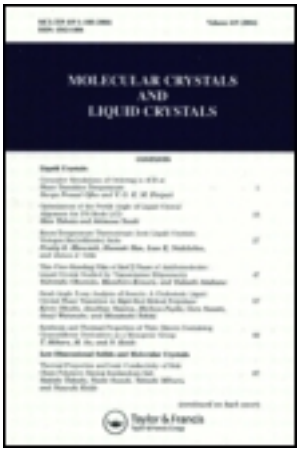

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50 Years Liquid Crystal Research-A Personal Retrospective

Dietrich Demus [a] [b]

[a] International Scientific Consulting Office, Halle, Germany

[b] Chisso Petrochemical Corporation, Ichihara, Chiba, Japan

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50 Years Liquid Crystal Research-A Personal Retrospective

DIETRICH DEMUS[1,2]

[1]International Scientific Consulting Office, Halle, Germany
[2]Chisso Petrochemical Corporation, Ichihara, Chiba, Japan

The reporter has worked for 50 years in LC research. In 1960 only few specialists in several countries treated this topic. In Halle University since that time the system of classification of LC phases has been elaborated, and after the invention of LC displays new materials for practical application have been developed. Success and problems of this period are discussed, which ended in 1991 after unification of Germany. Since 1992 the author had a stay of more than 2 years in Japan as special scientist in Chisso Corporation, followed by a permanent activity as Scientific Consultant for this company.

Keywords Chisso Corporation; Halle University; history; liquid crystal research

1. Introduction

This invited lecture should have occurred 28 years ago. In 1982 the ILCC was to be organized in Krakow, with Prof. Jerzy Janik as chairman. The elder people of this audience will remember that in this time the military regime in Poland was installed, the planned conference could not take place and was shifted to Bangalore in India. Our liquid crystal group from Halle University had since the beginning of the seventies a cooperation agreement with the Krakow liquid crystal group including regular exchange of several scientists, and there was close cooperation using the mutual instrumental facilities and materials. There was not only scientific cooperation, but many of the involved persons became friends. In this sense it is a great pleasure for me to be invited by my Polish friends, especially chairman Prof. S. Urban, to perform this speech and to meet them again.

2. The Situation in the Sixties

About 50 years ago, exactly in the beginning of January 1960 I started my career in liquid crystal research [1,2]. I was a bachelor of chemistry and began to work as doctoral student and scientific assistant in the Institute of Physical Chemistry

This paper has not been published before and it has not been sent simultaneously for publication elsewhere.

Address correspondence to Dietrich Demus, International Scientific Consulting Office, Veilchenweg 22, D-06118, Halle, Germany. Tel.: +49-345-5232697; E-mail: dietrich.demus@t-online.de

Figure 1. The old chemical institute of the Martin-Luther-University Halle about 1985. (Figure appears in color online.)

at the Martin-Luther-University (Halle) in the group of Prof. Horst Sackmann. In the first weeks I was not allowed to do experimental work about liquid crystals, because the director of our Institute (Figure 1), Prof. Franz Sauerwald, urged me to do measurements of the contents of mercury in the air of the rooms of our Institute. We worked in an old building, stemming from the 19th century, and generations of chemists had used mercury for their experiments and sometimes accidentally spread out this metal in the rooms. I found remarkable amounts in the air, and as a result the floor covering in some rooms was replaced.

The University Halle had an old tradition in liquid crystal research. Around 1900 Daniel Vorländer started exactly in our old building his very successful synthetic work and we can say he was the founder of liquid crystal chemistry [3,4]. Most of his more than 2000 compounds were still preserved in the collection of the chemical institute, and some of his materials formed the basis for the beginning of our own research. After Heinrich Arnold in Sackmann's group had started the liquid crystal research by doing miscibility studies [5], I was the second doctoral student in this field. My task was to include as many compounds of different chemical structure as possible in order to check how many types of liquid crystalline phases exist, using the now well known miscibility criterion.

50 years ago two hypotheses existed concerning this matter: According to the famous work of Friedel [6,7] there were nematic, smectic and cholesteric phases. In contrast to this opinion, Vorländer insisted in his finding of several smectic phases in certain compounds, results which had been confirmed by Herrmann [9], Bernal and Crowfoot [10], Weygand [11] and after the second world war by the results of George Gray [12]. Compounds with up to 3 smectic phases, now known as smectic A, B, C, have been found. Step by step the number of identified phase types increased, in 1970 we knew 7 types, and including discotic, amphiphilic, polymeric and bent shaped compounds the actual number is difficult to be defined, may be one order of magnitude higher.

Figure 2. Original liquid crystal compounds from Vorländer. (Figure appears in color online.)

About 1960 liquid crystal research was an absolutely exotic topic. There have been very few research groups in the world:

Canada – D. P. Benton, I. E. Puddington University of Ottawa (investigation of salts);

France – V. Luzzati, A. Skoulios, University of Strasbourg (polymorphism and structures of salts)
Pierre Chatelain, R. Cano, M. Germain, J. Falgueirettes University Montpellier (optical properties, orientation of nematics by rubbing);

GDR – H. Lippmann, K. A. Weber University of Leipzig (NMR investigations)
H. Sackmann, University of Halle
Heinz Dunken, University of Jena (salts in lyotropic systems);

F. R. Germany – Wilhelm Maier, Alfred Saupe, Gerhard Meier, University Freiburg (physical properties and respective theories);

Great Britain – G. W. Gray since about 1950 synthesized liquid crystals. In 1960 he was Senior Lecturer in the University of Hull. He mentioned having had strong difficulties to become a professor which occurred not earlier than 1978, because the liquid crystals had not been accepted as honest scientific topic by his colleagues. In 1962 Gray published his important book "*Molecular Structure and Properties of Liquid Crystals*", the first modern book about liquid crystals [12];

India – J. S. Dave, J. M. Lohar, University of Baroda (miscibility investigations);

Poland – J. A. Janik, Jagiellonian University, Krakow (neutron scattering);

USA – Edward F. Carr, University of Maine, Orono (dielectric properties),
Kent State University, G. H. Brown. In cooperation with W. G. Shaw in 1957 he published the very influential paper *The mesomorphic state. Liquid Crystals* [13];

USSR V. Tsetkov, University Leningrad (dielectric and optical properties)
I. G. Chistyakov since 1961 at Ivanovo State University (he now is considered to be a pioneer of liquid crystal research in Russia).

This list may be not complete, but contains the scientists who worked with liquid crystals for a longer period.

Nobody believed in any application of liquid crystals, the situation was still as 1924 when Vorländer said: 'I was asked if liquid crystals can be applied in technique. I do not see any possibility for that' [14]. The number of publications about liquid crystals which appeared at that time was about up to 30 per year, in the world, not in our group (which in fact occurred later). In the last years it was about 9400 [15].

Quite secretly in the beginning of the sixties in the American company RCA several scientists (G. Heilmeier, J. Goldmacher, R. Williams, L. Zannoni, J. A. Castellano) investigated the interaction of liquid crystals with electrical fields, with respect to possible applications. The story of this American research is reported in much detail in the nice book 'Liquid Gold' in 2005 by Joseph A. Castellano [16]. The first effect they described in detail, the so called dynamic scattering effect, had already been found in 1918 in Uppsala (Sweden) by Yngve Björnstahl [17], but because it was not scientifically interesting enough and applications as far as the moon, his results became completely forgotten. This is another example for the fact, that inventions made too early are not recognized at all. The Americans started publications proposing LCs for use in displays in 1968 [18]. This was the starting shot for a race in the world which became broader, quicker and more and more successful.

After I had finished my PhD in 1963 I should not have had a chance to remain in the university because by the officials I had been considered to be politically unreliable. Just in this time, I had been already dismissed, a polymer producing company came to our institute asking for help in urgent problems of application. Since nobody else was willing to work about this matter, it was offered to me and I worked about it 6 years, finally elaborating my habilitation work from the results. Because the results were considered to be important for the industry, I did not obtain the permission for publication [19]. I may mention a fact which now looks like a curiosity. Within my work I had to perform a great number of numerical calculations. Of course, in this time electronic calculators had not been available. Simple mechanical calculation machines existed but were quite clumsy and time consuming, another choice would have been the extremely time consuming logarithmic tables. So I was forced to use as a quick tool the slide rule, which for younger people may be completely unknown. During these calculations I lost a substantial amount of accuracy of the data obtained by the experimental measurements, but I have been able to finish the work within a limited period.

In addition to this work I never stopped LC research and I have been supervisor of several diploma and PhD students.

3. Applied Research in Halle

In 1969 in our institute we saw the visit of officials of the "VEB Werk für Fernsehelektronik" Berlin. They had read the papers of RCA and wanted to know our opinion and to have support in providing liquid crystal materials, in order to

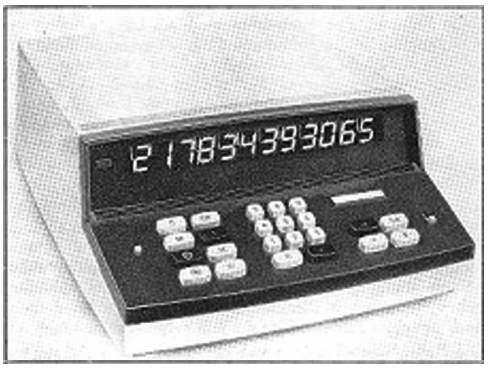


Figure 3. Exhibit at the Leipzig Fair 1972: Electronic calculator with liquid crystal display.

make experiments about the matter. A research group was established under contract with this company and I became the leader, because Prof. Sackmann was not interested in applied research and he preferred to continue his basic investigations. It was a quite hard time for me, because just when I had to start the organization of the new research group I had to finish the manuscript for a general textbook of chemistry, in which I was responsible for the part physical chemistry [20]. In editing this book a curious story happened. In 1969/70 in the GDR a reform of the academic education was running. In the frame of this reform the natural sciences should be included in the general Marxian-Leninist system by referring to respective publications. I was called to a commission in the city of Leipzig, that criticised my text and demanded to include references of Marx, Engels, Lenin, Stalin etc. I was not in the situation openly to resist this request, but in fact I did nothing. Some years later it was a great surprise for me, when the Chief reader of the publishing company thanked me for doing nothing. Asking him why he thanked me, he said that the West German publisher Verlag Chemie just signed an agreement for a license edition [19a], which yielded urgently needed 'hard currency' for the publisher and would not have happened with a text containing Marxian references.

Our work about application of liquid crystals within few months was successful, because by having reinvestigated hundreds of Vorländer's compounds in advance, I had a lot of experience concerning the relations of chemical nature and properties of compounds – this is transition temperatures, chemical and thermal stability.

And from the beginning it was clear that the problem of room temperature materials could be solved only with mixtures [21], with which we had already a lot of experience within Sackmann's research and special investigations. We developed a series of room temperature mixtures [22]. Soon we were able to establish a close cooperation with Hermann Schubert's group of synthetic chemists in our university, who started work about liquid crystals in the beginning of the sixties. Later Professor Horst Zaschke became the leader of the group responsible for the synthesis of applied liquid crystals. Our cooperation with the industrial partner was nice in so far, as we had to deliver every year a fixed quota of patent applications and samples of materials, and having fulfilled the quota we had freedom for additional research and there was much less bureaucracy than in the actual situation in Germany.

In 1973 I have been very busy with a lot of duties in the university and industry, and it was difficult to find time to finish the first volume of our table book "Flüssige

Kristalle in Tabellen''. Fortunately I caught the convenient illness – mumps together with both of my children-which allowed me to work at home several weeks without being disturbed, because of danger of infection I was not permitted to go into the Institute. This was the situation as our former director of the Institute used to say: 'Nothing is so bad that it does not include something good'.

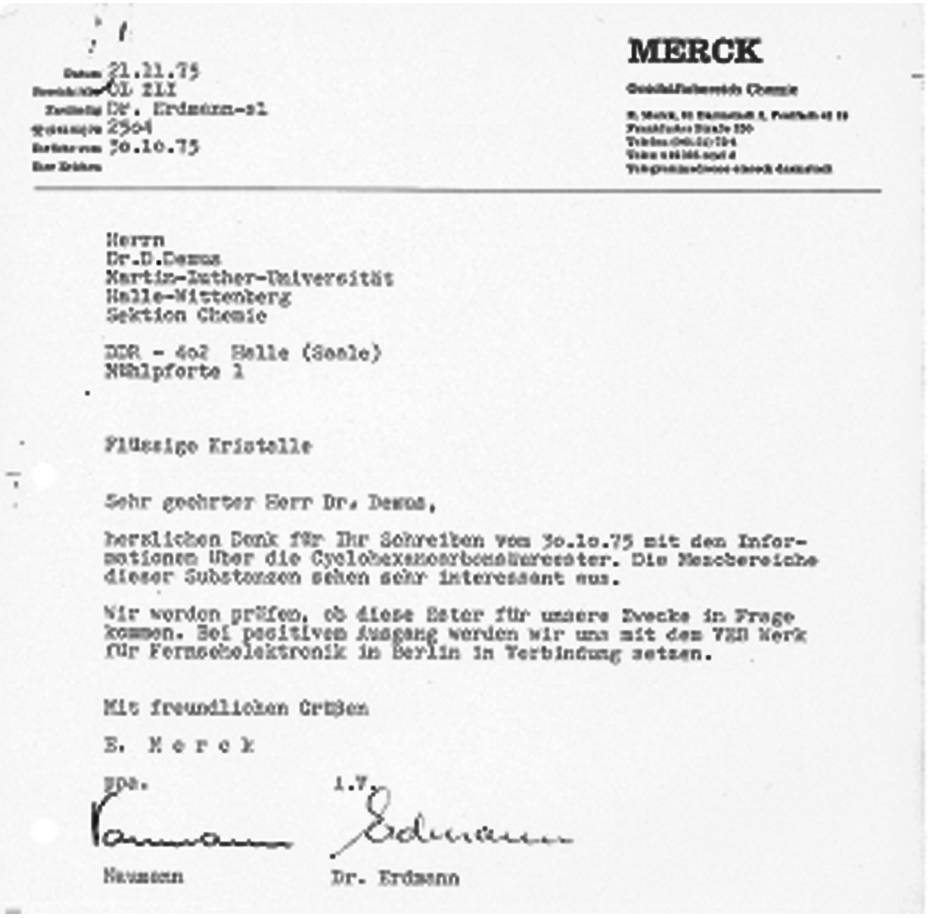
In 1972 our exhibit common with the VEB Werk für Fermsehelektronik Berlin at the International Leipzig Fair was honoured with a gold medal – our display using esters was much more stable than products of competitors, who used Schiff's bases and had secretly to exchange their displays every evening because of deterioration. In long term experiments we found the mixtures unchanged even after 8 years treatment with direct current, in this period only the electrodes made from ITO became dark.

Our R&D work started on the basis of the dynamic scattering mode (DSM), which was internationally up-to-date until 1971. Unfortunately our industrial partner was stick to this effect even after the twisted nematic effect was invented in 1971 by Schadt and Helfrich [24]. This effect needed much lower voltage than the DSM. The government of GDR in 1973 made a contract with the Soviet Union concerning the delivery of DSM displays with a working voltage of 10 V. In reality we were unable to deliver such displays, because the threshold voltage of DSM is about 7 V and reasonable contrast needs about 15 V. A great disaster followed, our contract with the industry was cancelled, the industry abandoned liquid crystal R&D completely and recommended the same for us. Fortunately the director of research of our University was wise and clever enough not to follow this recommendation, and in fact the VEB Werk für Fernsehelektronik Berlin one year later restarted liquid crystal R&D, now on the basis of twisted nematic displays, and signed a new contract with us.

Having felt the bad experience of the former contract with the Soviet Union, our industrial partner became very cautious. All instructions for making mixtures had to be elaborated in much detail. I remember that our partner criticized that we did not indicate how the materials in order to prepare mixtures had to be stirred – in left sense or right sense. At that time we laughed about this criticism, which in fact was unreasonable. But you never should underestimate the scientific progress. Quite recently I found a paper describing substantial differences in the optical properties of mixtures – depending on stirring in left or right sense [25].

A breakthrough in our international relations has been a conference organized in 1975 by the Brown Boveri company in Baden, Switzerland. In this conference for the first time I reported about the cyclohexanoates we had developed some years ago [26]. After my lecture I had a long talk with Dr. Erdmann from the company E. Merck/Darmstadt, who was highly interested to hear more about the introduction of cyclohexane in the LC chemistry. As a sequel we granted licenses to companies in several countries, and companies like Merck, Chisso, Hoffmann-la Roche and others started the systematic development of cyclohexane derivatives [27].

In the following years Hermann Schubert and his co-workers from our University introduced a large variety of heterocyclic rings in the LC chemistry [29], Gray had developed his famous low melting cyano-biphenyl derivatives [30], polymeric LC materials were synthesized in several groups and the physics of liquid crystals had been investigated experimentally and theoretically in several places in academic institutions and the industry with great success, with a leading position of the Orsay group under P. G. de Gennes, who in 1991 was honoured for his liquid crystal research with the Nobel Price. The number of publications about liquid crystals



MERCK

Datum 21.11.75
OL ZII
Dr. Erdmann-sl
2504
30.10.75

Herrn
Dr.D.Demus
Martin-Luther-Universität
Halle-Wittenberg
Sektion Chemie

DDR - 402 Halle (Saale)
Mühlpforte 1

Flüssige Kristalle

Sehr geehrter Herr Dr. Demus,

herzlichen Dank für Ihr Schreiben vom 30.10.75 mit den Informationen über die Cyclohexancarbonsäureester. Die Meßbereiche dieser Substanzen sehen sehr interessant aus.

Wir werden prüfen, ob diese Ester für unsere Zwecke in Frage kommen. Bei positivem Ausgang werden wir uns mit dem VEB Werk für Fernsehelektronik in Berlin in Verbindung setzen.

Mit freundlichen Grüßen

E. Merck

ppa. i.V.

Naumann Dr. Erdmann

Figure 4. Letter from Dr. Erdmann (Merck) to Dr. Demus.

increased to about 2000 per year. It was at that time that several leading researchers believed that the liquid crystal science basically was clear, and in future only routine work in synthesis and mechanical measurement of data could be done and it would be time to switch to other topics. As I already mentioned, never underestimate the scientific development. At the end of the seventies the discotic liquid crystals have been detected, unconventional liquid crystals with branched molecules, twins and oligomers, polymers, ferroelectric and antiferroelectric materials were developed and investigated, and there was remarkable progress in the field of amphiphilic materials.

In the application of liquid crystals large progress occurred, especially in the development of larger and more complicated displays on the basis of new effects like STN and ferroelectrics, cholesteric materials have been used for thermography in the medicine.

In the University Halle we did not work without success, but we experienced a lot of difficulties. Especially, because of the lack of so called 'hard currency', we were not able to buy necessary equipments and chemicals, especially chemicals for metal organic syntheses. The situation demanded that I should publish new books, because

there was no money for buying books from the international market, but own books have been a valuable currency in obtaining books from foreign authors in natural exchange. Another affair occurred when I travelled to the Raman Institute in Bangalore in 1978. I did not obtain any foreign currency in GDR in advance. The officials said I should obtain money in Bangalore. But there was no money, and no official available whom I could ask for help. I just had finished my book "Textures of Liquid Crystals" and I had a copy with me. By the good offices of Prof. S. Chandrasekhar I transferred the book to the Institute's library, in exchange for free food and some rupees.

I remember that once in our institute we only had money for repairs but not for buying new furniture, but we urgently needed a new table. Then our very clever joiner proposed, please bring me only one leg of any table and ask for repair, then I shall construct a new one according to your wishes. This philosophy then was applied more often to solve different problems.

In about 1987 the roof of our institute was defect and the rooms became wet. The appropriate craftsmen stated that the whole wood construction was out of repair and should be completely renewed, but for this purpose neither money nor craftsmen and material were available. The Rector of our University prohibited everybody to enter the roof because of danger of life. This was a really critical situation, in which I discussed with my colleague Prof. Frank Kuschel who was director of the Institute in this time. We came to the conclusion that only professors could resist the prohibition of the Rector. A major problem for this purpose was: we needed quite a lot of wood, but wood in this quantity was not available on the market. We were able to overcome this problem by working actions of the staff of the Institute in a forest. The forester as compensation delivered the necessary wood and the professors were able to make the repairs. I have checked some time ago that our repair still was stable. But quite recently the old building has been abandoned by the University and the Institute now is in a modern completely renovated house in the actual university campus.

Figure 5. Working action of the staff of the Institute of Physical Chemistry in the forest.

Figure 6. Profs. Kuschel and Demus working on the roof of the Institute of Physical Chemistry.

In the Halle University the research groups for applications of liquid crystals with increasing importance of these materials for application in displays became larger and larger and finally comprised about 50 people. Liquid crystals in the GDR were part of a strategic state program of microelectronics, which were like a toy for the country's leader Erich Honecker. The program was controlled by several ministries in Berlin, among them the secret service of GDR STASI, which was formally a ministry. As I know from documents (about 250 pages) of this ministry now laid open, I have been observed systematically for a long period. In 1985 we performed investigations about ferroelectric liquid crystals, which were considered by

Figure 7. Visit of Prof. S. T. Lagerwall in the Chisso Research Center Goi. (Figure appears in color online.)

Figure 8. ILCC 2000 in Sendai: Drumming during the banquet with Prof. G. W. Gray. (Figure appears in color online.)

Rangfolgen der zitierten Autoren (RAU) und zitierten Erfinder (RIN)

Recherche: Flüssigkristalle / liquid crystal, STN-Datenbank: SCISEARCH, Nachweise:18.688 (3/94), 28.401 (3/98)

1994		1998	
RAU	RIN	RAU	RIN
DEGENNES P G	CALUNDANN G W	DEGENNES P G	CALUNDANN G
GRAY G W	KWOLEK S L	GRAY G W	KWOLEK S L
PERCEC V	SCHAEFGEN J R	PERCEC V	JACKSON W J
FINKELMANN H	**DEMUS D**	CHANDRASEKHAR	SCHAEFGEN J
DEMUS D	JACKSON W J	MAIER W	FERGASON J L
CHANDRASEKHAR	FERGASON J L	FLORY P J	**DEMUS D**
FLORY P J	KUHFUSS H F	CLARK N A	KUHFUSS H F
CLARK N A	COTTIS S G	MEYER R B	COGSWELL F N
MAIER W	COGSWELL F N	FINKELMANN H	ISAYEV A I
	BLADES H	**DEMUS D**	COTTIS S G
	KLEINSCHUSTER	GOODBY J W	KLEINSCHUSTER

Ergebnis

Wenn wir als Top-Techniker jene Wissenschaftler bezeichnen, die besonders häufig sowohl als Autoren als auch als Erfinder zitiert werden, dann ist auf dem Gebiet der Flüssigkristall-Forschung D. Demus der Top-Techniker, auch wenn sein Rang nach 1994 sinkt.

patentanalyse-einf-2010-beispiel Prof. Reinhard Schramm - Adam Bartkowski - TU Ilmenau / PATON Seite 12 von 28

Figure 9. Result of the analysis of the German Patent Office.

the army of the GDR (Nationale Volksarmee) to be important for their purposes. Research programs connected with the army have been subject to a high level of secrecy. This was the reason why finally I was urged directly to report to representatives of this ministry about progress and difficulties in research and development [31], a fact that after the unification of the two German States in 1991 led to my summary dismissal from the University and cancelling of my prepared position as a director of a research group of the Max-Planck Society, a position which then was taken over by Alfred Saupe. After Germany's unification 76% of the academic teachers of Halle University have been dismissed [32a], a number typical for the changes in all Eastern German scientific institutions at that time [32b].

In a climate of witch-hunting at that time it was impossible for me to find a position in any academic institution in Germany, and despite former good relations to several companies also the industry refused to grant an employment [33]. In contrast to the communist regime in the GDR which in spite of political reserve (e.g., I never have been a member of a party and I always have been politically suspect because two of my brothers were living in West Germany) did not stop my scientific work, under the new conditions in the united Germany political burdens became much more important than scientific quality [35]. In order to continue scientific work [36] I was forced to go abroad.

4. Working with Chisso Corporation

Since about 1978 Dr. Inukai from Chisso corporation Tokyo regularly visited us in the University Halle and Chisso acquired several licenses for liquid crystal materials developed in Halle. As a sequel long lasting and good relations to Chisso Corporation had been established. Since 1992 I had the opportunity to work several years in Japan in the Research Center of Chisso Corporation as special scientific adviser and lecturer, and since my return to Germany I continue my work as an external scientific adviser for the same company. I am highly indebted to the colleagues of Chisso, especially Dr. T. Inukai, Dr. R. Tarao, Dr. Y. Gotoh and Dr. S. Inaba, for their warm friendship, and their help and patience by introducing me and my wife into the Japanese style of life. I became a member of the Japanese scientific community, often I have been invited for cooperation or to present lectures. Curiously probably I have been the only person, who was within the International Planning and Steering committee for the International LC Conferences resp. the ILCS representative for 3 countries – first GDR, then Germany and finally Japan.

5. Conclusion

Summarising, I must say, it is fascinating having been involved 50 years in a scientific topic which developed from a scientific curiosity to a topic acknowledged by a broad international scientific community and in application evolved to the leading technology in producing displays. When in about 1970 our director of research said, with liquid crystals we shall construct flat displays hanging on the wall, we said yes, we shall do, but all people laughed and in fact nobody really believed in it. And now LCDs are a market of billions of dollars. It is a seldom privilege to be involved in such a gigantic development, and despite of all difficulties I experienced in my life I am grateful for this privilege.

Acknowledgment

I am indebted to Prof. Frank Kuschel for his kindly contributing some photographs and for critically reading the manuscript.

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[29] A short overview is presented in ref. 3 page 46.
[30] Gray, G. W., Harrison, K. J., & Nash, J. A. (1973). *Electron. Lett.*, *9*, 139.
[31] A report about this matter, prepared by the historian Schrödter, E., can be found in the book *Beiträge zur Geschichte der Martin-Luther-Universität 1502–2002*, ed. by H.-J. Rupieper, Mitteldeutscher Verlag: Halle 2002, pp. 658–660. This report shows that Demus's connections with the Stasi have not been devoted to spying on colleagues or destroying careers of them, as in Germany automatically is alleged, but it shows that it is related to the research on liquid crystals. On p. 658 of his article Schrödter states: 'The preserved reports ... prove the efforts to improve the difficult boundary conditions for the research. This mainly concerns scientific relations to Western partners and the latent shortage of technical equipments and chemical basic materials.' This report of a historian is a seldom case. At present by a respective law persons concerned are not permitted to see the documents related to their own case. Therefore it is difficult to perform legal proceedings in order to clarify eventual accusations. By the side, the good working atmosphere in the Institute of Physical Chemistry Halle was famous, not at least due to the professors who guarded the institute against unpleasant political influences in the frame of their facilities.
[32] Hecht, A. *Die Wissenschaftselite Ostdeutschlands. Feindliche Übernahme oder Integration?*, Faber & Faber: Leipzig 2002, a) academic teachers except medical faculty see p. 52, b) the average of all dismissed academic teachers was 72%, see p. 59. Dismissals of academic teachers in Germany already had a sad tradition: in 1933–1936 by the National-Socialists in average 14.3% were dismissed [33]. According to Michael Grüttner (Spiegel Online Unispiegel 9. August 2010) after 1945 about 50% of the academic personnel was dismissed by the Allies, but most of them returned to their academic positions in the next years in the Western zones of Germany. In the Soviet zone the number of dismissals was higher; many of the dismissed persons went to Western Germany.
[33] Kuschel, F., & Ringsdorf, H. (2010). *Nachrichten aus der Chemie*, *58*, 759–763; Hartshorne, E. Y. (1937). *The German Universities and National Socialism*, George Allen & Unwin Ltd.: London, p. 95.
[34] The well known German historian Arnulf Baring, who still is frequently invited for public discussions in the leading German TV stations, in his book *Deutschland, was nun?* Siedler Verlag: Berlin 1991 p. 59 characterized the situation in East Germany as follows: 'The regime has nearly half a century the people made dwarfish, and their bringing up and education ruined ....if people now call themselves jurist or economist, educationalist, psychologist, sociologist, even physician or engineer, it does not matter: His knowledge is largely totally useless'. In this climate it was difficult to be acknowledged, despite positive judgement [35].
[35] The German Patent Office in an international study (http://www.paton.tu-ilmenau.de/pdf/vorlesung/lb4_analyse_beispiel.pdf) searched for the 'Leading Technician', a title which was granted according to the number of citations of scientific papers and patents. In the field of liquid crystals Dietrich Demus in 1994 and 1998 was identified as leading technician (Fig. 9).
[36] In 1991 in Halle I have been formally registered as jobless person in 'Early Retirement' (Vorruhestand). Most of the people in this situation did not find an employment again.