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

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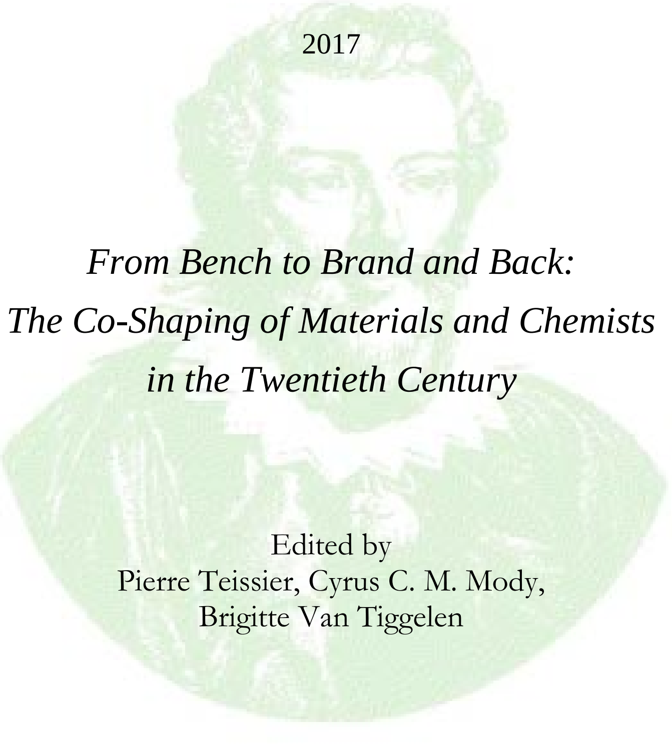

From Bench to Brand and Back:
The Co-Shaping of Materials and Chemists
in the Twentieth Century

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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, différentiation 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)[1] 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. Leydersdoff (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 expanse 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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[1] 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 expanse of local and global aspects.[2]

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 *disciplinarities* 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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[2] 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.[3] 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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[3] 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 expanse 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 "perceived 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.[4] 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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[4] 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"[5] was made of three elements: high temperature synthesis, making of bulk crystals, and structural analysis by X-ray diffraction. French and German chemists agreed.[6] 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 Wilhem 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).[7]

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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[5] 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*.

[6] 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).

[7] 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".[8] 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.[9] 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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[8] 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.

[9] 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.[10] 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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[10] "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: MMo_NS_{N+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.[11] 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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[11] 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[12] 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 Rannou, 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

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[12] 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 ($Zr - UF_4 - ZrF_4$) were combined in different proportions to form unexpected products: $UZrF_7$ and UZr_2F_{11} (Fonteneau & Lucas, 1974). Secondly, in a well-known compound like fluorozirconate ($MZrF_6$), 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 ($NdZrF_7$) 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 colorless solid instead of the usual smaller and darker compounds. When analyzed by X-ray diffraction, only rays of neodymium fluoride (NdF_3) appeared, which suggested that this reagent did not react. The lack of other signals suggested that the three other reagents (ZrF_4, BaF_2, 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 (BeF_2, AlF_3). They performed more systematic syntheses and drew a ternary diagram (ZrF_4 – BaF_2 – 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.

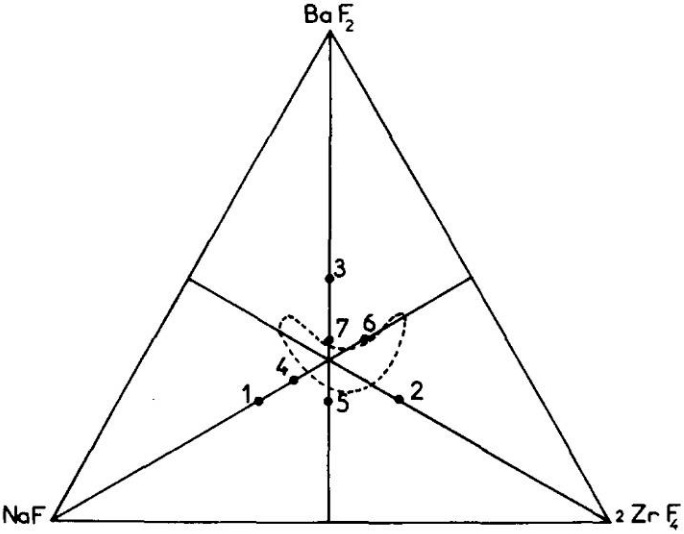

Figure 2 - Ternary diagram of ZrF_4 – BaF_2 – NaF and amorphous domain (Poulain et al., 1975)

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 opportunities. 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.[13] Fiber optics increased the information flow by comparison with electrical wires traditionally used for telegraph and telephone systems.[14] They could also reduce the

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[13] 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).

[14] 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.[15] 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.[16] It was also ignored by the solid-state

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[15] The attenuation was due to "extrinsic" impurities in the core fiber as well as "intrinsic" vibrations of the silicon-oxygen bond in silica (SiO_2), which forbade transmission longer than 80 kilometers without amplification.

[16] 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.[17] 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.[18] 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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[17] 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).

[18] 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.[19] 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.[20] 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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[19] The multiplication of characterization apparatus in chemistry laboratories was characteristic of the "instrumental revolution" in chemistry during the twentieth century (Morris, 2002).

[20] For example, the Poulains (2015, p. 6-9) added 3 to 4% of aluminum to increase the stability of fluorozironate 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.[21] 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.[22] 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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[21] 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.

[22] 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.[23] 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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[23] 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.[24] The case study shows how the

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[24] 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.[25] 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.[26] 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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[25] 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).

[26] 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.[27] 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 *NGOS 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.[28]

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.[29] 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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[27] *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.

[28] 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).

[29] 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).

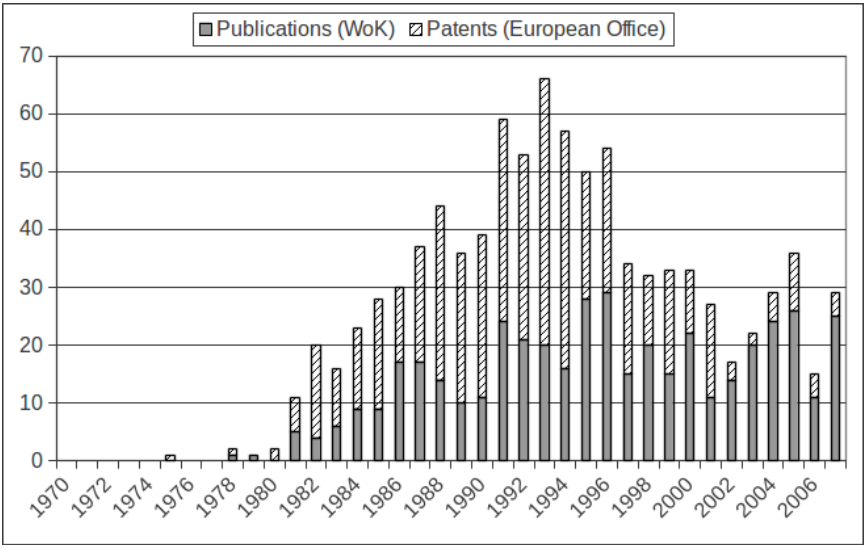

Figure 3 - Quantitative evolution of worldwide publications and patents on fluoride glasses

- *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 $53ZrF_4 – 20BaF_2 – 4LaF_3 – 3AlF_3 – 20NaF$. ZBLAN was strikingly close to the chemical composition of one glass published in the seminal paper of Poulain (*et al.*, 1975): $50ZrF_4 – 20BaF_2 – 5NdF_3 – 25NaF$. 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 expanse 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 thesilica 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.[30] 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.[31] The reading of old-fashioned

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[30] 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.

[31] 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.[32] 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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[32] "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 (Chevrel'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 national 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 characterization 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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[33] 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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