cellulose nitrate plastics in the late 1990s (Springate, 1997), the focus of explaining destabilization remained on the loss of nitrate from the cellulose. Some researchers suggested residual acids from manufacture as a possible reason (Selwitz, 1988; Reilly, 1991). Investigating this manufacturing residue as a cause of random breakdown in old cellulose nitrate required not only that the materials of the artifacts be studied, but also a better understanding of quality issues in past production. This would entail a different conservation science research approach, combining the polymer chemistry of historical plastics artifacts with original technical manufacturing information, and co-using primary evidence from the plastic itself and historical industrial documentation. For this endeavor, I initiated an interdisciplinary collaboration between chemistry and conservation science, which led to the doctoral

study by chemist Robert Stewart (1997), jointly funded by the Engineering and Physical Sciences Research Council and the Scottish Conservation Bureau of Historic Scotland. This interdisciplinary research is the focus of my discussion.

Manufacturing chemists of the early 20th century talked about stability issues of plastics. A critical step for end-product quality was the removal of trace sulfuric acid and sulfates following the reaction between cellulose and a nitric acid mixture with sulfuric acid to facilitate the nitration. Inadequate washing at this stage resulted in a poor quality plastic. This once-common knowledge had been lost and forgotten with the demise of the cellulose nitrate industry in the 1960s (Meikle, 1995, p. 28) and overlooked by conservation scientists trying to understand the behavior of the historical material. Rediscovering the impact of residual sulfuric acid helped focus our attention on inherent manufacturing problems that explained the odd behavior of the historical plastic. It also transpired that past manufacturers viewed high levels of residual sulfate content as indicative of a poorly made product. Thus historical plastic with a detrimental acidic content was primary material culture evidence of quality control in the earliest of the man-made plastics. This casts a different light and novel value on degraded plastics in heritage collections, as windows into past industrial processes.

- *Bench Making of Cellulose Nitrate*

To understand the relevance of production quality and its relationship to the stability of historical cellulose nitrate plastic, we first need to understand its making. Manufacture operated within material boundaries imposed by the raw materials, the level of control over the chemical process, and the skill of the maker. All three had a physical impact on the material in terms of its mechanical and chemical durability and stability. This resonated in the shaky start of the first commercial production of cellulose nitrate plastic by Alexander Parkes in London in 1866 under the name of Parkesine. By 1868 Parkesine production had ceased because of poor quality resulting from cost-cutting measures to produce too much plastic with cheap materials (Friedel, 1983, p. 10). Customers complained that it distorted within a few weeks (Mossman, 1994, p. 15). When John Wesley Hyatt and his brother Isaac Smith started making their version, called Celluloid, in 1872 in the United States of America, they used camphor as a plasticizer and ethyl alcohol as the solvent. These were two ingredients that Parkes included in his 1865 patent for Parkesine, but deemed unnecessary to use himself until working for Daniel Spill in London to make Spill's version, called Xylonite, in the early 1870s (Friedel, 1983 p. 10-12).

Celluloid, Xylonite and other commercial brands of cellulose nitrate were more stable than Parkesine, and went on to commercial success as simulants of ivory, pearl, coral, jet, marble, tortoiseshell, amber, horn and onyx as well as in transparent form. The consumer market was favored by the dependable supply of the plastic compared to the natural materials (Friedel, 1983, p. 64). Cellulose nitrate plastic was produced in Europe and in the USA until the 1960s, and made into a wide range of household goods, decorative items, and industrial parts. The versatility of cellulose nitrate in sheet, extruded rod, and molded forms led to a broad and diverse range of applications over its production lifetime, including Victorian hair combs, Constructivist art sculptures in the 1920s, parts for planes and cars, pearlescent casings and finishes for accordions and other musical instruments, ammunition casings, and table tennis balls (Katz, 1985; Meikle, 1995). Additionally, there were cellulose nitrate films, lacquers, explosives, and, for a short time, fibers. It is little wonder, then, that cellulose nitrate has made its way into so many public museums, galleries, archives and historic houses, and private collections (Lavédrine *et al.*, 2012).

At its simplest constituent level, cellulose nitrate plastic is a polymer, which gives physical structure to the material, mixed with a plasticizer, which imparts flexibility. It was classed as a semi-synthetic because the polymer was made of cellulose from cotton and wood that was chemically modified by a nitrating acid mixture. Cellulose is composed of long chain molecules of carbon and hydrogen atoms with many hydroxyl (-OH) side groups, and it is these hydroxyls that are replaced with nitrate groups by an esterification reaction involving an aqueous acidic mixture of nitric acid, water and, importantly, sulfuric acid. With different formulations of the acid mixture, different degrees of nitration substitution of the cellulose hydroxyls were possible. The nitrogen content determined the physical properties of end-product: 10.5% for moldable plastics; 11.5% for films; and up to 13.5% for explosives (Boschan *et al.*, 1955; Reilly, 1991).

- *Manufacturing Problems*

Sulfuric acid played an essential controlling role in the first stage of the polymer-modification reaction pathway by forming cellulose sulfate esters, which were then substituted with nitrates. The right strength and proportion of sulfuric acid in the acid mix was crucial for regulating the substitution rate and number of nitrate molecules, which impacted on the nitration content and hence the end-product. Reaction conditions and quality of the starting materials influenced side-reactions, which also affected the end-result. The reaction solution was always a complex mix of cellulose, nitric acid, sulfuric acid, water, cellulose sulfates, cellulose nitrates, sulfonic

and nitrosulfonic acid esters, oxycellulose and hydrocellulose (Worden, 1911).

A well-documented stabilizing step was repeated post-reaction washing of the esterified cellulose to remove unwanted traces of sulfuric acid and sulfate esters. From the earliest days of Hyatts' process and throughout the production decades of cellulose nitrate, this removal of acidic residues was a critical stage (Friedel, 1983, p. 17). It was alerted to in many publications, including key works on cellulose esters by the American chemist Edward Chauncery Worden (1911, p. 595-596), publications by industrial chemists, like Foster Sproxtton (1938), manager of the British Xylonite Company, and many others well into the 1950s (Miles, 1955). All noted that the quality of the end plastic depended on effective washing. The reason was that residual sulfuric acid would attack the structure-giving cellulose polymer backbone of the plastic, while the sulfate esters could form free acids, which catalyzed the degradation if not removed. Washing was done in large heated vats with boiling water until the overall acidity was reduced to 0.2% sulfate content or less (Worden, 1911). This proved to be a critical factor for the degradation susceptibility of historical cellulose nitrate.

Unstable cellulose nitrate plastic was always a concern of the manufacturers, and deemed a sign of a poor quality product. Problems included warping and distortion (Meikle, 1995, p. 23), and a yellow or brown color forming during 'seasoning' after processing or upon long storage (Worden, 1911). In the late 1920s, Ellington, a polymer chemist, investigated the problem with chemical analysis of fourteen transparent cellulose nitrate sheets manufactured in Germany, France, Britain, America, Switzerland and Japan which had yellowed and degraded (Ellington, 1929). His study showed that the two key destabilizing factors were the percentage (%) content of sulfate and of cellulose sulfate. The stable plastics had less than 0.1% total sulfate content whereas the unstable ones had 0.80% to 0.99% free sulfate and 0.24% to 0.63% cellulose sulfate. This chimes well with Stewart's modern analysis by ion chromatography of degraded historical cellulose nitrate plastic objects with varying visual signs of active degradation, such as discoloration, cracks, and characteristic square pattern crazing (Quye & Williamson, 1999, p.122-135; Shashoua, 2009, p. 151-184). The deteriorated aged plastics studied by Stewart all had a minimum of 0.5% total sulfate content. This was remarkably close to the 0.2% threshold maximum for a good quality plastic advocated by Worden a century earlier, demonstrating that manufacturers had the analytical capability and chemical understanding to measure and monitor the residual acid content of cellulose nitrate from the start of the 20th century, if not earlier. Indeed, they



acknowledged the importance of bench chemistry to control the properties of brand materials.

- *Opaque versus Transparent Plastic: A Clear Question of Quality*

Besides residual acids in degraded historical cellulose nitrate plastic, Stewart investigated another significant chemical composition factor linked to the common observation by conservators and curators - that transparent forms of the plastic tend to be more degraded than opaque forms. Again using ion chromatography, he found a high correlation between clear artifacts with visible cracks or yellowing and more than 0.5% sulfate content. However, if the plastic was opaque there were few visual signs of active degradation even if it was over the critical 0.5% total sulfate threshold. Why was this? Was there another quality relationship? The answer lay once again in the manufacturing chemistry for the plastic.

A lucrative consumer market for cellulose nitrate plastic was as a simulant of luxury natural materials. Imitation ivory, jet, pearl, coral and amber were popular forms (Böckmann, 1880, p. 97-100; Worden, 1911, p. 687-697), and it is under these guises that the plastic is often present in heritage collections or fashion, art, technology, social, and even natural history. To make imitation ivory and other opaque forms, the manufacturers added zinc oxide, zinc carbonate, or calcium carbonate to the cellulose nitrate dough (Sachs & Byron, 1921). Worden commented that "Transparent plastic is harder to keep stable than translucent and opaque, due to the stabilizing action of the zinc oxide and carbonate and other pigments present in the latter, and usually in large quantities" (Worden, 1911, p. 595).

Stewart readily detected zinc in historical samples of 'ivory' cellulose nitrate using X-ray fluorescence spectroscopy, and titanium from titanium dioxide, which was a common opacifier in many industrial applications from 1916. Stewart's sulfate analysis of these same artifacts confirmed that the minerals had maintained a protective effect over the decades in the plastics with over 0.5% of the detrimental sulfate content because they showed no sign of degradation. The chemicals added during manufacture to opacify the plastic were having a stabilizing effect on historical cellulose nitrate.

Cellulose nitrate manufacturers referred to their stabilizing chemical additives as antacids. Tellingly, the antacids were a safeguard against residual sulfuric acid and sulfates, and sometimes added even if deemed unnecessary at the time of production. The opacifying inorganic mineral were also classed as antacids, so their dual role as stabilizers was known. Antacids for transparent plastics were organic compounds, like urea (Worden, 1911). There were differing opinions about whether antacids for transpa-

rent cellulose nitrate covered up a poorly-manufactured product. Antacids were encouraged in a book about European cellulose nitrate production in the 1910s (Masselon *et al.*, 1912) while a book about the American cellulose nitrate industry, published at the same time, endorsed thorough washing and advised against antacids (Worden, 1911). Washing was the industry-wide preference on both sides of the Atlantic. In Ellington's research of the different makes of cellulose nitrate sheet (Ellington, 1929), he classed the stable plastics with low sulfate content and little urea or mineral content as high quality, viewing the low sulfate levels as good production control. Samples with high quantities of sulfate contained appreciable levels of urea (0.2% to 1.2%), which Ellington deemed "objectionable" to him as a polymer chemist. He referred to the urea as "artificial stabilisation" because manufacturers would have been aware that the sulfate in their material was an "undesirable impurity". Studies of urea in historical cellulose nitrate plastics have not been published yet, but urea content should be investigated to see if it is detectable and correlates with the stability of the historical plastics.

Herein lies an interesting quality question with implications for the interpretation of historical collections. If it took better production control to make a stable transparent cellulose nitrate than it did for an opaque form because the opacifying minerals acted as antacids, were lower quality plastics used to make the expensive-looking simulants like ivory, pearl, coral and onyx? If so, the technological value and quality of clear cellulose nitrates would be higher than the simulants despite the simulants having more aesthetic appeal and looking like a better class of material. Of course not all clear cellulose nitrate plastics were necessarily high quality, as evidenced by the many instances of degraded historical drawing instruments which tend to be transparent, but it does open up a new area for discussion about intrinsic and implied material value of historical synthetic simulants of natural materials between historians and curators of design and technology.

## Chemistry Matters

While Stewart's analytical study of degraded cellulose nitrate plastic links long-term stability to residual acids and added opacifiers from manufacture, it is only a partial insight into the chemical complexities of the end-product. Making cellulose nitrate was a multi-stage chemical balancing act. With the industry spanning from the 1860s to the early 1960s, it covered a monumental period of increasing chemical understanding as well as technical and social change. Manufacturing transitioned from an arena of expe-

rimentation and trade secrets to targeted research and greatly enhanced chemical knowledge of the materials and product. Yet the basic chemistry of the process did not change. What impact did this have on the material quality? And what are the implications for historical collections? Do the longevity and stability of old cellulose nitrate plastics correlate with date of production? To begin finding answers, the role of chemistry in the industry needs to be examined more closely.

Chemistry was intrinsic throughout the whole process of making cellulose nitrate. From its earliest days, the cellulose nitrate industry acknowledged the necessary input of chemists. Raw materials, solvents and additives had to be selected, purity-tested and prepared. The nitrating acid mixture needed specific formulations, while the esterification step required monitoring and control. Spent acid had to be removed and recycled. The right type and amount of solvent and plasticizer had to be added to the nitrated cellulose to make a 'colloidon' of the required viscosity for handling and shaping. Chemists were employed as in-house analysts and managers to select the best materials and to control the process. This included solvent solubility tests for the degree of cellulose nitration, and viscosity measurements to assess physical quality for processing (Schüpphaus, 1915; Partidge, 1929). Hyatt said he was "allowed to employ a chemist [Mr Frank Vanderpoel] for determining our acids and to systemize our nitration, instead of merely using hydrometers and thermometers" (Hyatt, 1914).

The era between the 1870s and early 1900s was one of empirical venture for the makers, but driven more by tacit technical experience and commercial enterprise rather than systematic scientific advances (Friedel, 1983). The molecular structure of cellulose was not deduced by Cross, Bevan and Beadle until 1895, although as it turned out advancements in polymer and macromolecular theories over the following decades had little effect on improving the quality of manufactured cellulose nitrate plastic. The best raw materials and additives were found early on because of industrial trials and observations (Friedel, 1983). This included a good plasticizer to soften the nitrocellulose polymer for shaping and molding, and a good solvent (Ott, 1940; Friedel 1983). The Hyatts and Daniel Spill used camphor, a natural extract from the wood and bark of the Japanese *Formosa* tree, as a plasticizer from the outset for their cellulose nitrate plastics in the 1870s. The undesirable pungency of camphor and its cost at the turn of the 20th century led to the testing of no less than 44 chemicals and many derivatives as substitutes (DuBois, 1907, p. 40-41), while oil of turpentine was used in World War I because of camphor supply shortages (Mork, 1917). Other alternatives were also trialed periodically (Sachs & Byron, 1921; Durans & Davidson, 1936), but camphor remained the best choice. Hyatt also

decided on ethyl alcohol for the solvent and patented the important process of 'seasoning' the finished product to allow all solvent traces to evaporate for stabilization (Meikle, 1995).

With good choices of camphor plasticizer and ethyl alcohol solvent from the outset, and awareness of residual acids and the benefits of antacid stabilizers in place by the start of the 20th century, the industry had established in its early days what chemists at that time considered to be the four strong pillars of material stability for the plastic. The main advance for the cellulose nitrate plastic manufacture in the 20th century was not so much the chemistry of the material, but rather controlling the many variables mentioned above during the production stages. Old industrial processes were revisited and re-evaluated (Lunge, 1901), advancements made in cellulose chemistry (Briggs, 1915), and the benefits of systematic applied chemistry advocated to help solve industrial problems (Bacon & Hamor, 1919). In 1920, Staudinger's macromolecular theory classified plastics as polymers. The crystalline structure of cellulose was revealed by X-rays one decade later (Clark, 1930). By the end of the 1920s it was agreed that cellulose was a polymeric chain of cellobiose monomers (Badgley *et al.*, 1945), but the direct impact of these major theoretical chemistry advances on cellulose nitrate plastic quality was far less than might be expected. The chemical process approach to esterification had changed very little since the beginning (Yarsley *et al.*, 1964, p. 173). Instead, the developments were more advantageous to manufacturing processes for the new related plastics made from cellulose acetate and other cellulose derivatives.

By the 1920s interest was growing in colloid chemistry to measure and characterize the viscosity of colloidon (Bancroft, 1922). This was driven further in the 1930s by the advent of fiber extrusion and injection-molding for cellulose acetate, although this did not benefit cellulose nitrate plastic much because these mechanical processes did not suit its flammable tendencies. The advent of the ultracentrifuge in 1938 improved viscosity measurements for cellulose nitrate plastics (Kraemer, 1938). However, the chemical complexity and control over minute changes throughout the whole process of making cellulose nitrate could not be overcome with the extent of knowledge about colloid chemistry at that time (Conaway, 1938). By the time polymer chemistry had matured in the 1940s, it was of more value to the expanding fiber-making industries for filament extrusion of viscose rayon and cellulose acetate, and for tailor-making cellulose derivatives rather than improving cellulose nitrate plastics (Tinsley, 1948). The rise of the more controllable petrochemical plastics proved too much competition for the variances of cellulose nitrate (Meikle, 1995, p. 23). Cellulose nitrate plastic was by now less appealing because its preparation was so sen-

sitive, with even small changes in the equilibrium having unpredictable effects (Conaway, 1938).

Despite a steady increase in chemical research for commercial cellulose nitrate manufacture from the 1910s to the 1930s, with a move from small factory works to scientific institutions and industrial labs (Morris, 2015, p. 242-252), plus commercial and academic investment in research, practical issues of variable chemical reaction parameters for cellulose nitrate plastic could not be resolved. Eventually commercial manufacture started declining in America in the mid-1950s amid competition from other better-controlled synthetic plastics (Meikle, 1995, p. 28). In 1963, the few European companies still making cellulose nitrate plastic were working with old equipment, while Japanese manufacturers used advanced technology (Kaufman, 1963). By this date, cellulose nitrate was no longer produced in the USA, but was still available and continued to be used for brush handles and spectacle frames (Yarsley *et al.*, 1964).

What does this overview of past cellulose nitrate plastic manufacture offer to the conservation science of cellulose nitrate plastics? The upshot is that despite progressive chemical understanding and a rise in research investment, there were surprisingly few major chemical step-changes for the manufactured material. With regards to the common heritage science application of material analysis to provenance the origin or date of an historical object, the chemical composition of commercial cellulose nitrate plastic can only enlighten us a little. The presence of a titanium opacifier would indicate a date post-1916 and, with more research, camphor substitutes used by different manufacturers, for example oil of turpentine derivatives, could be linked to specific periods. Other factors like the design and style of the object and trademarks would be more informative. Nonetheless, material information is still important to collect for preservation needs. For example, oil of turpentine derivatives discolored imitation ivory (Sachs & Byron, 1921), so its presence in an aged object would predict or explain changes to its appearance.

Gaining better appreciation of the quality challenges that the historical commercial makers faced to control vagaries in the process makes the random degradation between similarly dated or produced objects more understandable. It is an inherent vice, yet this does not detract from the benefits of analyzing degraded objects materials with well-known provenance, instead enhancing further the material picture of production quality effects and connecting material evidence to past written observations and tests.

## Changing Values of Brand Materials

The correlation between the chemical composition of artifacts and their physical condition by Stewart was made possible by the direct analysis of various artifacts, from good to poor quality. This invaluable primary source research relied on collectors and curators appreciating that, in this instance, de-accessioning and sacrificing a small number of historical objects would answer greater questions about stability to the benefit of many more in heritage collections. Some de-accessioning decisions were justified on the grounds that material breakdown had reached a critical point such that the artifact no longer had significance or value in the context of the collection and was also putting other parts of the collection at risk from the emissions of its degradation products.

In this way, these historical materials inadvertently acquired a new value for industrial heritage. While on one hand the degradation of material culture can result in irretrievable or irreversible loss of the form or function of artifacts, on the other these collections of historical materials enter a new phase of historical value, becoming “monuments of history” as material culture objects that reveal history and passage of time (Muñoz Viñas, 2005). Thus, un-degraded and degraded historical cellulose nitrate plastics both come to share significance and a material culture value for the conservation scientist and industrial historian, where there are mutual interests in product, production and quality. From its primary use as a brand material to one as an historical object in a heritage collection, an artifact experiences its first shift of significance. When it is removed from a collection because of degradation, the same artifact acquires a second and new value, as an invaluable material for experimental conservation science research into the processes of aging and deterioration.

Especially valuable for direct primary source evidence from the past are materials with well-documented provenance: where, when, and how they were made. For conservation scientists, company archives of products and production records provide significant historical clues. Detailed information is also essential for reconstructions of historical processes as another invaluable resource for technical history research (Stauber mann, 2009). It is as important to preserve and understand not just the manufactured end-product but the raw materials and the manufacturing processes, and to preserve manufacturers’ samples and associated knowledge through business archives. Increasing digitization allows on-line access to publications from the late 19th century and early to mid-20th century, such as *Industrial and Engineering Chemistry*, where much was published about the early plastics industry and now becomes invaluable for documenting its growth and changes. Access to these publications has significantly aided and enhanced

research to connect artifacts and modern production for conservation science, revealing an abundance of information from other chemical industries, such as the related synthetic fibers (Quye, 2014) and synthetic dyes (Quye, 2016). Likewise, it is essential to preserve the physical evidence of the products and documentation of production, and for conservation understanding to grow about materials for informed “interventive conservation”<sup>1</sup> (Shashoua, 2016), and for collection management of artifacts and archives (Brokerhof & Bülow, 2016). Uniting industry and historical material culture in this way offers a potent reconnection between maker and product.

### **Conclusion: From the Preservation of Materials to Interdisciplinary Research**

While there is an obvious desire to keep old cellulose nitrate plastics ‘alive’ so that their function, form and aesthetic can be appreciated and understood, their ‘death’ brings an unexpected insight into their material composition and manufacture, with the process and products of degradation providing invaluable pieces of primary chemical evidence of past production. Within the degraded plastic itself is a direct connection between material stability, the chemistry of the manufacturing process, and quality control during manufacture. Linking the chemical evidence in degraded and un-degraded cellulose nitrate historical artifacts with contemporaneous scientific accounts of their manufacture from those who understood the scientific principles of manufacture brings those historians interested in 19<sup>th</sup> century and early 20<sup>th</sup> century chemical manufacturing closer to direct primary evidence of quality control.

This a tale to emphasize that preservation of material culture makes knowing and understanding industrial techniques valuable and necessary. As observers with the gift of hindsight, we witness in cellulose nitrate plastic a threshold amount of a known malignant acidic residue that was just acceptable when made but has now become a destabilizing inherent vice with time. That there was a need to rediscover a well-known phenomenon first reported over a century ago and common knowledge until just 60 years ago says much about how easily and quickly information is lost with the decline of a commercial manufacturing industry. Research like Stewart’s reconnects the material evidence in the historical object with past manufac-

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<sup>1</sup> “Interventive conservation” deals with the physical treatment of objects, like cleaning or repair, whereas “passive conservation” seeks to control environmental conditions such as temperature and humidity.

turing method information, and revives the understanding to recognize the significance of objects and their contextual information. In this case, a quality issue inherent in a past manufactured product has resurfaced as a consequence of the material being kept by museums and collectors for longer than the manufacturers could have expected.

Researching historical materials for conservation science entails three essential aspects for meaningful and progressive insight: interdisciplinary collaborations; access to digitized, searchable archives; and an understanding of the chemistry of materials. The research described in this chapter for cellulose nitrate would not have been successful without cooperative understanding between an analytical scientist, a polymer chemist, and a conservation scientist. Our multidisciplinary discussions gave insight into the past industrial production of a material and connected the research to the history of science. In short, the breakdown of an inanimate material brought a new community of people together in a dialogue where chemistry, conservation and history had to be articulated and interconnected.

The study presented is by no means a unique example of how preservation brings insight to past technology and production quality. Collaborative research between conservation scientists at the Kunst Historische in Vienna and historians revealed that the unexpected and unlikely corrosion of gold coins minted in the 19th century. The problem transpired to be the dies, carrying traces of contamination iron from other coins onto the surface of the gold coin (Traum & Griesser, 2006). Taking a look beyond what is happening to the aged material now and placing its present chemical condition in the context of its production takes historical materials research beyond issues of current preservation state into the realms of technical production and industrial quality.

The multidisciplinary collaboration of material chemists and heritage scientists, and knowledge exchange with curators and historians of technology and industry is enlivening, indeed vital, when the maker's voice is lost. Access to historical manufacturing information greatly assists conservation scientists and conservators in their quest to understand more about original modern industrial materials. At this point in time there are many examples of historical cellulose nitrate, but with loss through degradation, preservation of these once common mass-produced objects becomes even more pressing especially if other sources of related information disappear (Muñoz Viñas, 2005).

While any loss of material culture is lamentable to its collector and custodian, especially when the object loses significance because it is no longer physically intact nor accessible in its broadest sense, or becomes a health hazard or is detrimental to other artifacts, it can attain a new role



within historical and socioeconomic frameworks. An historical object travels different paths in its journey through the material culture world where it will be judged by our changing perspectives on value and significance. It may seem that the end of its useful 'life' will be the day when the object loses its material coherence and physically breaks down. To the materials scientist, this point can be the start of a new journey of discovery. Even if an object can no longer be used or understood, like the spectacles in Figure 1, its degraded material composition is a bridge to an otherwise distanced world of its creation.

In the context of stabilization of cellulose nitrate plastics, the endeavors of the industrial chemists testing the quality of the material for the consumer lifetime of the material are similar to the conservation scientists' testing of the composition of aged material to extend the artifact's lifetime. The connections among chemists, polymer scientists, engineers, and industrialists in the historical production of brand plastics are mirrored in the knowledge exchange community of chemists, conservation scientists, conservators, historians, and curators for the promotion and conservation of material collections. When quality matters for industrial heritage, historical objects benefit from new conversations in history of science for material significance and preservation.

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## **Part II**

*Knowing by Making and Making by Knowing*

## Twentieth Century Fertilizers in France from Natural Mixing to Artificial Making (1890-1970)

Philippe Martin \*

### Abstract

*Compound fertilizer is a material composed of a mixture of primary nutrients. The physical constitution and presentation of compound fertilizer evolved from the nineteenth to the twentieth century. This material was originally the subject of purely empirical knowledge, but later it gradually attracted chemists, who developed it in confrontation with agronomists and farmers. In return, in the interwar period compound fertilizer gave the chemical community a sense of mission: to solve the “urgent need” to increase fertilizer consumption and to make the product “rational” with respect to transport costs, storage stability, ease of use, and, of course, agronomical efficiency. This paper traces the confrontation of actors and technical and industrial changes that guided the development of compound fertilizer in France from 1890 to 1970.*

*Keywords: fertilizers, agriculture, adulteration, industry, productivism, chemical innovation.*

### Résumé

*Mélange d'éléments fertilisants majeurs, l'engrais composé est un matériau, qui évolua dans sa constitution physique et dans sa présentation du XIX<sup>e</sup> au XX<sup>e</sup> siècle. Initialement issu d'un savoir-faire technique empirique, ce matériau est progressivement investi par les chimistes, qui le façonnent en confrontation avec les agronomes et les agriculteurs. En retour, ce matériau oriente la communauté des chimistes, qui se sent investi, dans l'Entre-deux-guerres, d'une mission face à l'impérieuse nécessité d'accroître la consommation d'engrais : fabriquer un produit « rationnel » en termes de coût de transport, de stabilité au stockage, de facilité d'épandage et bien sûr d'efficacité agronomique. Cet article retrace les confrontations des acteurs et les changements techniques et industriels qui guident l'évolution des engrais composés en France de 1890 à 1970.*

*Mots-clés : engrais, agriculture, falsification, industrie, productivisme, innovation chimique.*

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IN THE FIELD of fertilizers at the turn of the 20th century, chemists felt that they were the bearers of a benevolent mission, especially through the development of superphosphate fertilizers. This was explained to farmers by the Compagnie de Saint-Gobain, one of the two biggest French fertilizer manufacturers (along with Etablissements Kuhlmann). “The use of chemical fertilizers chemical is no longer in effect a simple convenience for agriculture: it is an absolute necessity.”<sup>1</sup> With superphosphate, a straight fertilizer, the heavy chemical industry had gained a foothold in the fertilizer industry, yet chemical fertilizers still only played a supporting role. The major fertilizer remained farmyard manure, a “natural” fertilizer compound *par excellence* for the farmer – a farmer who often prepared his own mixed fertilizers. Manufactured compound fertilizers were suspected of adulteration and were virulently hated by French agronomists. As one agronomist, Achilles Müntz, said in 1890: “The decrease in the purchase of fertilizer formula is the true measure of the spread of agricultural science throughout the countryside” (Müntz & Girard, 1891, p. 407).<sup>2</sup> Yet eighty years later, in the 1970s, manufactured compound fertilizers accounted for 67% of consumption of fertilizers in France. Today, agronomists recommend them. The production plants of fertilizers are imposing, for chemical fertilizers have become commodities across multiple continents and the volume of fertilizer consumption has become massive.

Compound fertilizer is a mixture of the primary nutrients (nitrogen, phosphoric acid, and potassium), in contrast to straight fertilizer, which consists of a single element. From the nineteenth to the twentieth century, fertilizer materials have evolved in their chemical compositions (organic, mineral and synthetic compounds) as well as their formulations (powders, granules, pellets). Such an evolution raises issues associated with the social construction of a product: agronomists, chemists, and industrialists all confronted each other in negotiating product quality, standardization, and the opposition between natural and artificial (Jas, 2000; Cohen, 2011). This case also highlights the collaboration of chemists and industrialists in technology transfers to solve a series of “reverse salients” to advance the industry and best meet demand (Caron, 2010; Hughes, 2004).

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<sup>1</sup> “L’emploi des engrais chimiques n’est plus en effet désormais pour l’agriculture une simple convenance : c’est une impérieuse nécessité (Saint-Gobain, 1911).” All the translations of the quotations are from the author with slight revisions of the editors.

<sup>2</sup> “La décroissance de l’achat des engrais à formule est la véritable mesure de la diffusion des sciences agricoles dans les campagnes.”



This chapter tackles the co-shaping of fertilizer materials and the professional identity of chemists, industrialists, agronomists, and farmers. I examine, first, the origins of compound fertilizers. This reveals the cause of the distrust between agriculturalists and industrialists and the posture of chemists in this confrontation. I then ask how chemists inserted themselves into the manufacture of compound fertilizers and how they appropriated the needs of farmers in order to offer new products. I explore, finally, the solutions that were proposed by manufacturers and chemists to meet surging demand from farmers in the 1950s and 1960s, in the context of productivism among the French government and agricultural authorities.

### **Guano, Fish and “Organo-Mineral” Fertilizers: The Building of Trust in Compound Fertilizers (1890-1920)**

- *Mixed Fertilizers: Organic Origin and Empirical Knowledge*

In the 1830s, manufacturers produced fertilizer from mixtures of industrial and urban waste. One of the first “artificial” compound fertilizers in the 1840s was Peruvian Guano, a nitrogen and phosphate fertilizer. However, guano was not manufactured by industry but rather marketed by merchants. Thus, in 1845, the German chemist Justus Liebig partnered with the British industrial James Muspratt and took out a patent for six different fertilizers tailored to six different types of crops in the hope of replacing guano. It was a fiasco: the fertilizer formed a hard crust on the surface of fields. Indeed, Liebig worked in his laboratory and was distant from the field. In addition, he excluded any nitrogen fertilization (Bensaude Vincent & Stengers, 2001, p. 225-226; Jas, 2000, p. 36). The idea nevertheless caught on and in the 1850s manufactured “artificial guano” appeared in France. This was developed by individuals with industrial, agronomic training, such as Edouard Derrien, in Nantes on the Loire estuary in the western part of France (Martin, 2015). Abendroth, a doctor of philosophy and industrialist in Dresden, clearly defined in 1855 the challenges of “artificial guano” in terms of efficiency, consistency, portability, handling, cost, and industrialization of its production:

1. That this fertilizer can be provided in sufficient quantity; 2. That it is easily transportable and handling is easy and convenient; 3. That it always contains the main fertilizer ingredients in equal proportions; and 4. The goods,

having all of these conditions, can be established at a reasonable price and in any case lower than the Peruvian Guano.<sup>3</sup>

This definition, contained in a patent, is very interesting because it establishes the main issues that would guide the development of compound fertilizer throughout the twentieth century.

However, it was not until the late nineteenth century and early twentieth century that links were actually woven between chemists and fertilizer. The fertilizer industry at that time relied on the bulk chemical industry for the production of an intermediate required in the production of superphosphate, a product that makes inorganic phosphates assimilable by plants: sulfuric acid. With this product, chemistry entered the arena of compound fertilizers. Besides superphosphate, new forms of fertilizers appeared: “organic-chemical” fertilizers and “dissolved organic” fertilizers, including “guano dissolved” fertilizers in which guano is attacked by sulfuric acid in order to attach ammonia (Couturier & Lucas, sd, p. 49-50). With the discovery of mineral fertilizers, fertilizers made from organic mixtures were gradually displaced by “organic-mineral” fertilizer, a mixture of organic substances, minerals (Chile sodium nitrate, calcium phosphate, potash), and ammonium sulfate. The chairmanship of the Société des Agriculteurs de France by the Marquis Charles Jean Melchior de Vogüé, chairman of the Compagnie de Saint-Gobain from 1901 to 1916 is symbolic of this rapprochement of chemists and agronomy (Anonymous, 1965, p. 76). The manufacturers have guided farmers in their use of fertilizers by providing instruction manuals specifying the dose and the period of application, sometimes with recommendations for spreading, as did, in Nantes, society Pilon Frères, Buffet, Durand-Gasselin for its fertilizer bone (Anonymous, n.d.).

- *Adulteration and Product Quality: Compound Fertilizers Discouraged by the Agricultural Elite*

Since the early nineteenth century, the agricultural elite<sup>4</sup> intensively promoted modern agriculture among farmers in order to cope with an in-

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<sup>3</sup> “1° Que cet engrais puisse être fourni en quantité suffisante ; 2° Qu’il soit facilement transportable et que le maniement en soit facile et commode ; 3° Qu’il contienne les substances principales d’engrais dans des proportions toujours égales et que 4° La marchandise, présentant toutes ces conditions, puisse être établie à un prix modéré et en tout cas plus bas que le Guano du Pérou” Patents data base of the Institut National de la Propriété Intellectuelle (INPI) <http://bases-brevets19e.inpi.fr/>, cote 1BB25599.

crease in the urban population. This agriculture was based on the abandonment of fallow in favor of the “mixed farming/cattle breeding” system. The objective was to increase yields per hectare with cattle-manure and, in addition, “artificial fertilizers” (manufactured fertilizer or imports such as Peruvian guano). Although they encouraged the use of fertilizers, these authorities remained wary of manufactured compound fertilizers offered by manufacturers. Compound fertilizers were not well regarded by agronomists: they were seen as including unnecessary ingredients, their prices were high relative to fertilizing capacity, their ready-made formulas were not adapted to all cultures, and they combined ingredients which agronomists felt should be used separately or at different times. In the 1890s, Achille Müntz, Chemistry Laboratory Director of the Institut National d’Agronomie in Paris, recognized the value of mixing fertilizers for the farmer: “With straight fertilizers, nothing is easier than to respond to all cases of agricultural practice; they can be used individually or combined in pairs, in threes, in the desired proportion to obtain maximum results with minimum expenditure”<sup>5</sup> (Müntz & Girard, 1891, p. 392-394). Yet Müntz also condemned manufactured compound fertilizers:

[Compound] fertilizers offered by businesses must be rejected by the farmer. The farmer seeks to give the soil really useful elements in varying proportions, without having products imposed that do not meet this condition. Agricultural education will increasingly reduce their sales, and already in areas where culture is advanced, its use is restricted. The decrease in the purchase of fertilizer formula is the true measure of the distribution of agricultural sciences in the countryside. (our translation from Müntz & Girard, 1891, p. 407)

Above all, fertilizers, and particularly compound fertilizers, were the subject of fraud and adulteration in the nature, origin, quantity, and quality of components. The road to recognition of compound fertilizers was long and stretched throughout the nineteenth century. The farmer’s representations of natural and artificial was continually confronted (Cohen, 2011). In France, chemists such as Adolphe Bobierre (1850) made combating fraud

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<sup>4</sup> Agronomists, members of agricultural societies, landowners, large landowners, the readers of the *Journal d’Agriculture Pratique* of Alexandre Bixio... (Duby & Wallon, 1976, p. 105-107).

<sup>5</sup> “Avec les engrais simples, rien n’est plus facile que de répondre à tous les cas de la pratique agricole ; on peut les employer isolément ou les combiner deux à deux, trois à trois, dans la proportion voulue pour obtenir le maximum de résultats avec le minimum de dépenses”.

their workhorse. Appointed “chimiste-vérificateur en chef” in Loire-Inférieure, Bobierre was in charge of controlling the trade in fertilizers by application of the prefectorial decrees of February 23 and April 6, 1850. Through his many books on the fraud of the fertilizers, he sensitized the public authorities to these questions and contributed to the establishment of the fertilizer investigation of 1864, which gave rise to the first French law of 1867. While not immediately restoring confidence, the law of 4 February 1888 corrected malfunctions in this first law by making it possible for the farmer to analyze a sample of fertilizer in experiment stations. This law created a new transaction mode in the fertilizer market in which the alliance of science (chemistry in this case) and the French state played a major role (Jas, 2000, p. 294-310). Chemists were no strangers to agronomic experiment stations; rather, they intervened downstream from the design of fertilizers by improving analysis and quality control among manufacturers.

With the need for sulfuric acid to produce superphosphates and dissolved guanos, chemists were increasingly present in the manufacture of fertilizers. Many factories joined a workshop for the production of sulfuric acid with the process of lead chambers, which required the presence of a chemist. The product quality constraints would, moreover, lead the most manufactured factories to install a chemical analysis laboratory headed by a chemist. The importance of the role of chemists in the branch of compound fertilizers would take a new turn in the interwar period.

### **Complex Fertilizer Pellets: Chemists’ Recognition of and Slow Progress Down the Path Towards an Integrated Product (1920-1950)**

- *Farmers’ Strong Demand for Fertilizers around 1920*

After World War I, the demand for fertilizers from French farmers strongly increased. This need was linked most of all to material shortages due to the war, but it was also based in a need to compensate for the lack of labor (dead, wounded, rural exodus) (Dumoulin, 1988, p. 175-180). The trend that began before the war continued and was strengthened with the need to increase agricultural productivity using mechanization, seed selection, and fertilizers (Duby & Wallon, 1977, p. 178). In the department of Loire-Inférieure, the Société d’Agriculture de France and local agricultural unions, such as the Syndicat central des agriculteurs de Loire-Inférieure, supported an approach to increasing yields and production intensification that involved purchasing fertilizer together and mechanizing fertilizer application (Anonymous, 1928). The fertilizer distributors were promoted for small farming by agricultural unions. By the late 1920s, agronomists awaited

fertilizer in granular form to facilitate mechanized spreading: “We must hope that the industry strives to produce all fertilizers in granular form, which greatly facilitates their distribution.”<sup>6</sup> These compounds provided fertilizer to farmers, savings in transport costs, handling, storage, and spreading. They were all the more desirable given that the available labor was less.

With fertilizer production becoming an important branch of chemistry, major chemical groups moved closer to the world of agriculture. Manufacturers set up “experimental fields” (*“champs d’expériences”*) as demonstration plots of the effect of fertilizers (Cerf & Lenoir, 1987, p. 32). So, Compagnie de Saint-Gobain created, in 1926, the Bureau Central de Renseignement Agricole et de Propagande and organized cropping trials systematically from 1927 onward. The borders among agronomists, chemists, and industrialists began to dissolve as their responsibilities began to interfere and overlap.

- *The Slow Appropriation of Pellet Fertilizers by Chemists*

In the 1920s and 1930s, the issue of synthetic ammonia was solved (Travis, 2015), opening new perspectives in the field of chemical fertilizers. With nitrogen now available and cheap it was possible to consider binary or ternary compound fertilizers. To facilitate the consumption of fertilizer and expand its market, the issues raised by Müntz in the late nineteenth century, were placed on the agenda of the chemical community: how to remove inert substances and lower prices? The result was an increase in the concentration of fertilizers and limitation of the use of sulfuric acid which requires expensive handling and processing of iron pyrites. These issues were discussed in France in several meetings of the Congrès de Chimie Industrielle (Industrial Chemistry Congress). The inorganic chemist Camille Matignon (1930) explained the task of chemists and industry:

The current trend in the fertilizer industry is to eliminate all inert substances. These contain substances which are often expensive because of their origin and transport, or of no or insignificant interest for plants. Thus the industry is oriented toward the search for concentrated fertilizer with high-analysis materials, formed from phosphoric acid itself, by its union

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<sup>6</sup> “On doit souhaiter que l’industrie s’efforce de produire toutes les matières fertilisantes sous cette forme granulée, qui facilite beaucoup leur distribution” (Anonymous, 1929).

with ammonia or with potassium hydroxide and ammonia.<sup>7</sup> (Matignon, 1930, p. 84)

He added that this was the international issue that “dominates the heavy chemical industry”<sup>8</sup> (p. 84), especially in the United-States, Germany, England, Russia, Poland, and Italy. Indeed, the question of nitrogen had been replaced by that of phosphoric acid (Hackspill, 1929). These questions were accompanied by three issues: granulation, hygroscopicity, and effectiveness (Ross *et al.*, 1927). To use the terminology of Thomas Hughes (2004), it was a “reverse salient” that chemists sought to overcome. Renowned chemists with expertise in nitrogen, such as the Frenchman Georges Claude or the Italian Giacomo Fauser (1934), tackled the problem. Matignon, a scientist renowned for his work with fertilizers, took part as well. He proposed several synthetic processes used in the composition of fertilizers. As a professor at the College de France he also organized a course, and numerous meetings, on issues affecting agriculture and fertilizers (Lestel, 2008, p. 363-367).

In the 1910s, several chemists proposed solutions regarding the ammoniation of superphosphate, notably Wilson and Haff in the US (Keenen, 1930) and Von Gerlach in Germany (Matignon, 1923). But all faced a disadvantage: retrogradation of phosphoric acid. The chemists of Compagnie de Saint-Gobain invented and put on the market in 1924 a phospho-nitrogen fertilizer named “superam”. Their “homogeneity [was] far greater than that of a simple mixture, and [their] dryness of characters and upper friability comparable to those of the best dried and ground superphosphate”<sup>9</sup> (Matignon, 1923, p. 216). In the US, the American Cyanamid Company acquired Ammo-Phos Corp, which produced “Ammophos” (phosphoric acid and cyanamide) (Haynes, 1949, p. 21-25). These lines of research highlight the competition among chemists, with national antagonisms in the background. Camille Matignon contrasted the creation of “superam” against German research which resulted in a product that was not

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<sup>7</sup> “La tendance actuelle, dans l’industrie des engrais, est d’éliminer de ceux-ci toutes les substances inertes qu’ils contiennent, substances souvent coûteuses par leur origine et leur transport, d’un intérêt nul ou insignifiant pour les plantes. Aussi est-on orienté dans la recherche des engrais concentrés, formés à partir de l’acide phosphorique lui-même, par son union avec l’ammoniaque ou avec la potasse et l’ammoniaque.”

<sup>8</sup> “domine toute la grande industrie chimique.”

<sup>9</sup> “homogénéité beaucoup plus grande que celle de simple mélange, et des caractères de siccité et de pulvéulence supérieurs à ceux des meilleurs superphosphates séchés et broyés.”

as good. Similarly, he highlighted the ways in which ammoniac was nationally specific to the US: as he said, it “has the disadvantage of a phosphoric acid concentration which is in opposition to the customs of French agriculture”<sup>10</sup> (Matignon, 1923, p. 217).

This initial line of research led to a high concentration product (highest percentage of nitrogen and phosphoric acid), which limited the use of sulfuric acid but did not remove it completely. It was only later, with the use of nitric acid, that this was achieved. One solution was the direct reaction of nitric acid with calcium phosphate, but that presented technical problems (foam caused by a byproduct of the reaction, calcium nitrate) (Gardinier, 1974, p. 84-86). The intermediate solution of Saint-Gobain chemists was to implement a process called “sulfonitrique” in which sulfuric acid transforms lime into calcium sulfate and prevents the occurrence of calcium nitrate and foam. For their part, the Etablissements Kuhlmann exclusively used nitric acid, but employed a particular highly concentrated Russian phosphate from Kola instead of Moroccan phosphate (Ross, 1931). In Europe these were known as “complex fertilizers” since at least two elements were combined in a chemical reaction.

Research was also done on combinations of potassium nitrate (NK), in particular by the German firms Thorssell and Kristensson (IO) and Kali-Industrie Aktiengesellschaft, and by Whittaker and Lundstrom of the Bureau of Chemistry and Soils in the US Department of Agriculture (USDA) (Ross, 1931). For his part, the French chemist Georges Claude invented “potazote” which became famous among agronomists and agricultural unions, as shown by the *Bulletin du Syndicat Central des Agriculteurs de Loire-Inférieure*: “science itself has not disdained to address the problem of combined fertilizer, since the great scientist Georges Claude, to whom we already owe the most elegant method of making synthetic ammonia, has also endowed us with a remarkable combined fertilizer” (Anonymous, 1934).<sup>11</sup>

But with these new fertilizers with high-concentration materials, it was also more difficult to maintain the hygroscopic properties when in powder form, which led to the development of granular fertilizers (Slack, 1967, p. 19). Fertilizers in granular form also appeared for other technical reasons: handling hazards, unstable products, and poor preservation in

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<sup>10</sup> “présente le désavantage d’une concentration en acide phosphorique qui heurte les coutumes de l’agriculture française. ”

<sup>11</sup> “la science, elle-même, n’a pas dédaigné de s’occuper du problème de l’engrais combiné, puisque le grand savant Georges Claude, auquel nous devons déjà le procédé le plus élégant de fabrication de l’ammoniaque synthétique, nous a également doté d’un engrais combiné remarquable.”

stores. The first work on granular fertilizers was carried out in the US in 1922 by the Bureau of Soils (Hardesty & Ross, 1938). For cyanamide the handling of the powder is dangerous for the fingers, so the presentation in pellet form was intended to aid its passage through mechanical distribution apparatus (Daviet, 1988, p. 596-597).

As explained by Raymond Berr (1930), ammonium nitrate and phosphate would provide excellent nutrients to produce compound fertilizers. They gave pellets of a “complete” ternary (NPK) fertilizer, as did Alvin Mittasch, head of German Oppau laboratories of BASF with the “Nitrophoska” (Thompson *et al.*, 1949). The creation of Nitrophoska in Germany pushed the Mines Domaniales to undertake research, at the request of the French Agriculture Minister, Henri Queuille (Anonymous, 1927, p. 541). One can see here, clearly, that competition between France and Germany guided the research strategies of French chemists and which would elevate them as national heroes if they succeeded. It also led them to explore other ways to limit the use of sulfuric acid. A joint subsidiary of Mines domaniales and Kali-Sainte-Thérèse, the Société d’Étude pour la Fabrication et l’Emploi des Engrais Chimiques, was created in 1928 to conduct research and industrial tests for the manufacture of chemical fertilizers derived from potash. Pierre Jolibois, professor of chemistry at the École nationale supérieure des mines de Paris, became the Scientific Director of this research society (Lestel, 2008, p. 272). One of the first results obtained was the development of a method allowing the use of hydrochloric acid produced by the manufacture of potassium sulfate for the manufacture of dicalcium phosphate (Torres, 1999, p. 78).

- *The Mechanization of Chemical Industry: The First Production Units of Complex Fertilizer Pellets*

In the compound fertilizer sector, building production units for compound fertilizer in granular form was the most promising innovation. Forming a compound fertilizer with separate pellets for each nutrient caused additional costs compared to complete granulation all at once. The French chemical groups developed their own granulation processes, but they also relied on technology transfers for techniques that were more efficient than their own processes. Saint-Gobain placed its first granulated complex fertilizer factory in Rouen (Seine-Maritime) in 1932 (Daviet, 1988, p. 589-601). In its ammonium phosphate production unit, Saint-Gobain used the American Dorr process to produce phosphoric acid and ammonium phosphate. The start of the unit was very laborious and eventually the process was abandoned in favor of the production of phosphoric acid alone (7 tons per day) (Detuncq, 1966, p. 3-8; Nielsson, 1986, p. 228-229). Subse-



quently, in 1934, an ammonium nitrate production unit was put into service to produce ammonium nitrate, but also to enrich nitrogen compound fertilizers (Daviet, 1988, p. 589-601). We see a technical system take shape here, which would develop rapidly in the 1960s in France: ammonium nitrate and complexes fertilizers. From the Société d'Étude pour la fabrication et l'Emploi des Engrais Chimiques, the Société Chimique des Potasses d'Alsace (SCPA) gave birth to the Potasses et Engrais Chimiques (PEC) plant in Grand-Couronne (Seine-Maritime) in 1929 under the direction of Marcel Massenet, to manufacture, among other things, bi-calcium phosphate. From 1933, it began producing ternary fertilizers containing nitrogen. Liquid ammonia was converted by oxidation of nitric acid, used to prepare the ammonium nitrate which, added to the chloride or sulfate of potash and bi-calcium phosphate, allowed the manufacture of compound fertilizers. Continuing his research, the technical team led by Jean Dessevre developed, in 1937-1938, a new process for obtaining a high-concentration fertilizer (38% nutrients) (Torres, 1999, p. 78, p. 104-105).

Innovation of fertilizers in granular form was therefore the result of a cluster of innovations of technological processes (Caron, 2011, p. 30): synthesis of ammonia, phosphoric acid manufacture, manufacture of nitric acid at lower cost thanks to inexpensive ammonia, manufacture of ammonium phosphate, but also mechanization of agriculture with fertilizer distribution apparatus.

- *Agronomists Change their Views on Compound Fertilizers*

In France, the control of fertilizers came from the fraud department<sup>12</sup> and the increasing demand by growers for systematic analyses by agronomic stations, which forced the industry to improve the quality of the composition of fertilizers (Roux, 1933). The agricultural engineers of the Services Agricoles Départementaux eventually came to promote compound fertilizer in preference to straight fertilizers (Gardinier, 1974, p. 100-101). In an exchange with the Académie d'Agriculture in 1939, agricultural engineers recommended them (Lenglen, 1939). The professor of École d'agriculture de Grignon, Lucien Brétignière explained that "while we still taught at the beginning of this century the prohibition of compound fertilizer, today we recognize, without question, the benefits of these fertilizers provided, of course, they are honestly made, affordable, and that the for-

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<sup>12</sup> Foundation of the fraud department in France by decree of 21 October 1907 for the application of the law of 1 August 1905 (Jas, 2000, p. 317-320).

mulas of these fertilizers are simpler and more straightforward”<sup>13</sup> (Lenglen, 1939). Agronomist Albert Demolon further described their agronomic efficiency:

recent experiments in fertilization highlighted the key idea that there is a close solidarity in cooperative action among the various nutrients. Thus the increase of nitrogen that would have brought disappointment if there had been no wider use of potash and phosphate fertilizers, and vice versa. We can therefore consider that the compound fertilizer, binary or ternary as appropriate, shall normally provide the maximum manure effect.<sup>14</sup> (Lenglen, 1939)

Chemists have succeeded in offering compound fertilizers that meet the industrial constraints of cost, transport, and storage and the farmers’ need for concentration and simplification of spreading. Now favorable to compound fertilizers, agronomists supported their approach. The way was open for a ramp-up of compound fertilizers in the 1950s and 1960s.

### **Rise of Compound Fertilizers: Chemists and Engineering Companies (1950-1970)**

- *Mechanization and Agricultural Productivism*

The trend that started in the interwar period increased in the 1950s in France: farmers wanted to simplify crop operations and searched for high-concentration fertilizers which would reduce transportation costs, handling, and spreading. The extension of motorization, reducing the presence of horses, further reduced the amount of natural/animal manure on

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<sup>13</sup> “alors qu’on enseignait encore au début de ce siècle la prohibition des engrais composés, aujourd’hui, on reconnaît, sans conteste, les avantages de ces engrais, à condition, bien entendu, qu’ils soient honnêtement fabriqués, à un prix abordable, et que les formules de ces engrais soient de plus en plus simples.”

<sup>14</sup> “les expériences récentes sur la fertilisation ont mis en relief cette idée force qu’il y a une solidarité d’action étroite entre les divers éléments fertilisants. C’est ainsi que l’accroissement des apports d’azote n’aurait donné que des déceptions si parallèlement il n’y avait pas eu utilisation plus large des engrais potassiques et phosphatés et inversement. On peut donc considérer que l’engrais composé, binaire ou ternaire suivant les cas, assure en principe à la fumure son effet maximum.”

farms (Anonymous, 1946). Agricultural authorities<sup>15</sup> strongly encouraged farmers to take this path. Industrialization discourse in farming was part of the modern and productivist postwar movement (Pessis *et al.*, 2013): “Compound fertilizer is the ambassador of rational fertilization”<sup>16</sup> (Chambre Syndicale Nationale des Fabricants d’Engrais Composés, 1952, p. 43). This product was “rational” in terms of transport cost, storage stability and ease of spreading (figure 1).

This increase in dose was made possible, in particular, thanks to progress in plant breeding and mechanization. In the interwar period, wheat varieties with long straw lacked the rigidity to withstand heavy fertilization rates. By the end of the 1940s, new varieties were selected with solid straws which would not fall due to heavy fertilization (Pambrun, 2009, p. 35). The need for ternary compound fertilizers can also be explained by the expansion of spring crops (barley and corn), which have a short growing cycle and need to receive the three primary nutrient elements together rather than separately (Chambre Syndicale Nationale des Fabricants d’Engrais Composés, 1962, p. 48-49).

This discourse of intensive agriculture was not unanimously shared and increasingly received a rough ride. At the end of the era we are examining, one of the champions of productivism in the 1950s, the French agronomist René Dumont, renounced his positions on intensive use of fertilizers (Séjeau, 2004; Dumont et de Ravignan, 1977, p. 268-270). The organic movement emerged, particularly in England in the 1930s with Albert Howard (Conford, 2002), and in the late 1960s it moved in step with the development of the counterculture (Hughes, 1989, p. 443). This movement again raises the question of natural and artificial. In France, organizations such as the Fédération Nationale des Syndicats de Défense de la Culture Biologique et de Protection de la Santé des Sols, advocate a return to the origins of organic compound fertilizers. The federation condemned the “use of all chemicals that are synthetic pesticides or mineral fertilizers and promotes the full and exclusive use of organic fertilizers and products derived from them” (Anonymous, 1974).<sup>17</sup>

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<sup>15</sup> The Institut National de Recherche Agronomique (INRA), established in 1949, the Centres d’Études Techniques Agricoles (CETA), on the initiative of farmers, appeared from 1944 (Cerf & Lenoir, 1987, p. 34).

<sup>16</sup> “l’engrais composé est l’ambassadeur de la fertilisation rationnelle”.

<sup>17</sup> “l’utilisation de tous les produits chimiques qu’ils soient pesticides de synthèse ou engrais minéral et prôn[ant] l’utilisation intégrale et exclusive des engrais organiques et des produits issus de leur transformation.”

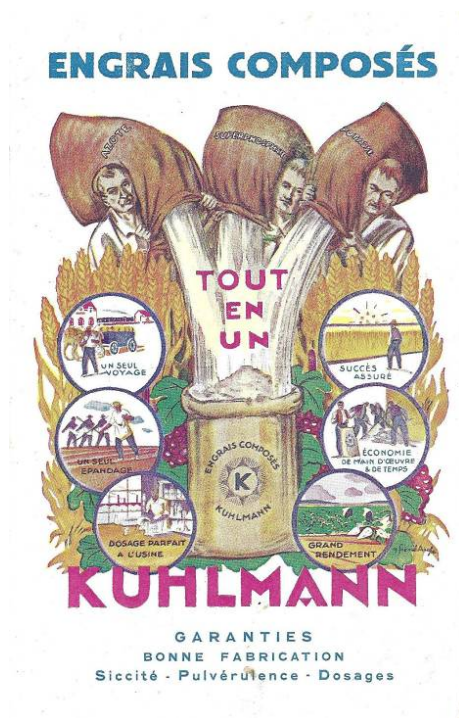


Figure 1 - Promoting the rationality of compound fertilizers of Etablissements Kuhlmann. (Source: Advertising postcard. s. d. Author's private collection)

- *Expansion of Manufacturing Units of Fertilizer Pellets Compounds*

Research on compound fertilizers which was undertaken by chemists in the interwar period led to the first production units in France just before World War II, but only fully bloomed after the war. Industrial achievements expanded due to the initiative of chemical engineering companies under the leadership of the French government and the Plan de Modernisation et d'Équipement<sup>18</sup> and using new materials such as hydrocarbons for nitrogen components (Anonymous, 1950).

Developed in the United States in the interwar period, chemical engineering took off in France in 1950 after the return of US productivity

<sup>18</sup> The Plan de Modernisation et d'Équipement was a governmental administration set up in France after 1946 to plan the economic development of the country. Its role was to coordinate the actions of private and public industries with a view to achieving the economic and industrial objectives set by the French state.

missions under the Marshall Plan (Ndiaye, 2001, p. 77). In the 1950s and 1960s, chemical companies gradually expanded their industrial research laboratories (Anonymous, 1953) and created chemical engineering conglomerates. In 1949, the Établissements Kuhlmann constituted a chemical engineering company as a subsidiary, the Société Technique d'Entreprise Chimique (STEC) (Léger, 1988, p. 130-131). They justified this creation thus: "Because of the considerable development of the chemical industry, various companies are continually called upon to use specialized design offices, with an experienced technical staff to design and implement projects related to the expansion and the creation of factories" (Kuhlmann, 1958, p. 48).<sup>19</sup> It was the same for SCPA, who in 1958 decided to create an engineering subsidiary to sell the "process PEC" manufacturing complex fertilizers, which was designed before the war (Torres, 1999, p. 222).

The manufacture of compound fertilizers, which remained the main market for smaller manufacturers who mainly produced fertilizer mixtures of organic and inorganic materials, expanded strongly in the 1950s and 1960s as large chemical groups invested heavily in the promising market for fertilizer compounds in granular form. Their chemical engineering companies provided this technical change through competing granulation processes. Apart from ammonium nitrate and potassium chloride, which are outside the actual granulation process, the two main fertilizers involved in granulation processes are ammonium phosphate and phosphate nitrate. The latter two products were aimed at different markets in the 1960s, which led to different geographical distributions, technology transfer and different competitive strategies associated with different processes. In Europe, phosphate nitrate grew more than in the United States (25 plants producing 200 to 600 t/d to 1965) with the involvement of many large chemical groups (such as Saint-Gobain or PEC in France, and Norsk Hydro in Norway) (Slack, 1967, p. 121-124). In the US, ammonium phosphate was dominant, with Dorr-Oliver processes in the 1930s and from 1959 the ammoniator-granulator process of the Tennessee Valley Authority<sup>20</sup>, which competed with, among others, the "Spherodizer" method of the Chemical and Industrial Corporation (Slack, 1967, p. 111-121).

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<sup>19</sup> "En raison du développement considérable de l'industrie chimique, les diverses sociétés sont appelées à avoir recours sans cesse davantage aux Bureaux d'Etudes spécialisés, disposant d'un personnel technique expérimenté, pour concevoir et réaliser les projets afférents à l'extension et à la création d'usines".

<sup>20</sup> In the US, Tennessee Valley Authority, a public body set up under the "New Deal" in 1933, played a major role in the development of the use of fertilizers (Sheridan, 1979).

The strong combined demand for complex compound fertilizers and nitrogen fertilizers was embodied in France in the building of major plants producing both complex fertilizers and ammonium nitrate. This massive demand induced technical changes in the synthesis of ammonia with the development of cracking processes for obtaining hydrogen from hydrocarbons (petroleum residues and natural gas), instead of from coke oven gas. It was accompanied by construction of new networks for transportation of raw materials, such as the Lacq gas pipeline network which supplies the French territory, which was deployed under the leadership of the state (Collective, 1998, p. 44). In 1963 the Société Chimique de la Grande Paroisse (Fay, 1969) started a plant in Montoir-de-Bretagne (Loire-Atlantique) to synthesize ammonia and produce ammonium nitrate.<sup>21</sup> In 1973, the plant increased its production capacity by adding a production unit for ternary complex fertilizers with a capacity of 150,000 t/y through the transfer of US technology.<sup>22</sup> For granulation it, in fact, used the "Sphero-dizer" method of the American Chemical Industrial Corporation (Slack, 1967, p. 117; Hignett, 1985, p. 255). But technology transfer was also made from France to the United States. In 1962, the US company Ortho California Chemical built a plant in Iowa to manufacture 1,000 t/d of complex fertilizers (Anonymous, 1962). This was the third Ortho factory (the first two were in Richmond, California and Kennewick in Washington State) which used nitric acid instead of sulfuric acid to attack the phosphate using the French PEC method.

The growth of large granulated compound fertilizer units around the world has been achieved through the principles of standardized workshops, a market for granulation processes and an abundant and inexpensive source of hydrogen.

- *New Formulations of Fertilizer Production on a Small Scale: "Bulk Blending"*

Apart from the big factories, small units still held on. A dual industry structure existed with a tendency towards concentration: small fertilizer mixing units on the national territory near agricultural areas and large complex fertilizer units, oriented in part to export markets. In 1965 production of compound fertilizers was provided by 224 companies totaling 293 production units. However, production by small plants was low; almost 80% of compound fertilizer produced in France came from 27 companies totaling

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<sup>21</sup> AD Loire-Atlantique, 281 W 20, Notice descriptive. AD stands for the archives of one French administrative department, here Loire-Atlantique.

<sup>22</sup> AD Loire-Atlantique, 1373 W 152.

73 plants.<sup>23</sup> This was especially the case for binary phosphate potash fertilizer (potassium slag, super-potassium, phospho-potassium, etc.). “Bulk blending” based on the intermediate product production capacity of large chemical groups, would strengthen the small units in geographic proximity to agricultural production while also providing a tailored response to the needs of the farmer.

The “bulk blending” method of manufacturing compound fertilizer developed quickly in the United States after 1955 (Slack, 1967, p. 20; Hignett, 1985, p. 5-6). It used a simple mechanical mixture of high-concentration elements in pellet form to produce high-analysis fertilizer. The materials used (ammonium sulfate, ammonium nitrate, triple super-phosphate, ammonium phosphate, potassium chloride, etc.) were all manufactured as pellets by large chemical companies. The advantages of “bulk blending” were the cost and the proximity of the farmer and his requirements, which offset some of the original disadvantages of homogeneous granular fertilizers that we have seen, namely lack of homogeneity and a tendency toward caking. This industrial model returned to Müntz’s idea of designing a custom compound fertilizer for the farmer. In the US, between 1959 and 1964, the number of plants adopting “bulk blending” went from 201 to 1536.

In France, the SCPA decided to engage in “bulk blending” for potash granulation in 1960 (Torres, 1999, p. 158-159). From 1961 onward it operated a binary phospho-potassic granulating production unit in its Strasbourg facilities. Soon after it launched commercialization of that product in partnership with Établissements Delafoy from Nantes and the SCPA production unit installed at Teil (Ardèche). The technology transfer process innovation allowed Delafoy to achieve production. The company Delafoy in Nantes appealed to the engineer Carbona at the Reno Company’s Tréport (Seine-Maritime) factory, which had developed and patented a granulation process which “constitutes a considerable technical and commercial progress in enabling not only the maintenance, but also the development, of the market for simple phosphate fertilizers and photopotassiques”.<sup>24</sup> Gradually, SCPA developed small regional units for

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<sup>23</sup> AN IND 19771633/107, Rapport de M. de La Rochefoucauld, V<sup>e</sup> Plan de Modernisation et d’Équipement, Commission de la chimie, Groupe des engrais, sous-groupe des engrais composés, avril 1965. AN stands for French national archives.

<sup>24</sup> “constitue un progrès technique et commercial considérable, devant permettre non seulement le maintien, mais aussi le développement du marché des engrais phosphatés simples et photopotassiques”, AN IND 19771633/008SCPA, Note

manufacture of compound fertilizer, designed in partnership with local players.

## Conclusion

Initially only the domain of industry and empirical expertise, compound fertilizers were subject to adulteration and fueled farmers' mistrust of agronomists. Chemists gradually intervened in this area through the role of sulfuric acid, which could make certain organic components more assimilable. Placed far downstream from process design and manufacture of the material, they mostly played a role for analysis and control. In the interwar period they took control of the compound fertilizer field from design to production. The question of straight fertilizers versus compound fertilizers became a major issue for the chemical community (both academic and industrial).

Positioned between agriculture and industry, chemists were responsible for restoring confidence in compound fertilizers and bringing about conditions for the growth of consumption. They showed that they understood the needs of farmers by making "compound fertilizer" an integrated material, combining the major nutrients (nitrogen, phosphorus and potassium acid), as well as by solving problems with the concentration, cost, handling, and storage of fertilizers. They did so, in particular, by modifying the presentation of fertilizers. French chemists in academic research, such as C. Matignon or P. Jolibois, or those closer to industrial research, such as G. Claude, were interested in the issues of compound fertilizers. Without being dominant in their research, this work nevertheless reoriented their careers as teachers (C. Matignon's conferences), or stimulated them to new careers in industry (P. Jolibois became scientific director of Potasses et Engrais Chimiques) or revitalized their industrial research (the "potatoze" by G. Claude). Finally, after World War II, the massive development of the production of compound fertilizer in granular form was permitted by the development of a cluster of innovations in industrial processes of granulation which were disseminated and implemented by chemists in chemical engineering companies, and by the availability of hydrocarbon raw materials used to make large volumes of ammonia necessary for the synthesis of nitrogen elements. But in reaching its limits, the system also distanced itself from the consumer. In contrast, the "bulk blending" production model de-



veloped in parallel addressed the cultivator's needs afresh by offering tailor-made industrial fertilizers and geographic proximity.

Over this 80-year period extending from 1890 to 1970, we saw the confrontation of actors and technical changes that have guided the evolution of several kinds of compound fertilizer in France, from a heterogeneous mixture of organic materials to an integrated product, and from a powdered form to a granular form. This evolution has taken place at the global level with the development of granulated compound fertilizer plants depending on the country. Through academic exchanges or technology transfer, chemists and chemical engineering companies from different parts of the world have contributed to this evolution. These changes were accompanied by a changing role for chemists – who have become preeminent in the fertilizer industry – and industrial structures.

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## **Chromatographs as Epistemic Things: Communities around the Extraction of Material Knowledge**

Apostolos Gerontas<sup>\*</sup>

### **Abstract**

*Automated chromatography – gas chromatography and later high performance liquid chromatography – played an important role in the transformation of chemical analysis during the 1960s and 1970s. This chapter presents the historical narrative of the production and dissemination of chromatographic technology, and discusses the effects of the automation of separation at the social and epistemic levels. Emphasis is given to materiality, not only of chromatographic technological knowledge, but also of the knowledge produced by application of this technology in research.*

*Keywords: gas (GC) and high-performance liquid chromatography (HPLC), instrumentation, epistemology of things, research technology, scientific identity.*

### **Résumé**

*La chromatographie automatisée – chromatographie gazeuse et par la suite liquide à haute performance – a joué un rôle important dans la transformation de la chimie analytique durant les années 1960 et 1970. Ce chapitre présente le récit historique de la fabrication et circulation de la technologie chromatographique et discute les effets de l'automatisation de la séparation aux niveaux sociaux et épistémiques. L'accent est mis sur la matérialité, non seulement de la connaissance de la technologie chromatographique mais encore de la connaissance produite par l'application de cette technologie en recherche.*

*Mot-clés : chromatographie gazeuse (GC) et chromatographie liquide à haute performance (HPLC), instrumentation, épistémologie des choses, recherche technologique, identité scientifique.*

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“Every scientific advance is an advance in method.”  
Mikhail Tswett (1910)

CHEMISTRY was “revolutionized” during the 20th century by the introduction of a multitude of instrumental techniques of analysis – and by the industrialization of their production, maintenance, and promotion (Baird, 2002). On the one hand, the changes in chemical analytical practices had a stark epistemological and cultural dimension, affecting not only chemical praxis *per se* but also chemical theory, world-view, and sense of meaning and position of chemistry relative to other disciplines and the world. On the other hand, the new analytical methods had a significant effect in the organizational structures of modern chemistry. As Egon Fahr was already observing in the mid-70s, while the pre-automation “classical” analytics before World War II mainly focused on reaction phenomena and chemical properties, the analytical branch that evolved after the war was carried out mainly through the utilization of physical properties of the bodies analyzed (Ettre, 2008).

In the specific case of the professional practice of analytical chemistry, the changes were of a nature and magnitude that can be seen as fundamental. The emergence of instrumental techniques at the fore dramatically shifted the focus – and, therefore, the very meaning – of analytics from “separation” to “identification” of compounds. What had been the analytical chemists’ job, as late as 1940 – namely to “separate” and “quantitatively manufacture” compounds utilizing their reaction properties – was downgraded to a job for research technicians, since the new instrumentation made this possible.

The analytical chemist of the post-war decades became a professional manager of sub-professionally educated personnel (a distinction reflected in the academic curricula) that would necessarily free him or her from what was previously seen as the “dull” work of separation. Indeed, the analytical chemist of the 1950s was able to focus on the elemental properties of compounds, in what an external observer could probably describe as a process of chemistry finally becoming a “science” – of the kind that physics was. This closing of the distance between chemistry and physics in practice, focus, and scope not only made chemical practice faster or more effective; it created an “identity crisis” too. This change of nature of course became visible to professional chemists and specialists as early as 1947, and both its “positives” and “negatives” have been commented upon (Baird, 1993).

Thus, although analytical chemistry remains the discipline that focuses on “signal production and interpretation”, (Lewenstam & Zytkow, 1987, p. 308)<sup>1</sup> the very nature of the signal read and interpreted changed after the 1940s, and, therefore, the image that each interpretation paints has become increasingly different. Indeed, we witnessed a key event in the general history of chemistry: the shift in the focus of chemical analytics from the purely chemical properties of chemically defined substances to the properties of “molecular species”. Today “molecular structures are no longer considered properties of substances; they are now the species whose identity is to be determined and which are subject to chemical classification” (Schummer, 2002, p. 202). On the theoretical-field level this process reflects the transition from “classical” organic chemistry to physical chemistry, to physical organic chemistry, and then to the actual theoretical chemistry of today.

Despite the fact that the bases of the new methods developed and utilized during this period were all set before the war, their domination of analytical praxis was only possible after the means of modern electronics and optics were available, and after a market for those methods existed at a critical level. The rapid development of related industries (pharmaceuticals, biomedicine, and food) offered the necessary market for the commercial viability of these methods from the 1950s on.

By the 1970s the literature reports two different terms that tend to describe what were considered largely different branches: *chemical analysis* (corresponding to “classical” analysis based on reactions), and *instrumental analysis* (corresponding to the “new ways”). From the same decade on, we can register an attempt to cover both terms by the much broader umbrella term “*separation science*”, with claims to a separate disciplinary status, under which instrumental analysis is actually the dominant power and classical analytics have been reduced to the status of poor relative.

In the driver’s seat in the new era, not coincidentally,<sup>2</sup> we find mostly specialists of the new chromatographic instrumentation. Barry Karger, Lloyd Snyder, and Csaba Horváth (1973) co-authored a hand-book entitled *An introduction to Separation Science*. There, although the significant differences

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<sup>1</sup> According to Yuri A. Zolotov (cited in Danzer, 2007, p. 2), the chemical, physico-chemical, and physical methods of analytical chemistry do share a basic common epistemological characteristic: “All of them [...] have the same feature: it is the dependence of signal on analyte concentration. The important task of analytical chemistry is therefore the discovery and implantation of these dependencies into analytical procedures.”

<sup>2</sup> As E. Lederer and M. Lederer (1955) wrote: “No other discovery has exerted as great an influence and widened the field of investigation of the organic chemist as much as Tswett’s chromatographic adsorption analysis.”



between different methods and processes of chemical separation are acknowledged, the authors write in the preface:

we believe, however, that common underlying principles of separation exist and that the understanding of these fundamentals can result in a fuller appreciation of the advantages and disadvantages of the specific methods. We also believe that these principles lead to the logical establishment of a field of separation science. (Karger *et al.*, 1973, p. 9)

And for building up of the necessary connections in the already-recognized fields, the authors say that

these [separation process] systems are often, beyond their practical usefulness, excellent examples to illustrate the underlying physico-chemical principles. An introduction to separation science, therefore, is an introduction to thermodynamics and transport phenomena as well. (Karger *et al.*, 1973, p. 11)

This “separation science” was then, and is still today, a term with an ambiguous definition. While it clearly contains chromatography at its current center, its claim of unifying methods based on common “physico-chemical principles”, over and through traditional disciplines, is in doubt – and was never fully acknowledged by the broader community of chemists and chemistry-related professionals. Its existence as a term however does represent, as we shall see, an important epistemic shift in modern chemistry, as well as changes of a social nature that modern instrumentation brought to the chemistry-related disciplines. On the one hand, instruments of modern chemistry have an independent epistemic value: they are “epistemic things”, as defined by H. J. Rheinberger (1997), embodying phenomena and leading through their manipulation and evolution to the production of new knowledge. On the other hand, instrumentation reconfigured the position of modern chemists inside their institutions and vis-à-vis their own professional practice.

### **Chromatography: From Bench Design to Brand Instruments**

Chromatography appeared as a technique of separation at the turn of the 20th century, created as a separation tool by Mikhail Semenovich Tswett (1872-1919) to tackle the then-current issue of chlorophyll isolation. It was recognized and canonized, after a latent period, as a chemical tool in the mid-1930s through the research of Richard Kuhn and Edgar Lederer on carotenoids and the subsequent work of other researchers primarily in

Germany (Gerontas, 2014). Both of these were critical periods for the establishment of a series of chemistry-related disciplines and sub-disciplines, the re- distribution of relative disciplinary weights and spaces, and the re-formation of older academic milieus. While the “race for *chlorophyll*” was starting on one side of the European continent in 1901, the word “*biochemistry*” was not yet officially introduced, and the discipline that today we identify with physical chemistry had only recently become autonomous from the broader chemical world.

Chromatography, as invented by the physiologist Mikhail Tswett, was a physical-chemical technique built to solve a biological problem – namely, the isolation of chlorophyll, which was considered by chemists of the day to be solvable only through traditional organic analysis. Despite being a separation technique, chromatography was created to serve the needs of the discipline of physiology: separating, but not interfering with or destroying the molecular structure (Gerontas, 2014). Thus, we could consider chromatography’s appearance as a bridge between two world-views, the strictly mechanistic-constitutional view that organic chemists held about living matter, and the more holistic one, which physiologists had to hold. Functionality of a molecule – and, therefore, its position in a biological cycle – did not need to be destroyed or altered for its separation. This evolutionary step can be considered as of vital importance for the establishment of experimental physiological chemistry and the disciplines which are today perceived as standing on it.

The technology that offered the basis for the automation of the procedure became available in the mid-1950s. The first complete gas chromatograph apparatus was built in 1952 in Austria (Hinshaw, 2003; Bobleter, 1990) and, finding a ready market awaiting, the machine gave birth almost immediately to a vibrant industry that continued expanding during the following decades.

At the 1954 National Meeting of the American Chemical Society, H. W. Patton of Tennessee Eastman Co. presented what is reportedly the first American paper in GC. In it he described a self-constructed system using an adsorption column in the elution chromatography mode, an inert carrier gas, and commercially available thermal conductivity cells that played the role of the detector. Another person present at the meeting, L. V. Guild of Burrell Co., realized the possibility of changing this setup into a full GC apparatus for commercial production. The new instrument was announced next year, under the commercial name Kromo-Tog Model K-1 (Ettre, 2008). The machines that followed shared all the main characteristics that made gas chromatography successful: they were user-friendly and versatile – planned to be useful from the very start. They did not de-

mand deep knowledge of chromatography to produce data, but they could grow together with the experience and practice of their user (and expanded with the purchase of peripherals and applications). Quite importantly, they did not demand the user and the manager of the data and the organizer of the research to be the same person.

By the end of the 1960s gas chromatography was the analytical method most considered as dominant among all the available methods of instrumental analysis. As a method, GC was from the beginning characterized by its protean abilities: capacity of analyzing samples across a broad qualitative range, easy adaptation for preparative work, and the possibility of use on different scales of quantity and precision from the miniscule to the mass industrial. Probably a more important characteristic for our subject, however, was the complexity of the machinery necessary to perform all these functions with minor adaptations. In a single apparatus, by the mid-1950s, micro-column technology was being used for adsorption, while ultra-sensitive sensors of different kinds were combined with pumps, pressure controllers, and micro-furnaces – all coupled to printing machines and the necessary lamps and switches. Apparently, GC was a chemical creature that demanded much more than chemistry to live. All these “externals” to the technique were built upon theoretical constructs that, even if they offered a rather crude description of the phase kinetics in the machines, were effective enough to support the stone-upon-stone creation of functional apparatus. Quite importantly, as figure 1 demonstrates, the particulars of chromatography were decisively hidden from the view of its users and essentially black-boxed.



*Figure 1 - Fe&M Model 700 Dual-Column Gas Chromatograph, ca. 1961  
(Source: Courtesy of the Chemical Heritage Foundation)*

The fact that the production of the apparatus would have to be delegated outside, away from the chemical laboratory *per se* was only expected. The usefulness of the machine, its potential as a product in a more or less secure market, coupled with the possibilities of variations of technology that would act protectively against patent restrictions were sure to attract companies with a relative know-how in one or more of the technologies involved in the complete apparatus. Meanwhile the delegation of this work to industry at the same time made the machine more available to interested researchers, and afforded a drive towards standardization which could not be reached through the alternative “do-it-yourself” strategy. Although this process is largely familiar to any chemist nowadays – who usually has an inbuilt psychological distance from the instruments that he or she utilizes, formed already from his early years of study – it was a relatively unexplored path in the 1950s,<sup>3</sup> and one that would show some unexpected dimensions.

On the one hand, industrial players not only utilized expertise transferred from academia, they also built significant R&D structures themselves, which were soon to play an important role in the evolution of the instrumental culture of modern chemical research. On the other hand, the “outsourcing” of the construction of GC apparatus to industry turned these tools into commercial objects like any other and created a vibrant market. The companies had the understandable motivation to compete for control of this market, not only through improved technology and products, but also through service structures, advertisement, “lobbying”, and “special relations” with the “clients” – in this case, universities, hospitals, public institutions, states, etc.

The first commercial steps resulted in the rapid expansion of available technology, the multiplication of available instruments, and the expansion of available solutions suited to an increasingly larger proportion of research requirements. The second phase had effects which were more pronounced in the long-run. After all, since the primary interest of the

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<sup>3</sup> Several of the companies that participated in GC production (especially in its first commercial steps) were companies that had built their technological bases in instrumentation – mostly optics and electromagnetism – and their connections to academia during the World Wars (mostly during the 2<sup>nd</sup>, but not only) or by inter-war momentum and incentives (which included the Great Depression). To offer examples, the Varian brothers built their first klystron at Stanford University with the help of Prof. William H. Hansen, while aiming “to invent a source of strong microwave signals in order to improve air navigation and warn of potential Nazi bombing raids” (Varian Associates Edition, *50 years of Innovative Excellence*), while Burrell Corporation’s interest in gas analysis and adsorption originated in the World War I effort concerning gas masks and chemical warfare.

companies was expansion of the available market, the proportion of researchers working with GC instruments had to be raised. This could be achieved only if the “practice” of gas chromatography were disconnected from its theory and the connected understanding of the technique, in a process that history of technology was to observe several times since.

By the end of the 1950s, the industrial editions of guides to “practical” gas chromatography would multiply, soon to be followed by relevant courses too. Industry offered not just “practical” solutions to already existing problems of research, it also “suggested” problems that could be solved by utilization of GC, and tutored young chemists (and not only chemists) in how to “practically” utilize gas chromatography apparatus. Side by side with the manuals of the machines, industrial guides appeared, offering tutoring in their use. Courses were planned and offered on industrial grounds and at universities (but not run by university personnel), while advertisements in specialized journals of analytical chemistry – first in the US, later also in Europe – made a special point of the “simplicity” and the speed of the new machines. The “practical and convenient” character of the technique was aggressively promoted by the interested companies as a strategy of widening their available market and their percentage of control over it (Gerontas, 2013).

This process of disconnection between theory and praxis was of a magnitude (and of a suddenness) that disturbed more traditional chromatographers, not least because it significantly weakened their – then newfound – claims that chromatography was a “*scientific field*”, distinct from the other fields of chemistry (Wixom & Gehrke, 2010). While the expansion of the applications of GC through the chosen industrial strategies was indeed rapid, this very expansion had significant effects on the grounding of these very applications in solid theoretical facts. The comprehension level of the newly expanded pool of users of the technique was on average lower – and a significant part of the “science of chromatography” was being transformed into an empirical “craft”.

For a concise view of the community’s complaints and concerns, itself a compilation of similar concerns over probably one and a half decades, we find for example an editorial in the specialized journal *Chromatographia*, written by L. Szepeszy (1970, p. 253) under the title “Software must be developed”:

[T]he instrumentation in gas chromatography and in data processing i.e. the hardware, has made a very fast progress. Will the development of basic knowledge and theory, i.e. the software, keep level with that? I think the answer is no and the gap will be ever wider. In my opinion we can hardly claim at the present time, that chromatography is an exact science. We have

insufficient basic knowledge for the description of the elemental processes of flow, diffusion and mass transfer taking place in a chromatographic column. [...] We are now witnessing a development in liquid chromatography similar to which took place in gas chromatography in the fifties. The hardware for efficient application of liquid chromatography is making fast progress.

In a similar tone, the noted chromatographer V. Heines complained in 1971 that, concerning the theory of chromatography, “there has been no fundamental breakthrough since 1944” (Heines, 1971, p. 280-281). Indeed the “general theory of adsorption” which Tswett (1906a; 1906b, p. 238) imagined for his original chromatography not only had not materialized, it had become somewhat of an impossibility. If in the early 1940s, J. Norton Wilson (1940) and Don Devault (& Libby, 1943) wanted to write a “theory of chromatography”, by the end of the 1960s the only interesting aim for researchers was writing surveys concerning the “theories” of chromatography – loosely using the word “theory” to mean a multitude of models describing optimal molecular kinetics. The veteran chromatographers were concerned: not only about the “software” of the already existent and successful gas chromatography, but also about the fact that the then new-born high performance liquid chromatography was following exactly in GC’s steps.

### **Social and Epistemic Hierarchies: Turning Liquid Chromatography to High Performance**

The most persistent shortcoming of GC, and the one most bound to this technique’s very nature, was the fact that not all the analyzable substances can be readily vaporized. Even among the ones that can, not all of them can be vaporized without significant losses, damage to their molecular structure, or even production of unwanted by-products. This holds true generally in the chemistry of organic macromolecules; but it becomes crucial in the chemistry of biological substances, where the functionality of a molecule in a process is as important as the isolation of this molecule *per se*.

The new focus on protein structures and their newly comprehended economic significance in the 1950s attracted the attention of several researchers in the chromatographers’ community. The then developing ion-exchange chromatography offered the basis for what was in fact the first LC instrument. The amino-acid analyzer of S. Moore, W. H. Stein and D. H. Spackman – a direct result of research funded, organized, and executed at the Rockefeller Institute for Medical Research – was first described in 1958 and entered commercial use one year later (Ettre, 2008;

Moore *et al.*, 1958). Despite the fact that the amino-analyzer was indeed the first instrumental LC, it was still a long way (and almost a decade removed) from the possibility of an actual high-speed liquid chromatograph. It was absolutely specialized, and the technology available at the time gave no possibilities for expansion of its scope. Its creation however occupies a position in this narrative for two main reasons: it was the first instrument utilizing LC that actually worked, thus giving an encouragement to researchers in both academia and industry who were thinking of taking this course; and it managed successfully to enter mass production, proving to the interested industrial players that a market-to-fill indeed existed.

When the actual liquid chromatograph appeared, it was less a result of automating liquid chromatography, and more an adaptation of the gas chromatograph to liquid phases – despite the physical difficulties that such a transformation had. Gas chromatographs were immensely successful. They had set the standard for what should a chromatography apparatus be able to do, and – most importantly – around them there was already a network of specialists, companies, institutions, and journals that had both the interest and the funds to expand. Not surprisingly, as the noted chromatographer Istvan Halász noted in retrospect (Halász in Kirkland, 1971, p. 211): “Most of the workers developing high-speed liquid chromatography were outstanding experts in the field of gas chromatography who tried to ‘translate’ and to apply their theoretical knowledge and experimental skill to this new field.”

By the summer of 1965, Csaba Horváth and Sandy Lipsky at Yale had a full instrument that could actually go to the production line, if not for the fact that its parts – especially the columns of the newly created packing material – were not yet individually in production. The machine was presented at the 6th Symposium for Gas Chromatography (September 1966 in Rome, Italy). However, Horváth and Lipsky decided that since they still had work running concerning the behavior of nucleic acids in the apparatus, they would proceed with the presentation of an interim report – a full paper would have to wait. The final system, as described in a publication of 1967, contained a Hitachi-Perkin-Elmer Model 139 UV-Vis spectrophotometer for detection with a 5 $\mu$ L cell, which was individually constructed by Horváth himself from a Swagelok GC fitting (Ettre, 2008). Csaba Horváth was the godfather of the new apparatus. The “P” in HPLC initially stood for “pressure but was later replaced with the word “performance” – which probably had a better ring in an era quite fascinated by performance.<sup>4</sup> Later,

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<sup>4</sup> The reason for the change from “pressure” to “performance” is unclear; and still in several languages the name of the HPLC apparatus is translated from English as

in the 1970s, and due to the initial investment that was necessary for a new HPLC machine, the chromatographers' community started joking that the "P" in HPLC stands in reality for "price". After all, the new machines were significantly more expensive than almost any other piece of equipment that a laboratory could have (Gerontas, 2014).

### **To Build the Science of Chromatography or the Science of Separation**

The networks that gas chromatography initiated were the primary instruments of knowledge transfer and education of the new specialists of the field of instrumental chromatography (and, partially at least, "separation science") – specialists who themselves were no longer definable through the previously acknowledged disciplinary barriers. From the 1950s on, the new group of specialists involved people from almost every field related to chemistry, and some that indeed had nothing to do with chemistry altogether. In the new, growing forest, chemical engineers, mechanical engineers, pharmacologists, physicians, electricians, and mathematicians could all find a niche and, while finding it, re-define their own selves as "chromatographers" and specialists in the new techniques. The borders separating the "natural" categories of knowledge as they were represented by the existing scientific fields of the time proved to be too thin in all cases of chemical instrumentation – and in the case of chromatographic instrumentation too.

Quite importantly, the mechanization of chromatography created for the first time a distinction between the "chromatography-users" and the "chromatography-producers". Not all the new "chromatographers" were in a position to understand the technology involved in the new machines even down to the basic level, and not all of the producers of this technology were actively involved in any kind of research other than the production of the technology. With the appearance and expansion of chromatographic apparatus, an important number of chromatography specialists were now "research-technologists" (Shinn, 2002; 2004). The term "research technologies" should be taken to mean the instrumental-technological means for research which operate in the grey zone between "science" and "technology" without explicitly belonging to one of the two, and – even when specia-

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high *pressure* liquid chromatography. Horváth stated at least once (Ettre, 2008) that the initial name gave the impression that pressure was the only difference that the new apparatus had with traditional column chromatography. Yet there were other factors, such as offering super performance. However, considering the fact that it is indeed high pressure which is the heart of the HPLC machine, it might just be that the word "performance" had a better and more market-oriented sound to it.



lized – they serve academic research equally as well as industry, forensic services, the police, the military, metrology, technical and engineering players, or whoever else might need this service.

Since there was no direct correspondence between the new technologic means for practicing chromatography and an established field of study, while the design and construction of the new machines suggested multi-disciplinarity, automated chromatography failed to find a position in the standardized academic curricula (Gerontas, 2013). It was not purely chemical enough to be taught in a chemistry department, it was useful to biology, medicine, pharmaceuticals, and of course, dependent on chemical engineering and all the fields that were connected to it, but it did not directly belong to any of them. Instead, automated chromatography could find a position in the already existing universe of instrumentation and the chromatography specialists could carve a corner of it for the sake of their self-identification. Since the 1930s research-technology had migrated massively from Europe to the United States and circuits, hubs, and networks of instrument specialists and companies had appeared quite quickly. Chemical instrumentation (or more correctly instrumentation for chemistry) had its own and important niche in this environment, visible, but not in any case independent from, the broader instrumentation field which as a whole had its own big field-representative.

The Instrument Society of America – later renamed the International Society of Automation (ISA) – was founded in Pittsburgh in 1945, as an attempt to unify the numerous local organizations of a similar kind in the US. It soon became an increasingly international body with members all around the world (a fact that more than anything signifies the need for such an instrument-specialized society and its lack elsewhere). Being almost from the start the single non-directly-industrial player broadly involved in education and certification of technicians and users of instruments, it played a significant role in forming the scientific “under-class” of laboratory technicians that fueled the rapid expansion of laboratory automation in the US. Designed from its very inception to be interdisciplinary in nature, it soon exceeded by far the subject of instrumentation and came to prominence worldwide in the more general field of industrial automation. The society published (and publishes) several journals – of which the most relevant to chemistry are *American Laboratory* and the *International Laboratory* – as well as books and digests, and provides courses, training and certification to professionals in selected locations all through the US.

The subjects and the organization of the material of the published digests of articles coming from the journals and conferences of ISA offer a picture of the diverse interests and sub-groups inside the society, and, pos-

sibly, the different weights that each group held and the positioning of chemistry among all the interest groups. The petroleum-related subjects seem to have been a standard heavy-weight interest of a significant part of the ISA membership especially in the late 1960s and early 1970s (the existence of the annual National Chemical and Petroleum Instrumentation Symposium and the full publication of all its papers from 1960 on indicates that). Analysis instrumentation had its own weight; there were indexes of proceedings from 1956, *American Laboratory* and the *International Laboratory* stably focused there, and there were annual anthologies of papers from these journals. However, not all instrumental techniques carried the same weight.

Many of the users and virtually all of the producers of the technology of gas chromatography were able to find a position in the broad range of people involved with ISA and modeled their own smaller groupings and practices according to the ones that ISA maintained. Chromatographic journals appeared, and conferences, meetings, and symposia of specialists were often organized. The means that ISA had devised for the promotion of instrumentation and automation among the interested publics became the ways of the chromatography crowd and the involved businesses as well, with the businesses at the steering wheel. Instrument-making companies organized workshops and teaching events, published handbooks, had their own training centers, and participated in the funding of groups and networking activities such as symposia and conferences that did a lot both to strengthen the shared identity of chromatographers and to attract new talents to chromatography. Industrially organized workshops and organized training at the course-centers of the big instrument producers became the primary means of education of the next generation of chromatographers.

Virtually all the big instrument-producing companies had their own course-centers and organized their course material primarily around the models that they were commercially producing. A survey of the material left from the Perkin-Elmer Corporation's<sup>5</sup> division in Germany reveals that the workshops organized and the training offered to new practitioners of

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<sup>5</sup> Perkin-Elmer, a company with a deep background in optics, was one of the corporations that entered the gas chromatography sector early, and became almost dominant in it in the 1960s. The background in optics obviously played a significant role in this success story. After the Second World War, the company took over a previously German owned factory at the Bodensee. There, next to the training centers and the production units, the company also operated its own museum of instrumentation. The greatest part of the archive material from this museum and a big part of the instrument collection are currently in the possession of the Chemical Heritage Foundation.

chromatography were rather diverse in character. Slides, demonstrations, short lectures, together with notes about specific issues and applications of the machines constituted these courses and workshops, and the relative weight of each medium often changed. However, all of them did share one basic characteristic: they were explicitly practical. The aim of the training of the courses and the workshops was invariably not to substitute “scientific” training but to create “plug-and-play” (to use the personal computer terminology) users of chromatographic machines as quickly as possible. Thus, in the course-material of Perkin- Elmer surviving from its Bodensee course-center, the material referring to issues concerning chromatographic theory is virtually non-existent. Similarly, the (hand) books that were produced by the company also focused on the practicality of the apparatus and not the underlying principles.

Furthermore, it was through the active advertising of new machines and methodology of these companies that more new chemists and chemistry-related specialists came into contact with the newly available analytical technology. Therefore, despite the absence of any means of formal training of any kind in the new automated chromatography, the dissemination of the technique, the multiplication of its possible applications, and of its practitioners grew in an explosive manner.

As the noted chromatographers Calvin Giddings and Roy Keller (1965) noted in *Advances in Chromatography*, the “explosive growth” of the field had already made it “difficult for any single individual to maintain a coherent view” of its progress. Between 1958 and 1963 the specialized *Journal of Chromatography* “swelled” from 563 pages to 1,698 pages and “the 1964 volumes contained 2,300 pages, an increase of some 300 % over 6 years,’ while the 1965 volume was expected to reach 2700 pages. As they continue,

Hais and Macek in their bibliography of paper chromatography covering the years 1943-1956 report 10.290 references. A continuation, which covers the period 1957-1960, lists 8.300 more. Preston as of October, 1965, published about 11,400 cards reporting papers, books, reviews, meetings, etc. that have appeared since the inception of the method. (Giddings & Keller, p. ix-x)

This “explosive” growth<sup>6</sup> of articles, publications, books concerning the chromatographic techniques reflected the equally explosive magnifica-

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<sup>6</sup> The term “explosive” has been repeatedly used by practitioners of chromatography in personal communications with the author—which may offer us an idea about how they perceived the rapidity of the expansion of their field. Quite interesting is the fact that chromatographers of different decades were each using the term for

tion and generalization of the application of chromatography and the continuous flow of new recruits to the ranks of chromatography specialists. A technique that was initially built for application in the physiological chemistry of the early twentieth century was transformed through mechanization into a broad cluster of techniques that could be applied in a variety of sub-fields of the analytical plateau: in research or in industry, in pharmaceuticals or radiochemistry.

This was the vibrant reality: the specialists of chromatography (and around them the specialists of all separation techniques) had their conferences, their journals, funding, companies, unions, and institutions. Research on the improvement of automation of research was a more than valid field and “making the fortune for many people”,<sup>7</sup> while the chromatography-producers were building up their common identity, narrative, and reproduction mechanisms (Gerontas, 2013). Giving to this identity a disciplinary name however, and demarcating it from other identities active at the chemistry-related plateau was not that easy – and still it is not fully resolved.

One suggestion came in the form of a claim that there was an independent “science of chromatography”. According to this claim (explicitly voiced in the title of the *Journal of Chromatographic Science* since 1963 and numerous publications through the decades up to today), chromatography is far more than a technique (or even a cluster of techniques) for chemical separations. Instead, chromatography is a scientific discipline, a phenomenon or a cluster of physical-chemical phenomena with numerous practical applications. As such, chromatographic science has chromatography as its theoretical epistemic object, while chromatographic applications (instrumental or not) have a double function. They are research techniques outside chromatographic science *per se*, and simultaneously the epistemic objects and the experimental procedures inside the field.

A competing suggestion was that chromatography, as a cluster of techniques, shared more with the other analytical techniques used for chemical separation than with anything else. According to this view, as mentioned, there are physical-chemical principles which are common and underlying for all the mixture separation techniques such as chromatography, electrophoresis, distillation, crystallization; etc. Thus, all of these should be included in one, unified “science of separation”. This term was

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their decade: so, for Ettre, Giddings and Keller there was an explosion in the 1960s, for Molnár in the 1970s, for current chromatographers in the biotechnology era of the 1980s, etc. If nothing else, that signifies that the “explosion” was a durable one.

<sup>7</sup> 2016 private communication of Jack Gill to the author.

significantly more widespread than the “science of chromatography”, as it was supported by the existence of a number of journals and academic curricula after the 1980s. The “science of separation” was also easier to fit into the more traditional sense of the discipline. After all, such a separation science would at least be reducible to an accepted physical-chemical “theoretical” basis. Yet, the term never became catchy enough for such a discipline to be fully recognized in the traditional sense.

On the one hand, this instrumentation science could not be fully grounded to theory. A great part of the instrumentation knowledge is never reducible to words and remains strictly “praxical” in nature, at least according to the meaning given by Heidegger (1954; Ihde, 2009). It is only transferable in the form of packets of technology: modules and whole instruments. On the other, the organizational structures of the instrumentation specialists remained at a pre-academic institutional stage, or a pre-disciplinary status (Hacking, 1983).

### **Instrumental Processing: Knowledge Production and Materials Distribution**

As mentioned, the chromatographers were *de facto* separated into two different categories: the chromatography-users, who did not need to have deep knowledge of the technology involved to practice chromatography; and the chromatography-producers, the research-technologists *per se*, who were the main motors of the development of new instruments, applications, and solutions.

For the first category of chromatographers, the technology was effectively black-boxed. This fact made the reproduction and continuous expansion of their class an easy matter. No academic curriculum was necessary, no elaborate scientific handbooks and training. The practical training at the industrial course-centers, apprenticeships with previous users of the machines, coupled with active assistance from the Application Groups of the instrument makers were more than enough for the machines to be immediately useful to their buyers. User-friendliness and fast problem solving were – for the basic and routine users – significantly more important than deep understanding of underlying principles.

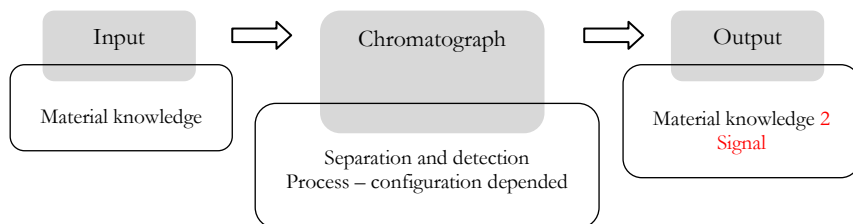
The type of knowledge that came to these consumers and which they put to use was largely “praxical” in nature with, however, significant episodic contributions to the outcome of their researches. Instruments effectively altered the world being observed (and the type of observations possible), creating thus a “different texture of the world” (Baird, 1993, p. 270). Practically speaking, the snapshots of reality offered by instruments could

be utilized as reality themselves – a radical and revolutionary identification in its own right. Since modern science cannot function without viewing the world through the technological window, the modern scientist can only perceive as objectively real what is represented as such by his or her equipment. Not surprisingly, this new objective reality built by instrumentation is highly related to the ability of the modern scientist to imagine reality through the instrumentation-generated snapshots of it. That is, the objective of this reality is easily turned into the mathematically supported products of the subjective of the scientist. As Alfred North Whitehead claimed :

The reason we are on a higher imaginative level is not because we have a finer imagination, but because we have better instruments. [...] a fresh instrument serves the same purpose as foreign travel; it shows things in unusual combinations. The gain is more than a mere addition; it is a transformation. (quoted by Ihde, 2009, p. 46)

Returning to the definition of chemical analysis as the discipline which receives and interprets a signal from states of material knowledge, chromatographic apparatus did not only alter the sensitivity or the speed of separation processes; they gave researchers access to new types of material knowledge that could readily be fed onto next-level processes. As we can see in the representation of figure 2, if the original mixture (to be separated) in a physical equilibrium state is the initial material knowledge of the separation process, then the collapse of the equilibrium by the chromatograph could be perceived as producing a new type of material knowledge next to the signal, which can be further used in the next levels of analysis and/or synthesis. Thus, in the analytical process, a chromatograph (of any kind) becomes an essential black box: its input being material knowledge, its output the signal (expressed by a chromatogram) and the separated substances (new material knowledge).

The difference between the two types of material knowledge in this process could be called the “epistemic contribution” or “epistemic content” of the chromatograph – with the chromatograph itself being essentially a black-boxed information-producing automaton for its base user. Since the epistemic contribution of the machine comes from the destruction of a physical equilibrium, it can for any given process be expressed in entropy units and/or information units.



*Figure 2 - The chromatographic process, with the apparatus as a black box  
(Source: picture processed by the author)*

Next to (and “over”) the basic routine users of the apparatus, however, was the “higher class” of the producers and the super-users of the machines. This group needed a deeper understanding of both the principles of chromatography and the technological laws which made the machines possible. Since academic pre-graduate training in chromatographic instrumentation remained rudimentary well into the 1980s, the training of this class was also dependent on apprenticeships and seminars – albeit at a significantly higher level. Lineages of research-technology producers appeared, great names of the field being the doctoral supervisors of the next generation of great names and the collaborators both in the academic and industrial sectors of other great names. Quite often, these personal relations would also take an “ethnic” and personal character, with lineages of researchers containing an important number of people of the same nationality, even while extending over different countries or continents. The most notable example of this was the “Hungarian School” of chromatography. The Hungarian sage of gas chromatography Halász was, in Germany, the supervisor of the, also Hungarian, father of HPLC Horváth, who was the childhood friend of the Perkin-Elmer senior scientist Ettre. The latter played an important role in bringing Horváth to Yale, where he constructed the first HPLC. There Horváth and another Hungarian, Molnár (who was sent to Yale from Europe by Halász too), developed the solvophobic theory of chromatography (Gerontas, 2013).

If this setting looks pre-disciplinary or a-disciplinary (or even pre-scientific) in nature, it should be remembered that these are exactly the characteristics often seen in periods of fast “revolutionary” changes as Hacking (1981, 1983) suggests. During these periods traditional institutions are re-configured, while a number of new groups appear and attempt to carve their niche – most probably starting their organization from the traditional

“guild” forms and structures. Specifically in analytical chemistry, the introduction of instrumentation brought with it an identity crisis which made the setting even more fluid (Baird, 2004, p. 99-103).

While the formal structuring and recognition of a field of “separation science” (or “chromatographic science”) was lacking, mostly because of its absence in academic institutions and curricula, this class of chromatographic instrument producers was really producing new knowledge in the form of technological packages and modules. Interestingly, in this process chromatographic apparatuses held more than one position, often simultaneously. Chromatographs were quite often the products of the process, but they were also the objects of experimentation; while, also often, the final research products were new applications, physical-chemical parameters, peripherals, and modular adaptations (Gerontas, 2014). What all the products shared was the essentially material nature of the knowledge produced, and the similar materiality of the knowledge transferred and distributed to the consumer class.

## Conclusion

Since separation process lies at the basis of any chemical process (being the first essential step), its automation after the 1950s had crucial effects on the overall practice of chemistry. The chromatographic apparatuses – initially GC, afterwards HPLC too – played a significant role in the transformations that are usually described by the term “instrumentation revolution” and stand, even today, at the center of any laboratory (either in their initial forms, or as hybrid apparatuses embodying other processes besides chromatography).

Reforming the practice of separation meant the subsequent reformation of the stratification of the laboratory micro-society and its relation to external players. The new laboratory, after the 1960s, was significantly more dependent on the logistical and technical support of the instrument makers than the laboratory of the past. Furthermore, the distance between managerial chemists and the laboratory technicians and personnel became more pronounced and more significant, socially and economically.

The new chemist does not only have a psychologically inbuilt distance from his or her instruments, but also a practical one: while he or she is responsible for the management of the laboratory in ways which were not necessary in the pre-instrumentation era, there is no longer a need for deeper involvement in the experimental procedures and setup per se. A great part of these procedures is automated; another great part is computer-modeled and simulated. The data is presented to the chemist already trans-



lated and ordered, by both machines and human technicians. Finally, a great part of the interpretation of these data can be semi-automated based on databases of accumulated knowledge of the past.

In practice, the introduction of the analytical instrumentation liberated a great amount of “creative force”, while at the same time assisting in reinforcing a type of micro-social stratification in the laboratory. As a result, highly qualified chemists take managerial roles, occupied primarily with planning of research and experimentation. On the other hand, a significant part of the work which was once tied to analysis – sample preparation, analysis per se, statistical processes, basic data interpretation and classification – is delegated to computers, students, and technicians.

For this new laboratory to exist, as we have seen, chromatographic apparatus had to be stripped from their theoretical content. Modern analysts utilizing their instruments for their research cannot always be sure about their functions (both in technical principle and in diagnostics). Thus, occasionally the act of collecting and processing the data that these instruments supply can be an act of sheer faith on the part of the scientist involved. Not knowing the details of the technology, heavily dependent on spare parts and technical assistance from “outside,” the modern scientist has been trained to use the high-tech equipment of his or her laboratory, but has often not been educated to do so.

In retrospect, this distancing between practice and academic theory had positive effects in the fast innovation, dissemination, and multi-adaptation of analytical technology, which significantly increased the epistemic output of virtually every chemistry-related discipline. Indeed, it could probably be argued that the weaker the “scientific” and theoretical background necessary for the functioning of the chromatographic apparatuses, the more effective and broader their application was. Predictably, this weakening of the cognitive element of the chromatographic machines did bring a strengthening of the cognitive element of the chemistry-related fields – that is, a strengthening of the cognitive element at a higher level of knowledge.

In many ways, chromatography as a case which can be examined next to other cases of technological knowledge and dissemination, some of them more known and notable due to the more generic character of the technology involved (i.e. computer and internet technologies). In these cases, groups of interested technologists (in the case of computers, enthusiasts of the Silicon Valley type) side by side with industrial players were the primary generators of both the knowledge and the translation-transfer processes towards a wider public. In those cases, too, we can observe the separation between the producers of technology and its consumers, accom-

panied by a weakening of the cognitive element of these technologies at the consumer level.

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## **The Exotic Glasses of Rennes (France): Local Knowledge-Making in Global Telecommunication**

Pierre Teissier\*

### **Abstract**

*This chapter tackles the question of local knowledge-making in changing scientific and economic environments in the field of advanced materials. It relies on a case study at the University of Rennes, in Western France, where the chemistry laboratory of Jacques Lucas conducted a program on non-oxide glass materials from the 1960s onwards. The chapter aims at explaining how the local production of these “exotic glasses” in Rennes was both shaped by a bench culture of solid-state chemistry and international R&D supported by the telecommunications industry. This case exhibits how research on materials was organized by a transatlantic division of labor in the Western world.*

*Keywords: materials science and engineering, solid-state chemistry, glass materials, differentiation of labor, bench culture, scientific disciplines, telecommunication R&D.*

### **Résumé**

*Ce chapitre aborde la question de la production locale de connaissance dans le domaine des matériaux, soumis à un environnement scientifique et économique changeant. Il s'appuie sur une étude de cas à l'université de Rennes (France), où le laboratoire de chimie de Jacques Lucas a conduit, à partir des années 1960, un programme de recherche sur des « verres exotiques », dépourvus d'oxygène. Il vise à expliquer comment la production locale de matériaux originaux à Rennes a été façonnée à la fois par la culture de synthèse de la chimie du solide et la R&D internationale des télécommunications. Ce cas montre ainsi que la recherche sur les matériaux a été organisée dans le monde occidental selon une division internationale du travail de part et d'autre de l'Atlantique.*

*Mots-clés : science et ingénierie des matériaux, chimie du solide, verres, différenciation du travail, culture de laboratoire, disciplines scientifiques, R&D des télécommunications.*

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THIS CHAPTER tackles the question of local knowledge-making in changing scientific and economic environments in the field of advanced materials. It relies on a case study around the University of Rennes, in Western France. There, a group of chemists from the laboratory of Jacques Lucas conducted a program on exotic glass materials from the 1960s onwards. The chapter aims at explaining how the local production of glass materials in Rennes was both shaped by a bench culture of solid-state chemistry and an international research and development (R&D)<sup>1</sup> environment which fostered optical fibers for the building of worldwide telecommunication networks. This case exhibits how multinational companies and national policy-makers organized a Western division of scientific work, by relying on local disciplinary opportunities such as Rennes to provide brand materials for the booming internet bubble. The techno-economic dynamics of telecommunications gather a wide diversity of agents from start-up to multinational companies, from academic researchers to financial investors, from materials to instruments and theories.

The historical complexity of such a case can be grasped through three types of analytic literature. The first type is the study of scientific practices in local contexts, including laboratories, which developed from the late 1970s onwards in Sciences and Technology Studies (STS). This “practice turn” shifted the attention of scholars from universality to locality, from explanatory frameworks to descriptive approaches and from the articulation of causalities to the mobilization of resources (Merz & Sormani, 2016, p. 1-9). Second, the case fits what H. Etzkowitz and L. Leydersdorff (1997) labeled the “triple-helix of university-industry-government relations”. Contrary to the “practice turn”, this second STS trend tends to over-estimate the global aspect at the expense of national determinisms and local differentiations, as recalled by T. Shinn (2002). Contrary to the first two types of literature, the third one, on industrial policies and science poli-

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<sup>1</sup> Here is the list of the acronyms found in the chapter: AT&T (American Telephone and Telegraph), CEA (Commissariat à l'Énergie Atomique), CNET (Centre National d'Étude des Télécommunications), CGE (Compagnie Générale d'Électricité), CNRS (Centre National de la Recherche Scientifique), DGA (Direction Générale de l'Armement), GNP (gross national product), MSE (materials science and engineering), NATO (North Atlantic Treaty Organization), NOGS (Non Oxide Glass Society) NTT (Nippon Telephone and Telegraph), OECD (Organization for Economic Co-operation and Development), R&D (research and development), STS (science and technology studies), STL (Standard Telecommunications Laboratories), UK (United Kingdom), US (United States).

cy for innovation and, convincingly elaborates mechanisms for national institutions to act, at the expense of local and global aspects.<sup>2</sup>

Thus, none of the three mentioned types of analytic literature provides a coherent theoretical apparatus that would encompass all the aspects of the historical case of Rennes. However, each of them points to one relevant scale of analysis: national administrations for science policy for innovation; specific places such as laboratories or start-ups for the “practical turn”; and global networks for the “triple-helix”. By following the glasses of Rennes over six decades (1960s-2010s), the chapter successively investigates these three scales of knowledge-making. The first part shows how national policy-makers shaped different *disciplinarity* for materials research, which organized a division of labor between Europe and the United States during the Cold War. The second part analyzes the local reconfiguration of research in Rennes, where the synthesis of non-oxide glasses at the bench and their mobilization by the telecommunications industry reshaped the practices of solid-state chemists towards a hybrid culture. These glasses were said to be “exotic” since they deeply differed from the mainstream glasses made of silica, a silicon oxide. The third part explores the “elsewhere” where bench materials would become brand products: the transnational triple-helix devoted to the building of fiber networks in competition with satellite communication. The fourth and last part goes back from brand to bench in a time of economic crisis to question the cultural changes in the knowledge-making of solid-state chemists through their connections with the telecommunications industry. The circulation of knowledge, instruments, and materials through the different scales of activity (local, national, global) provides a means for scientists to reshape their initial culture through the mobilization of economic, political and technological influences. Thus, the articulation between circulation and differentiation of materials and scientists can explain the making of knowledge.

The multi-scale narrative has required the multiplication of information sources, which explains the heterogeneity of the corpus: oral testimonies of scientists and administrators in materials research and fiber-optic communication; institutional archives from the laboratory of J. Lucas and a professional glass society in Rennes as well as from the Centre National de la Recherche Scientifique (CNRS) in Paris; scientific and

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<sup>2</sup> This is exemplified by the conclusion of an article by Ian Bartle (2002, p. 22-24), devoted to the two-decade process of liberalization of electricity and telecommunication sectors in Europe: “while national institutions have significantly influenced the pace and timing of reform [... it] is the international convergence of the norms of competition and privatisation that institutional theories of public policy appear particularly weak in explaining.”



technological literature, including selected readings and quantitative analyses from on-line databases of publications (Science Direct, Web of Knowledge) and patents (European Patent Office); secondary literature in the domains of history of science and technology, STS, and science policy. In spite of its patchwork nature, such an *ad hoc* corpus is liable to connect local specificities to global trends by gathering complementary information. On the local side, the epistemological study of knowledge-making is mainly extracted from oral archives and scientific articles. On the global side, the historical trends of telecommunication would have not been grasped without secondary literature. Between local and global approaches, the gap is sometimes big since business articles rarely go down to bench materials. Quantitative analysis provides a means to bridge the gap in-between.

### **National Policy-Making and the International Division of Labor in Materials Research**

Materials research was dependent on national contexts during the Cold War. It was framed by different “disciplinary structures” in the Western world with regards to epistemic methods, academic organizations and societal functions.<sup>3</sup> In the United States (US), advanced materials were given an important political function in the Cold War. This led to the building of a new interdisciplinary entity of materials science and engineering (MSE) and to the active support of solid-state physics. In Europe, the field was both shaped by industrial and academic dynamics. This favored a balanced, although sometimes conflicting, collaboration between solid-state chemists and physicists. These differences of “disciplinary structures” between the United States and Europe induced an international division of labor in materials research in the Western world during the second part of the twentieth century.

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<sup>3</sup> This chapter alternatively uses the three complementary ways to consider scientific disciplines listed by Rudolf Stichweh (1994, p. 55-56): a set of questions and methods, close to the “disciplinary matrix” of Thomas Kuhn (1970); a specialized system in interaction with the scientific environment, made of other disciplines; a scientific system in interaction with the society at large, including different spheres of human activity such as technology, industry, policy, and education.

- *The Cold War Policy of Materials Science and Engineering in the United States*

The US federal government implemented MSE as a new academic entity in response to the 1957 Sputnik success of the Soviets. Around \$200 million were spent by the Department of Defense over a decade (1961-1970) to fund fifteen Interdisciplinary Laboratories (later Materials Research Laboratories), as well as training programs in top-rank universities, including MIT and Stanford (Leslie, 1993). The idea was to foster fundamental solid-state research oriented towards industrial applications. It was modeled after the 1930s example of AT&T Bell Labs (Hoddeson, 1977). Collaborative research between chemists, crystallographers, electricians, engineers, mechanics, metallurgists, and physicists was organized towards the design of advanced materials for strategic domains. The epistemology of MSE defined an integrated tetrahedron of four elements: process, structure, property, and performance. In addition, materials scientists distinguished between “intrinsic” and “extrinsic” properties (Goodenough, 2001, p. 22). The former were induced by the composition and structure of inner matter while the latter were more related to the performance of the end product through the optimization of several parameters (shape, morphology, doping level, purity, etc.). Training programs taught these considerations to several hundred graduate students throughout the country. The annual number of awarded PhD in materials science multiplied ten-fold in two decades, from around 30 in 1970 to 300 in 1990, at the expense of metallurgy (Groenewegen & Peters, 2002, p. 129-130). In the same period, the number of MSE research centers multiplied five-fold, from around 20 to almost 100. Composite materials dominated the research field during the same period (Bensaude Vincent, 2001). A special emphasis was put on the study of solid-state structures, including structural defects. Industrial companies and state governments joined the military during the 1970s in funding the research field. Another institutional step was the foundation in 1973 of the Materials Research Society. Its membership increased from 300 at the beginning to around 1,000 in 1980 and 10,000 in 1990 (Philips, 2016). Fall and spring meetings gathered an audience of the same order of magnitude twice a year in the US.

In spite of the interdisciplinary rhetoric, MSE was under the symbolic domination of physics. In particular, solid-state physicists were widely supported by military agencies during the Cold War, even if they retained the latitude to perform fundamental research (Martin, 2013, p. 240-245). There were around 2,000 researchers according to the 1973 *American Men and Women of Science*. Between 200 and 400 PhDs were annually awarded in solid-state physics during the 1970s. On the contrary, chemistry was “per-

ceived as playing a supporting role in materials science, and a relatively unexciting one at that" (Whitesides *et al.*, 1987, p. 204). Chemists mostly performed optimization, purification, and design of well-known compounds. Indeed, less than 50 PhDs in ceramics were awarded per year in the last three decades of the twentieth century. The *American Men and Women of Science* identified around 70 solid-state chemists in 1973.

- *The Disciplinary Organization of Solid-State Research in Continental Europe*

Advanced materials were promoted by European states mainly through existing academic disciplines.<sup>4</sup> They were fostered by NATO conferences and publications, OECD incentives, and specific funding from the European Science Foundation, after its foundation in 1973. A European branch of the Materials Research Society was also established in 1983. However, there was no coherent policy in Europe to implement MSE as a university entity during the academic expansion and specialization of the Cold War. National policies towards MSE remained diverse. In the 1960s, Dutch scholars were influenced by materials science through the central role played by Philips Company in the Netherlands (Steggerda, 2004). In the 1970s, British metallurgists mimicked American orientations towards MSE (Cahn, 2001). In the 1980s, a French national initiative failed to establish MSE as a profession (Bertrand & Bensaude Vincent, 2011). In the 1990s, Germany seemed to achieve a higher degree of integration of MSE (Hentschel, 2011).

Materials research was mainly driven in Europe by industrial R&D and academic disciplines, including chemistry, crystallography, physics and metallurgy. In particular, solid-state physicists and chemists formed two equivalently strong communities of research and education in European universities (Teissier, 2014). They became institutionalized during the second part of the twentieth century. Solid-state physics was modeled on the US community (Pestre, 2004). Their disciplinary matrix was made of three elements: X-ray diffraction, structure-property relationship, and quantum theory of solids (Weart, 1992).

On the contrary, European chemists built solid-state chemistry without copying America, where chemists were deeply influenced by MSE. Dutch, French, German, and Swedish chemists were at the forefront of solid-state chemistry during the twentieth century. Sub-sections were grad-

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<sup>4</sup> For a national account of the development of materials science and engineering in Europe, see the case of Swedish universities from the 1960s onwards (Gribbe & Hallonsten, 2017).

ually established in the respective national chemical societies: 1963 for Germany, 1976 for France, 1998 for England. In 1978 the first “European Conference of Solid-State Chemistry” was organized in Strasbourg (Alsace), a symbolic place for the political history of France and Germany. It was under the supervision of two well-known professors from each country: Paul Hagenmuller (born 1921) from France and Rudolf Hoppe (born 1922) from Germany. Three years later, the International Union of Pure and Applied Chemistry (IUPAC) established its commission on “solid-state chemistry”. Most European solid-state chemists shared the same practices and representations of matter. Their “disciplinary matrix”<sup>5</sup> was made of three elements: high temperature synthesis, making of bulk crystals, and structural analysis by X-ray diffraction. French and German chemists agreed.<sup>6</sup> They developed “crystallochemistry” as the investigation of the relationship between synthesis and structure, which allowed the making of original solid compounds. It had been renewed by German inorganic chemists in the 1920s and 1930s (Klemm, 1955). In particular, the research school of Wilhelm Klemm (1896-1985) in Danzig specialized in the making of series of oxide and fluorine crystals by slightly changing the chemical composition from one compound to the following in the series. They played around with chemical structures like J. S. Bach made musical variations on a theme in *The Art of Fugue* (Hoppe, 1998, p. 178).<sup>7</sup>

In Continental Europe, materials research was driven by solid-state physics and chemistry, which tended to favor the study of “intrinsic” properties rather than “extrinsic” ones (Simon, 2005, p. 4). The institutional autonomy of both academic disciplines explained why their approaches differed from each other. Solid-state physicists, who were more interested in the characterization of “purified phenomena”, adopted a global description of matter. On the contrary, solid-state chemists, who were more interested in making “dirty materials”, preferred to focus on the local arrangement of

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<sup>5</sup> A disciplinary matrix was defined by Thomas Kuhn (1970) as a set of knowledge, methods, values and representations that is shared by a given community of research and education at a given time. There is a circularity in this concept since the matrix defines the community and *vice versa*.

<sup>6</sup> According to German chemists’ testimonies, “The typical work for a [solid-state] chemist was: 1) synthesis of a new compound, 2) chemical analysis, 3) determination of the structure, and then publication. Determining the structure represented the end-stop.” (Simon, 2005, p. 4). For the French case, see (Teissier, 2010).

<sup>7</sup> Interestingly, a French solid-state chemist who started his career in the 1960s also used the musical metaphor to explain crystallochemistry: “Crystallography allowed us to play; crystallochemistry allowed us to make the structures sing” (Férey, 2010, p. 3).

atoms, seen as geometrical blocks (triangle, tetrahedron, octahedron) composing crystals (Pouchard, 2004, p. 10). Such a differentiation allowed them to collaborate in a complementary way: physicists performed the most subtle characterization of properties and proposed theoretical models while chemists provided new solid compounds with original atomic arrangements. This academic organization was typical of continental Europe, even if materials research was also conducted by industrial companies, Philips being the most famous in the Netherlands. On the contrary, “in the English-speaking world, where academic ‘departments’ [were] normal, no departments of either solid-state physics or of solid-state chemistry [were] to be found” (Cahn, 2001, p. 46).

- *The Western Division of Labor of Materials Research in the Cold War*

In spite of national differentiations, an international field of materials research developed on both sides of the “iron curtain”.<sup>8</sup> Strategic needs for nuclear, space or electronic industries stimulated the emergence of academic publications. Ten new journals were established on the solid state and materials between 1956 and 1969 in the United States, the Soviet Union and Western Europe. The first one, *Physics and Chemistry of Solids*, published by Pergamon Press in Oxford, announced “the coming of age of solid-state science”. Its editorial board epitomizes the international dimension of solid-state sciences as well as its large scope, from industry to fundamental research.<sup>9</sup> The first two journals that mentioned “materials” in their title were successively published in 1966 and 1967, in Oxford and Moscow: *Materials Research Bulletin* from Pergamon, followed by *Fizika i Khimiia Obrabotki Materialov* (*Physics and Chemistry of Solid Materials*), from Nauka. The first of them encompassed the disciplinary variety of materials research in the Western World, from solid-state chemistry and physics to MSE. This was made clear by its editorial position, the composition of its board, and the disciplinary affiliation of authors. On the whole, the ten or so specialized

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<sup>8</sup> Historical studies on materials science in the Soviet Union are rather scarce. A case study on the nuclear industry (Holloway, 1998) suggests that the Soviet Union developed specialized research institutes to foster advanced materials.

<sup>9</sup> Harvey Brooks from General Electric was chief-editor. Five other top-rank scientists formed the editorial board: Hendrik Casimir (1909-2000), from Philips Eindhoven; George Dienes (born in 1918), from Brookhaven National Laboratory; Jacques Friedel (1921-2014) from the engineering school of Mines in Paris; Lev Landau (1908-1968) and Evgeny Lifshitz (1915-1985), theoreticians from the Soviet Academy of Science.

journals on the solid state provided an international space of quick scientific exchanges for the booming field of materials research.

*A posteriori*, they appeared to provide an historical tool to compare materials research on both sides of the Atlantic Ocean. In 1987, Francis Di Salvo (1987, p. 163), a former chemist from AT&T, then professor at Cornell, published a list of 18 new physical phenomena, which had been characterized during the two previous decades (1965-1985). According to his survey, 70% of these phenomena had been discovered by US materials scientists and physicists while 70% of the materials that exhibited these phenomena had first been synthesized by European chemists: German, French, Soviets, and Dutch. This made explicit an international division of labor between the United States and Europe during the Cold War: Europe was more focused on synthesizing new solid structures; the United States was more efficient at characterizing new properties. There was a double advantage for the United States: at the symbolic level, the characterization of phenomenon was more valued than the synthesis of solid compounds; at the economic level, new properties were the first step towards new advanced materials. This division of labor was a consequence of the differences between the social organization of research materials in Europe and the United States during the cold war. The disciplinary organization of solid-state chemists in Europe boosted the development of synthetic creativity through "cristallochemistry". This was under-estimated in the United States.<sup>10</sup> There, they mostly performed the optimization and purification of materials. This was a result of the organization of MSE under the guidance of physicists and of military and industrial goals.

- *The Bench Culture of Solid-State Chemistry in Rennes*

French solid-state chemists contributed to the international division of labor by making numerous original compounds. In particular, the University of Rennes, in Western France, published two star-materials. It hosted five small chemistry groups that studied inorganic solid compounds in the late 1960s (Ministère de l'Industrie, 1966, p. 258). It was a time of expansion and specialization in French academia. Science policy favored the integration of small groups into big centers. The CNRS was in charge of the reorganization. The CNRS was the national research agency established in 1939 to organize French academic research, both generally and at

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<sup>10</sup> "In the United States, synthesis of solid-state compounds has been considered out of date and a little dull, and few academic departments have even one professor involved in synthesis of new solid-state compounds." (Di Salvo, 1987, p. 164-165).

the laboratory level. Over three decades (1949-1982), its number of employees increased by a factor of 10, from 2,420 technicians and researchers to 23,000, and its budget by a factor of 40 (Picard, 1990, p. 214). The CNRS missions were to manage its own laboratories on specific research and to distribute its employees in university laboratories to strengthen French academic research. In 1965-1966, a new category of association with the CNRS was created to provide extra funds and means to university laboratories with sufficient size and quality.

This science policy led to the gathering of the five research groups of Rennes into one single unit of research and education devoted to “structures and properties of the matter”. This unit received the CNRS association label in 1975 to become the Laboratory of Chemistry and Crystallochemistry of the Elements of Transition (CNRS, 1975). Jacques Prigent was the laboratory director. However, each research group kept its autonomy under the leadership of a professor: Jean Lang (1927-2014), Dominique Weisel, Daniel Grandjean and Jacques Lucas (born in 1937). Each was specialized in mineral, physical, or crystal chemistry, which contributed to mixing these sub-cultures of chemistry in Prigent’s laboratory. There, two materials that became known worldwide were produced in the early 1970s.

First, in Prigent’s group, Marcel Sergeant and his PhD student, Roger Chevrel, investigated crystallochemistry. They learned to synthesize a new series of crystals of general formula:  $MMoN_{SN+2}$  (M stood for transition elements). In 1971, they published an article in French in *Journal of Solid State Sciences* (Chevrel *et al.*, 1971), where they announced the synthesis of “new phases of ternary molybdenum sulfides” and their structural analysis by X-ray diffraction. The article was read by some researchers at Bell Labs, who assumed that these new sulfide structures might have interesting electrical properties (Matricon and Waysand, 1994, 307). The group of Bernd Matthias replicated the syntheses and characterized superconducting properties at very low-temperature, around a few Kelvins, thanks to cryogenic electrical devices. They optimized the chemical composition of the different phases, by slightly changing the relative quantity of elements, in order to increase the critical temperature of superconductivity. They could thus go up to 15K, which allowed them to publish in *Science* in March 1972 the “first ternary system” providing “high-temperature superconductors” (Matthias *et al.*, 1972). The Bell Labs group’s approach approximated the MSE tetrahedron: optimization (process), phase analysis (structure), superconductivity (property) in order to increase the temperature of use (performance). Solid-state chemists at Rennes, on the contrary, relied on the synthesis of original crystals and their structural analysis. The symbolic gap between the *Journal of Solid State Sciences* and *Science* revealed the symbolic gap

between the synthesis of new crystals and the disclosure of new properties for application.<sup>11</sup> This exemplifies the first advantage of the United States in the division of labor in materials research during the Cold War: symbolic capital. The second case of star-materials from Rennes stresses their second asset: economic capital. It was developed in Lucas's research group.

### **The Local Reconfiguration of Solid-State Chemistry towards Glass Materials**

- *The Solid-State Chemistry Group of Jacques Lucas*

The research group of Jacques Lucas emerged from the French academic expansion of the 1960s (Picard, 1990, p. 209-234). In 1964, Lucas completed his PhD on uranium complexes under Prigent. He went on in inorganic chemistry during his military service. By chance, he was attached to the French Atomic Energy Commission (CEA), a public body that led both civilian and military research and development on atomic energy and materials, where he was in charge of a small research group in Saclay, near Paris (Lucas, 2005, p. 4). He was lucky to learn fluorine chemistry in a wealthy laboratory at a time when it was unusual for young draft scientists to do research. This two-year CEA experience oriented Lucas towards fluoride crystals when he was appointed associate-professor in Rennes in 1966. A charismatic leader, Lucas took advantage of the academic university to gather a dozen PhD students and technicians in the early 1970s. He also relied on CNRS funding to buy instruments, hire technicians, and find grants for PhD students.

However, the public abundance slowed down in France at the end of the 1960s. 1970 was the first year of decrease for the equipment budget of the CNRS and of stagnation for the salary budget (Guthleben, 2013, p. 279). French budget of R&D decreased in relative share, from 2.4% of GNP in 1968 to 2.1% in 1971. The slowing down of public funding was counter-balanced by a national policy towards the collaboration between university and industry during the 1960s (Duclert, 2004). Lucas's group took part in a national program funded by the CNRS and a private company, Compagnie Générale d'Électricité (CGE), to work on fluoride crystals

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<sup>11</sup> However, the symbolic imbalance between the two journals was counter-balanced by the number of citations of the two articles: 325 for (Chevrel *et al.*, 1971) and 180 for (Matthias *et al.*, 1972) according to Science Direct (November 2016).



for laser applications (Lucas, 2005, p. 1-5). It mainly focused on fluoride pyrochlore structures<sup>12</sup> as exemplified by figure 1.

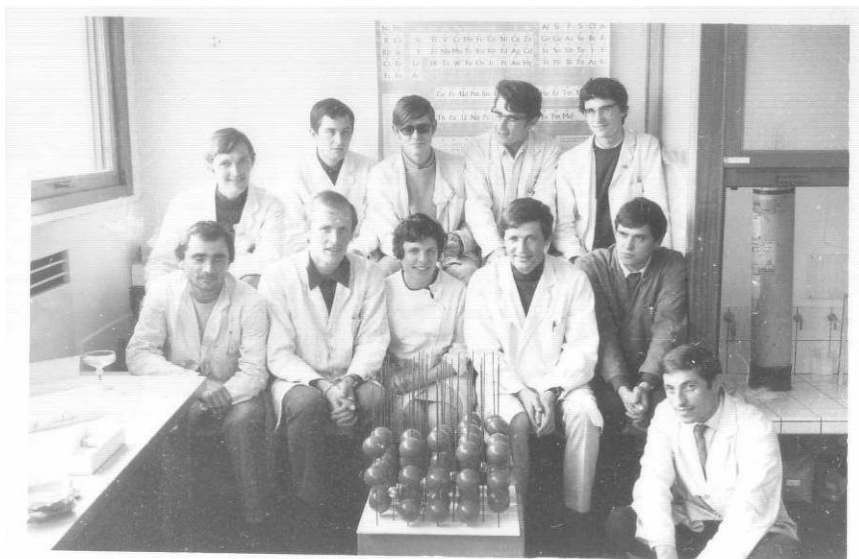


Figure 1 - Photography of Jacques Lucas and his research group from Rennes around 1970. From Left to Right: (top) Robert Rannon, Marcel Poulain, Hervé L'helgouach, Jean Yves Moisan, Jean Pannetier (bottom) Gilles Fonteneau, Daniel Laguitton, Odette Texier (ITA), Jacques Lucas, Jean Hamelin (ITA), Michel Poulain. (Source: institutional archives of Laboratoire Verres et Céramiques. Courtesy of J. Lucas)

The arrangement of the research group around a pyrochlore ball-and-stick model, which mimicked a soccer team around the ball, indicated the central place played by these structures. To study them, Lucas and his collaborators applied the “disciplinary matrix” of solid-state chemistry: high-temperature synthesis, bulk crystals, and structural analysis (Lucas, 2005, p. 15). Basically, mineral powders were mixed, put in a sealed nickel tube to prevent oxidation from air, and heated in a furnace for one to three days at around 1,000°C (Poulain *et al.*, 1972, p. 319). The cooling down allowed the melt to crystallize in one or several structures. Following the 1960s trends in crystallochemistry, Lucas’s group had two means to prepare

<sup>12</sup> Pyrochlores are natural structures characterized by the following chemical composition:  $A_2X'B_2O_6$ ,  $A=Ca, Na, Pb...$ ,  $B=Nb, Ti...$  and  $X'=F, OH...$

new series of compounds. Firstly, two or three reagents ( $\text{Zr} - \text{UF}_4 - \text{ZrF}_4$ ) were combined in different proportions to form unexpected products:  $\text{UZrF}_7$  and  $\text{UZr}_2\text{F}_{11}$  (Fonteneau & Lucas, 1974). Secondly, in a well-known compound like fluorozirconate ( $\text{MZrF}_6$ ), chemical elements were alternatively substituted for each other (M could be Mg, Ca, Cr, Mn, Fe, Co, Ni, Cu, Zn) (Poulain & Lucas, 1970, p. 822). Besides structural analysis by X-ray diffraction, crystals could also be characterized through physical measurements if specific magnetic or spectroscopic properties were expected.

- *The Local Making of Heavy-Metal Fluoride Glasses by the Poulain Brothers*

The solid-state chemistry routine for pyrochlore structures was disrupted by the tandem work of two brothers from Lucas's team: Marcel Poulain (the second one from the right in the top row of Figure 1) and Michel Poulain (the last one on the left at the bottom). The youngest one, Marcel (born in 1945), was the first to join Lucas's group in 1967 after a curriculum in electronics and chemistry in Rennes. He submitted a university thesis on earth alkali fluorozirconates in 1970 and a doctorate on transition metal fluorozirconates in 1973 under Lucas's supervision. When a technical position opened in the laboratory, Marcel advised his elder brother, who was jobless in spite of a physics degree, to apply (Poulain & Poulain, 2015, p. 2).

Michel (born in 1935) was hired as a technician for the operation and maintenance of X-ray and magnetic instruments. All worked so well that he had spare time to pass certificates in chemistry and electronics. He could even submit a university thesis in 1972 on the spectroscopic and structural characterizations of rare earth fluorozirconates (Poulain, 1972). The physical properties were studied through a multidisciplinary collaboration with Pierre Brun (born in 1934) from the neighboring Laboratory of Quantum Electronics (Brun *et al.*, 1973). Michel thus contributed to Lucas's group research on laser applications. Lucas let him conduct part-time research with his brother probably because they were as skilled as they were independent and stubborn. In spite of his physics background, Michel preferred chemical syntheses. He quickly learned how to screen hundreds of compositions a month by proceeding dirtily in the first round (Poulain & Poulain, 2015, p. 1-5). His intuitions and trial and error empirical methods led him to define the most promising compositions, on which he spent more time. From his thesis, he extracted neodymium fluorozirconates ( $\text{NdZrF}_7$ ) with fluorescent properties that sounded promising for laser applications. The lack of reproducibility of his results led Michel to work on this composition in 1974. After one trial, he got back from the furnace a centimeter long co-

lorless solid instead of the usual smaller and darker compounds. When analyzed by X-ray diffraction, only rays of neodymium fluoride ( $\text{NdF}_3$ ) appeared, which suggested that this reagent did not react. The lack of other signals suggested that the three other reagents ( $\text{ZrF}_4$ ,  $\text{BaF}_2$ ,  $\text{NaF}$ ) led to amorphous compounds. The colorlessness and the size of the product strengthened the suspicion: a glass instead of a crystal had been synthesized. The result was unexpected in a program devoted to pyrochlore structures.

However, the accident was attractive for two reasons. Firstly, the Poulain brothers were excited by having found a new type of heavy-metal fluoride glass while only two other cases had been reported with lighter elements ( $\text{BeF}_2$ ,  $\text{AlF}_3$ ). They performed more systematic syntheses and drew a ternary diagram ( $\text{ZrF}_4 - \text{BaF}_2 - \text{NaF}$ ). A ternary diagram provided a visual tool to mark out the stability domains of the different structures that could be made by from the variable compositions of the three components. Figure 2 shows the chemical and structural map of the compound with the amorphous domain in the middle of the diagram.

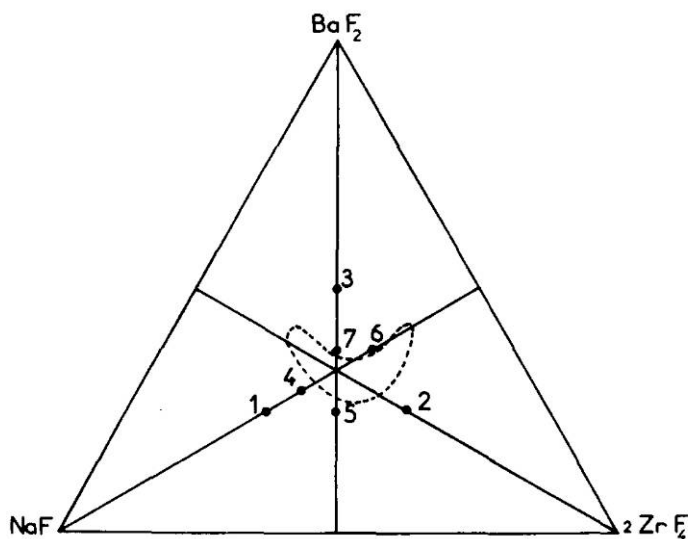


Figure 2 - Ternary diagram of  $\text{ZrF}_4 - \text{BaF}_2 - \text{NaF}$  and amorphous domain (Poulain et al., 1975)

Once he had been alerted, Lucas stressed a second reason to go on in this direction. As a team leader, he thought about the possible use of glasses for optical applications, which would provide new funding oppor-

tunities. Yet, while public funding had been stagnating since the early 1970s, the 1973 economic crisis had made the general context worse. More specifically, the academic competition was rough in French solid-state chemistry (Teissier, 2010, p. 239-248). University national committees and CNRS commissions were dominated by powerful mandarins such as Jacques Bénard (1912-1987), Robert Collongues (1924-1998), Michel Fayard (born 1928), Paul Hagenmuller, and André Michel (1909-2000). These elites tended to keep means, honors and positions for their own laboratories while small groups from the provinces like Lucas's team experienced hard times in the 1970s (Caro, 2005, p. 12). Both the epistemic search for originality and the marginality in a competitive academic environment led Lucas's group to shift from the crystal-based tradition of French solid-state chemistry to the unknown domain of optical glasses, while hoping for industrial applications. Their choice was strengthened by positive signals coming from the international telecommunications industry.

- *How the Economic Crisis and Industrial Hopes Turned Academic Chemists towards MSE*

The international mainstream of solid-state chemistry was still firmly grounded in the study of well-organized solids during the 1970s for both instrumental and industrial reasons. Indeed, X-ray diffraction had become the central tool for solid-state characterizations since the 1930s, which discarded less-organized solids like glasses. In addition, most high-technology industries relied on crystalline materials, including semi-conductors and composite materials. The "hope of applications" for amorphous materials slowly changed the situation from the 1960s onwards (Mazières, 1978, p. 10). The success of the *Journal of Non Crystalline Solids* established in 1968 highlighted the interest of the academic community at the end of the decade.

Telecommunications also contributed to fostering this hope for applications for glasses through the expanding market in silica fibers for commercial devices and military electronics in the 1970s.<sup>13</sup> Fiber optics increased the information flow by comparison with electrical wires traditionally used for telegraph and telephone systems.<sup>14</sup> They could also reduce the

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<sup>13</sup> The first silica fibers were sold around 1970 by Corning. In 1978, the US market in fiber optic systems was \$12 million, with \$4 million for commercial products (TV, computers) and \$3 million for military electronics (Montgomery, 1968, p. 1100).

<sup>14</sup> In the 1950s, one coaxial cable could thus deliver 600 telephone conversations through 600 different channels (MacChesnay and DiGiovanni, 1990, p. 3537).

tremendous quantities of (expensive) copper that were used in networks (Keck, 2004). The principle of optical fiber was to guide infrared light signals across a glass-core surrounded by a cladding to convey information. Theoretical and experimental problems had been solved in the 1960s, mainly by Standard Telecommunications Laboratories (STL), the research center of International Telephone and Telegraph, in Harlow, UK (Kurkjian and Prindle, 1998, p. 810). Around 1970, the US glass manufacturer Corning produced a silica fiber that met commercial needs with an attenuation of twenty decibels per kilometer (20dB/km) at a given infrared light length of one and half micron. Silica was cheap, transparent and easy to shape.

However, the oxide composition of silica induced an irreducible “intrinsic” attenuation.<sup>15</sup> Optical repeaters were thus required at regular intervals (around 1 km) to amplify the attenuated signal. On the contrary, non-oxide glasses appeared to have a better “intrinsic” transparency, which made them good candidates to increase the repeating distance. Chalcogenide glasses (made of S, Se or Te) had been extensively studied in the 1960s through military contracts for infrared detection devices (Copley, 1971, p. 26). Halide glasses (made of F, Cl or Br) were outsiders when the fluoride glasses from Rennes boosted industrial hopes in the 1970s. Indeed, Bell Labs theoreticians predicted that fluoride glasses could decrease the attenuation of silica by several orders of magnitude (Lucas, 2005, p. 6). The repeating distance was hoped to reach 1,000km, which was interesting with regards to transoceanic telecommunication systems. In addition, the broader infrared transparency of fluoride glasses (up to 7 microns instead of 2 for silica) made possible a multi-mode technology with several light wavelengths instead of one.

It was in this context of commercial expansion and prospective hopes for optical fibers that the Poulain brothers, Lucas and Brun published the making of unknown “fluorinated glasses” (*verres fluorés*), later renamed fluoride glasses (Poulain *et al.*, 1975). To do so, they chose the *Materials Research Bulletin*, a good quality journal where solid-state chemists were used to publishing. Their article attracted little attention from international scholars during the following years.<sup>16</sup> It was also ignored by the solid-state

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<sup>15</sup> The attenuation was due to “extrinsic” impurities in the core fiber as well as “intrinsic” vibrations of the silicon-oxygen bond in silica (SiO<sub>2</sub>), which forbade transmission longer than 80 kilometers without amplification.

<sup>16</sup> The article was credited with 9 citations between 1975 and 1980, only 2 from groups other than Lucas's (Google Scholar).

chemistry community to which Lucas's group belonged.<sup>17</sup> On the contrary, it attracted many visitors to Rennes from glass-making and telecommunication companies: Corning, of course, as well as AT&T, British Telecom, Denshin Kokusai Denwa, French National Center for Telecommunications Studies (CNET), Hoya Corporation, Nippon Telephone and Telegraph (NTT), etc. The industrial interest confirmed Lucas's will to jump into the making of fluoride glasses.

This opportunist strategy induced two shifts for Lucas's group: an economic shift towards contract-based funding and an epistemic shift towards MSE. The first shift was linked to general economic trends. Usually in French academia, the budget balance between salary and equipment was around the same in CNRS and scientific universities: around three quarters for salaries and one quarter for equipment (Picard, 1990, p. 212-214). This meant that no less than three quarters of the budget came from public funding. One consequence of the 1973 oil crisis was a decrease of the state budget and a relative decrease in R&D funding, from 2.1% of GNP in 1971 to 1.9% in 1981. This meant, for Lucas's group, that there was no new recruitment for a decade, from 1975 to 1985, while there had been three new positions during the previous decade (Adam, 2006, p. 5). The equipment budget was even easier to reduce. The decline in state finances was, to some extent, counter-balanced by the industrial boom in telecommunications. Lucas thus signed several contracts to make optical fibers for industrial and military institutions (NOGS, 1988, p. 1-2). Most of the contracts originated from French public agencies, either civil (CNET) or military (DGA), and from private companies (CGE). They allowed him to buy new equipment and hire PhD candidates.<sup>18</sup> Thus, the 1970s decrease in public revenue induced a partial replacement of tenure by three-year research positions in Lucas's group. Probably exceptional in the 1970s, the situation became normal in the 1980s France through the exponential growth of university-industry contracts: their number was multiplied thirty-fold for

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<sup>17</sup> In France, a few solid-state chemists, including Collongues, expressed their interest in the fluoride glasses from Rennes while the majority was either indifferent or hostile to the amorphous materials (Galy, 2006, p. 9; Serreau, 2004, p. 15). Outside Rennes, only one PhD in electrochemistry was submitted on the theme, in Grenoble (France), devoted to the "electrochemical study of a new class of glasses from zirconium fluoride" (Leroy, 1979).

<sup>18</sup> The two first university theses on fluoride glasses were submitted in the University of Rennes, in 1976, by Rosa Bugueno-Velasquez and Maydom Chanthanasinh. Both were probably foreign PhD students. None of them was mentioned in the main protagonists' testimonies.

CNRS in one decade, from 1982 to 1991, especially in the departments of chemistry and engineering sciences (Lanciano-Morandat, 1999, p. 119).

The epistemic shift turned the solid-state chemists in Lucas's group towards MSE. Lucas and the Poulains were excited by the investigation of fluoride glasses but they had no expertise. Their main instrument, X-ray diffraction, said almost nothing about amorphous materials. Their first reaction was to continue their multidisciplinary collaboration with their physicist neighbor Brun to perform spectroscopic characterizations (infrared and fluorescence). This gave them information about the optical transmission and local structure of the glass. Lucas's industrial contracts allowed his group to buy instruments to handle and characterize glass materials. They acquired spectroscopic apparatus to characterize the infrared transmission range.<sup>19</sup> Differential thermal analysis was needed to characterize the glass quality by measuring the glass transition temperature ( $T_g$ ). This helped them to practice the art of glass-making to decrease the number of crystalline grains by trial and error under experimental conditions: heating, cooling, composition, viscosity.<sup>20</sup> In addition, the design of materials required several new machines to hot-press and extrude the melt, and to polish, cut, and pull the fibers to meet industrial requirements (Adam, 2006, p. 1). Last, they needed some theoretical basis to understand the vibrational behavior of glass (phonons) during light propagation. Lucas (2005, p. 16) collaborated with a specialist in molecular dynamics, Austen Angell, from Purdue University, to model the local glass organization. In short, these solid-state chemists dropped crystallochemistry to investigate the relationship between composition and property of glasses to improve their performance. By doing so, they adopted the MSE tetrahedron and their investigation shifted from "intrinsic" to "extrinsic" optical properties of fluoride fibers.

- *From Bench to Brand: the Economic Activity of Le Verre Fluoré*

There was a third consequence in Rennes resulting from the shift towards optical fibers. Lucas and the Poulains, who wanted to turn academic finding into profits, established a start-up company outside the university in 1977: Le Verre Fluoré. The incorporated company was led by Gwénael

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<sup>19</sup> The multiplication of characterization apparatus in chemistry laboratories was characteristic of the "instrumental revolution" in chemistry during the twentieth century (Morris, 2002).

<sup>20</sup> For example, the Poulains (2015, p. 6-9) added 3 to 4% of aluminum to increase the stability of fluorozirconate glasses and operated in open air (instead of under a controlled atmosphere) because the oxygen destroyed impurities in the reagents.

Mazé, Marcel Poulain's friend. Local businessmen (B. Angon, Y. Le Met) helped them to start, before the stocks were shared by the Mazé family and the Poulain family (Poulain, 2015, p. 14-15). Two graduates in technological chemistry from Rennes, Vincent Cardin and Jean-Yves Carré, were hired to conduct in-house R&D. They could rely on the laboratory's expertise until Lucas and Mazé quarreled. Then, they were helped by the Poulains, who remained involved in *Le Verre Fluoré*. This made them isolated, if not in trouble, in Lucas's group. The gap between the laboratory and the company increased in the 1980s.

Besides individual quarrels, selling fibers was a different business than doing science. Cardin and Carré had to optimize compositions, purify glasses, shape materials, and draw fibers. Two or three contracts were signed with the CNET to develop the technology of optical fibers in the late 1970s (Poulain & Poulain, 2015, p. 16). Between 1981 and 1985, *Le Verre Fluoré* took three patents on the making of fluoride glasses and the design of fibers.<sup>21</sup> But the main strategy of such a small company was to keep know-how secret and in-house. Employees developed a good chemical and engineering expertise in fluoride glasses, from bench compounds to brand materials. They were able to manufacture customized fibers for NASA.<sup>22</sup> But the economic situation remained uncomfortable since the market was dominated by multinationals and the applications limited to short-distance high-technology applications: dental lasers (YAG-Erbium), astronomy interferometers between telescopes (Mont Wilson, Hawaï, La Silla, etc.), space detectors, etc. (Poulain & Poulain, 2015, p. 17-19). Over its whole history, *Le Verre Fluoré* never scaled up and remained stuck at two to three employees and a small turnover of around \$0.5 million.

The accidental synthesis of fluoride glasses in Rennes in the mid-1970s induced the local reconfiguration of an academic solid-state chemistry group towards materials science and engineering. This epistemic and sociological shift can be explained by the articulation of academic and economic trends: Lucas's group was a marginal solid-state chemistry group in

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<sup>21</sup> The 1981 patent on fluoride glasses was opposed by the CNRS in 1987, which sounds surprising. This unclear episode was not mentioned by the interviewed protagonists. This may be a clue to suggest that the reason to oppose the technology transfer was more linked to individual quarrels than institutional rules of the CNRS.

<sup>22</sup> In 1985, *Le Verre Fluoré* succeeded in designing one specific optic fiber for NASA Jet Propulsion Laboratory for around \$100,000. They learned afterward that they were the last company contacted by NASA after the other competitors had declined the offer because of too high specifications (Poulain & Poulain, 2015, p. 19).



the French provinces at a time when the academic competition got harder because of the relative decrease in public funding for national R&D in an international context of economic crisis. The group took the opportunity of telecommunications expansion to sign industrial and military contracts to develop new optical fibers. They had to turn to MSE and drop the solid-state chemistry matrix to fulfill commercial demands. The industrial contract-based organization drastically changed the everyday practices of the laboratory while slightly changing the budget balance between public and private funding. Indeed, then and now, the salary (from public funds) was three times the equipment budget.<sup>23</sup> The equipment, which was generally public funded in the 1970s, is now largely linked to industrial contracts. The case of Lucas's group gives a clear lesson for science policy-makers and STS scholars for national cases (such as France) where academic salaries are mainly paid by public funding: science policy is not driven by those that pay more (recurrent salaries) but by those that pay less (extra money for equipment and grants). This is a common case where private enterprises are free riders on public funding.

### **Global University-Industry-Government Triple-Helix of Non-Oxide Glasses**

The fluoride glasses of Rennes contributed, with other exotic glasses (chalcogenides, halides), to stimulate international R&D on non-oxide glass materials for optical fibers during the 1980s. Multinational telecommunications companies and US military agencies organized the triple-helix integrating universities, industries and states in America, Asia, and Europe. Conventional glass-manufacturers on the contrary did not provide much innovation for non-oxide glass materials (Kurkjian and Prindle, 1998, p. 810).

The triple-helix was framed by two competitions. Inside fiber-optic communication, exotic glasses competed with classical glasses made of silica. Yet Corning and other glass-manufacturers invested several \$100 million to design commercial fibers whose attenuation gradually decreased, from 20dB/km ca. 1970 to 1 ca. 1980 and 0.2 ca. 1990 (Cohendet *et al.*,

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<sup>23</sup> Over three decades (1971-2002), the balance of the budget of Lucas's laboratory was remarkably stable. The ratio of three quarters for salary which was true in the 1970s is still true today. The 2002 budget of 1,8 million euros was as follows: 72% for employees' wages (41% of university and 31% of CNRS), 7% of recurrent public funding (Education and Research) and 21% of military and civil contracts (Laboratoire Verres et Céramiques, 2002).

1987, p. 264). In addition, besides fiber-optic networks, the telecommunications industry built satellite communication networks (Marandi, 1988). The techno-economic choices were not just induced by the performance of single materials but by the “evolution of large technological systems” (Hughes, 1987). Indeed, in a given communication network, each part had to be tuned to all others. For fiber systems, optical cables linked emitting devices (lasers, diodes) to processing devices (electronic computers) through a complex network of nodes (amplifiers, repeaters) and microwave phenomena (Faltas, 1988). Such systemic competition might explain why telecommunications companies kept on asking for better performance: each “reverse salient” was thought to endanger the whole system. The triple-helix around exotic glasses benefited from this systemic competition.

This led experts to overstate the need for reducing fiber attenuation and increasing information speed. AT&T was driven by the example of the electronics industry where Moore’s law displayed decades of exponential growth in processor speed (Brock, 2006). In the early 1980s, it was in charge, with Standard Telephone and Cables and Alcatel, of installing the first transoceanic fiber cable: TAT8 would be in operation in 1988 between America (Tuckerton, NJ) and Europe (Widemouth, UK and Penmarch, France) for a \$300 million budget. AT&T advertised that TAT8 would carry the equivalent of 37,800 virtual voice channels with a 25% reduction cost per voice compared with the 1983 electric wire TAT7 (Jeffcoat *et al.*, 1984). If repeaters were put every 60km with TAT8, industrial experts and materials scientists announced that the replacement of silica fibers by fluoride glasses in “the next generation of transoceanic cables” might even avoid the need for repeaters (Westwood and Winzer, 1987, p. 257). The prognostications caused the silica fiber market to expand quickly to reach \$2,4 billion in the late 1980s.

- *Knowledge Circulation Channels in Exotic Glass R&D*

Industrial and military funding organized the scientists’ enthusiasm towards the research and development of non-oxide glasses for telecommunication applications. Between twenty and thirty academic and industrial laboratories were involved, coming from numerous disciplinary backgrounds: astronomy, ceramics, chemistry, engineering, glass, materials science, optics, and telecommunication.<sup>24</sup> The case study shows how the

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<sup>24</sup> An analysis of citation of the seminal article by Poulain (*et al.*, 1975) gives converging results. According to Science Direct (November 2016), the five main domains of the 288 citing articles are the following: materials science (196), physics and astronomy (123), engineering (62), chemistry (49), computer science (19).

international triple-helix, which was so heterogeneous in membership, skills, and expectations, and so linked to industrial and military competitions of the Cold War, shaped a common feeling of belonging resembling an ideal-type of the Mertonian norm of “communism”: the free flow of information for the benefit of the whole scientific community. This worked provided that money was pouring in. The importance of knowledge circulation in the case of exotic glasses led researchers to pay special attention to the main international channels of exchange: periodic symposia, clearing houses, scientific literature, and patent publications.

The first “International Symposium on Halide and Other Nonoxide Glasses” was organized in 1982 in Cambridge by John Gannon (STL, UK), and an international panel of six major researchers in the field.<sup>25</sup> It was funded by the British Society of Glass Technology and STL as well as US and European military institutions. The audience of one hundred participants was composed of industrialists (45%), academics (36%) and state administrators (19%). Three countries dominated the symposium with around thirty participants each: the US, UK and France. The symposium lasted four days and featured forty communications divided into eleven sessions.<sup>26</sup> It was framed by the chemical composition of glasses, half of them being fluorides, and the MSE tetrahedron. The following symposia were alternately organized in the US and Europe every other year.

Following the organization of the third symposium in Brittany (1985), Lucas established a clearing house in Rennes in 1986. The international Non Oxide Glass Society (NOGS) aimed at linking physicists, chemists, materials scientists and engineers interested in halide and chalcogenide glasses. All information related to non-oxide glasses were to be sent to

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<sup>25</sup> Martin Drexhage (Rome Air Development Center, US Air Force), Lucas and Marcel Poulain (University of Rennes, France), Cornillon Moynihan (Institute Rensselaer Polytechnic, USA), Peter MacMillan (University of Warwick, UK), and G. H. Sigel (US Naval Research Laboratory).

<sup>26</sup> The list of sessions was the following: n°1 “glass forming halide systems” (chairman: J. Gannon); n°2 idem (M. Drexhage); n°3 “halide glasses containing rare earths” (Marcel Poulain); n°4 “preparation and processing of halide glasses” (J. Lucas); n°5 “optical properties of halides glasses” (G. H. Sigel); n°6 “optical and physical properties of halides glasses” (P. C. Schultz, Corning); n°7 “structure and glass formation: theoretical approaches” (C. Moynihan); n°8 “structure and glass formation: experimental studies” (J. D. Mackenzie, University of California); n°9 “applications for halide glasses” (O. H. El-Bayoumi, Rome Air Development Center); n°10 “chalcogenide glasses: preparation and properties” (J.A. Savage, Royal Signal and Radar Establishment); n°11 “chalcogenide glasses: properties and applications” (P. W. Mac Millan) (ISNOG, 1982).

NOGS: conferences, events, publications, national research descriptions. They would be published every other month in *NOGS News*, a craft journal sent to NOGS members around the world.<sup>27</sup> Lucas (2005, p. 16) wanted to extend the valuable telephone communication he could have with his friends to the whole community. He was convinced that the commercialization of fluoride glasses could only be achieved through the collective sharing of tacit knowledge, trials and errors, and incremental steps. The first issue of *NOGS News* expressed this naive ethos close to “communalism”: “the free flow of scientific information [had] allowed non-oxide glass science and technology to grow so rapidly” (NOGS, 1986, p. 1). It was more probably a mix of cooperation and competition. After eight months, *NOGS News* (1987, p. 3) already needed 66,000 francs to complete the annual budget. Glass manufacturers and telecommunications companies paid the difference.<sup>28</sup>

A quantitative survey of articles and patents devoted to “fluoride glasses” (figure 3) show the evolution of the “triple helix” around exotic glasses from the 1970s onwards.<sup>29</sup> The seminal article from Rennes can be spotted in 1975. The five following years were active in Rennes (articles from the laboratory and patents from Le Verre Fluoré) and quiet elsewhere. The early 1980s marked the expansion of R&D on fluoride glasses.

It was the time when the International Symposium on Halide Glasses was launched. The number of publications and patents peaked a first time in 1988 (44 items) and a second time in 1993 (66). The analysis of *NOGS News* exhibited the same trend for non-oxide glasses: the number of related publications was multiplied by two and half from 1987 (180 items) to 1997 (450) (NOGS, 1987-1997). The decrease of 1988-1990 can be explained by the funding shift, especially in the US, toward the booming field of high temperature superconductors after 1986 (Poulain & Poulain, 2015, 18). On the contrary, the patenting process increased until 1996. It was led

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<sup>27</sup> *NOGS News* was edited by Christine Adam, the wife of Jean-Luc Adam, then a young professor. Her low half-time salary (3,500 francs) was not even balanced by individual fees (28,700 francs) and company memberships (11,200 francs). One dollar was worth around ten francs.

<sup>28</sup> The companies that sponsored *NOGS News* were the following (by order of arrival): Kokusai Denshin Denwa Co., Ltd (Japan), Corning Europe (France), Du Pont de Nemours (USA), Central Glass Technical Center (Japan), E. Merck (Germany), Saint-Gobain (France), Owens-Corning Fiberglas Corp. (USA), Galileo Electro-Optics Corp. (USA), NTT Corp. (Japan), CSELT (Italie), CNET (France).

<sup>29</sup> The diagram displays the annual number of articles and patents that held “fluoride glasses” in their title. The corpus was based on two global online databases: Web of Knowledge and European Patent Office.

by Japanese companies, which registered more than 60% of the 400 patents on non-oxide glasses during the 1987-1997 decade (NOGS, 1987-1997). From the mid-1990s, there was a decreasing trend in publishing and patenting on fluoride glasses: the number of both articles and patents fell by two thirds from 1996 to 2002 (from 54 items to 17).

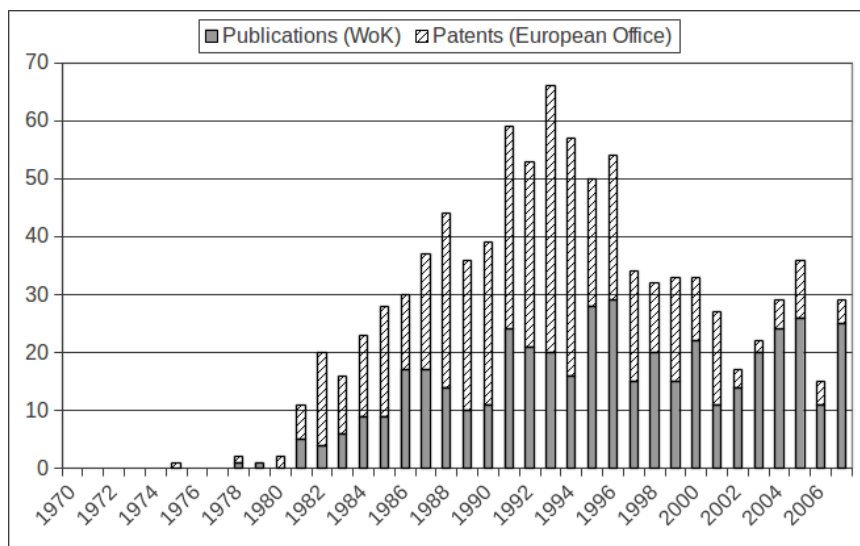


Figure 3 - Quantitative evolution of worldwide publications and patents on fluoride glasses

- *The Dispersion of the Fluoride Fiber R&D*

The 1990s decrease can be explained by two reasons. The first was that R&D efforts converged in the late 1980s toward a consensus on the composition of fiber glasses when the attenuation of silica fibers dropped around 0.2dB/km. The optimized composition of fluoride glass was named ZBLAN for the five elements involved: Zr, Ba, La, Al, Na. It was a delicate balance made of  $53\text{ZrF}_4 - 20\text{BaF}_2 - 4\text{LaF}_3 - 3\text{AlF}_3 - 20\text{NaF}$ . ZBLAN was strikingly close to the chemical composition of one glass published in the seminal paper of Poulain (*et al.*, 1975):  $50\text{ZrF}_4 - 20\text{BaF}_2 - 5\text{NdF}_3 - 25\text{NaF}$ . In-between, fifteen years of optimization turned fluoride glasses into commercial fibers with a broader transparency window (from 0.4 to 5 microns) and the required design. Several tens of millions of dollars had been spent in the same time around the world to foster non-oxide glass R&D (Poulain

& Poulain, 2015, p. 16). Fluoride glasses became brand products for high-technology niches in the 1990s: interferometry astronomy, laser medical applications, and military infrared devices. Profit expectations were reduced accordingly. In spite of their “intrinsic” properties, fluoride glasses proved difficult to purify and manufacture at low cost. Their “extrinsic” properties forbade them the mass-market contrary to silica fibers. Their study became less stimulating and the triple-helix diversified fiber-glass compositions, once dominated by fluoride types, towards other halides, chalcogenides, oxy-halogenides, and oxy-nitrides. The evolution was embodied by the 1994 renaming of the symposium to “International Symposium on Non-Oxide Glasses”. For the first time in 12 years, the symposium was organized in Asia, China being the host.

The second reason was linked to economic trends that favored short-term profits at the expense of R&D funding during the two last decades of the century: “financialization of the economy” (Pestre, 2003, p. 83); liberalization and privatization of telecommunications in Western countries (Bartle, 2002). The effect was enhanced in the early 1990s by the disintegration of the Soviet Union. Industrial innovation, which had been strategic in the Cold War, appeared less profitable. It was all the more the case in fiber optics, where the silica market for terrestrial and oceanic telecommunication networks had quickly expanded: around 100 million kilometers of silica fibers were installed up to the late 1990s (Kurkjian and Krol, 1998).

The rate of silica-fiber installation was around 5 million kilometers per year when the dot-com bubble, boosted by the world wide web, collapsed in March 2000. The burst of the bubble on the New York Stock Exchange sounded the death knell for fluoride glass R&D since major multinational companies withdrew. Large companies stopped their manufacture of fluoride fibers: AT&T, British Telecom, Galileo Electro-Optics, Naval Research Laboratory, NTT (Poulain and Poulain, 2015, p. 18). Small companies continued to compete in small space, military and medical niches: Le Verre Fluoré in France, ThorLabs in the US, FiberLabs in Japan. The “communism” feelings did not survive the lack of funding. The triple-helix around exotic glasses was sharply reorganized, which stimulated the circulation of researchers. In the US, when telecommunications companies like AT&T closed their high-quality R&D centers, dismissed researchers found academic jobs in university laboratories (Lucas, 2005, 14-15). In Rennes, when the brand optimization came to an end, chemists went back to the bench to carry on glass chemistry.

## Back to Bench: Chemical Skills for Materials Science in the Third Millennium

Lucas's group became an independent Laboratory of Glasses and Ceramics in 1992. In spite of its close acquaintance with the triple-helix of non-oxide glass materials, it never broke with chemistry. On the professional level, its members kept their affiliation with the chemistry department of the University of Rennes. On the epistemic level, MSE made them aware of the design of materials, including the importance of "extrinsic" properties, but they remained experts in the making of new compounds, not the optimization of well-known materials. Actually, the circulation from crystallochemistry to glass materials shaped new interdisciplinary practices between solid-state chemistry and MSE. The researchers from Lucas's group were bench chemists since they highly valued the synthesis of original compounds while chemists in MSE were supposed to optimize well-known compounds. They were also materials scientists since their syntheses were oriented towards the expected performances of brand products. The customized design of tellurium halide glasses (TeX) for military cameras and astronomy devices gives a good example to analyze the chemical creativity in an industry-driven academic research.

- *Bench Creativity and Interdisciplinary Practices*

The bench creativity of Lucas's group can be analyzed, *post facto*, by the articulation of three main tools: descriptive chemistry, periodic table, and crystallochemistry.<sup>30</sup> The first tool was the descriptive chemistry of the mid-century decades. Indeed, thousands of ternary diagrams had been published during the twentieth century without paying much attention to the amorphous domains since solid-state chemists and physicists mainly focused on crystalline compounds. Several types of publication were screened in Rennes to spot amorphous phases: chemical journals, encyclopedic books of inorganic chemistry, optics, etc.<sup>31</sup> The reading of old-fashioned

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<sup>30</sup> This methodology is a reconstruction based on the testimonies of J. Lucas (2005), J.-L. Adam (2006), and the Poulain brothers (2015). It is interesting to remark that, in spite of their sociological quarrels, they shared very specific chemical practices.

<sup>31</sup> Testimonies from Rennes respectively mentioned these three types of references: Soviet journals of chemistry; *Le traité de chimie minérale* in 12 volumes (1932-1934) and *Le nouveau traité de chimie minérale* in 20 volumes (1956-1964) edited by Paul Pascal (Pacault & Delhaes, 2007); *Fiber Optics: Principles and Applications*, written by the Indian-born physicist, N. S. Kapany, in 1968.

publications with fresh (glass-oriented) eyes gave clues to test original compositions with unpredictable results.

The second tool was the periodic table, which, one century after Mendeleev, remained the “catechism” of solid-state chemists (Lucas, 2005, p. 17). It was read dynamically by circulating along the lines, the columns and the diagonals of the table. The substitution of one element for another in the reagents was oriented by the relative position of their respective squares giving their properties (size, electronegativity). The bench success was linked to the chemist’s aptitude for reading the table according to his own memory of the past trials and errors.

The third tool was crystallochemistry. Indeed, if amorphous solids do not have a long-range order, they exhibit short-range arrangements of atoms. This local order was pictured by former solid-state chemists like the geometrical blocks (triangle, tetrahedron, octahedron) composing crystals. Let us remember the central role for Lucas’s team in the early 1970s of the pyrochlore structure by noting the ball-and-stick model in Figure 1. Lucas and his coworkers could thus imagine the modification of optical properties of glasses by modifying their local arrangements.

Each of these tools has been mentioned by other French solid-state chemists of the same period. Their articulation was oriented towards the comprehension of the relationship between the property of chemical elements and the geometrical organization of atoms. What makes the expertise of Lucas’s group original in European solid-state chemistry was twofold. The first one was the application of the solid-state methods to study amorphous glasses. Yet glass materials were too dirty and complex for the structure-property relationship to be clarified by theoretical models like crystalline materials.<sup>32</sup> This provided a kind of “modeling with hands” (*modèle avec les mains*) that stimulated chemists’ knowledge to invent new compounds. The second originality lay in the brand-orientation of bench practices.

- *Strategic Materials for Military Devices*

In 1984, the French military R&D agency (DGA) contracted Lucas to make a glass transmitting light in the infrared transparency window of the terrestrial atmosphere (8-12 microns). A Chinese student, Xiang Hua

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<sup>32</sup> “One has empiric models. With the means of calculation, one can model simple structures and some properties.” (Adam, 2006, p. 8). “We used simple molecular orbitals. We didn’t use the too complicated models of physicists. I think it would be big-sounding because we handle too complicated solids to give ourselves the illusion that were are great theoretical scholars.” (Lucas, 2005, p. 19).



Zhang, was hired by Lucas on a 3-year PhD grant (1984-1987). They found completely new tellurium halide glasses (TeX), whose transparency window was 2-20 microns, wider than the initial specifications (NOGS, 1988, p. 3). This was a new class of materials, known as TeX glasses or *Texglass*. Ten years after the fluoride experience, the laboratory held expertise in optics and MSE to design “molded lenses” for night vision infrared cameras. Just as for glasses, a decade (1986-1996) was necessary to complete the composition optimization and reach the performances required for optical, thermal and mechanical properties. Patents were taken with CNRS.

Zhang launched a start-up company, Vertex, with Lucas’s benediction. The context of technology transfer was better in France in the late 1990s than in the 1970s. The 1999 “law on innovation” of the minister of Education and Research, Claude Allègre, eased the founding of start-ups from academic research (Lucas, 2005, p. 10-11). Regional authorities (Bretagne), private investors (banks, joint venture, Umicore) and Lucas invested in the company capital. The business was profitable but the production limited. Umicore was the direct competitor of Vertex, through an alternative technology of infrared germanium lenses. It soon acquired Vertex. The multinational company would implement a change of production scale. The research program, commissioned and funded by the French State, through DGA, and carried out by public institutions (University of Rennes and CNRS), enriched both the first stockholders of Vertex (including the inventors) and one multinational company (Umicore). The infrared cameras based on the TeX lens found at Rennes would equip the French Army and expensive car models of BMW and Cadillac.

The accidental synthesis of fluoride glasses by the Poulains brothers in the mid-1970s was turned into an original program in the synthesis and design of non-oxide glass materials. The program hybridized the disciplinary matrix of solid-state chemistry towards the bench synthesis of new glasses (instead of new crystals) and the design of optical materials by adapting MSE practices to a chemistry laboratory. It was rewarded by the election of Lucas at the Academy of Sciences in 2004.

### **Conclusions: Chemical Skills, Division of Labor, and Innovation in Materials Research**

This case study on exotic glasses of Rennes exhibits three major features of materials research in the second part of the twentieth century: an international division of labor; an economic dynamic of innovation; and a disciplinary differentiation of knowledge.

Firstly, materials research was strongly framed by the science policy of national governments during the twentieth century. Materials research was mainly conducted through a disciplinary organization of solid-state physics and chemistry in continental Europe while the US built interdisciplinary programs in MSE to link fundamental solid-state physics to industrial requirements. These national differences in science-policy contributed to an international division of labor in the Western world during the Cold War: new solid compounds were more often synthesized by European chemists while new solid-state properties were more often characterized by American physicists and materials scientists. This provided two advantages for the US over their European allies: the symbolic capital to study “purified phenomena” instead of preparing “dirty materials”; and the economic and strategic capital to turn promising bench compounds into brand devices for industrial and military domains.

The University of Rennes exhibited two attitudes with regards to the international organization of materials research in the 1970s. The group of J. Prigent accepted the division of labor: it synthesized new crystals (Chevrete’s phases) and the group of B. Matthias at Bell Labs displayed their superconducting properties in the US. On the contrary, the group of J. Lucas synthesized an exotic glass and displayed its original optical properties. Then, it joined a triple-helix of university-industry-government around non-oxide glasses to escape its marginal position in French solid-state chemistry and contributed to the innovative design of exotic materials.

Secondly, the economic dynamic of innovation in advanced materials is based on the articulation of competition and cooperation, i.e. “coopetition”. There were two types of competition in the telecommunications race. On the one hand, US and NATO military agencies funded optic-fiber R&D until the end of the Cold War to beat the Warsaw Pact countries. On the other hand, multinational companies, from Asian, European or US origins, funded R&D on communication networks to beat their competitors during the dot-com bubble of the neoliberal age. The public image of Mertonian “communism” could not survive the funding decrease of the 1990s. Secrecy played its crucial role in partitioning knowledge.

However, secrecy went side by side with a quick circulation of knowledge, practice, equipment and money for medical, military, space, and telecommunications materials. The innovation backstage was full of actors with a huge variety of size, temporality and goals: the secretary of the Non Oxide Glass Society, who edits *NOG News* (budget of \$10,000 annually); the start-up companies with 2-3 employees (\$500,000); the academic laboratory (\$2,000,000); the International Symposium on Non-Oxide Glasses; R&D centers from several multinational companies (\$100,000,000) and na-

tional research centers (CNRS); military institutions (DGA, NATO); and states, including France, UK, and the US. The advancement of materials was reached through the collaboration between universities, industries, civil and military agencies.

The interaction between public and private agents played a special role in the collaborative process. The technology transfer from public academe to private industry was made easier from the mid-1970s (Le Verre Fluoré) to the late 1990s (Vertex). This was induced in France by the relative decrease in public R&D funding from the early 1970s, prior to the US Bayh-Dole Act of 1980. The impression given by Lucas's group is that the public revenue funded most of the total budget, including the salaries of scholars accounting for three quarters of the total, while industrial contracts, either civil or military, oriented research. The case of Vertex is of particular interest in this respect: the funding was 100% public (through Education and Army) while the start-up was bought by the dominant company on the market: Umicore. For the sake of strategic options and economic impetus, administrations supported material glasses for the benefit of private companies.

Last but not least, the division of labor and the dynamics of innovation relied on the disciplinary differentiation of knowledge. The choices of Lucas's group required the reinvention of the research portfolio along two epistemic shifts. On the one hand, *crystallo-chemistry* was turned into *glass-chemistry* to nourish a synthetic creativity in the making of glass compounds. On the other hand, the optimization and design of fibers became a routine activity to increase the optical performance of glass materials. The cross-fertilization of synthetic creativity and materials design favored the understanding of chemical canons (furnaces, old-fashioned literature, periodic Bible, crystallochemistry) with fresh eyes. In addition, thousands of trials and errors, thorough instrumental characterizations, contradictory discussions, and total failures were also needed for Michel and Marcel Poulain as well as Jacques Lucas and Xiang Hua Zhang to create – by imagination and actual making – new forms of glasses: heavy-metal fluorides and tellurium halide glasses. The chemical skills of the group cannot be understood without the subtle association of creative gestures (arts) and repetitive practices (sciences).

Contrary to Prigent's group, Lucas's laboratory modified its disciplinary identity of solid-state chemistry and changed its place in the international division of labor. From then on, it both provided new compounds and characterized them. It was emblematic of a wider evolution that worried American scholars and, probably, policy-makers. Indeed, European and Japanese solid-state chemists had increased their interest in physical charac-

terization in the 1980s (Di Salvo, 1987, p. 165). The interaction of academic chemists with MSE was stimulated by the decrease in public funding during the economic crisis of the 1970s. Since US chemists did not show much interest in the art of creation in chemistry, the dominant position of the US in the division of labor was threatened.

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<sup>33</sup> If no other mention, the interview was made by P. Teissier in French. Some of the interviews can be read on the following website: *Sciences : Histoire Orale*, <https://www.sho.espci.fr/?lang=en>

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## **Part III**

*Innovating and Recycling: Telling the Stories of Materials*

## Making Sense of Chemistry: Synthetic Rubber in German Popular Scientific Literature (1929-2009)

Jens Soentgen\*

### Abstract

*The chapter analyzes the popular literature on synthetic rubber between 1929 and 2009 and asks how popular science books explained and interpreted rubber research and development to a general (German) public. How does popular literature produce different narratives, including myths, of the same history? The invention and use of synthetic rubber were important topics in popular science literature in the Weimar Republic and during the Nazi period as well as after 1945 in the German Democratic Republic (DDR) and in the Federal Republic of Germany (BRD). Narratives and argumentative schemes of these books are analyzed, and it is shown how they constructed the social meaning of rubber and how the positive and negative resonance of this construction changed according to different political contexts.*

*Keywords: natural and synthetic rubber, Buna, substance histories, polymer chemistry, Nazi-period, Auschwitz, popular science, history of chemistry, myths of science.*

### Résumé

*Ce chapitre analyse la littérature populaire sur le caoutchouc synthétique entre 1929 et 2009 et questionne la façon dont les livres de science populaire expliquent et interprètent la recherche et le développement sur le caoutchouc à un public généraliste allemand. Comment la littérature populaire produit-elle différents récits, notamment des mythes, d'une même histoire ? L'invention et l'utilisation du caoutchouc synthétique furent des sujets importants dans la République de Weimar et durant la période nazie ainsi qu'après 1945 en République démocratique allemande (DDR) et en République fédérale d'Allemagne (BRD). L'analyse des récits et schémas argumentatifs de ces livres montre comment ils construisent la signification sociale du caoutchouc, qui change de résonance, positive ou négative, en fonction du contexte politique.*

*Mots-clés : caoutchouc naturel et synthétique, Buna, histoire des substances, chimie des polymères, période nazie, Auschwitz, science populaire, histoire de la chimie, mythes de la science.*

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ALTHOUGH synthetic rubber was produced on a small scale for research purposes in 1909, its social life did not begin before 1915. The first large-scale industrial production of synthetic rubber took place in Elberfeld (today a city suburb of Wuppertal, Germany), then (after 1916) in Leverkusen.

The following study investigates political narratives centering on this extendable substance. In the first place, popular non-fiction books will be evaluated. These contain what the science philosopher Ludwik Fleck (1980, p. 149-150) called exoteric knowledge, i.e., knowledge which is simple and convincing, not as complex as the expert's knowledge: "Out of the expert's (esoteric) knowledge arises the popular (exoteric). It appears thanks to the simplification, vividness and apodicticness certain, well-rounded, solid. It forms the specific public opinion and the world view and in this form reflects back onto the expert." For Fleck this popular knowledge is embodied above all in "popular books", against which he contrasts "textbooks". Popular science books have been a theme several times in science studies (Bell & Turney, 2014; Bertschik, 2008). Authors of such books are sometimes scientists, more often writers or journalists (often with a scientific training). These authors are not to be seen as creative inventors of completely new ways to think or write or act. They try to approximate with their narrations the attitudes, mindsets, values, world-views of the readers and thus to foster acceptance and demand for their book. Readers always include professionals and scientists, but the group of readers is much larger. Popular science books reach thousands, sometimes hundreds of thousands of readers. They are mostly seen as non-fiction literature, however most of these books include fictional literary strategies known from novels or even science-fiction literature. With these, the author tries to entertain his readers. He has to model his personalities also according to the expectations and according to the value system of the readers, in order for his story to reach acceptance.

Popular science books are important for science itself: without popular scientific books that he and others wrote, Einstein's relativity theory would hardly have become a societal event because not many people outside of the scientific community would have taken notice of this theory. Darwin's theory would also have hardly been socially influential without popular books. Popular science books put into perspective scientific research and research results for the greater scientific as well as non-scientific public. They give science a meaning, in the framework of stories, that also reflects back on the scientists themselves, as Fleck (1980, p. 149) already noted. They have at least an indirect impact on the course of science, because they attract young people and encourage (or discourage) them to be-

come scientists. They legitimate or de-legitimate scientific work and thereby influence the funding which society spends on science.

Popular scientific books are a space of transaction between the general public and science. Through them certain areas of science – or also science as a whole – are presented and are religiously, culturally, and politically positioned. My aim in this paper is to describe the mode in which this is done. How do these popular books make sense of rubber-synthesis and rubber-chemistry? Alongside this basic question more technical questions are also to be answered: What is told, and what remains untold in the popular literature? How is rubber-chemistry described and put into political and cultural perspective in the different Germanies that existed in the time-period analyzed?

As I have already explained what I mean by popular books, it remains to discuss the other central concepts that are important for my paper: discourse, narrative, chronicle. By narrative I mean a narration which integrates (struggling) persons, groups of persons, situations, events, and things and which is written or told with the intention to entertain and to inform the reader or the audience. A chronicle is a written sequence of events. Discourse has to be explained a bit more thoroughly. Popular books on certain sciences (chemistry) or on certain scientific achievements are not isolated productions; rather, they form a discourse, a side-discourse to the scientific discourse. They react critically or affirmatively on each other, use the same *topoi*, i.e. the same narrative and argumentative schemes, because the authors of popular books on synthetic rubber read other popular books. But they are not only discourses in the sense of the linguistic discourse analysis, but also in the stronger political and epistemological sense which Foucault inaugurated (Foucault, 1981, p. 74). They create a certain perspective on the historical situation and the role that science plays within it. They attribute a certain political sense to the work of a certain group of people. They have also an economic impact. However, they should be distinguished from mere economic advertising which may be intended to create brands.

The discourse of the popular science books on rubber which analyzed here is a part of the more general public discourse on chemistry and industry. There is lot of evidence that chemistry was already perceived in the German Reich, but especially from the Weimar Republic on, as the most modern and most fascinating natural science, though certain critical accounts were already published (Woker, 1925). This public appreciation of chemistry reached its absolute climax in the “Third Reich”. Schenzinger’s novel *Anilin* was the most successful novel in Germany in the Nazi-period. After WWII, industrial chemistry, although deeply involved in the Holo-

caust, was still highly esteemed: as highly in the Federal Republic of Germany as in the German Democratic Republic.

This discourse is not only linguistically, but also politically nation-focused. Science – in this case, rubber-chemistry – was related to Germany, but not to an isolated Germany. Germany and German science were seen in a European, transatlantic, and colonial context. However, as already mentioned, there were many Germanys in the time-period that I will analyze: The German Empire (1871-1918), the Weimar Republic (1918-1933), Nazi-Germany (1933-1945), and then the Federal Republic of Germany (since 1949) in the West and the German Democratic Republic (1949-1990) in the East. Synthetic rubber was produced in all of these states, and books on synthetic rubber have been written since the Weimar Republic. That makes an analysis of popular rubber-literature even more intriguing. I collected a corpus of 28 popular German books which deal, sometimes only within a single chapter, with synthetic rubber. The criterion whether or not to include a book in the corpus was that it signaled explicitly or indirectly (by its style, through the use of pictures, absence of scientific details etc.), that it was written for the general reader, not for a limited audience of, say, rubber scientists. The search for these books could not use a systematic method but had to proceed by means of serendipity. Books that had been translated into German were not included systematically. However, these books were also collected and have been taken into consideration, as they influenced the German literature. Thus, the popular books on “red rubber” and the “congo-state” by Arthur Conan Doyle, Edmund Dene Morel and others which were translated into German before WWI proved to be important for the German literature on synthetic rubber.

The analysis of the corpus<sup>1</sup> exhibits ruptures and continuities of discourses during the 20th century. On the one hand, the deep political changes induced several narrative changes in the popular literature on synthetic rubber. On the other hand, there are two striking continuities in the industrial production of rubber in Germany and the hero-making of rubber chemists during the whole period.

Popular books on science always present narrations. A narration not only deals with processes, but also with the actions of people who have characters, friends, and foes. These are modeled according to the values of the either imagined or factual readers of the book. The narrative often follows a certain scheme, for example the “hero saves his people”. Especially through the narratives, and somewhat less through arguments, figures, and

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<sup>1</sup> Not all of the collected books are quoted in this paper. The author will provide the full list of his corpus on request.

facts, the authors of these books try to ‘make sense of rubber-chemistry’. This sense-making means that ideas, experiments, inventions, and industrial activities are integrated into a greater context. They receive a political and cultural significance. They tell the readers something about the meaning of the enigmatic activities of the chemists in the laboratory. Such sense-making would not be successful if it were a mere construction. It uses historical facts, but it combines them in a way that would not be the way a rubber-historian would deal with these facts. They are combined in order to produce a certain emotion, a certain attitude towards chemistry. In the literature investigated here, this attitude is positive. The popular rubber-literature in Nazi-Germany in particular shows this, in that the chemist is stylized as hero and liberator. It is suggested that with his hard, lonely laboratory actions he wants to serve world peace, understanding among peoples, and universal justice. Chemists and rubber chemistry are presented in this manner in the works of Anton Zischka, Karl Fischer and others in the 1930s and 1940s as well as later.

The popular discourse on synthetic rubber in Germany must be distinguished not only from rubber-science itself, but also from the professional historical discourse on the rubber industry. Although the popular rubber books also deal with history, they deal with it in a quite different way than historians of economy or technology or science do. The presentation is much more emotional and does not aim at answering historical questions, but aims at creating motivating images and emotions.

In order to understand exactly why German synthetic rubber – made out of the materials coal and limestone, which are both available in Germany – achieved national importance, the first half of this paper will be devoted to orienting rubber on the political and historical map. We need at least a short chronicle of rubber and rubber-chemistry, a short table of events with the least interpretation possible. Without such a table, it is not possible to interpret the popular rubber literature properly. The following can be seen in this sense as a contribution to a cultural and political history of science.

### **A Short Historical Chronicle of German Rubber**

- *The Colonial Context of Natural Rubber*

Produced from the milky sap (latex) of specific tropical plants, caoutchuc was already known in the cultures of South and Central America long before the arrival of the Europeans. As Las Casas reports, Christopher Columbus brought such a (caoutchuc) ball, as “large as a jug”, from the

New World to Seville. This ball was the very first caoutchuc product that reached the soil of the Old World.<sup>2</sup>

At first only the indigenous peoples, who had access to the latex sap and knew how to process it, had a quasi-monopoly on the material because back then the milky sap as such did not transport well from America to Europe. Only the goods that the Indians made in a complicated process that included a biological vulcanization were traded, not the latex sap itself. The discovery of the possibility to make rubber shoes and rubber flasks plastic again and to form and to make the product preservable with sulphur enabled the build-up of a European and American rubber industry.

As natural rubber, *caoutchuc*, *India rubber*, *gum-elastic*, *borracha*, *hule* and under some other names from the middle of the 19th century, caoutchuc rapidly spread to all of Europe and worldwide. At first primarily used for watertight shoes and coats, it soon served mobilization: In 1888 John Boyd Dunlop developed an air-filled rubber tube for bicycles and later for the manufacture of automobile tires (by Michelin, 1894). Natural rubber was also indispensable as an insulator for the developing electric industry. Rubber became a key substance of the industrial revolution.

Although latex-producing plants and trees also occurred in the Old World, the *Hevea brasiliensis*, which primarily grows in the Amazon region, provides an especially valuable caoutchuc in ample quantity. The most important route of the rubber thus went from the upper Amazon via Manaus to Belem-do-Para and; from there over the Atlantic. The flow of rubber corresponded to a flow of money in the reverse direction which made some people in jungle cities, above all Manaus, rich for a short time.

- *Brand Materials of the German Industry: Buna-S and Buna-N*

The monopoly position of Brazil was uncomfortable for the industrial nations which were increasingly dependent on rubber (especially because of the rise of automobile-industry). In 1876 the English planter Henry Wickham secretly shipped to England a huge number of seeds of the *Hevea brasiliensis* which had been collected for him by indigenous tribes (Jackson, 2008, p. 288). They were raised there and later shipped to tropical colonies of the United Kingdom. From 1889, caoutchuc also came from the British and Dutch colonies in Southeast Asia, whereby the monopoly position of Brazil as a supplier of pure natural rubber was ended.

Other industrial countries without colonies could either buy rubber on the world market or produce caoutchuc artificially out of other, more

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<sup>2</sup> For one history of caoutchuc, see (Soentgen, 2013), which provide extensive literature/references.

easily accessible substances. Although the molecular structure of caoutchuc was unknown at the beginning of the 20th century, several countries found ways to produce synthetic types of rubber. In the Soviet Union, synthetic caoutchuc was produced out of ethanol, according to a recipe of the chemist Sergej Lebedev; the spirits were produced from potatoes (Lewis, 1979; Plumpe, 1990, p. 355). The US stockpiled huge rubber reserves and later produced synthetic rubber out of oil (Morris, 1989; Plumpe, 1990, p. 355). In the conflict-rich era of imperialism, marked by competition among the great powers, one did not necessarily want the energetically best synthesis or the synthesis that was technically the most elegant, but rather one that could not be blocked by other powers. Therefore, the Germans chose coal and lime as starting materials, because they were certain to have enough of these in their own country. Fritz Hofmann (1936, p. 424), the inventor of the first German synthetic rubber, emphasized this in his 1936 retrospective:

“From raw materials, which in any amount at any time sufficiently cheap stand at our disposal, we had to proceed if we wanted to come closer to our goal. Of such raw materials in our zone we do not have many. The potato scarcity in the World War has shown us, that even this in normal times abundantly available fruit for the purpose of nourishing animals and people, will be completely claimed if our borders are threatened or even blockaded. But exactly in this situation we must have caoutchuc freely at our disposal. Therefore we have not, as Russia did this, built up our butadiene out of potato spirits, although we knew this route for a long time, but rather we have stayed with coal, of which for many generations forth the most abundant amounts are available.” (Hofmann, 1936, p. 424)<sup>3</sup>

German synthetic rubber takes its place in a long line of substitutions and syntheses through which since the 19th century Germany achieved increasing independence from imports from colonial powers and transformed from a dependent recipient of colonial commodities (which foreign colonial powers produced) to an export nation. Beet sugar, camphor, indigo and other dyes, vanillin, ammonia and with it nitric acid and saltpetre (Plumpe, 1990, p. 203-243) – they were all expensive trade goods that had to be imported until, thanks to chemical research, they were henceforth produced in the country itself and exported. During WWI, the Haber-Bosch process, delivering reactive nitrogen, was particularly decisive for maintaining the German battlefield, which would otherwise have broken down from lack of ammunition as early as 1915. Altogether one can speak

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<sup>3</sup> All the translations of the German quotations are from the author.



in retrospect of a compensation strategy, because the syntheses and substitutions replaced the lack of colonial production locations. This nexus was also noted abroad by the American Chemist Edwin Slosson :

“Long ago it was said that the British ruled the sea and the French the land so that left nothing to the German but the air. The Germans seem to have taken this jibe seriously and to have set themselves to make the most of the aerial realm in order to challenge the British and French in the fields they had appropriated.” (Slosson, 1921, p. 23)

Synthetic rubber joins in here: it was christened with the name Buna, shortened from the starting materials *but*adiene and *na*trium (German for sodium), which was used as a catalyst. A variant is Buna-S, a so-called mixed copolymer, in which styrene is mixed in. This Buna-S is even today by far the most important synthetic rubber internationally, because it is especially suited for automobile tires. These still consist, for the most part, of this material. Besides Buna-S, Buna-N is also produced, which is similarly very wear-resistant, but in addition is resistant to organic solvents and oils. Buna-SS is even more wear-resistant than Buna-S and similarly is especially suited for tires. Lastly Buna 85 and Buna 115 are produced, the so-called numbers Buna (“Zahlenbuna”), which are distinguished by their heat resistance (Treue, 1955b, 256). Altogether world rubber production is nowadays split into two thirds synthetic and one third natural rubber.<sup>4</sup>

The initiative for rubber synthesis came from the German chemical industry: The Bayer management conference of 18 October 1906 offered a prize of 20,000 Marks as a reward for the chemist who could find a process for synthesizing rubber or a substitute before November 1909 (Plumpe, 1990, p. 342). The chemist Fritz Hofmann took up the challenge and had success. In the laboratory in Elberfeld (today a city suburb of Wuppertal, North Rhine-Westphalia) he succeeded in polymerizing the hydrocarbon isoprene. The German Imperial patent office issued the dye factories previously known as Friedrich Bayer & Company in Elberfeld the patent Nr. 250690 for the “process for production of synthetic rubber” (Lanxess 2009a, p. 6). Later Hofmann developed a further synthetic rubber, methyl rubber. The German Kaiser Wilhelm II demonstratively supported this German material: In 1912 he outfitted his state limousine with automobile tires out of methyl rubber. Hofmann’s methyl rubber was, however, too expensive for peacetime. In addition the Continental (company) in Hannover, a major tire company, declined to process it further because the quality was too poor (Plumpe, 1990, p. 343).

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<sup>4</sup> Personal communication from Dr. Ernst Schwinum, Leverkusen.

- *Industrial Production of Rubber in Wartime Germany*

Nevertheless, in the war year 1915 it went into large-scale production, since the German Empire was cut off from the supply of natural rubber during World War I (Plumpe, 1990, p. 343-349). Rubber materials were of strategic importance, because, among other things, they provided insulation in the batteries of German U-boats. They were also necessary for gas-masks. The rubber-shortage in Germany made gas-masks frail and leaky (Slosson, 1921, p. 153). Until the end of 1919 the plant in Leverkusen produced 2524 tons of synthetic rubber. By today's measures this is not much, but it was sufficient for the German U-boat fleet of that time. After the war's end production was again discontinued because it was not profitable and in addition methyl caoutchuc's quality was too poor for use in automobile tires. Instead, natural rubber was used again. Between WWI and WWII, rubber research was taken up again in Summer 1926 by the IG Farbenindustrie AG (Plumpe, 1990, p. 349).

On the eve of World War II synthetic rubber, now in the shape of Buna-S, was again placed at the center of the national agenda. This substance was developed by Bayer in 1929, building on the prior work of Hofmann. At first they had not thought of industrial production, although Buna-S had many advantages over the methyl caoutchuc of World War I. This synthetic rubber had what it takes to be able to replace natural rubber in many important applications. But it was much more expensive than natural rubber. In October 1930, IG stopped the synthetic rubber project. Thus, as it seemed at first, this synthetic rubber would be excluded from having a societal and political life. The "salto mortale" which, according to Marx's analysis, every ware on the market has to make in order to transform the invested labor value into exchange value (Marx 1983, p. 67), failed, and indeed fundamentally.

With the Nazi seizure of power things changed. The Reichswehr was interested in synthetic rubber and established contact with the IG (Plumpe, 1990, p. 357). In Hitler's secret memorandum to the Four Year Plan, written in August 1936 and which only Göring, Blomberg, and later (1944) Speer received, the material was ordered, whatever its cost might be. Hitler had a very strong interest in synthetic rubber. It is speculation whether this connects to the biographic fact that he was poisoned with mustard gas in October 1918, perhaps due to an inefficient, rubberless gas-mask. In any case, he knew that for modern, highly motorized war rubber was indispensable. And chemistry was to deliver it. In *Mein Kampf* he had already emphasized that the present "is reigned by technology and chemistry" (Hitler, 1943, p. 469). In his memorandum he wrote:

It is just as obvious to organize and secure the mass fabrication of synthetic rubber. The claim that the process is perhaps still not completely clarified and similar excuses are from now on to be silent. [...] The question of the cost price of this raw material is similarly completely irrelevant, for it is still better we produce expensive tires in Germany and can drive on them [...].<sup>5</sup>

Hitler pushed through 'his thing' against strong doubts, such as those expressed by Hjalmar Schacht (Treue, 1955a, p. 195-205). The German synthetic rubber became a reality in the very same year.

The goal that Hitler's order for rubber production should serve was clear, if one considers that rubber is essential for military vehicles, tanks, planes. More than 10 percent of the weight of a U-boat consisted of rubber (Klemm, 1960, p. 53). Hitler expressed his secret goal in 1936, which he endeavored to reach via synthetic rubber and the Four Year Plan at the end of his document: "I therefore set the following tasks: I. The German Army must be combat-ready in four years; II. The German economy must be war-capable in four years" (Treue, 1955a, p. 210). Synthetic rubber came into the world as part of the Nationalist Socialist mobilization, together with the Volkswagen, the autobahns, and finally the *Blitzkrieg*. Because Buna-S was three times more expensive than natural rubber, it would presumably have never gotten off the ground if not for its political godfather and the lack of German colonies. But when power politics necessitated ramping up production of synthetic rubber on a large scale for the purpose of war preparations, the innovation process took effect and the necessary technical knowledge grew apace, until synthetic rubber could compete with natural rubber (Streb, 2003, p. 97-132). The capabilities of the chemists and engineers zealously followed Hitler's desire (Maier, 2015). The entire rubber industry was "bunized", for the new peoples' comrade could not be processed with the machines used for natural rubber. Not only was the industrial technology in no way designed for the stuff, but economic concerns were also expressed. The rubber processing industry resisted, explained that the new material required five times, if not eight times more processing effort than good old natural rubber (Treue, 1955b, p. 256). This did not hold up the order. Thanks to new machines and sales paths, numerous solutions to technical problems (Erker, 2005, p. 423-445), inventions, and patents, whole new factories grew up with breathtaking speed around the substance. Hitler's authority allowed the previously only

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<sup>5</sup> From (Treue, 1955a, p. 208). Treue (1955b) writes "augenblicklich" ("immediately") instead of "augenscheinlich" ("obviously").

dreamed of synthetic rubber to become a reality. In a monograph the economic historian Jochen Streb (2003) has thoroughly analyzed the successful National Socialist innovation politics and compared it, at least from a purely technical standpoint, with the United States in the same epoch.

Also in 1936 the IG Farben concern began the construction of a major industrial plant for production of synthetic rubber in Schkopau in Saxony. Later large plants were also set up near Marl, Ludwigshafen, and Auschwitz. All these plants still produce synthetic rubber today. Above all, Buna-S was produced since it was best suited for tire production (Streb, 2003, p. 99). The build-up of the synthesis route per order was successful; in Germany, Buna-S production exceeded domestic consumption in 1943.

### **The Making of Rubber Narratives in German Popular Literature**

I dealt so extensively with the chronicle of the German rubber industry because it is only possible to determine what is lacking in German popular books on the topic if one has a sufficient background of sound historical information. The narratives presented there create their specific perspective mostly by the emphasis and amplification of a selection of facts. We do not find, for example, any mention in the popular literature of the reluctance of the rubber industry, to work with the expensive synthetic rubber. Instead, everybody works together to achieve the noble goal. Not only the machines were “bunized”, but also peoples’ minds. The Führer’s national emotionalism was so strongly projected onto domestically-produced rubber, even before the construction of the Buna plants, that it experienced a sort of second, ideological vulcanization. Home-made synthetic rubber was perceived as a valuable national product. Yet at the same time, it was seen as a contribution to the progress of all mankind because synthetic rubber liberated the oppressed and was said to bring more justice to the world. In this way users’ acceptance was fostered and its legitimacy was underscored with regard to Germany’s distrusted neighbor-states.

- *“Red Rubber” in the Colonies of the European States*

Just as politics motivated and steered work on rubber synthesis with its goal setting, thus now the scientific-technical result was reformulated into a political victory. German synthetic rubber was embedded in the great national myths, so that not only the invention was celebrated as a patriotic feat, but also its use was virtually equated with a fulfillment of duty. It was taken as a demonstration of where the new Germany stood and where it wanted to go. It is important to note that these national goals were not un-

derstood as an outflow of hubris, but rather as a specifically German contribution – completed with science and technology – to world peace, to reconciliation between peoples, and to the liberation of the oppressed.

In particular, the ‘cleaner’ character of the new production methods was often pointed out. In the view of authors in that era, this ‘cleaner’ production rose above the cruel production methods of the colonial rulers. German non-fiction books brought out the gruesome dark sides of natural rubber, the rubber of the others.

These dark sides were well known in those days. They had come to light through popular books. In 1906 the British-French journalist Edmund Dene Morel published his attention-arousing work *Red Rubber* (from which parts were translated into German), in which he denounced the conditions in the Congo, which since 1885 was a “free state” in possession of the Belgian king Leopold II. At the center of Morel’s critique stood the system of forced labor introduced by the Belgian monarch, which required the natives to collect caoutchuc for the agents of the king, who afterward sold it on the European markets. Gatherers who refused to participate in the production or who did not deliver enough caoutchuc were victims of sadistic punishments. The merciless exploitation by the Belgian colonial rulers, who in no way spared women and children, led to such a massive decimation of the local population, that today it is referred to as a genocide. These events were not just described by writers – such as Joseph Conrad (1902), whose *Heart of Darkness* reflected the experiences of a journey in the Congo – but were also denounced by missionaries, travelers and politicians. Horrors were also known from the Peruvian Amazon area. In 1909 Roger Casement reported on the orgies of cruelty in the caoutchuc areas on the upper Amazon (Taussig, 1984). In 1913, Walter Ernest Hardenburgs published *Putumayo – The Devil’s Paradise*, in which he reported on a system of forced labor on the Putumayo River in northwest Amazonia that was shockingly similar to that which Morel had described (Taussig, 1984).

In Europe it was generally known that natural rubber was a blood-stained thing. One might wonder whether the decision of the Bayer management conference in October 1906 to foster research on synthetic rubber might have been influenced by Dene Morel’s revelations concerning the red rubber, which were published the same year and immediately sparked discussions internationally but also in Germany. This is not very plausible, though, as there were strong enough economic reasons: “the world was willing to pay \$2,000,000,000 a year for rubber and the forests of the Amazon and Congo were failing to meet the demand” (Slosson, 1921, p. 146).

- “Buna – Victory of Reason!”: *Science and Technology for a Peaceful World*

In the popular literature, Germany's lack of colonial experience was turned from a deficit into a plus, in order to equip the synthesis program with an aura of moral superiority. Germany's separate scientific-technical modernization was put into perspective as an exemplary German and morally superior route. It is no accident that the depiction of the horrors in the Congo as well as those on the Putumayo (Fischer, 1938, p. 37-87; p. 118-148) appears at the beginning of a political-technological vision in Karl Fischer's non-fiction work *Blutgummi* (*Blood Rubber, Red Rubber*). That vision imagined the industrial rubber synthesis developed by the German scientists leading away from the cruelties of colonial caoutchuc production. Cleaner science and technology legitimized Germany's claim to leadership. In this ideologization synthetic rubber adopted an identity-stabilizing function: synthetic rubber materializes the moral and intellectual superiority of the Germans.

The chapter dedicated to the new substance in the book *Blutgummi* is called: “Buna – Triumph of Reason” (Fischer, 1938, p. 207). In it the German substance is portrayed not only as technically superior, but also as the fulfillment of humanitarian values. Indeed, it is conceded that the British plantation caoutchuc was a first step to more humane production methods. But one had stopped halfway: “If now there was no more struggle over bloody rubber and no-one need any longer lose health and life, however, thus continued the monopoly reign of the rubber barons – only that instead of the whip the price tag stepped in” (Fischer, 1938, p. 18).

In this perspective overcoming “the capitalistic and geographic monopoly through the strengths that are awarded to the human understanding and not their economic power: through the synthesis of mind and nature” (Fischer, 1938, p. 19) was reserved for synthetic rubber. Toward the end of his book Fischer declare festively: “But as in those days the first ton of plantation caoutchuc already meant a victory over red rubber, so already the first Buna tires mean a triumph of moral and reason over the speculative economy, a triumph of mind over money and the market” (Fischer, 1938, p. 241). At the same time he emphasized the nationality of the substance: “What is here produced is German rubber, rubber whose development one regulates, whose characteristics one can adjust to the requirements of its later life, that one can make more wear resistant or more oil resistant or more aging resistant than the natural rubber” (Fischer, 1938, p. 240). Synthetic rubber thus reveals itself as a Prussian soldier, duty conscious and resistant, scientifically structured through and through, exact, obedient, and mission-ready at any time. Rather than a bad substitute for natural rubber, it confidently appears as perfection become substance.

The writer Anton Zischka also celebrated the German rubber story in his work *Science Breaks Monopolies*, which was printed more than 600,000 times and also translated into 16 languages (Weber, 1999, p. 219). The rubber chapter of the book was entitled “Rubber from lime and coal versus ‘red rubber’ (Blutgummi)”. Zischka (1937, p. 185), whose works were also printed in large numbers outside Germany (on Zischka with further references Weber 1999, Hahnemann 2008), explained: “rubber from lime and coal versus ‘red rubber’, this is only a single building block in the great building of the new world. But it is also a symbol. Peace and progress instead of war and plunder. Science will make a reality out of an utopia. German science especially”. In Zischka’s logic the fight against monopoly leads, with the help of science, not to war but by logical necessity to peace: “If we [...] break monopoly... then we overcome also the fear of hunger and exclusion. Then we fight jealousy and enviousness. Then we work for a *lasting* peace, for who would fight for something that *all* have?” (Zischka, 1937, p. 185, his emphasis). The researchers thereby get a key role: “Monopoly upon monopoly was broken, step by step the forward-probing researchers conquered ever new living space, *ended fights over raw materials, by making them available to all.*”

Here is sketched, in contrast to other forms of global economics, a specifically German modernization ideology that is based not on exploitation, but on science and technology. This should be, so it is claimed, the best politics of peace, because with the lifting of scarcity the reasons for war are also removed. This ideology is not German in the sense that it is a German “invention”. We find it already fully expressed in the works of, for example, Marcelin Bertholet, the most influential French scientist of the late 19th and early 20th century. Bertholet, the inventor of the term “chemical synthesis”, already imagined a clean world of synthetic wonders in the year 2000, where not only materials but also human food would be produced synthetically out of carbon dioxide and water, the necessary energy being delivered by the sun. Human minds would be made peaceful by certain chemicals... (Bertholet, 1896, p. 508-515). The topos could even be traced back to the alchemists of the early modern era who, like the Rosicrucians, combined a chemical utopia (production of gold out of other metals) with a social one... But let us return to popular literature.

Karl Aloys Schenzinger (1937, p. 375-376), one of the most successful German authors of the first half of the 20th century, similarly used the topos in his novel *Aniline*. Schenzinger, however emphasized above all the national increase in power that would become possible through the synthe-

sis.<sup>6</sup> The widespread influence of Schenzinger's novel can hardly be overestimated. *Aniline* was the most sold book in the Nazi period in the German Reich, with a printing of over 920,000 copies alone until 1944 (Schneider, 2004, p. 80-81), around one tenth of the number of *Mein Kampf* copies.<sup>7</sup> It formed the chemical understanding of multitudes of people and was even successful after the end of the war in a lightly cleaned-up edition. The book was reprinted in Germany into the 1970s.

The chemist is often depicted in Schenzinger's novel as exemplifying the "Nordic performance type" in the sense of the colorful Husserl-student and race-theorist Ludwig Ferdinand Clauß (1929, p. 1-10). These men are performance-oriented, they do not make a fuss about themselves, they are objective: one contrast to the Nordic type in Clauß' typology is the audience-oriented "Mediterranean show-type" (Clauß, 1929, p. 11-15). This idea of a typical German character who is tough, does not give up, etc. was also prominent in the self-perception of German chemists (Duisberg, 1933, p. 207).

As most of the quoted books were published in the Nazi period, it is important to note that the central narrative of the scientist who liberates his country with his inventions (Soentgen, 2014) has older roots. It was already present in a seminal state in the science-fiction novel *Kautschuk* by Hans Dominik, which was published during the Weimar Republic in 1930. There, the chemist Dr. Fortuyn invented the "electro-synthesis" of rubber, which surpasses even chemical synthesis. Spies from other countries (USA, France, Great-Britain) try, with a great deal of criminal energy, to get hold of the secret. But they fail. In the end, the foundations are laid for gigantic synthetic rubber plants. The rubber-plantations in tropical countries will be stubbed out...

Carl Duisberg, the head of the supervisory board of IG Farben and the most powerful chemist in the Weimar Republic (and at that time probably worldwide) also had the scheme in mind when he explained in a public

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<sup>6</sup> The following passage is deleted in the post-war edition: "No naphtha source, no oil, no rubber in one's own country. No colonies. Dangerous sums threaten to flow out to foreign countries. We are hemmed in, geographically, scientifically, politically. We want to live! Ever louder is the support for the artificial material. The artificial material today determines the future of the German nation. The artificial material has become a question of German existence. But there now the German chemist is already aroused, [...] From coal and lime one came to calcium carbide, from there to acetylene, from acetylene to butadiene through polymerization to Buna, to synthetic rubber." (Schenzinger, 1937, 375 f.)

<sup>7</sup> Hitler's *Mein Kampf* had been printed 10,240,000 times by 1943 (Hitler 1943, cover).



speech in December 1929 that the chemical industry had a special significance for Germany because only that industry could transform poor German raw materials into valuable products (Duisberg, 1933, p. 208). Fritz Haber had very similar thoughts of (1927, p. 9): in a speech in Argentina in 1923 he emphasized that chemistry was a typical German thing, as in Germany chemistry compensates for the lack of raw materials with “innovative mind”.

The quotations make clear that the chemical synthesis was presented as an ethically motivated compensation strategy of a people, who fell short in competition for colonies due to their tardy national unity and consequently based their economic – and thereby also political – power on chemical synthesis. Chemistry, however, was supposed to not only free the Germans and make them powerful, but also bring peace, justice, prosperity, and freedom to all mankind. German scientists would solve the resource-scarcity-problem and allow everyone to live in peace. The history of German synthetic rubber in the subsequent Nazi Period is as far from this claim as one could possibly think. German synthetic rubber did not and was not meant to serve peace and justice and freedom. We have to turn to the chronicle once again.

- *The German Red Rubber: IG Farben Buna in Auschwitz*

The IG Farben Company – attributed such an important role as a liberator of mankind by Schenziger – instead used the Holocaust for its own purposes. The IG Farben plant Buna IV, set up at the desire of IG Farben after 1941 in the camp Auschwitz-Monowitz under the supervision of the SS, had the purpose of supplying synthetic rubber (Lautenbach, 1995). This decision, made in the IG Farben headquarters in Frankfurt, contributed to the fact that the camp in Auschwitz was built up into the central death camp in the system of National Socialist concentration camps. Through this decision, Himmler’s attention was directed to the location (Wagner, 2000, p. 285). For Himmler the IG Farben decision was welcomed; it offered him the possibility to take part in armaments projects. The chemists responsible on site and at the IG Farben headquarters in Frankfurt soon determined that the totally exhausted and emaciated camp inmates were not very productive. They did not, however, draw the conclusion that they should insist on better working conditions for the Jewish prisoners. Rather they proposed to more quickly replace the ‘used up’ prisoners with new ones.

The “used up” prisoners were gassed in Birkenau. Individual punishments of prisoners were also requested by IG managers and promptly carried out by the SS. Thus, the librettist and author Fritz Lohner-Beda,

who had composed a sad “Buna song” in Auschwitz, was beaten to death after IG Farben managers complained about his, in their eyes, meager work performance (Schwarzberg, 2000, p. 158-171; Hilberg, 1978, p. 596). Out of a total of around 35,000 camp inmates who worked there, more than 25,000 died as a result of their work for the German Buna (Steinbacher, 2004, p. 42 & 47). The life expectancy of the camp inmates was on average three months, sometimes only a few weeks. The single steps of the synthetic route to Buna became to them stations in their way of suffering and death:

The Carbide Tower, which rises in the middle of Buna and whose top is rarely visible in the fog, was built by us. Its bricks were called “*Ziegel, briques, tegula, cegli, kamenny, mattoni, téglak*”, and they were cemented by hate; hate and discord, like the Tower of Babel, and it is this that we call it – *Babelturm, Bobelturm* – and in it we hate the insane dream of grandeur of our masters, their contempt for God and men, for us men. (Levi, 2006, p. 78-79).

The Buna-plant, which shaped the life of the chemist Primo Levi in a horrible way (Maier, 2015, p. 554-555), did not produce one single ton of synthetic rubber during WWII. However, it was put to work after WWII by Polish authorities in Upper Silesia and is working still.

After the end of WW II German synthetic rubber production was in good shape technically despite war damage and dismantling, thanks to years of sponsorship by the National Socialist state. However, it needed economic support for a transition period, because it could still not compete in price with natural rubber. In the young Federal Republic of Germany (BRD) a compensation fund was set up into which all caoutchuc-importing businesses would pay a specific sum per imported kilogram. Federal economics minister Erhard signed the decree PR Nr. 42/52 on 17 May 1952 (Kränzlein, 1980, p. 114-115) The fund existed until 1958. Eventually German synthetic rubber became competitive enough that it could exist in the market without political support. The German Democratic Republic (DDR), meanwhile, possessed a large production location at the plant in Schkopau. In the DDR’s planned economy synthetic rubber was surrounded by political objectives for much longer, namely until the fall of the Berlin Wall.

Some IG Farben chemists were convicted as war criminals in the Nürnberg trials for their participation in IG Auschwitz. They were soon set free and again continued their careers in the chemical industry. In addition, IG Farben, before it was disbanded, paid an indemnification to the survivors of the camp. From this point on the popular Buna stories separated into two lines. The one was spun in the DDR, the other in the BRD.

- “Elaste” in the DDR: *Return to Utopia*

The DDR was founded in 1949. Within its state territory it possessed the Schkopau Buna plant, a huge factory for synthetic rubber. With the reactivation of production the bloody history of this rubber had to be dealt with. This was reflected in the popular literature, which showed an interesting and surprising continuity with the story-lines of the Nazi-period.

The story of the fight against monopoly was easily accepted from the Nazi period and for the most part continued with a few changes. The National Socialist state had not overcome the red rubber, that was evident. But why? The answer was given in popular books: because it was directed by capital. The actual overcoming of red rubber was reserved for socialism. This line made possible an important continuity between the National Socialist-histories and the DDR stories: the chemists remained on the side of the good, they remained bringers of progress. They were not guilty. Only guilty were the capitalists, the IG Farben directors. Thus the lines between friend and foe were drawn anew.

When Johannes Kropf (1949) published a short story *From Red Rubber to Buna*, there were scarcely any changes with regard to the stories from the National Socialist period. Synthetic rubber would be the solution against exploitation and cruelty. Rubber chemists were thus turned into the heroes of the working class, instead of the German people. Then, however, the lines were adjusted to the new situation. Peter Klemm, whose book *Detroned Gods – Stories about Raw Materials* begins with the chapter “Red Rubber to Buna”, described the “lords of the IG Farben” not as apostles of clean and fair rubber, but rather the opposite: as warmongers, promoters of fascism who wanted nothing other than war. They ardently desired war, according to Lenin’s teachings about imperialism as the last stage of capitalism: “The German imperialists not only counted on it [the war], they strived for it, because, yes, they still wanted to reach the old goals that they had not reached in the first World War – the new partitioning of the world” (Klemm 1960, p. 47). While in non-fiction books of the National Socialist period the IG Farben managers, without exception, were positively portrayed as real chemists who at the same time were great businessmen, popular authors now drew a distinction between the capitalistic bosses and “their chemists”. It was the capitalists who had really caused the war, they and their “Nazi generals”. Chemists were now portrayed as victims, robbed of the fruits of their labor, indeed, tangled in a new war that

threatened to destroy their entire achievement.<sup>8</sup> As Manfred Künne argued in his novel *Buna*, the third part of his great caoutchouc trilogy which appeared in 1985, four years before the fall of the Berlin Wall: Only Socialism is summoned to break monopoly without setting up a new one.

Synthetic rubber and the stained white smocks of the chemists were washed clean in this literature. With that, the past, as far as the DDR was concerned, was “overcome”. With this synthetic rubber (‘Elaste’) could again become a utopian substance, a substance which would be part of a “better world”:

“Out of the stinking material, out of rubber, profit and blood, by us out came a material that one can take in the hand without dirtying it. It combined the knowledge of the learned, the bitter experience of the working class under capitalism and the élan of the youth. It is a product out of applied natural laws and the laws of societal development – out of chemistry and socialism.” (Klemm, 1960, p. 62)

Thus in the DDR literature the story from the National Socialist period was continued, if with important changes. In this literature synthetic rubber still serves to overcome colonialist oppression, has a global mission as a symbol of peace, freedom, and justice and is not just an instrument of national power strategies. Yet whoever would think that these Buna writings from the DDR are embarrassing efforts, that they drip with ideology and have hardly anything to do with reality has not read the writings published by the chemical industry in West Germany. Compared with the BRD-stories one can consider the DDR writings in all their eccentricities as pure enlightenment. Of course the hypothesis in all of the socialist synthetic rubber books that war guilt lay not with Hitler, but rather with the capitalists who had wanted the war (in view of IG Farben’s enormous export market) is not convincing. But at least in these DDR-writings the attempt is made to take issue with the German red rubber. To be sure, the result is distorted and one-sided, but at least the central facts are determined and the names of the chemists responsible for the IG Auschwitz are given. In contrast the specific texts meant for a broader public published in West Germany nearly completely ignore the topic.

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<sup>8</sup> “Now, the IG Farben in fact broke the monopoly, but the concern at the same time attempted to erect a new one. It wanted the synthetic rubber, the result of the scientific efforts of the chemists. But this monopoly was already broken, even before it could even be set up, even before there was Buna or wheels that rolled on Buna for war. The reason for this was the Soviet Union and socialism.” (Klemm 1960, p. 48).

- *The 'Jack-of-All-Trades': Buna in the BRD*

After the break-up of IG Farben by the allies the unteachable Karl Aloys Schenzinger (1953) wrote an IG Farben tragedy novel, in which he, in a strongly glossed over depiction, once more put its splendid work into the limelight and even praised, of all things, synthetic rubber (alongside synthetic fertilizer and synthetic gasoline). Nowhere in the book is there mention of IG Auschwitz and the heads and chemists of IG Farben are without exception depicted in a positive light. IG Farben's Buna plant in Auschwitz is spoken of nowhere. Nor is Auschwitz missing only in Schenzinger's book. Even the historian Wilhelm Treue does not mention it in his solid, Hitler-critical monograph on "Rubber in Germany", although with 600 million Reichsmarks it was one of the largest investment projects of WW II (Steinbacher, 2004, p. 37). These memory lapses were not only a literary phenomenon but also a social one. These lapses highlight that those in the German chemical industry who were responsible for IG Auschwitz, such as Otto Ambros, Walter Dürrfeld, Heinrich Bütefisch, and Fritz ter Meer were very soon again busy in the chemical industry after a short incarceration in the Landsberg war criminals prison (Maier, 2015, p. 256).

Historical research has thoroughly reappraised the interconnections of chemistry and politics in the synthetic rubber industry and especially IG Auschwitz (Lorentz & Erker, 2003). In popular books dedicated to synthetic rubber however, the topic is not addressed. If one looks through the synthetic rubber histories of Bayer AG, Hüls AG, or the newest, Lanxess AG – the word Auschwitz is not found therein. Directly after WWII, the town-name Auschwitz also became a taboo in German chemical journals (Maier, 2015, p. 555-556). Instead, the individuals responsible for IG Auschwitz, such as Otto Ambros, received an honorable remembrance.<sup>9</sup> But industrial publications were not only places where the interconnections of synthetic rubber and the Holocaust were inadequately explored. Also in the volume *Rubber – the Elastic Fascination*, in which the Berlin technology museum participated, out of 383 pages only a single one is dedicated to Auschwitz (Giersch & Kubisch, 1995, p. 155).

This must be even more remarkable as *The Periodic Table*, a book by the Buna survivor Primo Levi, is read by many chemists. However, his Auschwitz Buna book, *If This Is a Man*, seems to be less well-known among

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<sup>9</sup> See for example the picture insert in Kränzlein (1980). Kränzlein was involved in Buna production in WWII, but not in Auschwitz (Maier 2015, p. 93). On Ambros, who thought he was an innocent victim, see Westermann (2007, p. 87-96) and also, with new sources, Maier (2015, p. 113).

German chemists. In contrast to the DDR literature, in the later BRD histories caoutchuc is no longer represented as a political substance. There is a noticeable break in the rubber myths.

In the West Germany industry publication put out shortly before and after the fall of the Berlin Wall caoutchuc is instead portrayed as a faceless, apolitical, technically perfect substance. Synthetic rubber is politically relevant only insofar as its production contributes to economic growth. This sounds very modest and in a way also a bit boring. But this new, humble rubber myth integrated itself into a general trend in the Federal Republic, for which economic success stood at the center. Indeed, the historian Werner Abelshäuser (2004, p. 11) wrote that “German history since 1945 is above all economics history” and that the “West German Federal Republic [...] for a long time was like a successful economy in search of the purpose of its political existence”. Synthetic rubber’s depiction has shifted from Fischer’s (1938) “Triumph of Reason” to the “Triumph of Chemistry” presented in a Bayer commemorative publication from 1988 (Verg *et al.*, 1988, p. 248).

Similarly, Lanxess AG (2009a), at this time the world’s largest producer of synthetic rubber for tires and seals (and a spin-off of Bayer AG) put out a commemorative publication for synthetic rubber’s “100 Year Anniversary” which depicted it as a “tailored” material whose characteristics can be adjusted much more exactly to its technical functions than would be the case with natural rubber. Innovation was now the key term of the narrative for the 100th anniversary of the invention of synthetic rubber.<sup>10</sup>

In other words, without synthetic rubber, no “modern world”! Synthetic rubber is now an internationally-active “many-faceted problem-solver” (Lanxess 2009a, p. 15): a “Formula for Success” or simply a ‘jack-of-all-trades’. It is useful everywhere – a *materia universalissima*. This peaceful rubber fearfully avoids acting aggressively: rather it is a confirmed pacifist which is everywhere where people have fun. Possibly for this reason the company’s anniversary commemoration was moved to the year 2009, so that they could celebrate synthetic rubber as an innocent scientific discov-

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<sup>10</sup> “without modern rubber species out of the retort as for example Therban(R) (HNBR = hydrated acrylonitrile-butadiene rubber), Levapren(R), Levamelt(R), Baymod(R) L (EVA = ethylene-vinyl acetate rubber), Bapren(R) (CR = chloroprene rubber), Krynac(R), PerBunan(R), Baymond(R) N (NBR = acrylonitrile-butadiene rubber), Krylene(R) and Krynol(R) (styrene-butadiene rubber) as well as BUNA(R) EP (EPM/EPDM = ethylene-propylene rubber) would neither mobility nor machine construction, neither electricity transmission nor space travel, nor modern architecture or raw materials processing be possible in their present form” (Lanxess, 2009a, p. 13).

ery of the year 1909 while putting the connection to armaments in the background. The war year 1915 probably did not come under consideration for the company and certainly not the year 1936, although these dates could make a better claim as the real birth year of German synthetic rubber. On the web, history is centered on the year 1909, while the time between 1909 and 2009 remains diffuse:

“Hofmann and his successors encountered numerous setbacks in their quest for an economical and usable synthetic rubber, but by searching for new processes and building large-scale plant at great expense they finally succeeded.” (Lanxess, 2009b, slide 2).

That one of these “large-scale plants” built by Hofmann’s successors of Hofmann was part of Auschwitz, is not mentioned. Instead, the most important point seems to be the success of the substance.

Thus the new German rubber books are distinguished by the attempt to normalize the substance, to depict it as a useful, harmless invention and to blend out its terrible political past. Yet even this attempt to make the substance apolitical, is, however, a political positioning. It fit into a political atmosphere at the time in which the Federal Republic of Germany saw itself as a similarly pacifist, economically successful “problem-solver”.

## Conclusion

Popular books on science, as Ludwik Fleck pointed out, portray science for the general public. A certain simplification, as Fleck already noted, is indispensable for this. However, our short analysis of German popular books on synthetic rubber shows that there is not only simplification but also – so to say – complication. On the one hand, the complexity of the research and development process and of the historical context is significantly reduced. On the other hand, something is also added: a typical narration-scheme which presents the chemist as a heroic liberator. With his invention, he liberates first his country, and then in the long run all mankind. This narration scheme is especially abundant in popular books published in Nazi Germany, but it can be traced back to alchemical writings of the early modern period. Rubber chemists played different roles according to different political contexts in 20th century Germany: during the Nazi period, they saved their people; during the Soviet era in the DDR, they saved their social class; during the Liberal era in the BRD, they saved the economy. There, the narrative gradually depoliticized, as synthetic rubber

was presented as a peaceful substance serving any technical purpose and, thereby, economic growth.

Does the popular discourse on synthetic rubber have any relation to the “bench”, to the laboratory of the rubber-chemist? Fleck argued that popular books also influence the scientist himself. That is plausible, and the author knows rubber-chemists who state that they love Schenzinger’s books. Yet it seems hard to get more than such anecdotal evidence for the thesis. On the other hand, the books themselves are often influenced by the scientists, as they were sometimes involved in the process of creation of these books.

One function of popular books on synthetic rubber is the legitimization of rubber chemistry as a work of national importance and human significance. They intend to present rubber-chemistry as something great. In this respect, they have an important function in motivating young people to start a career in the chemical industry. All in all, their function seems to be less important on a theoretical level. But science is not only a theoretical and experimental and technical endeavor. It is also, and in a certain sense especially, a social endeavor which will cease if society does not accept and fund scientific research and development or if there are not enough young people who think it worthwhile to engage in it.

It should not be forgotten that alongside the positive discourse on chemistry, there also exists a critical public discourse on chemistry, a counter-discourse so to say. This discourse started with critical publications on the development of chemical warfare in WW I after 1918 (Woker, 1925) and was also later focused on dissipating substances, especially gases and aerosols. But that is a different story...

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## Point and Line to Plane: The Ontography of Carbon Nanomaterials

Sacha Loeve\*

### Abstract

*The carbons known today as fullerenes, nanotubes, and graphene were all observed or theorized well before becoming emblematic nanomaterials. However, by the 1990s, their mode of existence was shifted from bench or brand objects to technoscientific objects. After focusing on the separate life-stories of these carbons, this chapter recounts how, by eventually interweaving their trajectories and mutually referring to each other, these objects have reborn as a family of low-dimensional nanocarbons unfurling a space of indefinite technological possibilities saturated by promises of radical novelty: the “nanoworld”. The co-shaping of nanoworld and nanocarbons is reminiscent of that of the three basic figures composing the world of painting according to Kandinsky: point, line, and plane.*

*Keywords: carbon, nanotubes, materials chemistry, fullerene, graphene, modes of existence, nanomaterials, nanotechnology, objects, technoscience.*

### Résumé

*Les carbones aujourd'hui connus sous le nom de fullerènes, de nanotubes et de graphène furent tous observés et théorisés bien avant de devenir des nanomatériaux emblématiques, mais au cours des années 1990 ils changent de mode d'existence et passent du statut d'objets scientifiques ou de produits commerciaux à celui d'objets technoscientifiques. En partant des récits de genèse de chacun de ces carbones, ce chapitre raconte comment ces objets, en finissant par entremêler leurs trajectoires et à s'impliquer mutuellement, ont contribué à déployer un espace de possibilités technologiques indéfinies saturé de promesses de nouveauté radicale, le « nanomonde ». La co-constitution du nanomonde et des nanocarbons n'est pas sans évoquer celle des trois figures de base composant le monde de la peinture selon Kandinsky : le point, la ligne et le plan.*

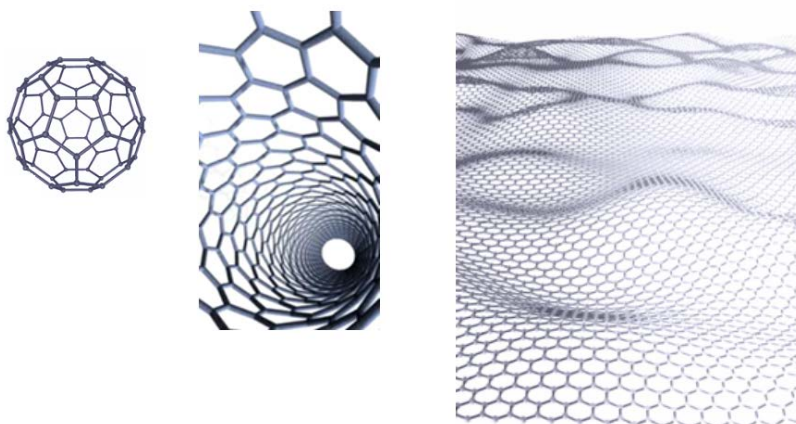
*Mots-clés : carbone, nanotubes de carbone, chimie des matériaux, fullerène, graphène, modes d'existence, nanomatériaux, nanotechnologies, objets, technoscience.*

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THE NANOSCALE science and technology funding initiatives launched in the 2000s were supported by grand narratives and slogans such as “there’s plenty of room at the bottom” or “shaping the world atom by atom” (IWGN, 1999). After the conquest of space, here is the conquest of the “nanoworld”. Populated with objects the size of few billionth of a meter in principle inaccessible to our senses, a distant world becomes a land of promises.

In this dizzying world, carbon is king. Fullerenes, nanotubes and graphene (figure 1) are the star materials of nanotechnologies, the stuff their dreams are made of. These all-carbon molecules with iconic shapes have the power to attract thousands of researchers and millions of dollars and to dangle miraculous solutions to all kinds of engineering problems from health to environment and from electronics to mechanics (Pugno, 2006).



*Figure 1 - Point, line and plan of nanocarbons  
(Source: picture processed by the author)*

But where do these nanocarbons come from? One often reads that carbon nanotubes popped up in 1991 under the electron microscope of Sumio Iijima, physicist of NEC corporation labs at Tsukuba, Japan. His short paper, “Helical microtubules of graphitic carbon” (Iijima, 1991), cited more than 40.000 times, is considered the dawn of a new era, a decisive

first step into the nanoworld.<sup>1</sup> However, nanotubes have been repeatedly characterized (and forgotten) since the 19th century under the name of carbon filaments. The isolation of graphene tells a similar story. While it is generally attributed to Andre Geim and Konstantin Novoselov in 2004, graphene was known as an “academic material” since the mid 20th century. As to fullerene, Harold Kroto, Richard Smalley, and their colleagues synthesized it incidentally in 1985 without knowing that spherical carbon was an object of speculation and chemical reverie for a long time. Moreover, generations of scientists and engineers have inventoried and exploited the characteristics, properties, powers, and behaviors of carbon in all its various forms for centuries (Walker, 1962; Bensaude Vincent & Loeve, forthcoming). Have these marvelous materials simply been revealed by new powerful scientific instruments like scanning probe or electron microscopes or forged by skilled molecular architects? In short, are they discoveries or inventions?

This chapter argues that carbon nanomaterials are the product of a shift in the pattern of existential relations or “modes of existence” that defines them as objects that matter. This notion of “modes of existence” belongs traditionally to the philosophical field of ontology. This field aims at explaining change by what persists through time so that we can hold true beliefs about the world. It usually considers that there is *first*, being (substance, reality, nature, or whatever) and, *only then*, modalization of being, i.e. different ways of saying something about the same existing thing. For instance, for Aristotle I can speak of the same substance according to the category of quality, quantity, locus, relation, etc. I can even vary the degree of existence of that thing from the potential to the actual, but without ever going so far as to change the *being* of this thing, which is assigned to a well-defined identity. So in classical ontology the notion of “modes of existence” is usually taken in a weak sense. It refers to modifications of dictum, not of being.

Here the notion of “modes of existence” is taken in a strong sense along the lines of French philosopher of art Etienne Souriau (1943), who advocated a “muti-realism”, which has recently been taken over by Bruno Latour (2010). Under this usage the modality attributes another way of being to that which it modifies. Carbon can be apprehended both as a chemical element, as a range of materials, of fossil fuels, as a tool for measuring

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<sup>1</sup> It is in the top ten percent of most-cited science papers (Ho, 2013) and the first most-cited paper for all of materials science. Ironically, those who contest this attribution are quoting the paper and thus contribute to enhance its citation score even more.

our ecological impact (“carbon footprint”), etc. Its multiplicity of modes is nicely suggested by the chemical concept of “allotropy” (“different ways”), but it goes well beyond that of different chemical objects. Each mode typifies a different ontology for carbon, a different way of being in connection with writing (*graphein* in Greek, from which the word “graphite” is formed), a different way for carbon to write itself (through us) (Loeve & Bensaude Vincent, 2017). So instead of ontology this perspective lends itself better to what can be called “ontography” (Lynch, 2013).

Between biography and ontology, ontography sets into narratives the modes of existence of singular things by focusing on the various inscriptions they afford to our material and symbolic practices. So for nanocarbons: they afforded a language of graphemes, surfaces of inscription, geometric structures, operations, and schemes that enabled multiple deterritorializations between bench and brand.

The first three parts retrace the separate life-stories of the turbulent carbon filaments, the academic graphene, and the speculative carbon balloon. The final three parts recount their nano-renaissance in a world of low dimensions: 0D fullerene, 1D nanotubes, and 2D graphene. The conclusion draws an analogy between them and the three basic figures composing the world of painting according to Kandinsky (1929): point, line, and plane.

### **Doomed to Oblivion: Carbon Filaments**

In 1826, Dr. Hugh Colquhoun (1826, p. 2) writes an enthusiastic notice introducing the discovery of “several highly interesting states of aggregation of carbon, one of which is not only of a very singular structure, but also an entirely new form”.

The action takes place in the castle of Crossbasket, Blantyre, near Glasgow. In the temporary absence of its landlord, the chemist Charles Macintosh – already famous at that time for his invention of the waterproof fabric – Colquhoun was in charge of superintending the implementation of a new “Macintosh process” for steel-making in a pilot plant apparatus.<sup>2</sup> The process consisted in bringing molten iron to react at high-temperature with a hydrocarbon gas in an airtight pressurized earthen vessel. When the gas is in excess a carbonaceous deposit forms. In this deposit Colquhoun found “capillary threads of carbon”, “a mineral hair”, whose “single lock seemed to contain thousands of thin filaments” that, “in thickness [...] are as delicate as the filaments of the lightest spider-web” (p. 3). After a series of tests, Colquhoun concluded that he was therefore facing a stable and entire-

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<sup>2</sup> English Patent n°5173 (1825).



ly new form of “pure metallic carbon” apart from graphite and diamond. But the story felt short because Macintosh was less happy with steel-making than with its famous raincoat. As the process involved too high temperatures for the state-of-art brickworks it could not be scaled-up. It was quickly abandoned, and the carbonaceous filaments forgotten.

However, carbon filaments resurfaced in two different and independent contexts. First, in the course of bench experiments on “*azotocarbores*” (nitrocarbons), Alsatian chemist Paul Schützenberger, director of the École municipale de physique et de chimie de la ville de Paris and his son Léon, a chemical engineer graduated from the same school, observed “certain facts worthy of interest that pertain to the chemical history of carbon” (Schützenberger & Schützenberger, 1890, p. 774). To achieve full decomposition of cyanogene ( $\text{N}\equiv\text{C}-\text{C}\equiv\text{N}$ ), they used a refractory vessel made of almost pure carbon (*charbon de cornue*) powdered with cryolite, a powerful dissolvent. The resulting carbon clogged the tube in a blackish felting presenting long thin filaments. Rubbed onto a sheet of paper, the substance leaves a dark trace reminiscent of “plumbago” (i.e. pencil lead: graphite). But which form of graphite was this “cyanogene coal”? Its oxidation products did not match with any graphite forms identified in the literature. So they cautiously concluded: “the filamentous carbon formed by the pyrolytic decomposition of cyanogene in the presence of cryolitic vapours constitutes a particular variety of carbon, neighbouring but not identical to electric graphite” (p. 777). But if the bench experiments on nitrocarbons were connected to the Schützenbergers’ views on organic pigments and artificial cellulose, filamentous carbon was not. Presenting no commercial value in a school and at a time when applications prevailed, the matter ends there.

Carbon filaments reappeared later on in coke ovens. A communication by two industrialists, Constant and Henri Pélabon (1903, p. 706-709), read at the Académie des sciences de Paris by Henri Moissan reports the meticulous observation of “certain deposits with a threadlike appearance and constituting, through the entanglement of their wires, a genuine wool of carbon”. This “filamentary carbon” (*carbone filiforme*) forms in the part of the oven exposed most directly to the highest temperatures. It is “generally cylindrical”, sometimes with “very thin and tight packages of wires that seem to have arisen in some points of other wires of larger diameter” and “some wires that seem formed of a succession of rings”. Their length varies from 5 to 8 centimeters and their thickness between 1 and 15 microns. For the two industrialists the formation of such filiform carbon is undesirable because it is a sign that “the pace of the oven is pushed too far”.

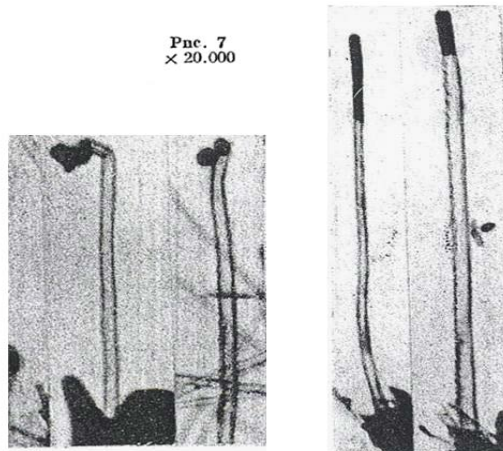
Although the Pelabon’s report quotes the Schützenbergers’ report, neither of them refer to the carbon filaments that Thomas A. Edison was

exploiting with relative success in his incandescent light bulbs. A surprising silence since Edison's invention had famously lighted the Paris World's Fair in 1889. Carbon filaments were selected by Edison at the end of an extensive research program where he spent \$40,000 testing 6,000 natural substances over the world before choosing a naturally fibrous organic material containing a large amount of cellulose, bamboo. In 1879, Edison filed a patent describing the processes for obtaining "metallized" carbon filaments by "flash carbonization" of bamboo, i.e., by means of the electric arc previously used for illumination in the first street lamps. "Metallization" also involved repeated electric arc treatment to get very pure ("metallic") carbon. Consisting of twists of several wires measuring about ten microns in diameter, these carbon filaments were featured in the first brand of "Ediswan" light bulbs commercialized in the 1880s and were also used in street lamps.

Thus, at the turn of the 20th century carbon filaments lived two parallel lives in two different technical systems. In coal and steel-making, where they could occasionally be transferred to the chemist's bench, their formation was undesirable, a sign that something went wrong. In early lightbulbs they had a luminous and widely public "brand" existence (Ediswan, General Electric, Shelby...). Electric light was the star of the celebrations of technical progress. But their success was ephemeral. During the interwar period they were supplanted by tungsten on the ground that overheated carbon is sublimed and blackens the bulb after thirty hours of use.

Carbon filaments fell into oblivion for a few decades before being recalled on stage thanks to Transmission Electron Microscope. TEM brought key information about their genesis not only because it is a powerful instrument (TEM provides rather static information) but because carbons bear the traces of their forming events (Rouzaud *et al.*, 2015). However, using TEM did not change the mode of existence of the filaments: it provided knowledge about their conditions of formation so as to *avoid* it. In the 1950s British ceramic chemists studying the wear of blast-furnace brickworks reported TEM observations of helical carbon "vermicules" and pointed out the catalytic origin of this "unusual form of carbon" (Davis *et al.*, 1953). The vermicules' growth was catalyzed by iron particles and a clear relationship was established between the initial particles' distribution and the growth and form of the filaments. A single metallic "speck" gives rise to a single carbon thread measuring as little as 10 nanometers, while a collection of "points" gives rise to many twisted threads forming a bigger "rope" of several microns so tough that it can penetrate deeply in the brick and provoke its rupture. The metal particles often stay attached to the filaments as a mark of their catalytic origin, as visible as black points in another

TEM study (figure 2) published in Russian at about the same time (Radushkevitch & Lukyanovich, 1952). The paper went unnoticed by Western scientists. In retrospect, many have claimed that the micrograph, which shows a 50-nm wide inner cavity, is the first image of multi-wall nanotubes ever seen (Monthieux & Kuznetsov, 2006).



*Figure 2 - Micrographs of iron-catalysed carbon filaments (Radushkevitch & Lukyanovich, 1952). (Original Russian Edition Copyright © by Nauka Publishers of the Russian Academy of Sciences. Copyright © 2010 by Pleiades Publishing, Ltd.)*

“Filamentary growth of graphite has recently been rediscovered” reads a 1958 crystallography paper (Hillert & Lange, 1958). Focusing an entire instrumental arsenal on the filaments (TEM, polarized light microscopy, X-ray and electron diffraction), the crystallographers make clear that the filaments are hollow, built up of lamellar units bent into cylinders, single or multi-wall, with thicker threads formed by radial growth. They display a variety of shapes while having all the same crystalline structure, graphite. The thicker threads are produced by a two-step mechanism: first, catalyzed growth of an individual filament; second, catalyst-free pyrolytic carbon deposition thickening the fiber.

In retrospect this 1958 paper reads like an anticipated description of carbon nanotubes. Some even claim that carbon nanoscience is only a re-discovery of phenomena that have already been observed but fallen into oblivion (Boehm, 1997; Monthieux & Kuznetsov, 2006; Monić, 2011;

Hoffmann *et al.*, 2016)<sup>3</sup>. By the 1950s, it had been established that a hollow carbon tube could be produced by catalytic growth. But did it constitute a matter of interest?

Actually, these retrospective readings highlight less some obscure forgotten precursors prone to reduce the revolutionary claims of nanotechnologists than the obscuring effects of research programs. Indeed, in the 1950s catalytically-grown carbon filaments could have become a research field in its own right if the thin filaments had not been eclipsed by the bigger carbon fibers.

The “graphite whiskers” obtained by physicist Roger Bacon by vaporizing hydrocarbons in electric arc discharge at high pressure and temperature were roughly the same objects (Bacon and Bowman, 1957). To Bacon, they were “scrolls”: concentric tubes made of a rolled-up graphite (figure 3). It has been suggested decades later that they were been multi-wall nanotubes, not “scrolls”. Bacon lucidly recognized “I may have made nanotubes, but I didn’t discover them”<sup>4</sup>.

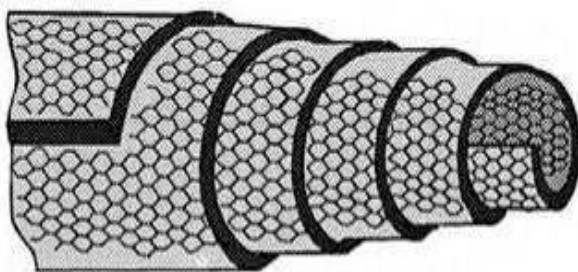


Figure 3 - Bacon's scroll model of graphite whiskers. (Source: <http://what-when-how.com/nanoscience-and-nanotechnology/carbon-nanotubes-and-other-carbonmaterials-part-1-nanotechnology/>)

One can observe without discovering. The famous Archimedean *eureka* does not proceed from mere visual evidence obtained through powerful instruments but from a context-dependent disposition of mind. Unlike chemists who examined the formation of the turbulent filaments in blast furnaces in order to get rid of them, Bacon was working for a chemical

<sup>3</sup> To remedy this situation a database of carbon allotropes with original bibliographic sources is now being established: <http://sacada.sctms.ru/>

<sup>4</sup> <https://www.eurekalert.org/staticrel.php?view=acslandmark090803>

company producing high-volume polymer commodities, Union Carbide. These filaments, which could bend and kink while remaining unbroken, could serve to make bigger and even more robust materials.

Yet reinforcing fibers became strategic during the Cold War period when major space and military programs required the manufacture of materials combining the lightness of plastics with the hardness of steel and the strength of ceramics. Bacon's whiskers served right away as precursors of the "carbon fibers" industrially mass-developed since 1963 as a reinforcing structure for composite materials. Dubbed "the new steel", carbon fibers won the competition among materials hands-down: with their high elastic modulus and low density and rigidity, when inserted in epoxy or polyamide resins they offered performance five or six times higher than aluminum or titanium alloys. Supersonic planes, helicopter tails and blades, rockets, Formula One cars, sports equipment... carbon fibers were strategic, high-performance and capitalistic – relatively expensive – materials for a proud industry employing armadas of materials scientists and engineers (Bensaude Vincent, 1998, p. 178-180).

The success of carbon fibers increased the clandestine status of their precursors, the carbon filaments. Hitherto encountered as anomalous by-products of steel production, they were now eclipsed by "the new steel". When found once again on carbon arc anodes (Wiles & Abrahamson, 1978), they were identified as "carbon fibers from about 4 nm to about 100 nm". They were not different objects, but smaller objects of the same kind. Of course, they kept on popping up, but in rather old-fashioned fields such as Soviet "metallic science" (Nesterenko *et al.*, 1982) or marginal research such as heterogeneous atmospheric chemistry (Buseck & Bradley, 1982). Again, they were quickly forgotten.

### **An Academic Material: Graphene**

By the mid-20th century graphite was a highly strategic material for the nuclear industry, with no less than 11 uranium-graphite-gas reactors built in France in the 1950-60s. While nuclear graphite occupied the forefront of the technomilitary scene (Walker, 1962), a more discrete but no less strategic existence opened up to graphite in the fabrication of intercalation compounds (Teissier, 2014, p. 253-254).

Graphite is made of stacked layers held together by weak van der Waals forces, each layer constituted of hexagonally-arranged carbon atoms linked by strong covalent bonds. Because of its lamellar structure, graphite is able to take up atoms, ions, molecules, or even metallic alloys by expanding the space between the planes while maintaining its structure unchanged.

Due to its chemical properties (both oxidizing and reducing) graphite exchanges electrons with its intercalated guest. These intercalation compounds are used in the manufacture of electrodes and batteries, such as the lithium-graphite compounds forming the negative electrode of lithium-ion batteries in our mobile phones and laptops.

The individual layer of graphite was isolated in the early days of intercalation compound research by German chemists Hans-Peter Boehm and his colleagues (Boehm *et al.*, 1962). While the preparation of these “thinnest carbon films” required astute chemistry, the resulting “lamellar carbon” was not a matter of interest for itself but rather a “test object” (Mody & Lynch, 2010) used for calibrating TEM lattice imaging<sup>5</sup> or possibly an interesting catalyst (Boehm *et al.*, 1963).

Boehm forged the term “graphene” (from *graphite* + *benzene*) in the 1980s for denoting a single layer of hexagonal carbon (Boehm *et al.*, 1986). His definition was formally adopted in a 1994 IUPAC nomenclature for graphite intercalated compounds and officially endorsed in the 1997 *Compendium of Chemical Terminology* (McNaught & Wilkinson, 1997).

Carbon filaments had no proper chemical name but were designated by a compound term (“graphite filaments”, “fibrils”, “vermicules”, or “whiskers”). On the contrary the IUPAC *Compendium* stressed that “it is not correct to use for a single layer a term which includes the term graphite, which would imply a three-dimensional structure”, and consequently that “the term graphene should be used only when the reactions, structural relations or other properties of *individual layers* are discussed”. Thanks to Boehm, the *two-dimensional plane* composing three-dimensional graphite had a name and a material identity of its own. “Graphene” became a common term to refer to the single sheet of graphite in the well-established and industrially-relevant field of carbon intercalation. Naming matters.

Moreover, the name denoted something that many solid-state chemists and physicists were familiar with: the perfect chicken-wire monolayer structure they were trained to *draw* for decades. Thus, before earning a name of its own graphene pre-existed as a theoretical paper model for understanding the chemistry and physics of graphite. It was used to calculate the band structure of graphite from the 1940s onwards and provided a paradigmatic case study to teach band theory (Pisanty, 1991). It was a “paper material”, a structural component virtually involved in higher-dimensional graphite and an abstract model used for many scientific purposes, but materialized only in paper drawings.

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<sup>5</sup> See [http://zfn.mpdl.mpg.de/data/Reihe\\_B/17/ZNB-1962-17b-0150.pdf](http://zfn.mpdl.mpg.de/data/Reihe_B/17/ZNB-1962-17b-0150.pdf) (p. 2).

This did not prevent researchers from getting graphene crystals at the bench, either as “overlayers” grown by epitaxy on a metallic substrate, “pancakes on a plate” etched or sliced into individual layers, or in the form of rolls, cones, or folded like an origami, which minimizes their surface energy – each time in a particular disposition or supported by a surface, never in its free state. Thus academic graphene lived a kind of dual or Platonic existence as an intelligible form and an imperfect copy.

### A Speculative Molecule: The Carbon Balloon

If graphene was an academic material, the nanomaterial known today as “fullerene” ( $C_{60}$ ) was first a speculative molecule. Structurally,  $C_{60}$  is a truncated icosahedron, a semi-regular polyhedron belonging to the class of the Archimedean solids, which grants it interesting mathematical properties and makes it resonate with an entire esoteric tradition devoted to mathematical cosmography. The figure appears in particular in the illustrations realized by Leonardo de Vinci in 1509 for the *De Divina Proportione* of Luca Pacioli and in Kepler’s treatise *Harmonices Mundi*.

The possibility of creating a giant hollow spherical carbon molecule was suggested in 1966 by a fictional inventor known as Daedalus (David Jones). Looking for a solution to bridge the gap between the density of solids and that of gases, he conceived of a hollow molecule that “would be a spherical shell of a sheet-polymer like graphite, whose basic molecule is a flat sheet of carbon atoms bonded hexagonally rather like chicken-wire” (Jones, 1966, p. 245). To do so he proposed to modify the high-temperature synthesis of graphite by doping carbon with defects which would introduce a curvature of the hexagonal plane and close the net into a spherical shell. But Daedalus did not tell exactly which defects would actually do the job. Coming back later to the spherical-molecule problem, he drew on a theorem known as the Euler-Poincaré characteristic<sup>6</sup> and on D’Arcy Thompson (1917), who applied the former to the structure of a microscopic sea creature depicted by Ernst Haeckel, *Aulonia Hexagona* (Jones, 1982). All this erudition served Daedalus to make a single statement: A *pentagon* would serve nicely as the required defect to transform a flat layer

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<sup>6</sup> Euler-Poincaré characteristic is an invariant describing the structure of a topological space regardless of the way it is bent. Denoted  $X$ , it is defined as the number of vertices ( $V$ ) less the number of edges ( $E$ ) plus the number of faces ( $F$ ). For spherical polyhedra  $X$  is always equal to 2 ( $X = V - E + F = 2$ ). It thus provides a simple rule to transform a sphere into a polyhedron of however many faces one wishes. The truncated icosahedron verifies such a characteristic:  $60 - 90 + 32 = 2$ .

of otherwise hexagonal lattice into a graphite balloon (figure 4), just like a soccer ball is made of 20 hexagons and 12 pentagons.

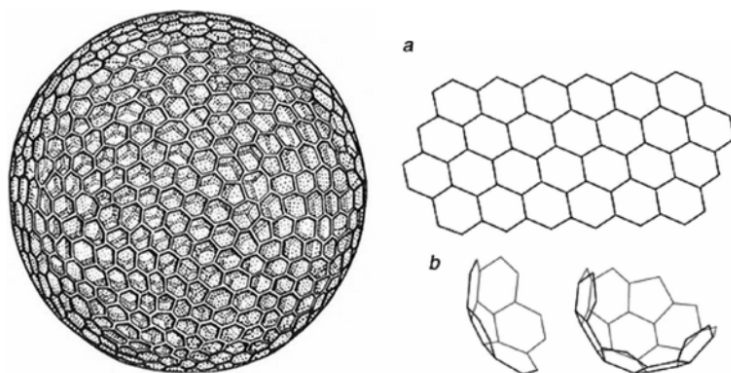


Figure 4 - Haeckel's creature and Daedalus' prediction (Haeckel, 1887, plate 111, fig. 1). (Source: Wikimedia Commons)

In addition to Daedalus' speculations,  $C_{60}$  and bigger polyhedral carbon clusters were repeatedly postulated and subjected to theoretical calculations. Japanese computational chemist Eiji Osawa predicted that carbon's structure would then be "superaromatic" (i.e., a conjugated aromatic structure that goes on and on and wraps back to itself) and thus probably stable (Osawa, 1970). He displayed a football image in the publication's front page. Soviet researchers D. A. Bochvar and E. G. Gal'pern (1972) presented the first Hückel calculation on  $C_{60}$  and came independently to the same conclusions as Osawa. Several other calculations followed (Davidson, 1981), including a paper entitled "Footballene: a theoretical prediction for the stable, truncated icosahedral molecule  $C_{60}$ " (Haymet, 1985). None of these theoreticians cited one another.

Thus, kicked off by the erudite speculations of a fictitious inventor, there was a strange football world cup involving mutually ignorant players. But the carbon balloon, theoretically possible and stable, also represented a synthetic challenge that many chemists believed would soon be at hand. As Cyrus Mody (2008, p. 166) reports, Orville Chapman set several generations of UCLA graduate students to "futile attempts to synthesize 'socchorene' [*sic*] ( $I_h-C_{60}$ )".

Footballene, soccerbalene, carbon balloon, soccerane, socchorene... Could we not find a more appropriate name for a molecule on the thre-



shold of existence? In a brainstorming exercise in nomenclature, chemists Josep Castells and Felix Serratosa (1983) attempted to forge a tentative IUPAC name for the hydrogenated species  $C_{60}H_{60}$ : Hentriacontacyclo[29. 29. 0. 0<sup>2,14</sup>. 0<sup>3,12</sup>. 0<sup>4,59</sup>. 0<sup>5,10</sup>. 0<sup>6,58</sup>. 0<sup>7,55</sup>. 0<sup>8,53</sup>. 0<sup>9,21</sup>. 0<sup>11,20</sup>. 0<sup>13,18</sup>. 0<sup>15,30</sup>. 0<sup>16,28</sup>. 0<sup>17,25</sup>. 0<sup>19,24</sup>. 0<sup>22,52</sup>. 0<sup>23,50</sup>. 0<sup>26,49</sup>. 0<sup>27,47</sup>. 0<sup>29,45</sup>. 0<sup>32,44</sup>. 0<sup>33,60</sup>. 0<sup>34,57</sup>. 0<sup>35,43</sup>. 0<sup>36,56</sup>. 0<sup>37,41</sup>. 0<sup>38,54</sup>. 0<sup>39,51</sup>. 0<sup>40,48</sup>. 0<sup>42,46</sup>]hexacontane!

But the quest for the synthesis of the carbon balloon never gave any result, until others simply fell upon it.

### Fullerene: The Third Allotrope Fallen from the Sky

As an astrochemist, Kroto got interested in the mysterious new diffuse interstellar bands (“the DIBs”) detected in the 1970s by radiospectroscopy in the dark clouds of interstellar space. The molecular carrier of the DIBs was – and is still – an astrochemical enigma. From the DIB’s spectra it was hypothesized that they might be long acetylic carbon-chained molecules ( $[C\equiv C]_n$ ) known for their controversial ability to form “carbine”, a hypothetical linear “third carbon allotrope” also known as chaoite or “white carbon” (Kasatochkin, 1967). The existence of carbyne as a solid was dubious because the longer these molecules are, the more unstable and even explosive they become. Kroto thought it was pure “carbon’s myth” (Kroto, 2010).

However, in a 1977 study using the Algonquin (Ontario) radio telescope in which he participated, the team managed to successfully detect the vibration frequency of polyynylcyanides  $HC_5N$  ( $H-C\equiv C-C\equiv C-C\equiv N$ ),  $HC_7N$  ( $H-C\equiv C-C\equiv C-C\equiv C-C\equiv N$ ), and  $HC_9N$ , “the largest molecule yet detected in interstellar space” (Kroto *et al.*, 1978). Kroto conjectured that their synthesis might originate from red giant carbon stars. To test this hypothesis he travelled in 1985 to Texas to work with Richard Smalley at Rice University. Smalley and his team had just built an apparatus that, Kroto thought, could nicely simulate the extreme plasma chemistry of carbon stars.

The machine, dedicated to the production and study of metallic and semiconductor clusters (crystals of the size of a few atoms), had no proper name. It was referred to as the AP2, “the second-generation apparatus, a supersonic nozzle in which a high-energy laser strikes a rotating disc of graphite. A chaotic plasma forms at the graphite surface, provoking the vaporization of carbon atoms into a dense and high-speed helium flow. The carbon time-of-flight mass spectroscopy detection of the helium gas emitted by the machine showed very neat spikes indicating the presence of “something” very stable made of 60 carbon atoms and other residual car-

bon clusters. In 1984, another group at Exxon got the same spikes in a larger spectrum with a similar apparatus, built at Rice (Rohlfing *et al.*, 1984). They were trying to understand the undesirable accumulation of carbon on catalysts. But they did not focus on these specific spikes.

Kroto *et al.* (1985) disclosed the newcomer in *Nature*. Instead of proposing a structural formula or a molecular model like any chemistry paper, they were content with a photograph of a soccer ball on Texas grass and named the molecule “buckminsterfullerene” by analogy with the geodesic domes designed by the architect Buckminster Fuller. How could a metaphor ventured on the basis of a unique experiment convince *Nature’s* editors and demanding referees? “It was such a beautiful and perfect structure, how could it have been wrong?” Kroto said later (Seeman & Cantrill, 2016).

Above all, the molecule imposed itself by its beauty. The scientific question – whether fullerene’s characteristics match the DIBs spectra – faded to the background. Still unsolved today, most fullerene researchers do not care about it – it just gives a nice aura of mystery to stage the molecules floating like celestial spheres in far out space. Although Kroto (1992) kept working on the problem, the mundane object itself – fullerene – has far overcome the scientific question that initially prompted its discovery<sup>7</sup>. Its popular name, associated with the notoriety of Fuller as a visionary architect, has also done much for it (Applewhite, 1995). What if the molecule had borne the impossibly awkward IUPAC name?

Right after the release of their *Nature* report, the group was told that the structure had already been postulated and calculated several times, although they were the first to have *claimed* (since there was no experimental confirmation of the structure) its synthesis. The discovery of Daedalus’ speculations attracted Kroto’s attention to the consequences of the Euler-Poincaré characteristic for fullerenes, namely that all fullerenes of any size have 12 pentagons – a magic number for all the family of fullerenes!

Fullerenes, however, were not so much hyped at the moment. They remained “a puff in a helium wind” (Harris, 1999), not a material but a trace detected in a gas obtained in a unique home-made instrument whose result could barely be replicated. Fullerenes could well have experienced a fate similar to the series of other putative “third carbon forms” like carbyne or hexagonal diamond (Bundy & Kaspers, 1967; Hoffmann *et al.*, 2016) – namely, oblivion. But they really took shape and became a hot topic five years later when Wolfgang Krätschmer managed to produce *solid* C<sub>60</sub>

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<sup>7</sup> At least for Kroto, since Smalley (1997) had other motivations and tells another story, related to semiconductor and metallic clusters for microelectronics (see Cyrus Mody’s chapter in this volume).

(Krätschmer *et al.*, 1990). Now the new form of carbon was materialized as a very pure (90%  $C_{60}$  and 10%  $C_{70}$ ) and beautiful plate-like crystal called “fullerite”. Since it was able to form a solid, a new carbon allotrope was added to graphite and diamond: fullerene, “the third man” (Kroto, 1993). Of course this “new” carbon was probably here long ago, widely distributed in the universe from stars to soot. But it did not exist as a technoscientific object until a method was established for producing macroscopic amounts of it with a simple technique accessible to any laboratory: a graphite electric arc.

From then on, it multiplied and contributed to engendering a new world of low dimensions: the world of nanocarbons. Fullerene, being zero-dimensional, can be considered its starting point. The genesis of nanotubes – 1D – will now be examined, before turning to graphene – 2D. All these materials existed before: the turbulent carbon filaments, the academic graphene, the speculative carbon balloon. But by the 1990s their mode of existence shifted to technoscientific objects. As they came to interweave their life stories and mutually refer to each other, these objects have helped to unfurl a space of indefinite technological possibilities saturated by promises of radical novelty also known as the “nanoworld”.

### Nanotubes: From Brand to Bench

Hidden in the bulk of the carbon fibers, the filaments came out of them transformed. But only with the help of fullerenes could they stabilize their new mode of existence.

First manufactured from coal or petroleum pitches, high-quality fibers were then industrially produced mainly in Japan from PAN (polyacrylonitrile  $[C_3H_3N]_n$ ), known as “acrylic” when used in synthetic clothes. The manufacturing process, which requires thermosetting, carbonization, and then graphitization, is quite complex and expensive. In the 1970s, Morinobu Endo was trying to find a cheaper alternative to the PAN process by starting from raw materials (benzene). In order to test his new process of “vapor-grown carbon fibers by catalytic decomposition of benzene”, he traveled to Orléans in 1974 to work with the French carbon materials scientist Agnès Oberlin and her TEM. The instrument required using very thin fibers by stopping their growth process at an early stage. Endo and Oberlin did so, and of course they rediscovered the filaments with their small opaque catalytic iron particles at the end of their tips.<sup>8</sup> They named this struc-

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<sup>8</sup> Additional testimonies and archive materials can be found in the website “Sciences : Histoire orale”: <https://www.sho.espci.fr/>

ture “hollow tube” or “hollow core” (Endo *et al.*, 1976). For Endo the hollow core was not an anomalous derivative of “real” carbon fibers but the very initial step of their formation process, the “central tube” before the thickening of the fiber. Instead of treating the hollow core as a defect, he viewed it as a crucial structural feature for the mechanical properties of the fiber, its strongest part which “never breaks when the fiber breaks” (Endo, 2002). Hollowness matters.

Simultaneously, the attention of researchers was shifted from the set of *causes* leading to the formation of the filament (the catalytic decomposition of hydrocarbon) to *the effect itself*. For instance, Baker *et al.* (1975) identified “a new mode of filament growth (...) in which the complete detachment of a catalyst particle from the surface of the metal was not a necessary prerequisite”. They established two modes of filament growth: “tip growth” (known since the 1950s) and “root growth” (newly characterized). The catalyst activated the filament’s growth but in the case of “root growth” it possessed its own dynamics. In other words, the catalyst acted as an *occasional cause* of the filament’s growth, a trigger. Then the growing filament starts a life of its own with different possible arrangements occurring during the rolling of graphene: circular, spiral, or helical arrangements controlled by chirality (Nesterenko *et al.* 1982). As a result, the tubular carbon structures came to be considered for themselves, partly independently from their generating causes. Effects overtake defects. Effects matter.

If catalytic triggering and thermal growth are two different processes, then it would also become possible to *decouple* them in order to better control the filament’s growth and produce well separated aligned tubules. This feat is claimed in the widely cited US patent claiming “cylindrical discrete carbon fibrils”, filed in 1984 and issued in 1987 to Howard Tennent of Hyperion Catalysis (Tennent, 1984). The patent covers a very large area, including multiple kinds of fibrils, compounds, and processes. In addition to applications in reinforcement of composites by embedding of the fibrils in a polymer matrix (a common, mainstream application of carbon fibers), other embodiments were claimed in which the fibrils could enhance the electrical or thermal conductivity of a material, increase the surface area of an electrode or a capacitor, provide a support for a catalyst, or shield an object from electromagnetic radiation. With such a large spectrum of applications (more or less reminiscent of the uses of nanotubes today), it is a root patent, referenced by more than 300 subsequent patents up to today – including by one of the many patents filed by Endo (2002) since his seminal 1986 vapor-grown carbon fiber patent (Endo, 1986). Since the mid 1990s, it has been referenced by many nano-related patents: “nanowhiskers”, “nanofiber”, “nanofibrils”, “nano-composites”, and then, by the turn of 2000,

mostly “nanotubes”. In contrast with carbon fibers, the main industrial target is no longer structural applications but electrical ones: conductive materials for microelectrodes, batteries, coatings and inks, and electrochemical cells.

When Sumio Iijima (1991) published the observations that led him to be credited for the “discovery” of nanotubes, their industrial uses were already widespread.<sup>9</sup> Nanotubes thus already had a rich “brand existence” at the factory, developed well before and rather independently of the emergence of an academic community gathered around them. So Iijima definitively did *not* discover carbon nanotubes in 1991, but he brought them *back* to the attention of a wider audience on the academic scene.

Fullerenes played a crucial role in the academic rebirth of nanotubes. Iijima had closely followed the irruption of the “third man”, Mr Buckminsterfullerene (Iijima, 1987). His 1991 paper starts with the invocation of fullerenes and reports having used the same graphite arc-discharge method as Krätschmer *et al.* (1990). The only thing Iijima did differently was to look not into the sooty deposit collected in the evaporation vessel but onto *the graphite electrodes* used to generate the arc-discharge themselves. As if the paper said “Hey, look over here, not there!” Then any researcher that had already tried to make electric-arc fullerenes according to the Krätschmer method could look again at the used graphite cathode discarded as junk to find similar tubes. The simplicity and wide availability of the experimental system affording nanotubes partly explains why so many researchers pay a huge tribute to Iijima’s 1991 report. Its popularity cannot be attributed to the magic power of the prefix “nano” since there was no “nano” in it.<sup>10</sup> It reads “helical microtubules”, “needle-like tubes”, or “graphene tubules”. The paper does not promise a bright future for industrial applications; rather, it simply describes tubes obtained by electric arc. This point is crucial: the thing of interest was to be found on the *generative part* of the experimental system – itself made of carbon (graphite electrodes) – not in the evaporation vessel where the *products* of electric arc discharge are usually collected. The thing coincides with its genesis.

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<sup>9</sup> Endo (2002) for instance was already mass-producing multi-wall nanotubes for electric batteries for more than a decade when he learnt – much to his stupefaction – that they had been “discovered” by his Japanese colleague.

<sup>10</sup> The term “nanotube” was coined as short form for “hollow graphitic tubules of nanometre dimensions” by the Franco-Norwegian physical chemist Thomas Ebbesen, working at the same time as Iijima in the same research institution (Tsukuba Fundamental Research Laboratories of NEC Corporation) in a 1992 paper reporting “large-scale” synthesis (gram quantities) of these objects (Ebbesen and Ajayan, 1992). Iijima only began using it in 1993 (Iijima and Ishahashi, 1993).

Of course Iijima did not discover carbon nanotube first, but in a way “he discovered them *best*” (Jones, 2011). His main accomplishment was a twofold operation of deterritorialization and reterritorialization<sup>11</sup> through which the tubes changed their mode of existence. The paper considered the tubes for themselves, regardless of their industrial uses, as unique individual entities growing without catalyst and hydrocarbons – only from graphite. He insisted on the relation between helical pitch, growth process, and electron diffraction patterns which suggested that the tubes might have unique electronic and mechanical properties with regard to their different helical conformations. The paper also broke the ties that bound carbon tubes to carbon fibers more than Endo did. Endo viewed hollowness as a structural feature responsible for the stiffness of a bigger fiber. Iijima valued hollowness as a functionality afforded by the tube, an affordance in the sense of a possibility of action offered to an agent by an environment (Gibson, 1979). Hollowness affords helicity, and helicity in turn affords new mechanical, chemical, and electronic behaviors. He also viewed Bacon’s “scroll growth” model (which explained the formation of the carbon filaments at the origin of electric arc-evaporation carbon fibers) as inadequate since no edge overlaps were observed at the needles’ surface, and replaced it with a “spiral growth” model aided by the chirality of the formative steps.

The divorce with Bacon’s growth model for carbon fibers did not just cut the hierarchical link between the two objects (deterritorialization); it also provided a new reinterpretation of their relationship (reterritorialization). Since Bacon’s scroll model is false, then Bacon’s “graphite whiskers” should have been multi-wall nanotubes, not scrolls.

Thus, Iijima deterritorialized the tubes from carbon fibers as well as from any field of application, and reterritorialized them onto fullerenes. His tubules were indeed finite structures closed by two hemispheres, and so fullerenes were both materially and symbolically capping the tubes. Thank to them their genesis was completed, looping back on itself. They could exist fully as individual objects in their own right (deterritorialization), while establishing structural and generative relationships with the other nanocarbons with which they appear in forming a family (reterritorialization). Fullerenes contributed to making the tubes more real and more attractive while, in turn, the tubes helped bring fullerenes the Nobel prize in 1996.

The tubes also helped make graphene more prominent. The existence of graphene was already implicated, *enveloped* in that of fullerenes and

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<sup>11</sup> Deterritorialization (Deleuze & Guattari, 1972) describes any process by which a set of hierarchical relations are broken and set free from their context of emergence to allow their actualization in different contexts (reterritorialization).

nanotubes as the virtual plane necessary for their mental reconstruction, the surface required for their design, their generative matrix. This is visible in the diagram displayed in Iijima's 1991 report (figure 5), as well as in two papers co-authored by Mildred and Gene Dresselhaus *et al.* one year later (Dresselhaus *et al.* 1992a, 1992b). The first explains the formation of fullerenes "by their projection on a honeycomb lattice" (i.e. graphene), and the second accounts for the structure (both topologic and electronic) of "*graphene* tubules" based on fullerenes.

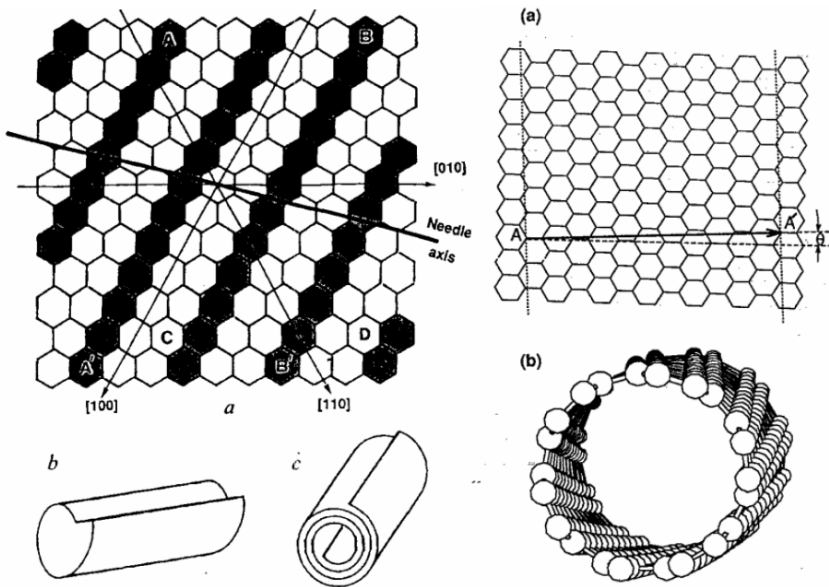


Figure 5 - From plane to tubes. Left: Spiral growth and scroll growth explained from graphene. Reprinted by permission from Macmillan Publishers Ltd: *Nature* (Iijima, 1991)<sup>12</sup>. Right: How to make a chiral tube from graphene. Reprinted by permission of APS <http://dx.doi.org/10.1103/PhysRevB.46.1804> (Dresselhaus *et al.* 1992b)<sup>13</sup>

<sup>12</sup> The Iijima's caption of (1991, p. 57) caption reads "a, Schematic diagram showing a helical arrangement of a graphitic carbon tubule, which is unrolled for the purposes of the explanation. The tube axis is indicated by the heavy line and the hexagons labelled A and B, and A' and B' are superimposed to form the tube (...). b, The row of hatched hexagons forms a helix on the tube. (...) c, A model of a scroll-type filament".

<sup>13</sup> "The vector AA' specifies a chiral fiber. We connect two dotted lines, normal to AA' at A and A' to form a chiral fiber" (Dresselhaus *et al.*, 1992b, p. 46).

It was in these 1992 papers that Dresselhaus transposed the topological “zig-zag/armchair” language from organic chemistry into carbon materials science. For nanotubes, zigzag and armchair are two specific non-chiral topologies located at the endpoints of an indefinite range of chiral topologies having various helical pitches. But this armchair-or-zigzag character also gathers together the three main nanocarbons within a generative topology by which they all shape each other. The way  $C_{60}$  fullerenes are cut affords armchair or zigzag tubes, and enucleating the end hemispheres of chiral nanotubes affords fullerenes of different specific geometries (figure 6). Similarly, rolling graphene in a zigzag or armchair way affords different specific tubes.

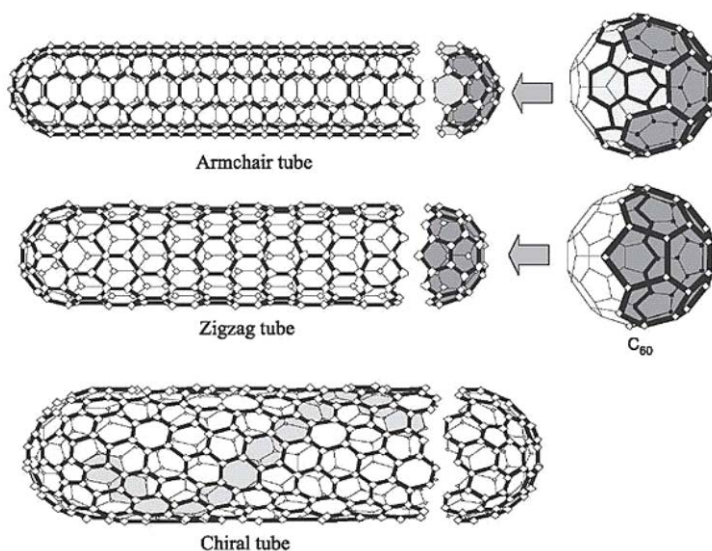


Figure 6 - The structure of armchair and zigzag graphene tubes (adapted from Dresselhaus et al., 1995). (Source: <http://what-when-how.com/nanoscience-and-nanotechnology/carbon-nanotubes-and-other-carbon-materials-part-1-nanotechnology/>)

In the period following the publication of Iijima’s observations a growing number of materials physicists and carbon scientists jumped on the electronic properties of nanotubes and embarked on the task of determining their band structure. But how to do so since the number of possible nanotube structures is theoretically infinite? Such was the role played by the nomenclature invented in 1992 by one of Iijima’s colleagues at NEC Semi-



conductors labs, physicist Noriaki Hamada (Hamada *et al.* 1992). Hamada's notation (figure 7) was a crucial contribution, used right away in most of the nanotube papers that would follow.

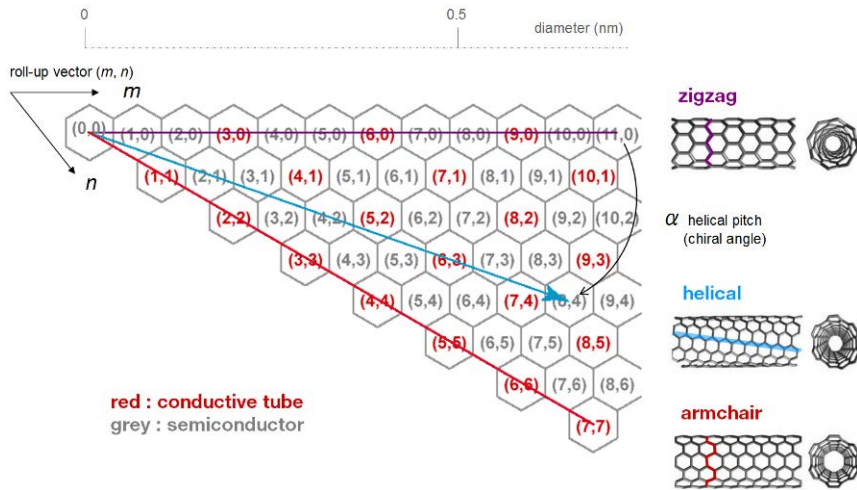


Figure 7 - Hamada's notation  
(Source: picture processed by the author)

A combination of two numbers,  $m$   $n$ , defines a vector on graphene lattice. This “roll-up vector” uniquely identifies all specific types of nanotubes. Zigzag nanotubes are those in which one of the two indices = 0, armchair nanotubes those where  $m = n$ , all other types are chiral. This vector determines the orientation of the tube circumference and its “helical pitch”  $\alpha$ . Thus, each type of nanotube of any circumference, length, and wrapping angle can be specified by a simple index of only two numbers.

But Hamada did more than establish a convenient nomenclature: he showed how theoretical predictions of electronic properties match this index. All armchair tubes are expected to be metallic; zigzag and helical tubes are semi-conductors except if  $m$  minus  $n$  is a multiple of 3 (then they would be metallic).<sup>14</sup> Thus, the electronic properties would depend sensitively on the wrapping angle. Moreover, it was also predicted that the band gap would depend on the tube's diameter, so that the electronic behavior could

<sup>14</sup> Dresselhaus (1992b) came independently to the same predictions with a less convenient nomenclature.

be tuned by pushing or bending the tube, which was later confirmed by experiments (Wilder *et al.*, 1998).

To understand the meaning and scope of this notation, it is instructive to compare it with the nomenclature established decades ago for intercalated graphite staging phenomenon, i.e. the fact that intercalated layers are periodically arranged in a matrix of graphite layers (Dresselhaus & Dresselhaus, 1981, p. 4). Like Hamada's notation, the symbolic index corresponds to a schematic diagram that can be drawn on paper.<sup>15</sup> Both languages are symbolic indexes linked with a structural representation of trivalent carbon structures. However, they are very different in that Hamada's notation is not a rigid language of *characterization* but a *design language*. In the former, substituting a  $\beta$  layer to an  $\alpha$  layer, for instance, is forbidden. The rigid rules of the stacking phenomenon would not allow for it. By contrast Hamada's language indicates how to roll a graphene sheet with a *chosen* lattice point superposed on the origin to form any nanotube having the desired geometry and electronic properties. It is both a nomenclature and a tuneable *recipe*, an invitation to make a virtually infinite number of possible tubes with predictable electronic behavior.

Allowing the specification of an infinite number of nano-objects having well defined properties, Hamada's notation opened a field of possibilities that is beyond imagination. This partly explains the blossoming of promises about a new revolution in nanoelectronics that arose during the 1990s.

## Graphene: Extreme Carbon Reborn

We have seen that graphene was not discovered in Manchester University by Geim and Novoselov. The Nobel Prize in Physics awarded in

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<sup>15</sup> The notation allows classifying graphite intercalation compounds by a "stage index"  $n$  denoting the number of stages graphite makes before finding an intercalated layer. Latin letters ABC refer to the three possible "profiles" of graphite layers,

A: OO–OO–O

B: O–OO–OO

C: –OO–OO–

Greek letters  $\alpha$ ,  $\beta$ ,  $\gamma$ ,  $\delta$  are the "stacking indices": the four possible configurations of the guest species contained in the intercalated layers. For instance, a stage  $n=1$  graphite intercalation compound is arranged in a periodical stacking sequence  $A\alpha A\beta A\gamma A\delta$ , a stage 2 compound is arranged in a  $AC\alpha CB\beta BA\gamma$  sequence and a stage 3 compound in a  $A\alpha ACA\beta ABA\gamma$ . There are thus definite relationships between the staging index and the stacking sequence.

2010 honors their “ground-breaking experiments regarding the two-dimensional material graphene”. What were the experiments that marked its entrance into a new existence, the rebirth of graphene?

The “Random walk to graphene” is nicely recounted in Geim’s Nobel lecture: two Russian physicists, tired about the monotony of solid-state physics and looking for fancier science in “Madchester”; an inexperienced Chinese PhD student; a Ukrainian scanning tunneling microscopist; and British scotch tape (Geim, 2010). Three initially unarticulated “thought clouds”: Geim’s own youthful dream of “metallic electronics”, his daze from all the fuss about carbon nanotubes, his reading of a thorough review of Dresselhaus on graphite intercalation compounds (1981), and his observation that despite the maturity of the field little was known about thin films of graphite. So Geim proposed a not-too-hard project for his PhD student: try to make graphite as thin as possible and see what would come from this idea. The suggestion to use scotch tape came from Oleg Shklyarevskii, an STM colleague from Ukraine working nearby on a fellowship. STM researchers customarily used highly-oriented pyrolytic graphite (HOPG) to prepare a standard reference test for STM imaging. In doing so, they would prepare a fresh surface of HPOG by removing a top layer with sticky tape, but they never paid attention to the stuff thrown away with the tape!

However, they did not get the Nobel Prize only for this “MacGyver exploit”. Whereas all previous incarnations of graphene were observational, they moved straightaway beyond observation and crafted a device showcasing the extreme tunability of its electric resistivity from a conducting to an almost semiconducting behavior. It was the end of the dual mode of existence of graphene: pure theory, impure incarnations. The two merged into a single new mode of existence. The “academic material” was turned into a technical device.

The Manchester team also claimed to have isolated *freestanding* graphene (Novoselov *et al.* 2005), although Peierls and Landau stated that 2D crystals cannot exist in their free state. According to them, instead of getting graphene in a particular and context-dependent incarnation, they showed and valued graphene “for what it really is”:

After all, we now know that isolated monolayers can be found in every pencil trace, if one searches carefully enough in an optical microscope. Graphene has literally been before our eyes and under our noses for many centuries but was never recognised for what it really is. (Geim, 2010, p. 88)

But what is it that graphene “really is”? First and foremost, graphene is a pure surface. Prior incarnations never involved freestanding graphene

as a self-sustained surface. Now the surface was deterritorialized, freed from the volume, freed even from the “*of*” (it is no more the surface *of* something... other than itself): a “surface in-itself” and no more an “over-layer” on the top *of* something or standing *on* something. As a scientific blogger nicknamed “Carbofiliac” put it:

It is clear that the graphene films that Boehm et al made are not freestanding graphene, since they are in a dilute alkaline solution – hence at best they are free-floating, not freestanding, and if you don’t know the difference, try standing on water, only few have accomplished that! (Carbofiliac, 2009)

What does “freestanding” mean for those who, like Geim, Novoselov, and many Carbofiliacs, sing this “ode to one” (Geim, 2010, p. 90)? Strictly speaking, it is not about standing in empty space like the Holy Ghost. In Carbofiliac’s joke, Jesus, unlike the Holy Ghost, can stand *on* something (walking on water) while still being “freestanding” (maintaining His walking behavior instead of swimming). Similarly, “freestanding graphene” refers to the ability of graphene to be reterritorialized in various contexts, transferred from one kind of substrate or environment to another while still maintaining its distinctive high “quality”.

Indeed graphene beats records in electron mobility, with conducting electrons behaving as massless particles much like photons, even in ambient conditions. For optics it absorbs light over a wide spectrum from infrared to ultraviolet. It is both nano and macro and can be engineered at both scales. It can potentially make membranes, flexible screens, conductive ink, transparent electrodes, magnetic shielding... In short, graphene fully meets the objectives of nanotechnologies as it is *par excellence* an “enabling material”. But even more is expected from it: a technological breakthrough not yet thinkable, for what distinguishes graphene from all known materials is less its performances taken one by one than the unique combination of “qualities” it affords.

Remarkably, graphene scientists often speak about its electronic, mechanical, magnetic, optical, or chemical “qualities” instead of using the more neutral “properties”. No doubt the use of this term shows a concern for valuating graphene and often goes with superlatives: astonishing, mystifying, counter-intuitive (Geim, 2010, p. 89). But beyond the hyperbolic effects this term emphasizes that graphene is *qualitatively* different. For instance, the extremely high amplitude of the electric field effect reported in Novoselov *et al.* (2004) – “thousands of times more than the few per cent changes observed previously for any metallic system” – is said to amount to a “qualitative difference” (Geim, 2010, p. 88). Or else, the properties of the individual monolayer are so different from those of the multiple stacked

layers composing graphite – starting with the fact that it is a surface with two sides, pushing to its extreme the “surface matters” motto characteristic of nanotechnology – that the variation is not a matter of more or less, but one of sameness and otherness.

“Quality” is also an old technical term of metaphysics. Unlike *essence* or the *nature* of being (*what* something is), qualities designate the *modes of being*, the *ways* by which something is (*how* something is). Etymologically, qualities (*qualia*) refer to the state of something that is *qua*, “that which is like that”. It differs thereby from *essential properties*, that which *define* something. 17th century mechanistic philosophers distinguished between “primary” (solidity, extension, figure, movement...) and “secondary” qualities (colors, odors, heat, textures, but also emotions, feelings and values). Primary qualities are caused by the essential properties of the substance; they are inseparable from its existence but separable from the existence of the perceiving subjects. “Secondary qualities” are deemed merely subjective, inseparable from the existence of their perceiving subject but separable from the substance. What about graphene’s qualities?

For a pure surface there is no more ontological difference between the material substance and its surface properties, between primary and secondary qualities, superficiality and depth. There is only a *superficial ontology* – an ontology of surface *qua* surface – wherein the stuff identifies itself with its qualities. Graphene has an intrinsic technical value as a pure surface, a “technicity” in the sense of Gilbert Simondon (1958), which is close to aesthetic beauty. When graphene champions speak of “recognizing graphene for what it really is”, they do not refer to an objective substrate that “stands under” (*sub-stare*) the surface and its properties. What graphene “really is” is not its essence but its high “quality”, a category that, in this context includes both being and value. The existence of a 2D structure in its free state is both an ontological reality and a technical opportunity, a mode of existence characterizing a “technoscientific object” (Nordmann, 2017). The objective and the subjective, the physical and the social meet on the same plane of existence – on the same surface, one might say.

## Conclusion

This chapter suggests that carbon has repeatedly attempted to attract the attention of researchers by deploying a variety of figures: filaments, whiskers, hollow tubes, soccer balls... but their dispositions and affordances remained buried in the black soot of furnaces and their small dimensions drowned in the mass of heavy industry. The nanotubes acceded to existence only in association with fullerenes as heads of a large family of

nano-objects captivating thousands of researchers. In this process, everything happened as if the three main nano-allotropes had mutually been “giving assistance” to each other – which is generally not the case with materials, which are often positioned in fierce competition to conquer markets. Each of them has become more real and more attractive by referring to each other in a space of emotional and material transformations comparable to the system of basic figures that makes the world of painting according to Kandinsky (1929): the point (fullerene), the line (nanotubes) and the plane (graphene).

The analogy conveys several meanings associated with this new mode of existence: that of *artistic quality*, the view of carbon as an artistic material instead of a dumb piece of matter. That also, of a system of *generative geometry*: just as for Kandinsky the line is the result of a living force applied to displace the point in a given direction and the plane is obtained by displacing the line, graphene can wrap into fullerenes or tubes, fullerenes can cap graphene cylinders into tubes, and tubes can be cut into graphene ribbons. *Interobjectivity* matters: just as the figures of painting can be described by their mutual relationships, the analogy allows one to pay – and to attract – attention to the relationships between objects, and not only between subjects and objects (Latour 1991, Harman 2011). Moreover, for Kandinsky, the point is not a mathematical abstraction; it has a certain shape, extension, and affective resonance with regard to its position relative to other objects. The line is a result of a living force with a certain direction and inflexion. As to the surface or “basic plane”, to Kandinsky it is not a mere substrate but a living matrix that requires the artist in order to be “fertilized” and to be felt “breathing”. Just as Kandinsky’s figures take on different affective tonalities shaping the sensibility of the beholder or the artist according to their mutual relationships, the configurations and dimensions of nanocarbons instantiate a *subjective geometry*. This emotional load may explain why fullerenes and nanotubes have (and surely will) remain molecules that matter even if they never deliver the tremendous applications promised over about twenty years of nanoscale research programs (Mody, 2008).

Despite all the proofs of concept for nanoelectronics and the indefinite field of possibilities opened for their design during the 1990s, nanotubes face great difficulty in finding their way to real-world applications while still maintaining their identity as individual nano-objects. Graphene supplants carbon nanotubes in this respect because it offers a homogeneous material, whereas sorting a batch of carbon nanotubes with specified wall numbers and helicity is barely feasible at an industrial scale. However, due to its lack of band gap graphene is not destined to replace silicon. Basic technoscientific research has made considerable progress in recent years

thanks to interdisciplinary mobilization but also because graphene has benefited from all the available nanoscale instrumentation as well as the know-how acquired in the chemical manipulation of carbon nanotubes. But graphene no longer has the right to exist as an academic material; it cannot remain a material on the shelf. Yet the number of cleanrooms, ultra-high vacuum instruments, postdocs, and European funding filling the “Home of Graphene” at Manchester University indicates the amount of means required to maintain graphene “freestanding”! Even if industrial production of large surfaces is possible, graphene cannot be introduced into macroscopic objects without a substrate and then it loses the affordances of the pure surface.

This is to say that the announced revolution may be delayed... However, graphene is also impelling a new movement back from brand to bench. Indeed it is perhaps unique but it is *generic*. Its mode of existence as a pure surface reveals to the possibility in other elements as well. Although it displays a unique combination of qualities graphene is neither perfect nor even optimal for the applications for which it is intended. Depending on the specifications of each sector, research begins to lurch towards other 2-D materials. Performances of the same order can be expected of any material displaying strong bonding within the plane and weak connections between planes. Thus graphene now appears as the head of a new family of 2-D “gigamolecules” including boron nitride, tungsten disulphide, and metal carbides, sulphides or selenides. Two-dimensional ceramic oxides are being made. After deploying a wide range of possibilities and opening the way to a new class of materials, carbon can fade into the background and give way to new competitors.

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## The Diverse Ecology of Electronic Materials

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### Abstract

*Silicon has been the dominant material in microelectronics for a half century. Other materials, however, have subsidiary roles in microelectronics manufacturing. A few materials have even been promoted as replacements for silicon. Yet because of silicon's dominance, none of these alternatives has gone from bench to brand; nor could any of them progress from brand to bench. For these reasons, historians have paid little attention to silicon and almost none to other microelectronics materials. I show, however, that we can better understand how the organization of the semiconductor (silicon) industry has changed over time by examining alternative microelectronic materials. I do so by presenting two case studies: one of a superconducting computing program at IBM, the most likely candidate to overthrow silicon in the '70s; the other of carbon fullerenes, the most likely candidates to overthrow silicon today.*

*Keywords:* Nanotubes, graphene, Josephson computing, Richard Smalley, IBM, Rolf Landauer, academic entrepreneurship, corporate research, historical alternatives.

### Résumé

*Le silicium a été le matériau dominant en microélectronique durant un demi-siècle. Cependant, d'autres matériaux ont des rôles complémentaires dans cette filière. Quelques matériaux ont même été promus en remplacement du silicium. Pourtant, en raison de la domination du silicium, aucune de ces alternatives n'est allée de la paillasse à la marque commerciale, et aucune d'entre elles ne pourrait retourner de la marque vers la paillasse. Pour ces raisons, les historiens ont prêté peu d'attention au silicium et presque aucun à d'autres matériaux de la microélectronique. Je montre, cependant, que nous pouvons mieux comprendre comment l'organisation de l'industrie des semi-conducteurs (silicium) a changé au fil du temps en examinant les matériaux microélectroniques alternatifs. Je le fais en présentant deux études de cas : l'un basé sur un programme d'informatique supraconductrice chez IBM, le candidat le plus plausible pour renverser le silicium dans les années 70 ; l'autre portant sur les fullerènes (carbone), les candidats les plus plausibles pour renverser le silicium aujourd'hui.*

*Mots-clés :* Nanotubes, graphène, informatique Josephson, Richard Smalley, IBM, Rolf Landauer, entrepreneuriat universitaire, recherche industrielle, alternatives historiques.

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THE DIGITAL electronic computer has been around for about seventy years. For most of that time, the majority of computer architectures have been built around transistors and other components embedded in integrated circuits composed primarily of silicon and silicon dioxide. Although there is some public awareness of the materials used in microelectronics (e.g. Gorilla Glass used in iPhones), professional historians have largely neglected the materials of computing. Instead, they usually profile individual mathematicians and theoretical physicists (Turing, von Neumann, Shockley, Bardeen) and/or big businesses (Bell Labs, IBM, Fairchild, Intel), neither of which are described as getting their hands dirty messing with chemicals and chemical apparatus. The biggest exceptions, by far, have been Christophe Lécuyer and David Brock (2006), who have consistently reminded us that the transistor would have been a footnote but for the expertise of chemists, metallurgists, and materials scientists who grew and purified crystals, developed novel photoresists, applied sophisticated acids and “dry etches”, invented techniques for cutting and polishing wafers, etc. Brock and Lécuyer have hammered on the point that materials innovation has been indispensable both in the creation of new *types* of gadgets and in the manufacturing of vast *numbers* of those gadgets.

The microelectronics industry both relies on materials innovation and also markets products that are used in materials innovation. Digital computing has been an important tool of chemical and materials research since at least the 1950s: in modeling of molecules (Francoeur, 2002), in more efficient circulation and searching of chemical abstracts (Rayward & Bowden, 2002), in creating a new field of computational chemistry (Johnson, 2006), in operating certain kinds of experimental apparatus (November, 2012). Thus, microelectronics offers a particularly clear example of the continuous circulation of people, materials, and ideas both from brand to bench and from bench to brand.

At first glance, though, that circulation might seem rather narrowly confined to a single material: silicon. Silicon integrated circuit transistors dominate the imaginary of microelectronics – even though little semiconductor manufacturing is done there any more, it is still *Silicon* Valley, not Gallium Arsenide Valley. And that is because silicon also dominates other material configurations of microelectronics in the marketplace. No other material has moved all the way from bench to brand in numbers of products that in any way rival silicon’s numbers. And because materials other than silicon hardly exist in “brand” form, they can’t move from brand to bench either. I will argue, however, that despite silicon’s dominance, the



material basis of modern microelectronics is actually quite diverse, and therefore that the flows of people and materials into and out of the microelectronics industry are quite complex. For all its dominance, silicon is hardly alone. More than half of the elements in the periodic table can be found in today's cell phones. In addition, if we look beyond just microelectronics *products*, and expand our perspective to include manufacturing *processes*, we can see that the production of silicon integrated circuits requires an even more diverse array of materials which draws on a dizzying array of expertise from plasma physics to organic chemistry.

Moreover, silicon integrated circuits have never gone unchallenged – there have always been other materials that have vied to replace silicon. Or, to put it less anthropomorphically, there have always been experts in silicon who have been dissatisfied with its performance and therefore sought out alternative materials. There have also always been experts in materials other than silicon who have sought to bring their materials into the mainstream of microelectronics. This article presents two case studies, one for each of these possibilities. The first looks at IBM's exploration of computer architectures based on superconducting, rather than semiconducting materials; this effort was widely considered the best possibility for the overthrow of silicon in the 1970s. The second case looks at attempts, particularly by the Nobel laureate chemist Richard Smalley, to make carbon the central element of microelectronics – either in the form of pure allotropes or as the main constituent of so-called “molecular electronics”. Molecular electronics and pure-carbon graphene are today widely touted as the best candidates for overthrowing silicon.

Thus, while the microelectronics industry is imagined (by the public, by insiders, by historians) to be a semiconductor industry (and specifically a silicon industry), the reality is that alternatives to and hybrids with silicon have played an important role in silicon's success. I therefore draw on the “historical alternatives” approach from business history, which pays close attention to the presence of alternatives and hybrids in the organization of manufacturing. In particular, the historical alternatives approach emphasizes the role of actors and organizations within an industry in proposing, observing, evaluating, and choosing among a variety of strategies. Although rarely applied to choices among technologies – much less materials – I argue that the historical alternatives approach can help us understand how the different parts of the research system generate and evaluate alternative materialities. By comparing my two case studies – one from the 1970s, one from the 1990s – I also show that we can observe how the roles of different constituents of the research system (corporate laboratories, universities, etc.) have evolved over the past half-century. Alternative materials

serve as a kind of probe to measure how different research institutions have changed in the past half-century.

### **Alternatives and Hybrids versus Epochal Breaks**

By focusing on the materials of microelectronics, I will transgress, though not entirely overturn, the conventional historical narrative of the development of digital computers and of the semiconductor industry. That standard narrative depicts innovation in electronic information-processing as having progressed through a series of discrete material-technological stages going back more than a century: the electromechanical switch yielded to the thermionic valve/vacuum tube, which then gave way to the discrete germanium (and later silicon) transistor, which was superseded by the bipolar (later CMOS) silicon integrated circuit, which in turn will someday surrender to a nanoelectronic architecture based on some other material: graphene, carbon nanotubes, DNA, charge transfer salts, or perhaps an as-yet-undiscovered molecule (Choi & Mody, 2009).

This kind of narrative has a compelling simplicity, yet we should be wary of such all-one-way-or-the-other stories. Among business historians, Jonathan Zeitlin has been particularly critical of narratives in which forms of business organization switch suddenly and completely from one mode to another: from the family firm to the multi-division corporation to the networked venture-labor enterprise. Instead, Zeitlin and Charles Sabel have proposed the “historical alternatives approach” to thicken the temporal boundaries between such transitions and to acknowledge actors’ uncertain and heterogeneous strategies in attempting to choose among different co-existing organizational forms. As Zeitlin (2007, p. 124-128) puts it in a review of his and Sabel’s framework:

the process of strategic reflection and hedging against risk gives rise to a proliferation of hybrid forms.... Hence the predominance of hybrid, mixed, and intermediate forms... over polar types has proved to be the empirical rule rather than the exception.... The interpenetration of strategies and practices within industries and national economies at any one time resulting from actors’ efforts to hedge their organizational and technological bets about future changes in the environment casts inevitable doubt on the possibility of drawing sharp distinctions between epochs.... [I]t seems more useful to distinguish historical epochs according to changing orientations towards [what is] regarded as normal or paradigmatic than to divide history into periods where social life was in fact thoroughly organized according to one or another master principle.

Zeitlin mentions “technological bets” in this passage, but the predominant use of the historical alternatives approach has been in tracing the evolution of organizational forms. As both business historians (including, surely, Zeitlin) and historians of technology would acknowledge, however, the distinction between a technological bet and an organizational one is often fuzzy. Organizations that last continually adjust, and adjust themselves to, the technologies they use.

With that in mind, it should be clear that the historical alternatives approach can help us undermine the stark polarities of the standard narrative of microelectronics. Electromechanical switches continued in use long after the invention of the vacuum tube, and are still one of the dominant and obdurate forms of interface between users and their electrical/electronic devices (Plotnick, 2012). For many years, vacuum tubes possessed decisive advantages over transistors for certain applications. Indeed, in a few niches, such as high-end audio, tubes still live on (Downes, 2009). The shift from tubes to transistors was therefore gradual and still only partial. So was the later shift from the discrete transistor to the integrated circuit. Even though integrated circuits are ubiquitous today, when they were invented in the late 1950s they initially only offered advantages for a few, mostly military, applications. For most consumer electronics products, such as “transistor radios”, the discrete transistor was the reasonable choice for many years. Moreover, the (still incomplete) transition from discrete components to integrated circuits featured a long period in which *hybrids* of the two were at the leading edge – rather similar to the long period of hybrid gas and electric cars in the late 19<sup>th</sup> century (Mom, 2004). Perhaps the most commercially important computer of all time – IBM’s System/360, first sold in 1965 – used a hybrid chip architecture known as Solid Logic Technology, in which various sub-circuits were baked together as discrete components, but the sub-circuits themselves were monolithic integrated circuits (Bassett, 2007, p. 67).

The development of alternatives in parallel with each other, and the emergence of hybrids between alternatives, characterizes the history of electronics and computing from the systems level (whole computers and their peripherals) all the way down to the materials from which those systems are composed. At the systems level, historians of computing are beginning to grapple with the fact that analog computers co-existed with – and in some applications outcompeted – digital computers for much longer than the old celebratory narratives acknowledged (Cohn 2013). At the level of materials, Brock (2009), Lécuyer and Ueyama (2013), and a few others have begun to flesh out the diverse ecology of electronic materials that complemented, competed with, undergirded, and extended the dominance of silicon.

Foregrounding that diversity might, to the uncharitable, simply seem like giving ribbons to the also-rans. The history of microelectronics is filled with side streets branching away from silicon, but so far all of those have turned out to be dead ends or, at best, *culs-de-sac* serving some limited “neighborhood” of applications. For instance, compound semiconductors – semiconductors composed of more than one element, such as gallium arsenide or gallium nitride – have been promoted for decades for their *theoretical* superiority to silicon. So far, though, compound semiconductors have found only niche service in lasers, light emitting diodes (LEDs), and some solar cells. For most microelectronics applications, silicon really has been dominant, in *some* sense, for more than a half century. My intent here is not to romanticize the unsuccessful underdogs.

Still, there are good reasons to pay more attention to the diverse ecology of electronic materials – the mutually supporting tools and materials such as lithographic steppers, advanced cleaning pads, and photochemical resists that combine to allow fabrication of complex chips. One reason is simply that the kinds of actors who pursued quixotic alternatives to silicon are intrinsically interesting. For those not inclined to arguments from intrinsic interest, however, I would point out that the same actors were also often people who made important contributions elsewhere in science, contributions that might not be fully intelligible without examining their interest in alternative electronic materials. David Brock and David Laws (2012), for instance, have examined the history of a superconducting device called the cryotron which competed with the transistor in the late ‘50s and early ‘60s, after which it fell from view entirely. The people who developed the cryotron included Dick Garwin, famous both in gravitational radiation physics and ballistic missile defense policymaking, and Ken Shoulders and Dudley Buck, early pioneers of electron beam lithography. John Bremer (2007) makes the case that the first “integrated circuit” was not the semiconductor-based circuit invented independently by Jack Kilby and Robert Noyce, but rather a superconducting cryotron circuit made by Buck and Shoulders.

Narratives that embrace a larger, more diverse ecology of electronic materials also allow us to see constraints on innovation that would not be apparent from an exclusive focus on silicon. A computer or gadget is more than a chip. The materials that go into user interfaces, such as liquid crystals (Gross, 2011), have often suggested certain applications or innovation pathways and discouraged others. So have the materials used in power systems, especially mobile power supplies incorporated in portable electronic devices (Hintz, 2009; Eisler, 2012). If one looks at microelectronics from the perspective of power supplies, the trajectory of innovation in making transistors draw less power becomes more obvious than the oft-celebrated

trajectory of making transistors smaller and faster. Similarly, if you look at microelectronics from the perspective of the materials in which a chip is packaged (rather than the materials in the chip itself), then the geographic focus of your story moves from *Silicon* Valley to industrial districts in Taiwan and elsewhere (Tinn, 2012).

I could point to other reasons to pursue an historical alternatives approach to electronic materials, and no doubt there are good reasons I've overlooked. For the rest of this article, though, I want to concentrate on applying this framework to the *organizations* that pursued quixotic alternatives to silicon. Acknowledging the diversity of the electronic materials ecology tells us a great deal about the organizations that fostered innovation in electronics in general (including in silicon). It also tells us a great deal about the evolution of R&D organizations in general, both in and out of electronics and at the thick boundaries between the center and periphery of the electronics industry. In microelectronics, new materials co-emerged with new organizational forms throughout the 20<sup>th</sup> century, and will continue to do so in the 21<sup>st</sup>. And, again, many of the individuals involved in promoting (or at least exploring) new materials for microelectronics have also promoted new ways of organizing research on materials for microelectronics.

To give a sense of why organizations matter in the search for new materials, let me juxtapose two extended quotes. The first is from Zeitlin (2007, p. 120-123) again, fleshing out the historical alternatives framework:

the hallmark of this approach is its emphasis on the salience of alternative possibilities, contingency, and strategic choice in the development of modern industry.... [T]echnology and organization should not be taken as fixed, given, or even latent parameters to which economic actors must perforce adjust, but rather as objects of strategic reflection and deliberate experimentation in their own right.... Economic actors ... are often at least as concerned with determining, in the double sense of figuring out and shaping, the context they are in – market, technological, institutional – as with pursuing their advantage within any particular context.... Crucial to this process of strategic reflection is the capacity of economic agents to imagine and weigh up alternative courses of action, connecting the present with both the future and the past through narratives which constitute their identities and interests.

My second quote comes from Rolf Landauer, an IBM physicist probably best known for his work on the theoretical minimum amount of energy required to erase a single bit of information. Within the wider physics community this result is usually remembered as (possibly) disproving the possibility of Maxwell's Demon (Wright, 2016). Within IBM, however,

Landauer is remembered as “an outstanding scientific and technical manager of IBM’s Watson Research Laboratory, guiding it from relative obscurity to become by 1970 one of the world’s two most important and innovative engineering and scientific laboratories” (Bennett & Fowler, 2009, p. 1).

Much of what Landauer did at the Watson Lab (more commonly referred to as IBM Yorktown) was to help determine – in Zeitlin’s double sense – the ultimate limits to the miniaturization of microelectronics in order to aid executives in choosing which material basis for microelectronic circuits would facilitate the firm’s progress toward those ultimate limits ahead of its competitors. One of the critical functions of people like Landauer, therefore, was to provide Zeitlin’s “strategic reflection” by “imagin[ing] and weigh[ing] up alternative courses of action”. After a whole career of this kind of work, Landauer (1993) looked back in this way:

There are many advanced technology proposals which become major thrusts, only to be abandoned again subsequently. An adventurous technological climate has to reward the taking of risk, and must allow failures.... Among the many supposedly broadly applicable logic proposals we have seen come and go, we can find Gunn effect logic, tunnel diodes, ferrite core logic, schemes utilizing combinations of electroluminescent devices and photoconductors, fluid logic, parametric microwave excitation and Josephson junctions. Some technological candidates, such as Josephson junction logic, magnetic bubble storage, or the battery powered automobile, did deserve real examination. When they are discarded, it is done with trepidation, and knowledge that the decision may not last forever.

Landauer’s quote supports my point that we can use the historical alternatives approach to understand the history of microelectronics, particularly in terms of the material basis of microelectronic technologies but also in understanding the function of microelectronics firms’ corporate research laboratories. That is, corporate labs in Landauer’s era served, in part, as places where “advanced technology proposals” could be incubated to the point where they could receive “real examination” and either be pursued or “abandoned”.

Landauer adds his own take, though. If you read the whole article I’ve just quoted from, he argues that these various alternatives often receive more attention than they deserve because their proponents vocally promote their advantages but not their shortcomings, whereas potential detractors (such as Landauer himself) have little motivation to weigh in negatively until very late in the game. It’s a point that goes a long way toward explaining IBM’s rich history of exploring alternative microelectronic materials, devices, and manufacturing processes at enormous cost. Yet the skepticism of

people like Landauer meant IBM only rarely adopted adventurous alternatives.

Indeed, in a few famous cases – most famously CMOS transistors – IBM invented and/or developed alternative microelectronics technologies, only to discard them before being forced by the rest of the industry to re-adopt the technology later on. Other giant corporate research labs experienced similar misadventures in the 1970s and 1980s, such as Xerox PARC’s “losing” the graphical user interface to Apple and Microsoft (Smith & Alexander, 1988). Such mishaps contributed to the long-term reorganization of corporate research since the 1980s in which industry has downsized in-house research and nearly abandoned in-house long-range or fundamental research (Khan, Hounshell, & Fuchs, 2015).

### **A Semiconductor Industry No More**

To give a sense of how the search for novel electronic materials contributed directly to the decline of corporate long-range research, let me focus on an episode that is obliquely alluded to in the Landauer quote above. Note how Landauer assigns a particularly ambiguous status to one “broadly applicable logic proposal”: the Josephson junction. On the one hand, Josephson junctions are thrown in with a pile of other failures. On the other hand, Landauer sets Josephson logic apart as the only such proposal to “deserve real examination”. He even hints that, though Josephson was abandoned, it might – unlike its peers – come back someday.

So what is Josephson logic? In the early 1960s, a young British graduate student, Brian Josephson, made a series of striking predictions regarding the behavior of certain kinds of superconducting circuits – work for which he shared the 1973 Nobel Prize in Physics (when he was just 33). Those ideas were quickly taken up by research labs at several large US firms, including AT&T, IBM, General Electric, and even Ford Motor Company. Interest at IBM focused on ways of applying Josephson’s ideas to constructing circuits with extreme rapid switching times – i.e., circuits that would be useful in high-speed computing.

The Josephson junction’s promise was quickly made evident to IBM management. The early work

culminated in 1966 in demonstration of subnanosecond switching of Josephson tunneling devices, and in 1967 in the operation of a thin-film Josephson device flip-flop, both indicating that Josephson switching devices could indeed be switched very fast and could be competitive with projected semiconductor integrated circuits. On the basis of these encouraging re-

sults, the pros and cons of Josephson devices were assessed and an initially small research program was launched in 1967 with the aim of studying technological and system aspects. (Anacker, 1980, p. 108)

The Josephson project was commissioned partly on the basis of the novel characteristics of superconducting circuits. IBM was committed to leading in basic research, and superconducting materials were attractive on that basis both as a topic of fundamental intrinsic interest, and potentially as key constituents of scientific instruments that could be used in space science, biomedical research, and other areas.

However, IBM was also keen to push into Josephson junctions because of worries that further circuit miniaturization would not be possible for much longer with silicon, and therefore that alternative materials needed to be explored. That view was most vigorously put forward by Robert Keyes, a physicist and close friend of Landauer's (he wrote one of Landauer's National Academies obituaries and is mentioned as a confidante in another). In "Physical Problems and Limits in Computer Logic" and "Physical Limits in Digital Electronics", Keyes (1969 and 1975) warned that power dissipation, in particular, was a rapidly approaching problem, and pointed to a variety of exotic technologies that might provide at least temporary relief.

The stunning success of silicon semiconductor technology for information processing has not completely stifled the search for alternative technological bases for memory and logic. In the first place, although progress in silicon technology seems certain to continue and to provide ever-more-capable and economic general-purpose computers, quantum leaps or revolutions cannot be predicted with confidence; if forthcoming it appears that they must be sought elsewhere.... Thus there has been interest in and research related to logic based on superconducting devices, fluid devices, magnetic bubbles, and even optical devices, in the past decade. Superconducting devices based on the Josephson tunneling cryotron appear to be the most likely candidate for logic that will make a much larger, faster computer possible; a Josephson gate that switches in only picoseconds and has a power dissipation of microwatts has been described. (Keyes, 1975, p. 760)

In other words, Keyes was urging his firm to experiment with – and to engage in "strategic reflection" about – a whole host of "historical alternatives" to the silicon integrated circuit. And of those alternatives, Josephson computing seemed to be the most promising.

IBM heeded that call, and through the early '70s its Josephson program gradually grew, generating know-how and patents that would protect IBM's position if the technology took off. Other firms, especially AT&T



and Sperry, established smaller Josephson groups as, in essence, fast followers behind IBM so that they could catch up if need be – again, exactly what one would expect in a world dominated by uncertain choices among historical alternatives rather than binary transitions from one technological state to another. The US National Security Agency, as well, became interested in Josephson computing as a potential enabler of ultrafast cryptography. A small group at NSA conducted in-house research shadowing IBM and other firms, and at the same time began contributing about a quarter of the IBM project's funding.

By the late '70s, Josephson technology had progressed far enough that upper management deemed it ready to transition from "R" to "D". The size of the project swelled, to about 125 personnel and a \$20 million annual budget in the early '80s. Yet as superconducting chips came tantalizingly close to production, the project became increasingly dependent on exactly the *semiconductor* personnel and expertise that Josephson technology was to supplant. As one of the leaders of the project put it in a review article,

A computer made up of Josephson junctions constitutes a radical departure from a well-established semiconductor technology. The fabrication of Josephson-junction components relies, however, almost entirely on methods learned in the development of semiconductor devices. The substrate material chosen for the Josephson-junction chips is silicon, not because of its conducting properties but because techniques for forming precise microscopic structures on silicon are well established. Circuit patterns are defined photolithographically, as they are in making semiconductor devices. (Matissoo, 1980)

In other words, the Josephson team needed IBM's silicon manufacturing experts to adapt their technology for mass production, at just the same time that Josephson technology was maturing to the point that its potential could be measured directly against that of silicon, with the possibility that silicon could lose.

In 1980, IBM's Director of Research, Ralph Gomory, convened an "Extendibility Study" to make that direct comparison between Josephson and silicon technology, with the aim of deciding whether the company would halt the project or continue following the Josephson path in parallel with silicon. In the end, though, the study estimated that with another decade of development Josephson chips *could* be three to six times faster than

silicon bipolar junction chips.<sup>1</sup> That forecast was ambiguous enough that the company continued on with the Josephson project, though its research-oriented leader, Wilhelm Anacker, was replaced by Joe Logue, a manager with deep expertise in semiconductor manufacturing and *product* development. Like most of IBM's silicon establishment, though, Logue was skeptical of Josephson technology's potential, and took the job only on the promise that it would come up for another moment of strategic reflection in two years' time (Logue, 1998).

Accordingly, in 1983 Gomory commissioned another extendibility study, this time to compare Josephson technology, bipolar silicon chips, and gallium arsenide, another of the perennial contenders to unseat silicon. This time, Josephson was found to be even less competitive relative to silicon than just three years earlier. Both Josephson and gallium arsenide circuits possessed *theoretical* advantages over silicon, and in small, simple devices those theoretical advantages had in fact been realized. But the 1983 extendibility study forecast that over the foreseeable future any mass-produced, complex chip based on either Josephson junctions or gallium arsenide would most likely have too small (if any) advantage over silicon to justify the cost of the firm's investment. IBM had reflected strategically, and concluded that it should not follow the alternative path of Josephson computing. And so, on September 23, 1983, the program was canceled (Robinson, 1983).

## Back to the Bench

The Josephson program was by no means IBM's only foray into alternatives to silicon, but it grew larger and progressed significantly further than similar efforts. Based on estimates in the trade press at the time and interviews with participants, I believe the whole program probably cost on the order of a quarter billion of today's dollars – not small change, but also not a risky expenditure for a company as dominant as IBM at the time. The firm's return on that investment is hard to specify, but it would have included: a substantial patent portfolio, some key personnel (including several who helped save IBM from bankruptcy in the early '90s), and some admiring press coverage that reinforced the company's image as innovator. Indirectly, the Josephson program helped four IBM scientists win shares in two separate Nobel Prizes for Physics. The first, in 1986 for the scanning tunneling microscope, originated in part as an attempt to characterize ultrathin

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<sup>1</sup> Emerson Pugh *et al.* (1980), Josephson Extendibility Study, in IBM Archives, Box 475, Folder 1 of 8 (# 8 in box), 1-1.

superconducting films used in the Josephson program. The second, in 1987 for high-temperature superconductivity, was inspired by the program's search for better superconductors, and was aided by conversations with the program's superconductivity experts. During the late-'80s frenzy over high-temperature superconductors, the Josephson program gave IBM a leadership position in the field and a stake in reactive policy initiatives such as the Consortium for Superconducting Electronics.

The Josephson program also generated two pieces of critical self-knowledge that IBM slowly absorbed over the next decade. First, it learned that Josephson junctions were *not* the way forward and therefore that – at least in terms of microelectronics – IBM would remain a semiconductor firm and not a superconductor firm. That might seem an expensive lesson, but it allowed IBM (and all of the other firms that had been carefully watching it) to more efficiently allocate resources, particularly during the early '80s semiconductor boom associated with the first personal computer craze. And, obvious as it might seem, Josephson's non-viability was a lesson other organizations – notably the NSA and Japan's Ministry of International Trade and Industry – would continue to pay millions to learn over the ensuing decades. The *theoretical* speed and power advantage of superconducting electronics over semiconductors tempted MITI and the NSA even after the IBM program, and continue to lure smaller research groups even today.

At the same time, IBM learned from the Josephson program that it needed to change the way its research and manufacturing arms worked with each other. Over the course of the '80s, IBM Research would slowly become more product-oriented and less attached to basic, long-range investigation. That lesson would only be fully absorbed, however, after the company's early-'90s brush with bankruptcy. To keep IBM from dissolving, Josephson project veterans such as Carl Anderson, Juri Matisoo, and Mark Ketchen were called in to move IBM from a Cold War business model to a post-Fordist, Third Industrial Revolution (Dosi & Galambos, 2013) model – though, nodding to Zeitlin again, it's important to acknowledge that that transition was not as sudden as the metaphor of “revolutions” implies, and that older and hybrid forms continue to compete with more purely post-Fordist models.

These were exactly the kinds of side-benefits that large Cold War era corporate research laboratories in the US were supposed to accrue from their curiosity-driven exploration of fundamental questions. From the '50s to the early '80s, labs such as IBM Yorktown and Bell Labs were well-resourced and loosely steered, with the expectation that not all – in fact, quite few – of the alternative paths they wandered down would yield viable

products or processes. Basic research allowed firms to hedge their bets by examining alternative technologies that might, potentially, displace their core products. But corporate basic research also helped firms train new generations of managers; it reinforced those firms' reputations for innovation (and, implicitly, a continually improving product line); and it saved firms money in the form of hefty tax incentives favoring basic research (Asner, 2006).

As the Cold War gradually wound down and the global economy became more competitive, however, all of those justifications for basic corporate research diminished. Ironically, that led some of the most vocal supporters of these labs' reflection on alternatives to become prominent skeptics of that strategy. As Landauer's obituarists (Bennett & Fowler, 2009, p. 10) put it, for instance, "he understood what was needed to build a computer very well and along with Robert Keyes tried to pass such knowledge to the promoters of every cockamamie scheme that emerged. As a result he took a dim view of optical computing, [and] logic based on threshold devices, such as Esaki diodes and Josephson junctions". Keyes (1992) made a similar point, somewhat less colorfully:

The differences between the environments in a large [information processing] system and in a laboratory are often not recognized with the result that the essential attributes are missing in device proposals [e.g., single-electron transistors, cellular automata, and molecular electronics]. Thus, although many proposals for devices have been put forward, only three, the relay, the vacuum tube, and the transistor, have proven able to meet the requirements and form the basis of large computing systems.

Here we see the harsh lessons of IBM's Josephson computing foray brought to bear on all non-silicon, non-transistor microelectronics.

And yet, many many varieties of non-silicon, non-transistor microelectronics are still being actively promoted as potential future replacements for silicon transistors. Vast programs run by individual manufacturers, industrial consortia, and government agencies such as DARPA exist to manage research into those alternatives and to incubate them until they might be ready to move into production. However, that research is organized very differently than it was in the era of the IBM Josephson program. Back then, semiconductor manufacturing was still quite vertically integrated, at least at large firms like IBM. That vertical integration included research – once IBM bet big on Josephson in the early '70s, its program depended disproportionately on in-house expertise. Today, semiconductor manufacturing is almost completely dis-integrated. Firms specialize in chip design, chip fabrication, packaging, tool development, even research – but virtually no

firm does it all (and few do more than one of those activities). Thus, manufacturers need to depend on outside actors to elaborate possible alternatives to silicon. Industrial research consortia are one possibility; government laboratories are another. Increasingly, though, firms look to academic researchers – or to consortia and government agencies that manage a portfolio of academic researchers (Khan, Hounshell & Fuchs, 2015) – to bring alternative microelectronics materials closer to the market.

### **C<sub>60</sub> and Fullerene Materials as/and Electronics Research**

To better understand the semiconductor industry's increasing dependence on academic research, I want to finish with a brief outline of a story which is justly famous in the history of recent chemistry – namely, the discovery of buckminsterfullerene (or C<sub>60</sub>), for which Harry Kroto (formerly of the University of Sussex in the UK) and Bob Curl and Rick Smalley (both of Rice University in the US) received the 1996 Nobel Prize in Chemistry. A small cottage industry of popular histories and professional historical, social scientific, philosophical, and literary studies of the fullerene discovery has accumulated in the thirty-plus years since C<sub>60</sub> was announced (Aldersey-Williams, 1995; Baggott, 1994; Ball, 1994; Bueno, 2006; Sparrow, 2007; Kaplan & Radin, 2011; Broadhead & Howard, 2011; Eisler, 2013; McCray, 2013; Moskowitz, 2016). This literature has delineated several contexts which fostered the discovery of C<sub>60</sub> and/or its discoverers' later work – most notably space science and futurist “visioneering”. However, virtually none of these studies has foregrounded fullerene chemistry's more mundane if equally extensive connections with microelectronics.

The standard story usually begins with the fortuitous advent of the Kroto-Curl-Smalley collaboration. As a postdoc at the University of Chicago, Smalley had invented an apparatus for making spectroscopic measurements of very small, very cold clusters of atoms. A version of that device which he and his students built at Rice – known as the AP2 – was the center of his research program and formed the basis for collaborations with Curl. When Kroto encountered Curl at a conference in the early 1980s, the AP2 came up in conversation and Kroto seized on it as the means to investigate the chemical makeup of matter in interstellar space – an environment he believed contained a variety of very cold, very small clusters of carbon atoms which he thought could be simulated in the environment of the AP2. In the late summer/early fall of 1985, Smalley finally agreed to generate carbon clusters with the AP2, Kroto flew to Houston, and over the next few weeks Curl, Kroto, Smalley, and the latter's graduate students stumbled on, and struggled to interpret, data indicating the presence of a molecule

made up of sixty carbon atoms forming a closed cage –  $C_{60}$ , the third known allotrope of pure carbon after diamond and graphite.

This narrative of heroic serendipity amidst curiosity-driven research leaves a great deal obscure. Elsewhere in this volume, Sacha Loeve suggests how we might situate this episode in the long history of carbon allotropes. What I'll do here, instead, is to situate Smalley's contribution to this episode within the long history of research on electronic materials. At the time Kroto first suggested examining carbon, Smalley was studying the reactivity of small clusters (today we would call them nanoclusters) of *semiconductor* materials. The AP2 was originally built to study metal clusters, but in the months before  $C_{60}$  was discovered Smalley's group (in collaboration with Curl and a Rice electrical engineering professor, Frank Tittel) had moved on to examining whether semiconductor clusters differ significantly from metal ones (they do). Because semiconductor properties can vary wildly in the presence of even minute amounts of impurities, Smalley was afraid that vaporizing a carbon disc in the AP2 would contaminate any future experiments with silicon, germanium, or gallium arsenide in the same apparatus. Therefore, he refused to do Kroto's carbon experiment until after he had finished working on semiconductor clusters with the AP2.

The AP2 semiconductor cluster experiments were, in a sense, the kind of incremental basic research that scientists sometimes describe as "picking the low-hanging fruit". Smalley had an experimental apparatus, and the periodic table provided a menu of elements and simple compounds to put in it. Semiconductor clusters, in that light, were epistemically no different from the metal clusters they followed or the carbon clusters they preceded. But epistemology is one thing; gaining the resources to do an actual experiment, and then using that experiment to gain further resources – what Bruno Latour and Steve Woolgar (1986, p. 231) referred to as the cycle of credit – is another.

Semiconductor research, of course, has a large, well-resourced audience with specific technological aims in mind which Smalley, *et al.* played to in setting up their experiments with semiconductor nanoclusters – as seen in the introduction to one of their articles:

Driven by the extreme technological importance of new breeds of semiconducting materials, there has been quite an active interest in theoretical models of III-V semiconductors.... Virtually all theoretical approaches to semiconductor surfaces and interfaces start with a relatively small cluster of atoms and... compare to bulk surface measurements... [T]here is still a potentially severe mismatch between the essentially microscopic theory and essentially macroscopic experiments. One appealing way out... is... by developing techniques for generating and probing the very clusters the theory

is best able to handle. Certainly this will not be a universal solution. Particularly for semiconductors (where the major device-driven interest often focuses on such intrinsically macroscopic phenomena as depletion layers, etc.), not all properties of bulk interfaces will be accessible through the study of microscopic clusters. But the crucial short-range phenomena... occur in the small clusters as well. (O'Brien *et al.*, 1986)

Notably, the large discs of silicon, germanium, and gallium arsenide used in these experiments came from Texas Instruments, and one of the Ph.D. students working on the project, Sean O'Brien, went on from Rice to work for TI. Indeed, TI probably assisted Smalley more because they hoped to recruit his graduate students than because they thought his research findings would help them manufacture circuits (which they didn't).

With the (initially contested) discovery of  $C_{60}$ , Smalley's work on semiconductor clusters was put on a back burner, and has been forgotten in the popular and scholarly historiography. No wonder – Smalley's most-cited semiconductor cluster article has received less than 1% of the citations of the article announcing  $C_{60}$ . Yet that disparity was still somewhat contingent. By the late 1980s, Smalley was satisfied that he had overcome all possible objections to the  $C_{60}$  model he, Kroto, and Curl had proposed, but he still could not make enough  $C_{60}$  to analyze using bulk characterization tools. The amount that could be learned about  $C_{60}$  seemed to be disappointingly constrained.

So Smalley began to wind down his fullerene work and returned to the semiconductor cluster research he had been working on before  $C_{60}$ . As the title of a talk by a Smalley student in 1989 put it (perhaps in deliberate contrast to  $C_{60}$  research), "Silicon Is Never Boring".<sup>2</sup> In the early 1990s, however, three discoveries convinced Smalley to return to carbon-cage materials (generically known as "fullerenes"). First, in 1990 Donald Huffman and Wolfgang Krätschmer discovered an astonishingly simple process for making larger quantities of buckyballs. Simply by running an electric arc across two graphite rods in a helium atmosphere at reduced pressure, they could make enough  $C_{60}$  to analyze with an infrared spectrometer. Suddenly, a lot more became known about buckyballs very quickly. Smalley (1991) referred to this as " $C_{60}$ , Chapter 2".

Chapter 2, as it turned out, moved quickly away from buckyballs and toward carbon nanotubes. Both were closed cages of pure carbon, but

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<sup>2</sup> Poster for RQI Informal Seminar/Discussion Series talk by Mike Alford (1989), "Silicon Is Never Boring: Some New Results of Silicon Cluster Ion Reactivity", September 22, Rice Quantum Institute information file, Woodson Research Center, Fondren Library, Rice University.

Smalley could foresee much more interesting electrical and mechanical properties for the elongated nanotubes than for their spherical cousins. The year 1991 saw the first production of macroscopic quantities of multiwalled carbon nanotubes. Then in 1993 came the discovery of single-walled nanotubes (or SWNTs) – notably, by groups at microelectronics giants NEC and IBM. Smalley dubbed single-walled tubes “the world’s most perfect material” and dedicated the rest of his career to making, understanding, and applying them.

But perfect for what? Most scholarly attention to Smalley has focused on the rhetorical connections he spun from nanotube research to futuristic applications of nanotechnology such as Eric Drexler’s “molecular assemblers” or a “space elevator” lifting people and goods from earth’s surface to geosynchronous orbit. Indeed, Smalley used both Drexler and the space elevator in his attempts to persuade federal policymakers and university administrators and donors to support nanotechnology research. Yet even a cursory glance at Smalley’s public and private writings shows that the microelectronics applications of nanotubes and other nanomaterials pervaded his outlook. Smalley used the social capital that he accrued from the buckyball discovery largely to persuade Rice to hire a cohort of physicists, chemists, and electrical engineers with expertise in exotic electronic materials such as quantum dots and so-called “molecular electronics”. In the wake of the discovery of nanotubes, he re-oriented his own research to figuring how to make large quantities of high-quality nanotubes for *any* application, yet he consistently maintained that it was the microelectronics applications of nanotubes which were most achievable and would be most profitable. Smalley was incredibly eager to form collaborations with groups both inside and outside Rice to develop microelectronics applications of nanotubes, to the point of submitting a proposal to the NSF in 1998 for a multi-sited Center for Carbon Nanotechnology with “nanoelectronics” as its top objective.

By 1998, Smalley was also preparing to found a company to manufacture tubes. In anticipation of that move, he spoke with *Business Week* about nanotubes’ commercial potential, focusing almost exclusively on their microelectronics applications:

Q: What makes buckytubes compelling to industry?

A: Take a look at the preface and introductory sections of Sematech’s *National Technology Road Map for the Semiconductor Industry*, 1997.... The notion that they will have to leave silicon was discussed in depth. They see so many problems on the horizon that they can’t get around. So now they are ready to think about things like carbon.

Q: That’s a big departure.



A: Yes. And this gets back to the old dreams of “molecular electronics”.... There is a huge electronics industry, well in excess of \$200 billion a year, with a great desire to maintain Moore’s Law for another 50 years. It’s likely that tens of billions of dollars will be spent on breaking the 100-nanometer barrier. And the only thing on the other side of that barrier is molecular electronics.... In the 1970s, there was much discussion of molecular electronics, but nothing came of it, mostly because people didn’t have good molecular metallic wires. But now it looks like we do, and the name is “buckytube”. (Anonymous, 1998)

Nor was Smalley alone in predicting that nanotubes would soon lead to the overthrow of silicon by molecular electronics – IBM and other big companies were also pursuing that goal, as were many academic researchers.

In fact, the main customers for Smalley’s company, Carbon Nanotechnologies Inc., were microelectronics firms. Samsung (Anonymous, 2008), for instance, repeatedly said it was close to marketing a display system incorporating nanotube emitters (some bought from CNI). Mobile phone makers, too, reportedly experimented with using nanotubes as additives in their glass touch screens (Hecht, 2009). In 2007, CNI merged with another firm (Unidym), which was then sold in 2011 to another Korean electronics company (Wisepower), “a leading supplier of Li-polymer batteries for mobile appliances” (Anonymous, 2011). Today, Unidym’s corporate tag-line is “carbon for electronics”. Its one market success seems to have come from Entegris, a maker of the trays on which silicon wafers are carried in semiconductor fabs – an application where even the tiny decrease in dust and flaking caused by incorporating nanotubes into the tray’s plastic matrix could justify the enormous expense of using one of the world’s most exotic materials (Anonymous, 2014). That is, the microelectronics industry is so vast and depends on so many different high-performance technologies, that any time a new wonder material such as the carbon nanotube is invented, the microelectronics industry can probably find multiple ways of using it.

The Smalley-fullerene case gives a good sense of how academic chemists, materials scientists, and allied researchers increasingly spin their work as relevant to – perhaps even as a panacea for – the microelectronics industry, to great effect. At every step, no matter what experimental material or apparatus or research institution he was working with, Smalley could successfully siphon resources from firms, donors, and government agencies by constructing a plausible narrative about how that apparatus, material, and/or institution was going to extend Moore’s Law and revolutionize electronics. But Smalley’s case also points to the parallelism and hybridity of

research on electronic materials. He helped discover  $C_{60}$  in the midst of work on semiconductor clusters – work he returned to when  $C_{60}$  research stagnated, then abandoned with the nanotube boom of the early '90s. Yet he used that boom to bring resources not just to nanotube microelectronics research but also to people studying other electronic materials such as quinones and quantum dots.

As Sacha Loeve's chapter explains, the fullerene research community Smalley helped found has continued to spit out new candidates for electronic wonder-material, most recently graphene (which yielded a Nobel Prize in 2010). And yet, all that activity has displaced silicon only a little bit symbolically, and not at all commercially. In fact, silicon is indispensable to research on graphene and molecular electronics, whether as the substrate on which those alternative materials rest, or as the material for which nanolithographic methods were first developed but which are now applied in graphene and molecular electronics research. Moreover, as the Unidym example shows, even when silicon outcompetes an alternative material such as carbon nanotubes, that material may still find an application in the larger ecology of materials used in the manufacture of silicon integrated circuits. The relationship between bench and brand is complicated indeed.

## Conclusion

What I've tried to get across in these two case studies is some sense of how the task of considering alternative materials for microelectronics was re-organized in the late Cold War and post-Cold War periods. During the Cold War, firms invented their own alternatives to silicon, observed alternatives to silicon that originated in competing firms, and pursued parallel lines of research in-house to aid in the process of strategic reflection as to whether to abandon silicon in favor of something else. Academic research on alternatives to silicon existed, of course, but firms prized that research primarily as a source of personnel who could be recruited into corporate labs, rather than as a source of *knowledge* that they could directly put to use.

Curiously, though, academic training was often only indirectly relevant to the future careers of corporate researchers. Many of the people who participated in the IBM Josephson program had virtually no experience with superconductivity – or even microelectronics – before joining IBM. If they did have a background in microelectronics, it was more likely to be in semiconductors than superconductors. Even previous corporate experience was poorly predictive of an individual's future industrial research. People like Richard Garwin bounced merrily from superconducting electronics to

meson decay to gravitational radiation research (Collins, 2010) to ballistic missile defense (Slayton, 2013). A few of the Josephson project personnel stayed in superconductivity, but most floated into other areas, often tacking among basic research, applied research, and technology management for the rest of their careers.

By the 1980s, that style of work was becoming more common in academia as well (Mowery *et al.*, 2004). Particular end-products (“brands”) became compelling imaginaries to motivate a wide variety of activity at the academic bench. The ultrafast computer, in particular, was an imaginary that stimulated bench-work on a tremendous range of materials. Increasingly, that imaginary shaped the work of individual researchers more than their expertise in any given material. Rick Smalley, for instance, moved easily from semiconductor nanoclusters to buckyballs to nanotubes. The importation of this industrial template for shaping the identity of the academic scientist has been a source of vociferous protest by some historians, philosophers, and even scientists themselves (e.g., Forman, 2007). Yet it is hard to see how, in our current moment, it could be otherwise (Mirowski, 2011). The increasingly competitive post-Cold War global economy has led many firms in the US and elsewhere to believe that they cannot sustain *any* activity that spans all the way from bench to brand, much less the Cold War model of an in-house R&D portfolio consisting of multiple arcs leading from bench toward brand *and back* at the same time.

Pathways from bench to brand within industry are now supposed to be short, singular, and have a high probability of success. Research that may or may not lead to a product over a long time horizon has therefore been increasingly outsourced to universities and to industrial research consortia. Meanwhile, research within academia is increasingly supposed to delineate a plausible path from bench to brand. When that pathway becomes *very* plausible, academic researchers such as Smalley increasingly take it upon themselves to patent their work (and license the patent to someone who will turn it into a brand), and/or found their own company to shepherd their ideas to the marketplace themselves. Whether this increasing emphasis on “translation” from bench to brand (and decreasing tolerance for moves from brand to bench) will prove a sustainable innovation model over the longer term is very much unclear.

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