Appendix B. Site Reports—Europe

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Date visited: 17 October 1997

WTEC: R.W. Siegel (report author), D.M. Cox, H. Goronkin, E. Hu, C. Koch, J. Mendel, D.T. Shaw

Hosts:
Dr. Catherine Bréchignac, Director General of CNRS
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Dr. J.F. Baumard, Director, CNRS Materials Program and Laboratory of Ceramic Materials and Surface Treatments, Limoges
Dr. Christian Colliex, Director, CNRS Laboratory Aimé Cotton, Orsay
Dr. Henri Van Damme, CNRS, Centre de Recherche sur la Matire Divise (CRMD), Orléans
Dr. François Gautier, CNRS, Strasbourg
Prof. Jean-Pierre Jolivet, Université Pierre et Marie Curie, Paris
Dr. Jean-Paul Pouget, CNRS, Department of Physical and Mathematical Sciences
Dr. Jacques Prost, Director, Institute Curie, Paris
Dr. Albert Masson, CNRS Research Director

BACKGROUND

The panel spent the afternoon of 17 October from 13:30 to 17:00 at the headquarters of the Centre National de la Récherche Scientifique (CNRS) in Paris as guests of the CNRS Director General, Dr. Catherine Bréchignac. Dr. Bréchignac had the previous July taken over this important position after being Director of Physical and Mathematical Sciences at CNRS (the post now held by Dr. Jean-Paul Pouget) and Director of the CNRS Laboratory Aimé Cotton in Orsay. She graciously and generously assembled a group of nine senior leaders in the area of nanostructure science and technology to meet with the WTEC panel. The CNRS programs in the area of nanostructure science and technology are very extensive and range nationwide, from Lille to Marseille and from Rennes to Strasbourg, with the heaviest concentration being in the Paris area.

RESEARCH AND DEVELOPMENT HIGHLIGHTS

According to Dr. Bréchignac, who began with an overview of the CNRS activities, about 300 researchers in 40 physics laboratories and 200 researchers in 20 chemistry laboratories are involved in nanotechnology research with annual funding of about FFr. 18 million, much of which (~ FFr. 12 million) comes from the interdisciplinary Ultimatech Program within the CNRS. An additional FFr. 9 million is spent annually via contracts for nanotechnology research in about 45 laboratories paid on a 50%/50% basis by industry and the CNRS. These latter efforts appear to be particularly effective with regard to eventual technology transfer toward commercialization, in that this research is carried out within industrial surroundings, such as Thomson, St. Gobain, Rhône Poulenc, and Air Liquide. Particularly impressive was the degree of interaction and awareness apparent among the various participants in the French nanotechnology network, something clearly fostered to considerable advantage by the CNRS structure and its excellent management.

The CNRS research program ranges broadly across the full spectrum of nanotechnology. Synthesis of nanostructures using molecular beam, cluster deposition, and lithographic methods is being carried out, as are more classical chemical and electrochemical approaches. In addition, soft chemistry and biosynthesis efforts are underway. A wide range of scientific instrumentation for characterization and manipulation of nanostructures is being employed and/or developed. These include synchrotron radiation, near-field microscopies—scanning tunneling microscopy (STM) and atomic force microscopy (AFM)—and spectroscopies (STM, optical), magnetic and electron microscopy, and atom manipulation. The scientific and technological contexts of the CNRS nanotechnology research range over such diverse topics as nanomagnetism, molecular electronics, nanotubes, large-gap semiconductors, oxide layers, self-organization, catalysis, nanofilters, molecular sieves (zeolites), hybrid materials, therapy problems, and agrochemistry. Utilizing a roundtable format for the remainder of the discussions, the French participants described the activities in their own spheres of interest.

Research on Nanoparticles and Related Technologies

Dr. J.F. Baumard, Director of the Materials Program at CNRS and the Laboratory of Ceramic Materials and Surface Treatments (ENSCI) in Limoges, described the activities associated with the Department of Chemical Sciences at CNRS. The main research issues, under investigation at numerous university and industrial laboratories around France, are concerned with nanoparticles and related technologies:

- nanomaterials—a number of interfacial problem areas:
  - solid-gas interfaces and their relationship to adsorption and catalysis applications
  - solid-liquid interfaces in relation to dispersions, (soft) nanochemistry, and membranes
  - solid-solid interfaces and interphases in nanocomposites, more conventional composites, and hybrid materials
- nanosystems for molecular electronics and handling of species at the nanoscale level
- ceramic matrix nanocomposites containing intergranular metal nanoparticles and/or high-aspect-ratio carbon nanotubes
- interdisciplinary research:
  - in the area of adsorption and catalysis, 7 different laboratories are synthesizing oxyfluoride compounds with nanoporous architecture
  - in the area of dispersions, about 25 laboratories are investigating (soft) nanochemistry, membranes, and colloidal mixed systems
  - about 10 laboratories are researching Si-based (Si/C/N) nanophase ceramic powders in nanocomposites, composites, and hybrid materials

Research on Carbon and Other Nanotubes

Dr. Christian Colliex, the new Director of the CNRS Laboratory Aimé Cotton in Orsay, described the major types of research under development in France on carbon and other (e.g., BCN) nanotubes. The synthesis, elaboration, purification, and integration of nanotubes are being carried out by various methods at a number of locations:

- arc-discharge at Montpellier and Peleiseau
- solar furnace at Odeillo
- laser ablation at Chatillon

- catalytic routes at Orléans and Grenoble
- microscopic and macroscopic characterization performed by means of electron microscopy (high-resolution imaging, analysis, and electron energy loss spectroscopy—EELS) at Chatillon and Orsay
- near-field microscopy (STM and AFM) at Toulouse
- Micro-superconducting quantum interference device work (microSQUIDs) at Grenoble
- Raman spectroscopy at Nantes and Bordeaux
- Nuclear magnetic resonance (NMR) at Montpellier
- measurements of physical properties of nanotubes are also being made:
  - of mechanical properties at Chatillon
  - of conductivity, transport, and magnetism at Saclay, Orsay, and Grenoble

In addition, a variety of nanotube applications are being investigated in the areas of nanoelectronics, nanocomposites, and storage media. Also, significant CNRS network research programs (so-called GdR or Groupements de Recherche) are underway or planned. One on fullerene research (1992-1996) ended recently, and another on single- and multi-walled nanotubular structures has been proposed at about FFr. 1.5 million. Both are led by P. Bernier at Montpellier, who is also the coordinator of a European effort on nanotubes for microstructure technology (NAMITECH) that includes laboratories from Montpellier, Stuttgart, Valladolid, Namur, Nantes, Orléans, and Dublin and is funded at about FFr. 1.5 to 2 million. In all these French efforts, research ideas come from scientists in laboratories—they "bubble up from the bottom."

A new NEDO (Japan) effort on production, characterization and properties of novel nanotubular materials, coordinated by Sumio Iijima (formerly with NEC-Tsukuba and now at Nagoya University), includes efforts at NEC-Tsukuba, Tokyo, MIT, Rehovoth, and Orsay.

Research on the Rheology and Mechanics of Nanoparticle Arrays

Dr. Henri Van Damme (CRMD-Orléans) discussed ongoing, broad-based French activities in the fundamental rheology and mechanics of ordered and disordered arrays of nanoparticles with controlled interactions. Various dispersed nanoparticle systems are being investigated:

- sol-gel soft glass and colloidal crystals for optical applications
- complex fluids such as ferrofluids and "smart" gels
- mechanical reinforcement of rubber, plastics, and concrete

- porous aggregates of nanoparticles as aerogels to make glass or for damping (localized modes) or for their plasticity in part-forming applications
- "aggregate engineering" with nanoparticles, in which the physics and chemistry of the aggregates is very important, and especially their fractal nature in many mechanical applications
- consolidated nanomaterials, such as ceramics, metals, and cements, for their increased hardness (metals), wear resistance, and ductility (ceramics and cements)

An exciting effort by a 6-laboratory network, with some additional funding by industry, investigating the possibility of making ductile cement ("nanoconcrete"), which could have a strong future impact in this field, was also described.

Research on Magnetic Nanostructures

Dr. François Gautier (Strasbourg) described the extensive work on magnetic nanostructures, including those for magnetic recording and sensors, being carried out in France through a wide-ranging CNRS network and EU-level interactions as well:

- synthesis of various new nanostructures and nanosystems consisting of superlattices, nanowires, quantum dots, and nanoparticles, including work with filled nanotubes and also with the "atomic sawing" of multilayers by dislocation shear to form multilevel nanostructures with various architectures
- investigation of a number of related physical properties and their applications, including giant and colossal magnetoresistance (GMR, CMR), tunneling and magneto-Coulomb effects, magneto-optics, and rapid spin-flip femtomagnetism
- study of fundamental magnetization processes such as nucleation, magnetization reversal, and hysteresis
- work on new experimental methods and instrumentation such as microSQUIDs and nanocalorimetry

Most of the GMR work is being done in a joint laboratory at Thomson CSF (Orsay) led by Prof. Albert Fert, the pioneer of GMR, and jointly funded by CNRS, Thomson, and l'Université d'Orsay. Such laboratories are a special and apparently very effective feature of the CNRS national research program.

Research on Chemical Synthesis of Nanomaterials

Prof. Jean-Pierre Jolivet (Université Pierre et Marie Curie, Paris) described the work on the chemical synthesis of nanomaterials being carried out in the area of chemistry of condensed matter at the University under Prof. Jacques Livage, Director. This research is jointly supported by the CNRS and various industries, such as St. Gobain and Rhône Poulenc. The main topics of this work are

- nanostructured organic-inorganic hybrids
- oxide nanoparticles
- nanocomposites
- bioencapsulation

Wide-ranging application fields include dispersions and coatings, high surface area materials, modification of bulk properties (mechanical, optical, electrical, and magnetic), and immunology. For example, hybrid networks of organic and inorganic materials interpenetrated at the nanometer scale (e.g., a Zr metal network and a polymerized organic network former) could be synthesized using a sol-gel route from heterofunctional molecular precursors and engineered to yield improved mechanical properties of coatings by varying the proportions of the two constituents.

Also discussed were research efforts on the mechanisms involved in the interactions between the precursors and the control of the size and dispersion of various nanostructured entities. The mean particle sizes of nanoparticles of magnetic spinel iron oxides, for example, have been controlled by means of controlling the chemistry of the oxide-solution interface to make composites in polymers or silica glasses; the magnetic behavior of the dispersed state in the nanocomposite is thus controlled.

Research on Self-Organized Soft Nanostructures

Dr. Jacques Prost, Director at the Institut Curie (IC) in Paris, described work in progress at the IC on a variety of self-organized soft nanostructures, including lyotropic liquid crystals (softeners, detergents), block copolymers (polymer alloys), stealth vesicles (for drug delivery or gene therapy), and Langmuir-Blodgett films with grafted antibiotic surface layers. He went on to discuss a variety of related areas in analytical nanostructures, made by patterning surfaces with filled nanocavities, and in DNA-based bionanotechnology used to form nanoelectronics (wires, single-electron transistors), much of it done in the United States or Germany. He cited strong efforts in France at the IC, École Nationale Superieure, and Strasbourg on DNA molecule system micromechanics and on molecular motors for motility assays.

Research on Growth of Nanstructure Materials by Direct Cluster Deposition

Finally, Dr. Albert Masson, CNRS Research Director and a close colleague of Drs. Bréchignac and Colliex at the CNRS and Laboratoire Aimé Cotton, then described extensive and impressive work on the growth of nanostructured materials by means of direct cluster deposition from molecular beams, research done only by the CNRS in France at Orsay and Lyon. A variety of nanomaterials, including metals, semiconductors, and insulators, have been synthesized and studied for a number of their properties. They have been well characterized by several methods, including transmission electron microscopy, Raman and electron spectroscopies, STM, AFM, EELS, and grazing-angle X-rays, along with simulations.

CONCLUSION

The WTEC panel's visit concluded with a general discussion among the participants. It was clear on the scientific side that much more theory, in both the modeling and *ab initio* areas, would be extremely useful to the future development of the nanotechnology field, in France and elsewhere. In terms of the functionality of national research efforts, in nanotechnology and otherwise, there appears to be excellent (and rather unique) networking in France across disciplinary lines and traditional areas, which seems to be largely a result of the highly effective GdR research groups set in motion with money from the CNRS. There is some concern about the effectiveness of transferring science to engineering and then to manufacturing, but the joint CNRS programs with industry can often (but not always) overcome this problem.

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Date Visited: 14 October 1997

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Hosts: Dr. André Chatelain
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Dr. Jean-Philippe Ansermet
Dr. Klaus Kern

BACKGROUND

The École Polytechnique Fédérale de Lausanne (EPFL) is one of three federally funded research institutions in Switzerland. The other two are the Paul Scherrer Institute and Eidgenössiche Technische Hochschule (ETH) in Zürich. EPFL was founded in 1853 as a technical institute of the University of Lausanne. The Laboratory of Experimental Physics was founded in 1947 and has traditionally explored the boundaries between pure and applied science. Starting with activities in piezoelectric properties of crystals, nuclear magnetic resonance, and surface and thin films, EPFL turned its attention to nanoclusters in the 1970s. Today, the main focus is on nanoscale physics of clusters, surfaces, and nanoscaled materials.

RESEARCH AND DEVELOPMENT HIGHLIGHTS

André Chatelain has been involved in cluster physics research for 30 years. He asks the questions, “How many atoms are needed for such bulk-like properties as melting, magnetism, conductivity?” “How many atoms are required for the Curie Temperature to be exhibited?” Clusters are size-selected from a molecular beam and characterized with a Stern-Gerlach magnet, after which the deflection is measured and the clusters are accelerated through a column in which time of flight measurements are made. Connections between cluster and bulk properties such as hysteresis and coercivity have not yet been made.

Chatelain's group has developed techniques for fabricating carbon nanotubes in higher concentrations than previous methods. By fixing a single nanotube to a scanning tunneling microscope (STM) tip, currents as high as 1 mA have been obtained from the tube tip. Typical currents are in the 10 pA to 1 nA range. Using a phosphor screen, fluctuations in the spot position have been related to fluctuations in electron density over the surface of the nanotube tip. It is not known whether this arises from thermally induced structural alteration of the tip or changes in electron density due to local charging.

Klaus Kern has a large group working on self-organized growth of nanostructure arrays. The novelty of his approach lies in his use of periodic dislocation arrays that serve to isolate nucleating adatoms. This has been demonstrated using a Pt(111) substrate precovered with 1.5 monolayer of silver that forms a pseudomorphic layer, and a second Ag layer that forms a trigonal dislocation network. Subsequent Ag adatoms are repelled by the dislocations and form into a network of regularly spaced individual islands. Deposition is performed below 110 K. Kern states that these experiments open a new method to create almost monodispersed, regularly spaced, superlattice nanostructures using the natural properties of crystals.

Jean-Philippe Ansermet uses polycarbonate membranes with $6 \times 10^8/cm^2$ pores with gold sputtered on the back side as a template for magnetic nanowires. Magnetic material is plated into the pores, which are 20-200 nm in diameter and about 6 µm high. Ni or Co is plated in order to study anisotropic magnetoresistance (AMR), and layered materials are used for giant magnetoresistance (GMR) structures. A structure consisting of 300 ten-nanometer layers of Cu and Co gave a 40% GMR ratio at room temperature. One of the difficulties of this approach is making contact to individual wires. Ansermet masks the top of the membrane and introduces gold into the plating solution. Plating is halted when contact is made to one wire, or perhaps, a few wires.

Ansermet is considering how AMR is related to GMR. His approach uses the curling spin wall to separate domains in the wire. He explains that the curl avoids surface charge along the wire. He claims that if the spin flip length (~50 nm in Co/Cu) is less than the length of a domain, the system is an appropriate analog-to-GMR structure. His prototype experiments show that the AMR ratio is enhanced by using the curl domain wall.

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Date Visited: 15 October 1997

WTEC: E. Hu (report author), H. Goronkin, M.C. Roco, D.T. Shaw

Hosts: Prof. Dr. Klaus Ensslin (principal contact)
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Dr. Hans von Känel

BACKGROUND

Eidgenössiche Technische Hochschule Zürich (ETHZ), the Swiss Federal Institute of Technology in Zurich, was founded by the Swiss government in 1854 as a polytechnic university. Until 1969, it was the only national university in Switzerland; today, it is part of an ETH domain comprising ETHZ, EPFL (École Polytechnique Fédérale de Lausanne, the polytechnic institute at Lausanne) and four national research institutes. The ETH itself comprises about 12,000 registered students, 330 professorships, and 700 lecturers. Each year, about 1,250 students receive an ETH diploma and another 450 students complete a doctoral thesis.

RESEARCH AND DEVELOPMENT HIGHLIGHTS

ETHZ is carrying out a broad spectrum of nanoscience research, spanning synthesis, processing, and characterization, ranging from fullerenes to ferroelectric to magnetic materials and encompassing electronic devices and nanorobots (*Nanowissenschaften* 1996). The funding sources seem largely to emanate from the Swiss National Science Foundation, often under the auspices of a *National Research Program* (such as NFP 36) or a *Swiss Priority Program*; funding is also provided by industrial sources such as IBM Rüschlikon or Ciba-Geigy.

Specific Project Highlights

Professor Dr. Ensslin, Professor of Physics at ETHZ, is also formally head of the Paul Scherrer Institute Laboratory for Micro- and Nanostructures; a position specifically designed to enhance close collaborations between the two institutions. A long-time contributor to the study of functional nanostructures, Professor Dr. Ensslin described some current projects undertaken in his laboratory at ETHZ:

- Use of an atomic force microscope (AFM) to locally oxidize a structure, forming a "quantum point contact" directly, without need for electron-beam lithography or extensive alignment procedures. Professor Dr. Ensslin noted the ease of use of commercially available AFMs and their ready adaptability to lithography and materials modification at the nanoscale (Held et al. 1997).
- Establishment of low temperature (1 K), high magnetic field (10 tesla) capabilities to carry out scanning tunneling microscope (STM) spectroscopy of semiconductor nanostructures in the quantum hall regime.
- Wave function spectroscopy in specially tailored quantum wells grown at the University of California in Santa Barbara.

The WTEC team also visited the laboratory of Dr. Hans von Känel, who has developed an ultrahigh vacuum system for in situ growth and processing of Si, Si/Ge materials that allow monitoring of the growth process; low-temperature, controlled materials modification; and STM analysis. The system is also used for characterization of nanomechanical properties through the ability to prepare and "load" (sputter-deposit materials) cantilever probes in situ.

REFERENCES

Nanowissenschaften an der ETH Zürich. 18 May 1996.

Held, R., T. Heinzel, P. Studerus, K. Ensslin, and M. Holland. 1997. Fabrication of a semiconductor quantum point contact by lithography with an atomic force microscope. *Applied Physics Letters* Nov.

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Date Visited: 13 October 1997

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Dr. Heinrich Rohrer, Science and Technology Department
Wanda Andreoni
R. Allenspach
James Gimzewski
Peter Vettiger

BACKGROUND

The IBM laboratory in Rüschlikon employs approximately 200 people. The WTEC team visited the Science and Technology Department, formerly called the Physics Department. This department has about 50 people, of whom approximately 40 are scientists (including pre- and post-docs). In recent years, the lab’s scope has evolved beyond mostly basic research to a problem-oriented mission in support of existing and alternative exploratory technologies that are on IBM’s radar screen.

Heinrich Rohrer provided an introduction to the Science and Technology Department, pointing out the need for new ideas to move beyond the limits of present day technologies and devices.

The laboratories the WTEC team visited are well equipped and of a size normally found in universities. Many of the experimental apparatuses are set up by highly skilled technicians who also take an active part in the operation of the equipment for experimental purposes. Although the mission of the group is technology-driven, it is clear that science remains a key component and that the staff strives for understanding of the experimental results at the most basic level.

Collaborations with universities are numerous. The center leverages productivity considerably while fulfilling an educational purpose and creating a network of relationships of great value to all concerned. Some of these collaborations are supported by European (ESPRIT) and Swiss national programs.

Scanning probe technology was born in the IBM labs, and it has opened numerous areas for research in Switzerland. Scanning probes are used for atomic and molecular manipulation as well as characterization. Since the technique is relatively inexpensive, it is ubiquitous and drives many of the country's research projects.

RESEARCH AND DEVELOPMENT HIGHLIGHTS

Peter Vettiger described the IBM Zürich approach to mass storage using silicon microcantilevers with tips to record bits in a polymer medium (e.g., PMMA). Writing is achieved thermomechanically by heating the tip and creating an indentation in the softened polymer. Erasing is done in blocks rather than in individual bits by heating entire storage subfields.

Vettiger demonstrated a 5 x 5 array of individually accessible tips. In this case, the tips were fabricated using anisotropically etched silicon so that the 5 x 5 array used a 5 x 5 mm area (KOH etching provides sidewalls with a 54^{o} slope). In order to increase the density of tips, a new etching process was developed that provides vertical sidewalls so that a 32 x 32 array can fit into a 1 x 1 mm area. Parallel operation of 1000 cantilever/tips is envisaged with x-y addressing achieved through multiplexing. Bits of 20-40 nm in size have been demonstrated. This extrapolates to more than 60 Gbit/in^2 of data.

The array was demonstrated to image a test surface. In this demonstration, each tip provided an independent image.

James Gimzewski described the well-known buckyball abacus in which an STM tip is used to move C_{60} molecules along well-defined linear paths. He pointed out that the buckyball molecule can also be used as an amplifier when it is compressed by a scanning probe tip. He is generally working on concepts for manipulating and assembling molecules with the STM to implement useful functions.

R. Allenspach described the center's magnetism activities. This work focuses on the study of ultrathin magnetic films and multilayers with Cu/Co as a model system and has a direct impact on the understanding of magnetic properties such as giant magnetoresistance (GMR), exchange coupling, and surface anisotropy. It has led to the discovery of anisotropy oscillations due to quantum confinement in a Cu overlayer on a Co film. This work also includes detailed studies of film growth and morphology and how these correlate with magnetic properties.

Hans Biebuyck is a former student of George Whitesides at Harvard University, who is well known for microcontact printing using elastomeric stamps to transfer a pattern of self-assembling molecular layers to a substrate. The project he described encompasses both the science and pretechnology assessments of various high-resolution contact processing techniques like microcontact printing and microfluidic networks. The group has demonstrated the use of microfluidic networks for delivery of functionally distinct biomolecules onto targeted regions of a substrate and their application in localized biological assays. Stamps of very high quality and stability have been developed, and critical dimensions smaller than 50 nm have been achieved with microcontact printing.

Wanda Andreoni described the activities in computational materials science and the application of *ab initio* (Car-Parrinello) molecular dynamics techniques to various problems in science and technology, e.g., fullerenes, carbon nanotubes, and organic light-emitting structures for displays, catalysis, and chemical reactions. Some of this work is in direct support of ongoing local projects.

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Date Visited: 16 October 1997

WTEC: E. Hu (report author)

Hosts: Dr. Marc Van Rossum, Head VLSI Materials and Technologies, Advanced Semiconductor Processing Div.
Dr. John Randall, TI on assignment to IMEC
Dr. Wijm Magnus, PHANTOMS coordinator
Dr. Chris Van Hoof, Senior Researcher, Materials and Packaging Division
Dr. Jan Genoe, software specialist – 8-band solver
Dr. Jo deBoeck, Senior Researcher, Novel Materials and Components Group

BACKGROUND

IMEC (Interuniversity Microelectronics Center) was begun in 1984 by the Flanders government to strengthen the potential of Flemish universities and to strengthen the infrastructure in microelectronics. The general mission of IMEC is to strengthen local industry, set up new industry, and attract new industry by carrying out advanced, focused research. Initially, the lead-time on industrial technology was estimated to be five to ten years; actual operation has led to carrying out projects in the nearer-term future (3-10 years, or even under 3 years). The initial investment was ~ $80 million and 150 people. Currently, IMEC has >700 people, 100 of whom are nonpayroll (industrial participants, students with grants), and a >$80 million budget, of which ~ 50% derives from the government, and 50% is contract research.

RESEARCH AND DEVELOPMENT HIGHLIGHTS

The WTEC team's host at IMEC was Dr. Marc Van Rossum, the Head of VLSI Materials and Technologies in the Advanced Semiconductor Processing Division. He spoke of three main areas of research being carried out: (1) VLSI system design methodology; (2) materials and packaging; and (3) advanced semiconductor processing.

The advanced semiconductor processing area actually has 50% of the budget and less than 50% of the personnel. One-third of the capital expenditure in this area is associated with costs of running the pilot line. Currently, this area develops new process modules compatible with 0.25 µm and 0.18 µm lithographic design rules, as well as carrying out research in 0.1 µm. Assessments of the programs are carried out in five-year intervals; among the measures of success are the number of spin-off companies generated (an average of one per year) and the number of contracts generated with local industry.

Discussions with Dr. Chris Van Hoof, Senior Researcher in the Materials and Packaging Division, and Dr. Jan Genoe involved the following topics:

- InAsSb photovoltaic detectors operating in the 3-5 µm range, at 140 K (an improvement over InAs detectors) with CMOS camera readouts
- InGaAs (28% In) light-emitting detectors sensitive to 3.3 µm wavelength, with 10^{-4} efficiency, at >1 GB modulation rates

Dr. deBoeck is carrying out work on nanomagnetics and is the coordinator of an ESPRIT Program called SPIDER, on spin-dependent nanoelectronics, which looks at the possibility of combining semiconductor devices and ferromagnetic nanostructures. One approach in this regard is the formation of nanoscale MnAs ferromagnetic clusters in GaAs through the low-temperature MBE growth of MnGaAs (230ºC - 280ºC) and subsequent annealing at temperatures ranging from 625ºC–730ºC. Depending on the starting material composition and the annealing conditions, metallic clusters of 3 to 30 nm are formed, with saturation magnetization values that first increase and then decrease with annealing temperature. Photoluminescence studies of the predominantly GaAs surrounding matrix evidences good optical quality.

Discussions with Drs. van Rossum and Magnus centered on IMEC activities in new submicron electronic technologies, as well as on European-wide microelectronic and nanoelectronic programs. Dr. Wijm Magnus is the overall coordinator of the PHANTOMS (Physics and Technology of Mesoscale Systems) program, a network of institutions now including sites in Russia and Eastern Europe as well as in Western Europe. The strategic research domains comprising PHANTOMS are (1) quantum electronics, (2) nanometer-scale optoelectronics, (3) nanotechnology, and (4) novel circuit architectures. PHANTOMS meets twice yearly, and is attempting to put together a nanoelectronics roadmap. There has been an arrangement between 7 PHANTOMS institutes and the NRC at Ottawa through ECAMI (European-Canadian Mesoscopic Initiative), which was designed to facilitate short- to midterm visits. The initial program, which ended in 1997, was successful, and negotiations are now underway to seek an extension of such an arrangement, to involve 14 European labs and 9 Canadian labs. A similar arrangement between PHANTOMS and institutions in the United States would be highly desirable, but it has not yet been possible to negotiate such an arrangement. The National Science Foundation (NSF), for example, usually recognizes bilateral proposals rather than working agreements with networks of institutions.

There was also discussion of the ESPRIT Advanced Research Initiative in Microelectronics (MEL-ARI) that includes projects launched under nanoscale integrated circuits initiative. These projects are to contribute to the research of future electron devices, such as single-electron electronics (transistors/memories based on Si, SiGe, or GaAs), molecular electronic devices, single-flux quantum logic circuits (high T_c superconductor-based) or magnetoelectronic devices. For all projects, nanofabrication is a key issue, and e-beam lithography, scanning probe microscopy, and stamping techniques are to be explored as possible technologies to produce future devices. Three associated projects deal with (1) low-energy coherent electron microguns, (2) nanolithography using chemically amplified resists, and (3) vertical SiGe MOS devices.

ESPRIT is currently operating within its "Fourth Framework" (1994-98). The Fifth Framework of ESPRIT is currently in the definition phase. During the 1st–4th frameworks, about $13.6 billion European Economic Units (ECUs) were spent, of which 30% was spent on electronics. No figures were available for nanoelectronics specifically, but long-term research was ~10% of the total budget. Table B.1 provides a partial listing of ESPRIT projects in nanoelectronics.

TABLE B.1. ESPRIT Nanoelectronics Projects

Acronym	Project	Coordinator
SIQUIC	SiGe RTDs	D. Paul, Cambridge
FASEM	Single Electron Transistors	H. Launois
	4 x 4 memory based on Hitachi Cambridge device	CNRS, Bagneux
CHARGE	Coulomb Blockade	D. Haviland, KTH
QUEST	STM/AFM lithography	E. Dubois, ISEN, Lille
SPIDER	Spin Valve Nanoelectronics	J. deBoeck, IMEC
RSFQ-HTS	Single Flux Quantum Device	Rogalla, U. of Twente
QUADRANT	Cellular Automata, with University of Notre Dame	M. Macucci, Pisa
LASMEDS	Molecular electronics	P. Morales, ENEA
NANOWIRES	Nanowires	M. Welland, Cambridge

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Date Visited: 14 October 1997

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Dr. Winfried Brückner, IFW, Institute for Solid State Research, Thin Film Department
Dr. Jürgen Eckert, IFW, Institute of Metallic Materials, Leader, Department of Metastable and Nanostructured Materials
Dr. Karl-Hartmut Müller, IFW, Institute for Metallic Materials, Department of Superconductivity, Magnetism
Dr. V. Neu, IFW, Institute for Metallic Materials, Department of Superconductivity, Magnetism
Dr. Norbert Mattern, IFW, Institute for Solid State Analysis and Structural Research, Department of X-ray Structural Analysis
Dr. Martin Heilmaier, IFW, Institute for Metallic Materials, Department of Strength, Environmental Effects
Dr. Roland Scholl, Fraunhofer Institute, Institute for Applied Materials Research, Department for Powder Metallurgy and Composite Materials, Winterbergstrasse 28, D–01277, Dresden, Germany

BACKGROUND

The Institute of Solid State and Materials Research, IFW Dresden, was founded in 1992. As an institute of the Wissenschaftsgemeinschaft Blaue Liste (WBL) it is funded by the Free State of Saxony and the Federal

Republic of Germany. It has a staff of over 400 employees and is devoted to applications-oriented fundamental research. It is a member of the Materials Research Association, Dresden, and is associated with the Dresden University of Technology and the Fraunhofer Institute. In 1996 the staff consisted of 235 permanent and 180 temporary employees, of which 220 were scientists. The budget for 1996 consisted of about DM 30 million basic financing and about DM 11 million in projects. The scope of the research includes studies of the relationships between fundamental and applications-related characteristics of metallic and nonmetallic functional materials and thin films, investigation of structural properties and failure mechanisms, and studies of processing variables for property optimization. The WTEC team was hosted by Prof. Dr. Ludwig Schultz, who is director of the Institute for Metallic Materials. Research on nanostructures and nanostructure science is carried on in many of the groups in IFW, with about one-third of the groups partly or mostly involved.

RESEARCH AND DEVELOPMENT HIGHLIGHTS

Investigators in several fields of nanostructure science made presentations to the WTEC team, after which we toured the laboratory facilities. Brief descriptions of the presentations are given below, with scientific and/or technical highlights.

Prof. Dr. Karl Leo
Institut für Angewandte Photophysik, Technische Universität Dresden

Professor Leo gave a presentation on studies of electronic transport through single molecules in epitaxially grown organic heterostructures. This involves single-electron tunneling effects with single molecules. Common approaches using metal structures of ~ 30 nm size with capacitance of a few 10^{-17} F require temperatures of < 20K. IFW's approach is to use molecules providing stable and reproducible "bricks" with a typical size of 1-2 nm. Organic molecular beam epitaxy (OMBE) was used to deposit molecules of perylene-tetra-carboxylic-dianhydride (PTCDA) on Au (111), which is on a cleaved mica substrate. PTCDA orients on lines on the Au lattice. Coulomb blockade measurements are made at room temperature with an STM tip. This can only be accomplished on an "ordered" molecular lattice that is locked into the Au lattice so it does not move. An insulating layer of deconethiol is placed between PTCDA and the Au, and the S in the deconethiol binds to the Au. Preliminary I-V measurements have been made successfully.

Prof. Mark Golden
IFW, Spectroscopy Group, Department of Surfaces and Interfaces

Dr. Golden presented a review of his work on the electronic structure of fullerenes, nanotubes, and metal/fullerene multilayers. Spectroscopic methods are used for these studies and include X-ray absorption spectroscopy (XAS), angle resolved photoelectron spectroscopy (ARPES), X-ray photoelectron spectroscopy (XPS), and electron energy-loss spectroscopy (EELS). The facility for high resolution EELS measurements is a dedicated machine, i.e., not part of a transmission electronic microscope. Among the measurements made are charge states, bonding, plasmon dispersions, optical properties, and core level excitations. Various materials studied include C_{60}/metal multilayers, nanotubes, and doped fullerenes (off-ball doping-intercalation, on-ball doping such as $C_{59}N$, and in-ball doping-endohedral metallofullerenes such as Tm in C_{82}).

Dr. Winfried Brückner
IFW, Institute for Solid State Research, Thin Film Department

Dr. Brückner described his work on the electrical and mechanical properties of a resistive CuNi(Mn) thin film with a nanocrystalline structure. The CuNi(Mn) films, sandwiched between Ni-Cr films, had columnar grains about 30 nm in dimension, and were twinned. The temperature coefficient of resistivity (TCR) was a function of composition x of the $Cu_{1-x}Ni_x$ films and of the temperature of thermal cycling. The initial negative TCR changed to positive after heating to $\geq$ 500°C. This was explained by the changes in the mechanical stresses in the films, which were influenced by formation of NiO and grain growth at the higher temperatures.

Dr. Jürgen Eckert
IFW, Institute of Metallic Materials, Leader, Department of Metastable and Nanostructured Materials

The four major thrusts of this group are: (1) basic principles of mechanically alloyed nanocrystalline materials, (2) high-strength lightweight nanostructured alloys, (3) mechanically alloyed superconducting borocarbides, and (4) bulk metallic glasses.

In the first thrust the relationship to nanostructured research is that ball milling is a nonequilibrium processing method for preparation of nanoscale materials. In this regard the formation of nanocrystalline materials is studied by determining grain size as function of milling conditions such as temperature, milling intensity, and alloy composition. In terms of high strength lightweight alloys, Al and Mg alloys with mixed phases of nanocrystalline, amorphous, and/or quasicrystalline nanoscale microstructures are studied. Of special interest are Al-base (> 90 at.% Al) alloys with nanoscale quasicrystalline phases of 20-100 nm diameter surrounded by fcc Al phase of 5-25 nm thickness. The quasicrystalline phase comprises 60-80% volume fraction of the alloys. These alloys combine high strength (1,000 - 1,300 MPa fracture strength) and good ductility (6-25%). The suggested mechanisms for these excellent mechanical properties include the thin fcc Al layer around the quasicrystalline particles, a high density of phason defects and approximant crystalline regions with subnanoscale size, and the spherical morphology of the quasicrystalline particles with random orientations. The research is aimed at a better understanding of the mechanical behavior of these promising materials.

Bulk metallic glasses (e.g., $Mg_{55}Y_{15}Cu_{30}$) are prepared by solidification and mechanical alloying methods. The mechanically alloyed bulk metallic glass powders are consolidated at temperatures above T_g. Again some studies of mixed amorphous and nanocrystalline phases are carried out in these systems. That is, the nanocrystalline precipitates are used to strengthen the amorphous matrix.

Dr. Karl-Hartmut Müller
IFW, Institute for Metallic Materials, Department of Superconductivity, Magnetism

Dr. Müller described the research program on the hydrogen-assisted preparation of fine-grained rare earth permanent magnets. The technique used is “hydrogenation disproportionation desorption recombination” (HDDR). The final structure is fine-grained, 100-500 nm, rather than nanoscale, but during the disproportionation and desorption steps the structures can be ~ 100 nm in size. An example of HDDR for Nd-Fe-B is as follows: Original cast alloy of $Nd_{16}Fe_{76}B_8$ with Nd-rich and $Nd_2Fe_{14}B$ phases is processed in four steps: (1) hydrogenation forms $NdH_{2.7}$ and $Nd_2Fe_{14}BH_{2.9}$; (2) disproportionation reaction results in a fine mixture of Fe, $NdH_{2.2}$, and Fe_2B; (3) desorption provides a very fine mixture of Fe, Nd,

Fe_2B + $Nd_2Fe_{14}B$ nuclei; and (4) recombination yields fine grained $Nd_2Fe_{14}B$. Several rare earth permanent magnet alloys are studied at IFW using HDDR, including $Sm_2Fe_{17}N_3$ and $Sm_2Fe_{17-x}Ga_x$.

Dr. V. Neu
IFW, Institute for Metallic Materials, Department of Superconductivity, Magnetism

Dr. Neu described NbFeB magnet powders prepared by mechanical alloying. The goal of this work is to obtain high remanent, isotropic Nb-Fe-B powders for polymer-bonded permanent magnets. Mechanical alloying is used to obtain a nanoscale mixture of $Nd_2Fe_{14}B$ and aFe which provides for remanence enhancement via exchange coupling when the grain sizes are $\leq$ 30 nm. The mechanical alloying provides an amorphous + nc Fe structure which on annealing forms nc aFe + nc $Nd_2Fe_{14}B$ which behaves as a single magnetic phase. The powders can be bonded with polymers and form isotropic magnets with high remanence. In addition some Fe can be replaced by Co which increases the remanence (as well as the Curie temperature) and has provided $(BH)_{max}$ values up to about 150 kJ/m^2.

Dr. Norbert Mattern
IFW, Institute for Solid State Analysis and Structural Research, Department of X-Ray Structural Analysis

Dr. Mattern described work on soft ferromagnetic materials such as the "finemet"-like alloys (FeNiSiBNbCu) and FeZrB alloys. These materials are made by rapid solidification to obtain amorphous alloys, which are then partially recrystallized to give nanoscale (~ 50 nm) αFe particles in the amorphous matrix. Studies have included composition variations to influence nc grain size and studies of the crystallization kinetics. High nucleation rates and slow growth rates are desired and influenced by the alloy dopants. This research is funded by the federal government and by Vacuumschmelze and Siemens.

Dr. Martin Heilmaier
IFW, Institute for Metallic Materials, Department of Strength, Environmental Effects

Dr. Heilmaier described several projects involving dispersion hardening with nanoscale dispersoids. One project has the goal of dispersion strengthening of Ag to be used as casings for the BiSrCaCuO high T_C

superconductor. Mechanical alloying of Ag and Cr_2O_3 powders is followed by cold pressing, annealing in dry hydrogen, hot pressing at 500°C, and finally hot extrusion at 700°C. The mechanical alloying times were apparently too short in the initial study to provide a uniform distribution of the 40 nm Cr_2O_3 particles. A bimodal grain structure was observed with mean sizes of 10 µm and 0.3 µm composed of 60% pure Ag grains and 40% Ag grains with the nc oxide dispersoids. Even so, significant Hall-Petch hardening was observed at room temperature, along with increased creep resistance at 500°C in the dispersion-hardened Ag. Another project focuses on mechanical alloying of Ll_2–$(Al,Cr)_3$ Ti intermetallic with Y_2O_3 nanoscale dispersoids of 5 nm size.

Dr. Roland Scholl
Fraunhofer Institute, Institute for Applied Materials Research, Department for Powder Metallurgy and Composite Materials

Dr. Scholl described an in situ data acquisition and monitoring system for a planetary ball mill. This is important for studies of the mechanical alloying/milling processes used for formation of nanocrystalline materials. The device measures the temperature and pressure via a transmitter in the lid of the milling vial. This was done for a Fritsch "pulverisette 5" planetary mill; the work was partially supported by Fritsch. An example was given for milling of Ti and C powders to form TiC. Good time resolution, about 10 ms, is available to monitor the reactions, which can occur during milling and provide feedback to optimize the milling parameters.

After the formal presentations and discussions, the WTEC visitors were given a tour of the IFW laboratories. We observed very impressive state-of-the-art facilities for processing, characterization, and property testing. The dedicated EELS facility referred to in the work of Dr. Golden (above) was particularly noteworthy.

Site: **Institute for New Materials (INM)**
Im Stadtwald - Gebäude 43
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http://www.inm-gmbh.de/

Date Visited: 13 October 1997

WTEC: J. Mendel (report author)

Hosts: Prof. Dr. Rudiger Nass (principal contact), Head of Ceramics Department
Dr. Rolf Clasen, Director, Department of Glass Technology

BACKGROUND

Founded in 1988, the Institute for New Materials (INM) is located within the University of the Saarland. Currently, the Institute has 280 scientists and technologists who develop new materials that industry will need for the future. The institute's purpose is to further the utilization of new high technology materials on a large scale. It is a nonprofit limited liability company with institutional sponsorship.

RESEARCH AND DEVELOPMENT HIGHLIGHTS

At INM, research and development comprises basic research on highly innovative, high risk, long term programs as a basis for new technologies. The goal is to reduce the cycle time of 10-15-year programs (concept to commercialization). Products and processes are developed in close cooperation with industrial partners, who often provide the necessary financing. Since 1990, INM follows the scientific approach of integrating inorganic synthesis with chemical nanotechnology.

In addition to metals, nonmetal inorganic materials, and organic polymers of a singular nature, it is now possible to produce chemical composite materials on the molecular and nanoscale level. Processes such as sol-gel are used, in which liquid starting materials are utilized at low temperatures for nanoscale metal, ceramic, glass, and semiconductor particles. INM cites these high-interest features for preparing new materials as nanoparticles:

- small enough not to scatter light

- quantum effects—intrinsic properties of metal and semiconductor nanoparticles—for tailoring new properties
- large interfaces resulting from dispersion in a matrix so as to add another dimension for property tailoring

Specific Project Highlights

Dr. Rolf Clasen is currently preparing glass powders via the colloidal gel route. The advantages for this process are high purity powders.

Also in this same laboratory the following efforts are taking place:

- forming compacts of submicron silica particles by electrostatic deposition
- sintering behavior of submicron silica particles
- preparation of high purity silica glass tubes by centrifugal casting of colloidal gels

Role of Nanostructure Science within INM

INM is focusing on four basic areas for spin-off and adaptation towards commercialization:

1. New functional surfaces with nanomers: included are properties such as corrosion protection, wettability, coloration, micropatterned surfaces, porosity, or the ability for selective absorption of molecules.
2. New materials for optical applications: properties of lasers and ceramics are combined with those of polymers. Such features as optical filters, transparent conducting layers, materials for optical telecommunications, photochromic layers, and holographic image storage are under investigation.
3. Ceramic technologies: a simple precipitation process such as sol-gel provides for pilot-scale production of agglomerate-free powder.
4. Glass technologies: chemical incorporation of metal colloids with intelligent properties into glasslike structures are clearly possible.

Equipment

INM has available the following characterization tools:

1. HR-TEM
2. HR-SEM
3. EDXS
4. AFM
5. NMR
6. SAXS
7. X-Ray Diffractometry
8. GC/MS
9. Laser Lab
10. Rheology Analyzing System
11. Mechanical Material Testing Facilities
12. Optical Testing Services

Listed below are services INM performs:

- Consulting
- Project Definition
- Project Formulation
- Contract Assistance
- Patent Search
- Project Implementation
- Technical Development
- Quality & Certification
- Pilot Production
- Troubleshooting

CONCLUDING REMARKS

The emerging new technologies under study at INM will play a dominant role in the 21st Century. Nanomaterials will be incorporated into technical components and systems in most sectors of the technology. They thus become powerful tools in the preparation of specialized materials.

Site: **Max-Planck-Institut für Kohlenforschung**
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D45470 Mülheim an der Ruhr, Germany
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http://www.mpi-muelheim.mpg.de/

Date Visited: 16 October 1997

WTEC: D. Cox (report author)

Host: Dr. John Bradley
Fax: (49) 208 306 2987
E-mail: bradley@mpi-muelheim.mpg.de

BACKGROUND

The Max-Planck-Institut für Kohlenforschung (the Max Planck Institute of Coal Research) in Mülheim was founded in 1912 as one of the first institutes of the former Kaiser-Wilhelm-Gesellschaft as an independent foundation. It is one of more than 70 Max Planck institutes in Germany but has kept its independent legal status under private law and is a recognized nonprofit organization. It is well known for its discoveries of the Fischer-Tropsch process and the Ziegler catalysts. The patents from these discoveries as well as others have generated significant additional income for the institute over the years (particularly notable are the patents of Karl Ziegler, whose first patent for the low-pressure polyethylene synthesis was granted in 1953) and have allowed it to generously fund research efforts of the staff. The expiration of the Ziegler patents has reduced the outside income, and as a result, the funding levels are beginning to come more into line with those of other Max Planck institutes.

This institute has a history of exploiting the inventions made in the institute by retaining ownership of all patents through a trusteeship (the Studiengesellschaft Kohle mbH) which grants licenses producing this additional income. Presently the institute has about 30 nonexpired patents and published patent applications. The Studiengesellschaft Kohle also grants licenses for the usage of software developed within the Max-Planck-Institut für Kohlenforschung; for example, the mass spectrometry software MassLib® was developed at this institute.

As of the time of the WTEC visit, the staff has about 220 permanent employees, about 50 of which are staff scientists. In addition, there are about 100 graduate students, postdocs, and guest scientists distributed among about 24 research groups focusing in the following areas:

- Organic Synthesis
- Organometallic Chemistry
- Homogeneous Catalysis
- Heterogeneous Catalysis
- Supramolecular Chemistry
- Polymer Chemistry
- Coal Research

The institute has a rich history in the area of chemical catalysis, built in large part on Ziegler's Nobel Prize-winning work in ethylene polymerization. The present research areas at the institute focus on synthesis of novel materials for applications in catalysis, energy storage, and separations. The efforts that are particularly applicable to nanoscale science and technology are those involved in studying highly selective catalysts and in generating microporous inorganic oxide materials and high surface area materials for chemical energy storage. Research in these areas is centered in several of the groups at Mülheim, particularly those of Prof. Dr. M.T. Reetz, Dr. J.S. Bradley, Prof. Dr. W.F. Maier, and Prof. Dr. H. Bogdanovic.

The institute is very well equipped as are the individual research groups with world class capabilities in NMR spectroscopy, X-ray characterization and Modeling, Optical Spectroscopy, Mass Spectrometry, Electron Microscopies, and Chromatography as examples.

RESEARCH AND DEVELOPMENT HIGHLIGHTS

Research in the Group of Prof. H. Bönnemann

Prof. Bönnemann has developed widely applicable synthetic methods for the preparation of surfactant-stabilized colloidal metal nanoclusters (1-10 nm, mono- and plurismetallic) based on reduction of metal salts with surfactant-containing reducing agents and the use of surfactant cation salts of metal complex anions. These materials, which have high metal nanocluster content and high solubility (up to 1 mole of metal/liter) in organic solvents or water, have been applied as catalyst precursors both in liquid dispersion and in supported form for a variety of organic reactions, e.g., selective (including enantioselective) hydrogenation and oxidation. Further fields of application are bimetallic fuel cell catalysts, magnetic fluids, nanometal pigments for magneto-optical data storage, and magnetic cell separation in biological samples.

Research in the Group of Prof. Dr. M.T. Reetz

Prof. Reetz, in addition to his research effort, is one of two scientific directors at the MPI für Kohlenforschung. He uses electrochemical reduction of metal salts to prepare highly dispersed colloidal transition metal nanoclusters and supported nanoclusters, a process for which a patent has been granted. Variation of the current density and the temperature as well as the polarity of the solvent during the electrochemical synthesis allows control of the size of the nanoclusters. The stabilizing surfactant shell surrounding these nanoclusters can be visualized with a combination of STM and high resolution TEM. The clusters are evaluated as catalysts for selective organic transformations including carbon-carbon bond forming reactions (*Science* 267:367, 1995).

Research in the Group of Dr. J. S. Bradley

Dr. Bradley has long been involved in metal cluster and metal colloid chemistry areas of nanoscale science and technology. He joined the MPI für Kohlenforschung in 1995. His present emphasis in nanoscale materials focuses on the development of new synthetic methods for colloidal transition metal nanoclusters, their spectroscopic characterization (infrared, NMR, and extended X-ray absorption fine structure [EXAFS] spectroscopy), and the use of in-situ kinetic catalytic probes to define their surface chemistry. In addition, research is ongoing on the preparation from organometallic precursors of microporous nonoxide ceramics and their use in base-catalyzed reactions. For example, high surface area (400 m^2/g) silicon amidonitride with a mean pore diameter of 7 Å has been prepared.

Research in the Group of Prof. Dr. W.F. Maier

Prof. Maier's main research area is aimed towards design of new heterogeneous catalysts that will have isolated active centers in a microporous metal oxide matrix (amorphous microporous mixed oxides, AMM). Guidance for this approach is taken from the fact that enzymes and zeolites are the most selective catalysts, having in common an isolated active center and a shape-selective environment around the active site. Prof. Maier's group has developed techniques to prepare AMM materials by a special sol-gel process that allows control of the chemical composition, pore size (0.5-1.0 nm), porosity, and surface polarity in a single preparation step. AMM catalysts have a narrow micropore distribution comparable to those of zeolites, and a homogeneous elemental distribution. They have produced shape-selective catalysts based on microporous titania, zirconia, and alumina. The AMM materials have been shown to be selective catalysts for oxidation, hydrogenation, alkylation, and hydrocracking.

AMM membranes, prepared by dip-coating of asymmetric support membranes, are then used as catalytic membrane reactors. The catalytic membrane reactor allows the combination of catalytic activity with the permselectivity of the membranes to improve the selectivity of heterogeneously catalyzed reactions. Novel applications of AMM membranes include poison-resistant catalysis and complete suppression of secondary reactions with membrane catalysts.

Research in the Group of Prof. Dr. B. Bogdanovic

The nanomaterial research in Prof. Bogdanovic’s group focuses on the preparation of highly reactive, highly dispersed inorganic materials (metal and intermetallic cluster materials, metal hydrides, metal carbides) from molecular organometallic precursors. Materials based on active magnesium hydride were discovered for use as reversible hydrogen storage systems. Highly dispersed metals, intermetallics, and carbides have been evaluated in a variety of catalytic organic reactions.

Site:	**Paul Scherrer Institute (PSI)** **Laboratory for Micro- and Nanostructures (LMN)** **CH-5232 Villigen PSI, Switzerland** **http://www.psi.ch (or http://www1.psi.ch/www_lmn_hn/)**
Date Visited:	15 October 1997
WTEC:	E. Hu (report author), H. Goronkin, M.C. Roco, D.T. Shaw
Hosts:	Dr. Jens Gobrecht (principal contact), Head, Micro- and Nanostructures Laboratory Fax: (41) 56-310 2646; E-mail: gobrecht@psi.ch Dr. Werner Wagner, Head, Experimental Facilities Dr. Helena Van Swygenhoven, Experimental Facilities Dr. Thomas Jung

BACKGROUND

The Paul Scherrer Institute (PSI) is the largest Swiss-supported government laboratory. The approximate allocation of funding and division of personnel (1996) is as follows:

• government funding	158 million SF
• external funding	~28 million*
• PSI staff	880
• external staff	210
• doctoral students	230
• external users	650

*21% from industry, 29% from electricity generation industry and NAGRA for nuclear energy research, 8% from EU and Swiss National Fund

The allocation of budget (in SFr. millions) by field of research is as follows (parentheses show % of total allocation, including external funding):

• particle physics	13 (11)
• life sciences	14 (14)
• SS physics and materials sciences	39 (37)
• energy	34 (38)

Operated as a multidisciplinary national research lab, PSI provides research infrastructure and the operation of large scale facilities, such as the Spallation Neutron Source (SINQ), recently completed, and a synchrotron light source (Swiss Light Source), which was to begin construction in Spring 1998. PSI interacts broadly with universities and polytechnical high schools. University students may carry out their doctoral studies in PSI laboratories. PSI personnel also carry out radiation safety training and reactor education.

NANOSTRUCTURE SCIENCE WITHIN PSI

The WTEC team's host at PSI was Dr. Jens Gobrecht, the Head of the Laboratory for Micro- and Nanostructures (LMN) in the Department of Applied Solid State Physics. Dr. Gobrecht estimated that in his own area about one-third of the personnel budget originates from external funding, and about 50 people are involved in the nano field. The areas of research are organized into three categories as follows:

- Micro- and Nanostructuring Technology, which includes (1) electron beam lithography, (2) focused ion beam science and technology, (3) hot embossing lithography, and (4) LIGA. The first two areas are partially funded through the Swiss Priority Program (SPP) MINAST (Micro and Nanostructure Technology: with a four-year budget of ~ SFr. 48 million). Research area 3 is partially supported through the Swiss National Research Program (NFP 36) on nanosciences. Another subarea of research is titled "Zeolites as Materials for Nanodevices."
- Molecular Nanotechnology, which includes (1) biochemical recognition of individual molecules, (2) nanostructured electrodes for amperometric immunosensors, (3) immunosensor for penicillin in milk, and (4) neurite growth on biofunctionalized microstructured surfaces.
- Nanostructured Semiconductors Materials Research, which includes (1) near infrared Brillouin scattering, and (2) work on Si/Si/Ge and Si/Ge/C systems

Funding Profile

As mentioned above, the LMN participates in a number of national initiatives involving nanostructure science. *National Research Programs* (Nationale Forschungsprogramm, NFP) are directed towards solution of specific problems and are largely interdisciplinary in nature. In 1993, Nanosciences was chosen as a subject area of NFP 36 (1996-2001). The *Swiss Priority Programs* (SPP), were developed to ensure that strategic research in Switzerland is on a par with international research and that Swiss universities have the competence and financial means to play an important role in that research. SPPs are long term projects and can last eight to ten years, encompassing both basic research and practical problem solving.

RESEARCH AND DEVELOPMENT HIGHLIGHTS

Among the projects discussed in greater detail are the following:

- 50 nm plated metal structures by e-beam lithography
- stamping of 80 nm features in PMMA; also, fundamental studies of the polymer itself; possible application, heterogeneous catalysis
- nanoporous Au membranes for filtration: 100 nm Au membrane with 200 nm pores, 660 nm period, substrate etched off
- single-ion lithography to form nanoposts
- a-Si/a-SiN superlattice on single crystal Si: MBE/UHV CVD connected by a tunnel: recrystallize with emission in the blue
- biochemical sensor: immunological reactions taken on nanostructured surface; application: test for antibiotics in milk
- showed streptavidin immobilized on mica (see Figure B.1)

Dr. Thomas Jung described his work on the STM manipulation of molecules, rather than atoms, thus availing himself of preassembled building blocks. The building blocks can be prefabricated by chemical methods to possess specific structural, chemical, and physical properties. In addition to preassembly, a molecular approach has the advantage of being carried out at room temperature. The molecular building blocks considered include C_{60} (as was used to form the “abacus”), customized porphyrins, and bimolecular systems involving planar aromatic systems and fullerenes. In particular, Dr. Jung described work utilizing Cu-TBPP on Cu (100); experiments have elucidated molecular adsorption, surface movement, positioning, and stability of the assembled structures (for further information see Gimzerski et al. n.d.).

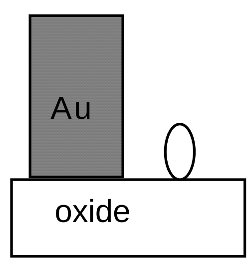

Figure B.1. Streptavidin immobilized on mica.

Team members also met with Drs. Werner Wagner and Helena Van Swygenhoven, members of the Department on Solid State Research at Large Facilities, associated with SINQ. In addition to providing support for outside researchers wishing to use the Spallation Neutron Source, members of this department also carry their own program of research in nanostructured materials, interacting broadly with a number of collaborators, both in Europe and the United States. Nanostructured materials are synthesized by inert gas condensation (IGC), and evaluations of such materials are made with respect to structural, magnetic (e.g., for Fe, Co, Ni), mechanical, and electronic (Pd, Pt) properties. Molecular dynamics computer simulations of structural and mechanical properties are carried out in order to explore conditions for improved materials properties, such as ductility or wear-resistance. Research is being undertaken with industrial collaborators in this last area (nanoscaled multilayered coatings for wear-resistant coatings).

In addition to SINQ, the Large Facilities Department of PSI makes available its IGC facility, hot compaction unit, indenter, X-ray diffraction and density measurements, small angle neutron scattering, prompt gamma activation analysis, TEM, and SEM with EDX. SQUID and NMR are carried out in the laboratories of collaborators at École Polytechnique Lausanne, and positron lifetime measurements are made at the University of Gent.

REFERENCE

Gimzerski, J.K., T.A. Jung, M.T. Cuberes, and R.R. Schlittler. *Scanning tunneling microscopy of individual molecules: Beyond imaging.*

Site: **Philips Research Laboratories**
Prof. Holstlaan 4
5656 AA Eindhoven, The Netherlands
Tel: (31) 40-27-42577; Fax: (31) 40-27-44282

Date Visited: 16 October 1997

WTEC: D.T. Shaw (report author), H. Goronkin, M.C. Roco

Hosts: Dr. L.G.J. Fokkink, Department Head, Physical Chemistry
Dr. Mark J. VanBommel, Senior Scientist in Inorganic Materials and Processing
Dr. A.W.M. (Ton) deLaat, Colloidal Chemist
Dr. Erik A. Meulenkamp, Research Scientist, Department of Physical Chemistry
Dr. Bianca M.I. van der Zande

BACKGROUND

Philips is one of the most important manufacturers of electronic consumer products in Europe, with annual sales of more than $40 billion. Roughly 70% of the Philips research comes from contracts from the company's product division; the rest is devoted to exploratory research, which typically does not lead to commercial products within three years. Among the exploratory research projects, an estimated 20% are related to nanoparticle/nanostructured materials. The laboratories have a wide spectrum of research activities that are of interest to this study, ranging from nanocrystalline materials synthesis, nanofabrication, and nanocrystal engineering to quantum transport in nanostructures and quantum theory of solids.

RESEARCH AND DEVELOPMENT HIGHLIGHTS

Dr. Mark J. VanBommel made a short presentation on nanoparticle coatings for consumer electronic applications (coatings for antiglare, antistatic, and antireflection uses). Depending on their specific applications, these nanoparticles need to have special properties (e.g., high electric conductivity, or low specular optical scattering). Antimony-doped tin dioxide ($Sb:SnO_2$) particles have been used for these applications, as an example. These nanoparticle coatings are typically produced by physical or chemical wet deposition processes. Spray pyrolysis, spinning, or dip coating techniques are usually carried out in a controlled ambient environment. To avoid dull-looking surfaces, the primary particle diameter is usually less than 2 nm. Figure B.2 shows an electron micrograph of these particles. The electrical conductivity can be regulated by controlling the antimony concentration in SnO_2.

Dr. A.W.M. (Ton) deLaat discussed briefly the production of nanometer-sized ceramic particles with very low sintering temperatures. Homogeneous green particles are important to prevent defects in sintered products; by using proper dispersants, dense and homogeneous layers have been obtained.

Dr. Erik A. Meulenkamp discussed size determination by absorbance measurements of ZnO nanoparticles and electrochemical properties of ZnO/ITO/glass electrode systems. ZnO nanoparticles produced by physical deposition are irradiated by light of various wavelengths. The shift of the absorbance peaks toward higher energy (or low wavelength) when the size of ZnO particles decreases (Figure B.3) clearly demonstrates the quantum size effect on bandgap energies. Figure B.4 shows the effects of electron accumulation for $E < -0.5V$ in a ZnO/ITO/glass electrode system.

Dr. Bianca M.I. van der Zande, who is on leave from Utrecht University, discussed the generation and optical properties of rod-shaped gold particles with diameters ranging from 10 to 30 nm. Aqueous dispersion of rod-like gold particles is obtained by electrode position in nanopores of anodized alumina. In the VIS/NIR absorption spectra, two absorption maxima are observed: one corresponds to the transverse plasma resonance, and the other to the longitudinal plasmon resonance, which moves to higher wavelengths when the particle aspect ratio is increased (Figure B.4).

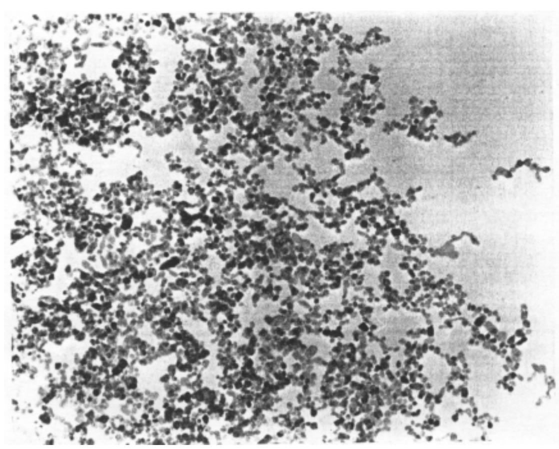

Figure B.2. Electron micrograph of antimony-doped tin dioxide particles (primary particle size ~20 nm).

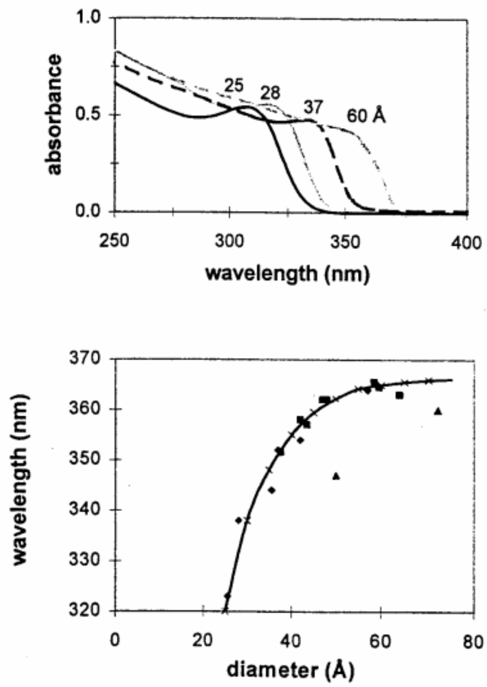

Figure B.3. Quantum size effect on the absorbance of ZnO.

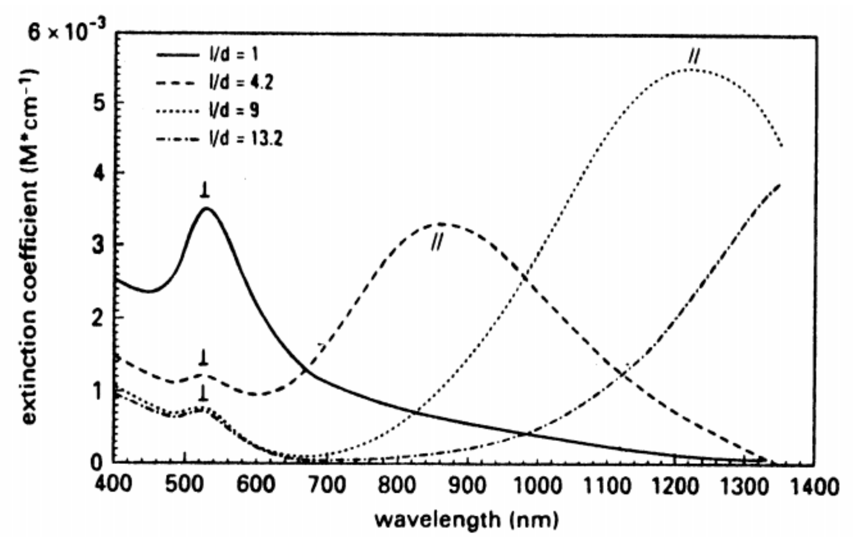

Figure B.4. Normalized experimental VIS/NIR absorption spectra of rod dispersions with aspect ratio L/d = 1 (spherical gold sol), L/d = 4, L/d = 9, and L/d = 13.

CONCLUDING REMARKS

From published papers, it is evident that Philips researchers are active in many other topics in nanostructures, including template synthesis of nanowires in porous polycarbonate membranes, self-assembled monolayers of metallic nanoparticles, and luminescence-tuning in semiconducting nanocrystallines.

Site: **Darmstadt University of Technology**
Department of Materials Science
Thin Films Division
Petersenstrasse 23
D-64287 Darmstadt, Germany
Tel: (49) 6151-16 6306; Fax: (49) 6151-16 6335
http://www.th-darmstadt.de/ms/fg/ds

Date Visited: 14 October 1997

WTEC: J. Mendel (report author)

Hosts: Prof. Dr. Horst Hahn, Head, Thin Films Division, Materials Science Department
Dr. Markus Winterer, principal contact

BACKGROUND

The Darmstadt University of Technology has recently focused on nanomaterials. Its new building for material science was completed in 1992 and was officially dedicated in 1996. Here work includes particles, films, coatings, and bulk phase. Interest is in pores, grains, and clusters. Preparation methods can include gas vapor phase condensation, chemical vapor deposition, liquid plasma studies, chemical deposition, and sputtering. Electrical, mechanical, and spectral properties are all of interest.

There are 22 scientists in Professor Dr. Hahn's group. Interaction with industry and other technical centers occur frequently.

RESEARCH AND DEVELOPMENT HIGHLIGHTS

1. Preparation of SiC by chemical vapor synthesis (Sylkie Klein). This work emphasizes both high purity and high production rates.
2. Synthesis of ZrO_2/Al_2O_3 by chemical vapor synthesis (Vladimir Srdic). Beginning with aluminum alkoxide, particles are formed in the 4-9 nm size range. Flow rate of helium influences properties.
3. Impedance spectra on nano Y-stabilized zirconia (Pia Mondal). Powders are prepared by inert gas condensation. The lattice conductivity appears to be independent of grain size.

4. Synthesis and characterization of nanofilms with chemical vapor deposition and synthesis (Stefan Siegfried). Both boron carbide and silicon carbide are deposited on a silicon substrate.
5. Synthesis of metal-matrix composite coatings (Andreas Moller). Results lead to improved hardness, wear resistance, and thermal stability.
6. Very thin films (Stefan Flege). This work involves layer growth at 3,000 degrees Celsius for vapor metal/ceramics applications.

ANALYTICAL EQUIPMENT

- Mechanical Creep Device
- HRTEM
- IR
- Diffractometer
- Molecular Beam Instrumentation
- Low Pressure Flow Chemical Vapor Synthesis
- SIMS
- Electron Probe
- XRD
- SEM
- TEM
- Sputtering Equipment

FUNDING PROFILE

About 60-75% of the equipment is paid through state support. Additional funding takes place through industrial collaboration.

CONCLUDING REMARKS

The Thin Films Lab at the University of Darmstadt is strongly positioned for preparation and characterization of nanoparticle systems. Their work reflects the University's commitment to expand the understanding needed for particles, films, coatings, and bulk phase.

Site: **Delft University of Technology (DUT)**
Faculty of Chemical Technology and Materials Science
Delft Inst. of Microelectronics & Submicron Technology
Rotterdamseweg 137
2628 AL Delft, The Netherlands
http://www.stm.tudelft.nl/ or http://dimes.tudelft.nl

Date Visited: 16 October 1997

WTEC: D.T. Shaw (report author), H. Goronkin, M.C. Roco

Hosts: Prof. Brian Scarlett, Department of Chemical Engineering
Dr. Cees Dekker, Faculty of Applied Physics

BACKGROUND

The nanoparticle/nanostructured materials/nanodevices research activities at the Delft University of Technology (DUT) reside primarily in the Faculty of Chemical Technology and Materials Science (STM) and Delft Institute of Microelectronics and Submicron Technology (DIMES). STM is one of the more research-oriented faculties of DUT, which is the largest, oldest, and most complete technical university in the Netherlands. About 40% of all PhD degrees awarded at the University have been in STM. Among the 3 research areas the program covers (biotechnology, chemical engineering, and materials science technology), the WTEC visit concentrated primarily on chemical engineering and materials science technology.

DIMES is a large national research center in microelectronics managed by the University. Funded by the government and accredited by the Royal Netherlands Academy of Arts and Sciences, DIMES was created in 1987 as a national facility for the fabrication of advanced integrated circuits and nanostructured devices. Its 300-odd staff members and graduate students actively explore the microelectronics field; studies range from carbon nanotube electronics to nanofabrication of single-electron tunneling devices. In addition to its vast (2,000 m^2) clean-room fabrication facilities, the center is also a training ground for scientific specialists in advanced microelectronics in the Netherlands. Since its inception, more than a hundred PhD students have finished their dissertations using DIMES facilities. The Institute offers some twenty graduate courses, as well as extensive training support for research students. It coordinates its graduate education programs with the University’s academic faculties, including the Faculty of Chemical Technology and Materials Science.

RESEARCH AND DEVELOPMENT HIGHLIGHTS

The Faculty of Chemical Technology and Materials Science (STM)

STM has strong programs in both nanoparticle generation and functional materials synthesis. Several professors in STM are active in the organization of an European Science Foundation program on Vapor-Phase Synthesis and Processing of Nanoparticle Materials. The principal objective of the program is to promote interdisciplinary collaboration between the leading research groups in Europe in aerosol and materials science. The program, with an annual budget of about FFr. 1.5 million (for meetings, workshops, and information dissemination), is coordinated by J. Schoonman of DUT and Prof. H. Fissan of the University of Duisburg, Germany.

Two major groups are engaging in nanoparticle/nanostructured materials research. One is led by Prof. J. Schoonman and the other by Prof. B. Scarlett, who was the WTEC team's host for the visit. Prof. Scarlett has several projects in nanoparticle technology, including the production of nanoparticles by electrospraying, electrostatic charging for micromixing, and nanoparticle formation in a laser-heated aerosol reactor. His group also developed photon correlation spectroscopy as a means to measure submicron particles in a gas, in situ. His coworker, Dr. J.C.M. Marijnissen summarized recent results on the generation of metal-oxide particles by bipolar mixing techniques. Different precursors (i.e., titanium tetrabutoxide, zirconium tetrabutoxide, etc.) have been used to generate metal-oxide particles. A project in collaboration with Prof. Schoonman concerns the generation of nanoparticles by laser-induced chemical vapor precipitation (LCVP). Although the LCVP technique is not new, the project's data in particle-generation parametric optimization are useful in scale-up production of Si, SiC, Si_3N_4, and SiC_xN_y nanoparticles. The research activities of Prof. Schoonman, have focused also on the synthesis of custom-designed structural and functional nanostructural materials. For example, his coworker Dr. A. Goossens reported the use of an electrostatic spray pyrolysis technique for the deposition of dense or nanoporous ceramic thin films for lithium batteries and other energy-related applications. A great variety of metal oxide nanocrystalline thin films have been synthesized, including TiO_2 and Li CoO_2. STM researchers have developed a technique called laser particle precipitation-aided chemical vapor deposition (LPPCVD), which produces thin films with very low substrate temperatures. Typically, the nanoparticle size is in the range of 10 to 30 nm.

Delft Institute of Microelectronics and Submicron Technology (DIMES)

Dr. Cees Dekker (Faculty of Applied Physics) made a short presentation on research activities in the area of quantum transport through nanostructures. Prof. Mooij (who was touring the United States at the time of our visit) heads the research program supported by DIMES. Four projects were discussed briefly:

1. Junction Arrays (Project Director, Hans Mooij) and Single Electronics (Project Director, Peter Hadly). The quantum behaviors of small circuits of superconducting tunnel junctions were studied experimentally and theoretically. A quality sample was designed and fabricated, in which quantum superpositions of charge states, as well as vortex states, have been experimentally observed. Quantum vortices were studied in one-dimensional arrays. Disorder was seen to lead to localization, while in periodic superlattices the vortices maintained their mobility. Fluctuations were studied in normal metals near a tunnel barrier.

In single electronics, there are three main efforts: (1) fabrication of small junctions for the study of charging effects, (2) understanding of high-frequency behavior of single-electron tunneling (SET) transistors, and (3) characterization of single-electron circuits. Figure B.5 shows an RS flip-flop consisting of four SET transistors, each with three gates fabricated by the group.

2. Quantum Dots (Project Director, Leo Kouwenhoven). Transport experiments on quantum dots were performed on a gated device, as shown in Figure B.6. Measurements of gate voltages vs. source-drain voltages show a shell structure corresponding to a 2-D harmonic confinement potential in normal atoms. Staff observed that the filling of a shell occurs according to Hund's rule: electrons occupying degenerate states prefer to have parallel spins, which lowers the total energy due to an increased exchange interaction.

3. Single Molecular Wires (Project Director, Cees Dekker). Single-wall carbon nanotubes were obtained from R.E. Smalley at Rice University for transport measurements. The nanotubes behave as coherent quantum wires at the single-molecular scale. The density of states appears to consist of well-separated discrete electron states. The approximate 0.4 meV energy separation corresponds to estimated 1-D quantum box where a 3 μm long nanotube constitutes the electron box. Electrical conduction through these discrete electron states appears to occur quantum coherently over micron-length distance.

For transport measurements of single metal nanoclusters, DIMES researchers showed results made on a 20 nm Pd cluster, which was trapped electrostatically between two nanometer-sized electrodes (Figure B.7).

4. NEXT Nanolithography (Project Director, Bart Geerligs). The Nanoscale Experiments and Technology Project is based on a facility for the fabrication and study of sub-10 nm structures. The project studies mesophysics on 10 nm to atomic-size structures and assesses the applicability of these mesophysical phenomena to future electronic devices. Main features characteristics of the NEXT system are: (1) working in an uninterrupted, clean, ultrahigh-vacuum environment, and (2) using maskless fabrication techniques based on scanning tunneling probes. On-going experiments in the facility include quantum electronic studies of metal quantum dots, 1-D mesoscale systems, and transport in molecules.

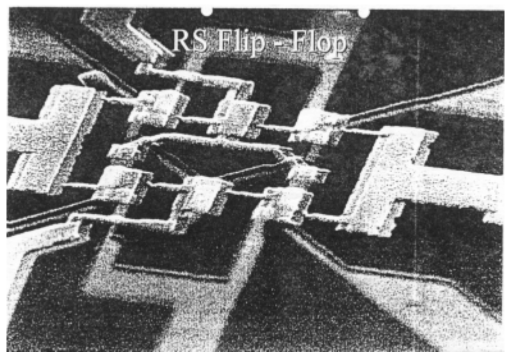

Figure B.5. RS flip-flop of four SET transistors, each with three gates.

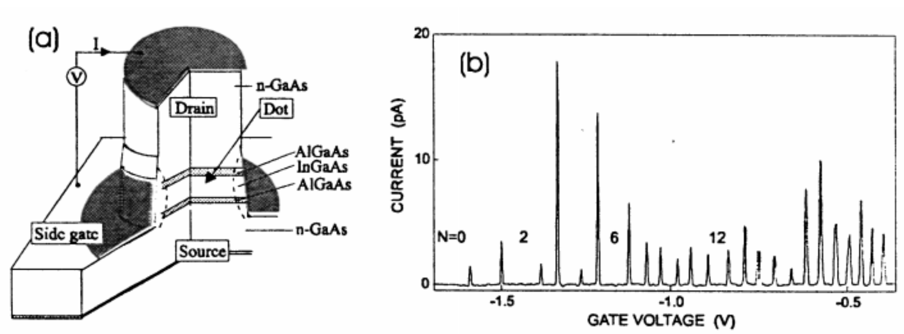

Figure B.6. (a) Schematic diagram of the gated quantum dot device and (b) the Coulomb oscillations in the current vs. gate voltage at B = 0T observed for a 0.5 μm diameter dot.

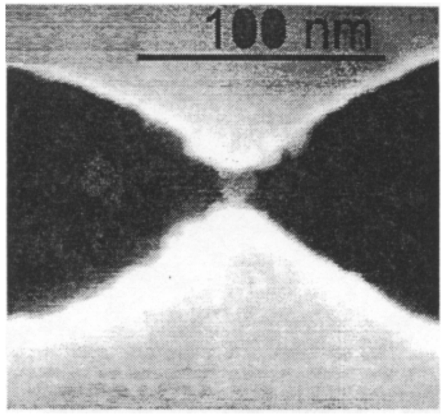

Figure B.7. Single Pd colloid cluster of 20 nm diameter that has been deposited between two nanoelectrodes.

CONCLUDING REMARKS

The research facilities in nanostructured materials at Delft University of Technology in general, and at DIMES in particular, are very impressive. Projects are carried out with close collaboration between researchers from industry and university. For many projects at DIMES it is common to have several industrial sponsors. Since the research is generally precommercial in nature, issues about intellectual property appear not to be a problem for these multi-industrial sponsorships.

Site: **University of Cambridge**
Cavendish Laboratory
Toshiba Cambridge Research Center (TCRC)
Madingley Road
Cambridge CB3 0HE, United Kingdom
http://www.phy.cam.ac.uk/ (Cavendish Laboratory)

Date Visited: 15 October 1997

WTEC: J. Mendel (report author)

Hosts: Prof. Michael Pepper, Director, TCRC
Dr. Neil Greenham, Conjugated Polymers and Molecular Solids Group (Cavendish Laboratory)
Dr. Andrew Shields, TCRC
Dr. Mark Leadbetter, TCRC

TOSHIBA CAMBRIDGE RESEARCH CENTER (TCRC)

Overview of the Center

The Toshiba Research Center (TCRC) was founded by the Toshiba Corporation of Japan as a laboratory pursuing fundamental research into the physics of semiconductor structures and associated topics. The intent is to provide an important part of electronics technology for the next century. This includes development of collaborative projects with academic institutions in the European community.

Accordingly, there is collaborative research with the Cavendish laboratory of Cambridge University on advanced growth and fabrication techniques. The Center maintains a close relationship with Toshiba Research and Development Center in Japan, and there are frequent reciprocal visits between the two centers.

Most research is for a 5- to 10-year horizon. Present staff totals about 18. All research is on semiconductors, on which ~ 50 papers have been published since 1991, the year the center was established. The work includes papers on electrical properties of quantum well structures where the electron wave function is controlled by application of a voltage to a controlling gate leading to a velocity modulated transistor. The center has a full in-situ cleaning chamber, which is used prior to regrowth studies.

Studies of electron propagation on curved and nonplanar surfaces are also taking place, partly as a result of observing the improved electrical properties of advanced structures. Accordingly, the need to develop an optical facility became readily apparent. Surface orientation as a function of a magnetic field has led to the field taking on different values; electron deflection can take place as a result of the electron velocity in the magnetic field.

Extensive investigations into infrared properties as well as excitonic effects led to the discovery of the positively charged exciton. Included here were investigations on both positive and negative properties of excitons.

Funding Profile

Funding is by the Toshiba Corporation.

Research and Development Highlights

- quenching of excitonic optical transistors by excess electrons in GaAs quantum wells
- one-dimensional wire formed by molecular beam epitaxial regrowth on a patterned pnpnp GaAs substrate

Concluding Remarks

The Toshiba Center is ideally positioned to explore electrical and quantum phenomena for semiconductor technology for the next century, due to its location, expertise of technical resources, and close association with collaborative communities such as Cavendish. Its path forward appears rewarding and certain.

CAVENDISH LABORATORY

Dr. Neil Greenham, associated with Prof. Richard Friend in the Conjugated Polymers and Molecular Solids Group, was the principal contact for this visit.

Overview of the Laboratory

Experimental and theoretical research in the Department of Physics at Cambridge is carried out in the Cavendish Laboratory. The laboratory was established 120 years ago by individuals such as Maxwell, Rayleigh, Thomson, Rutherford, and Bragg. From this noteworthy beginning the laboratory has evolved into three major sections: Condensed Matter Physics, Radio Astronomy, and High Energy Physics.

The funding for the investigations at the laboratory come from a combination of government and industry. There are currently some 63 companies that sponsor research at Cavendish.

Research and Development Highlights

The following areas of research are active in the area of optical and electrical studies:

- characterization of conjugated polymers
- Electrical and electro-optical properties of polymer semiconductor devices
- optical excitations of conjugated polymers and related materials
- optical microcavities
- optoelectronic properties of semiconductor nanoparticles

Discussion

Conjugated polymers with delocalized electron systems behave as model organic semiconductors. Here activities such as design and synthesis of new polymers are of interest for the semiconductor physics of these materials. They give strong electro-optical and nonlinear optical responses. There is concern for polymer light-emitting diodes as well as photovoltaic and photoconductor diodes. Subpicosecond time-resolved spectroscopy is included also. There is considerable collaboration with the Chemistry Department in order to tailor these materials to the appropriate properties needed for these studies.

Equipment at Cavendish

- X-ray diffractometer
- Low energy electron diffraction
- High resolution electron energy loss measurements
- Scanning tunneling microscope
- Optical spectrometers
- Fourier infrared spectroscopy
- Ion beam & electron beam lithography
- Atom scattering facilities
- Electron microscopy
- Angle-resolved photoemission
- Tunable dye lasers
- Laser Raman
- Molecular beam epitaxy

HITACHI CAMBRIDGE LABORATORY

Although this site was not visited, it is included in the report for the reader's interest (see also http://www-hcl.phy.cam.ac.uk/).

Key Personnel: Prof. H. Ahmed, Microelectronics Research Center
Staff: Six Post Doctorates, 17 Research Students

Overview of the Laboratory

The purpose of this laboratory is to carry out research into physics and fabrication of novel electronic devices. Activities include

- extensive electron beam lithography
- focused ion beam implantation
- electron-beam-assisted deposition
- thin film processing
- collaboration with Cambridge Physics Lab

Discussion

The Microelectronics Research Center has regular collaboration on quantum effect devices with the Cambridge Physics Laboratory. There is extensive equipment sharing. For example: a femtosecond laser system, an ultra-low temperature scanning tunneling microscope and a powerful system are shared for purposes of characterization on structures that are fabricated in the Center.

Also, a newly organized group within the Center is investigating sensor structures including an infrared sensor based on free-standing micro-thermocouples. Work is in process on microsensors in silicon and GaAs and on single-molecule sensors.

Site: **University of Oxford**
Department of Materials
Parks Road
Oxford OX1 3PH, United Kingdom
http://www.materials.ox.ac.uk

Date Visited: 16 October 1997

WTEC: R.W. Siegel (report author), C. Koch

Hosts: Prof. Brian Cantor, Cookson Professor of Materials, and Head of Department; Fax: (44) 1865-273738; E-mail: head.department@materials.oxford.ac.uk
Prof. David Pettifor, Isaac Wolfson Professor of Metallurgy, and Director, Materials Modelling Laboratory
Dr. Brian Derby, Director, Oxford Centre for Advanced Materials and Composites
Dr. Patrick Grant
Dr. Amanda Petford-Long
Dr. Kenya A.Q. O'Reilly
Dr. Alfred Cerezo
Dr. Paul J. Warren
Dr. Steve Roberts
Dr. John L. Hutchison
Prof. Peter J. Dobson, Department of Engineering Science
Prof. Malcolm L.H. Green, Head, Department of Chemistry, Inorganic Chemistry Laboratory
Prof. H.A.O. Hill
Prof. R.G. Denning
Prof. D. O'Hare
Prof. Paul Madden, Physical and Theoretical Chemistry Laboratory
Dr. R.K. Thomas
Dr. C.D. Bain
Dr. F. Marken

BACKGROUND

The full day of 16 October from 9:30 to 15:30 was spent at the University of Oxford visiting the Head of the Department of Materials, Prof. Brian Cantor, and various other members of the Department. It was a very interesting visit, with much to hear about in the area of nanostructure science and technology at this prestigious university. An additional one-hour visit at the end of the day was made to a group of faculty from the Department of Chemistry, Inorganic Chemistry and Physical and Theoretical Chemistry Laboratories. Although brief, the visit was quite effective, since it was held in a very informative roundtable format well organized by Prof. Malcolm L.H. Green and Prof. Paul Madden.

According to an initial overview of the University of Oxford and its Department of Materials presented by Prof. Cantor, the Department currently consists of about 330 people in total, with 20 academic staff and about 30 support staff, 80 research fellows and visitors, 80 postgraduate students, and the remainder undergraduate students. The Department's current annual research budget of ~£3 million, comes two-thirds from government and one-third from industry, with about 50% of this focused in nanoscale research activities. The research in general is quite broad-based and includes processing, characterization, and modeling activities in all classes of materials: metals, ceramics, polymers, semiconductors, and composites (see references list). The Department also houses the Materials Modelling Laboratory (Prof. David Pettifor, Director) as well as the Oxford Centre for Advanced Materials and Composites (Dr. Brian Derby, Director). In the important area of nanostructure characterization, the very impressive electron microscopy facilities here are particularly noteworthy, owing in part to the tradition left by Professor Sir Peter Hirsch, as are those in atom-probe field-ion microscopy. A series of visits with individual department staff ensued.

RESEARCH AND DEVELOPMENT HIGHLIGHTS

Dr. Patrick Grant reviewed the department's ongoing work in nanocrystalline sprayed coatings. This work utilizes a variety of spray processing methods including the Ospray process, electric-arc spraying, plasma spraying, and ink-jet spraying. He described some examples in more detail. For example, plasma spraying of titanium along with 100 µm diameter SiC fibers yielded multilayered structures with a matrix having a nanoscale substructure, owing to the rather stochastic process of oxide contamination. Work was planned to soon begin on spraying 600 nm diameter Al_2O_3 particles and 200 nm diameter SiC feedstock, as the research moved more toward the true nanoscale regime.

Dr. Amanda Petford-Long then described her work on nanolayered magnetic thin films. The primary focus of the work is on magnetic recording materials at the nanoscale. A significant collaboration is ongoing between Dr. Petford-Long at Oxford, Prof. Ami Berkowitz at UCSD, and Hewlett-Packard, Palo Alto, who together are funding three post-doctoral researchers in electron microscopy, band-structure modeling, etc., mainly on spin-valve materials. These are made by spraying multilayered films, such as NiFe/Cu/Co/NiFe/MnNi. Additional funding comes from small companies in the UK and the Science Research Council. Also described were unique capabilities for mapping local magnetization in devices using Lorentz transmission electron microscopy. They are also working on nanocomposite optical films, such as Bi nanoclusters in an amorphous Ge matrix, made by pulsed laser ablation techniques.

Dr. Kenya A.Q. O'Reilly then described her work on the nucleation of nanocrystalline secondary phases and the heterogeneous nucleation of droplets on matrix surfaces studied by TEM in various alloy systems. Melt spinning is used to produce composites of low and high melting point materials. The method is apparently limited to about 20 nm diameter particles, for example with Pb in Al. Melting (freezing) is observed via differential scanning calorimetry (DSC) at different cooling rates to examine kinetics. Studies of Al/Al_3Zr sponsored by ALCAN have shown that small particles melt first and then interface regions. The excellent electron microscopy facilities in the Materials Department (consisting of ~ 14 instruments, presently) are used in this work. A new high-resolution FEG-TEM will be added in a few months to upgrade further these facilities.

Next, Dr. Alfred Cerezo and Dr. Paul J. Warren reviewed some of their current work involved with investigations of nanocrystalline and amorphous materials, mainly alloys, using atom-probe field-ion microscopy and high-resolution TEM. The position-sensitive atom-probe (PoSAP) field-ion microscope was pioneered in this group and represents the ultimate in combined structural and chemical nanoscale analysis, since it has atom-by-atom sensitivity. Kindbrisk, Ltd. in Oxfordshire has commercialized the instrumentation through a technology transfer arrangement with the University. A variety of phase decomposition studies are being carried out in order to develop an understanding of the mechanisms and effects of different such processes in nanocrystalline materials, including Al-based (high strength), Fe-based (soft magnetic), and Zr-based (high strength) alloys. Pulse electroplated Ni-Fe alloys have also been investigated because of their interesting increased hardness and improved magnetic properties.

Dr. Steve Roberts discussed the ongoing research in the Department on nanocrystalline ceramics and ceramic-matrix composites, the latter with Dr. Brian Derby. Most of this work is being carried out on the Al_2O_3/SiC system containing ~ 5% SiC particles, similar to the materials studied by Prof. Koichi Niihara in Osaka. The work is funded by BRITE/EURAM and the Engineering and Physical Sciences Research Council (UK) with some inkind support from UK industry. Using 30-40 nm SiC particles well dispersed (intra- or inter-granularly) in 3-4 µm grain size Al_2O_3 yields a factor of 2 increase in strength (cf. the factor of 4 increase found by Niihara's group at Osaka), but a similar fracture toughness and 2-3 times greater wear resistance compared with conventional Al_2O_3. This material is found to be effective as a grinding medium, and good interactions with industry in the UK and abroad have resulted.

Dr. John L. Hutchison then discussed his work on supported metal catalysts and also on in situ high-resolution electron microscopy (HREM) observations of filling carbon nanotubes (multiwalled tubes now, but starting on single-walled tubes) with metals via reduction in the microscope. Dr. Hutchison is the director of the HREM facility, which has an environmental cell that has 0.25 nm resolution up to 20 mbar pressure and 500°C. He is also working on WS_2 and other related fullerene-like sulfides and selenides in collaboration with a group at the Weizmann Institute. These are found to give excellent lubricity in oil suspensions, since they apparently roll and don't slide.

The WTEC team's next visit on a very busy and interesting day was with Prof. Peter J. Dobson, Department of Engineering Science, who described the wide range of activities in nanoparticles and nanocomposites in his Department, much of it done in collaboration with colleagues in the Departments of Materials and Chemistry. They create a variety of nanoparticles via several methods, including colloidal, aerosol, gel/aerosol, sputtering, gas evaporation, and electrochemical routes. These nanoparticles (e.g., CdS, CdSe, ZnS, Ag/ZnO, etc.) are generally dispersed in a matrix to make a "high technology" paint or coating with a specific functionality. For example, semiconductor quantum dots with narrow size distributions for use as light emitters are being dispersed in a glassy or polymeric matrix to develop new display technology. Surface capping of the nanoparticles is also being investigated in order to optimize and control their dispersion and properties.

Finally, the last hour of the visit was spent with a group of eight faculty from the Department of Chemistry, Inorganic Chemistry and Physical and Theoretical Chemistry Laboratories in an informative roundtable format that was well organized and briskly guided by Prof. Malcolm L.H. Green, Department Head, and Prof. Paul Madden. First, Dr. R.K. Thomas spoke of neutron scattering studies of interfaces, for example the surfactant layer at an aqueous/silica surface. The interaction appears to be independent of silica particle size down to 5 nm, the smallest size looked at. Next, Dr. C.D. Bain described nonlinear optical studies related to tribology, work on crystal growth, and dissolution, as well as confined molecules trapped between glass ("lens") surfaces. Dr. F. Marken then discussed his work on electrocatalysis using small particles, emulsions in aqueous media. *Ab initio* molecular dynamics simulations of Na clusters in zeolite Y were then described by Prof. P.A. Madden in terms of what the Na cluster looks like in the super cage after the sodalite cages are filled. Grain boundary migration in Na with symmetric tilt boundaries was also being simulated and could be followed for tens of picoseconds at two-thirds T_M using a simpler molecular dynamics (MD) approach than Car-Parrinello with Kohn-Sham for materials, such as Na, with spherically symmetric bonding. Prof. H.A.O. Hill then presented his work on nanoelectrodes for sensor applications in which a different enzyme could be placed on each nanoelectrode. Profs. Dobson and Hill and others are now using carbon nanotubes with redox proteins in a tube of 3 nm radius for such sensors. Results of a new project on nanostructured polymers were then described by Prof. R.G. Denning in which one-, two-, and three-dimensional nanostructures were being created using optical interference methods. It is planned to fill the ordered voids created in these polymers with TiO_2 or other high refractive index materials. Prof. M.L.H. Green then spoke of his elegant results with filled carbon nanotubes opened by reduction with Nd_2O_3, $FeBiO_3$, or MoO_3, for example. Dr. D. O'Hare ended this session with a discussion of mesoporous silicates used for nanochemistry with organometallic catalysts. The general issue was then raised about funding for nanoscale science in the UK and, while EPSRC has an initiative in microstructure materials, it was felt that the monies were small and insufficient, with money for people and equipment easier to obtain than actual research support. It was perceived that even EPSRC is now being focussed toward "wealth creation" and the situation appears that it is not going to be getting better.

At the end of this long and interesting day, it was very clear that the time allowed could not possibly do justice to all of the excellent work being done on nanostructure science and technology at Oxford, and that an hour with the Chemistry Department could really at best only whet one's appetite.

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Date of Visit:	14 October 1997
WTEC:	D. Cox (report author)
Hosts:	Prof. Dr. Manfred Kappes, Physical Chemistry Prof. D. Fenske, Organic Chemistry Prof. H. von Löhneysen, Physics

BACKGROUND

The University of Karlsruhe has a strong effort in nanoscale science and technology. The effort encompasses several research groups in both the Chemistry and Physics Departments, as well as collaborative efforts with the Forschungzentrum Karlsruhe (FzK). The effort has both a strong experimental component and a strong theoretical component.

At present the formal structure is best exemplified by the two Sonderforschungbereiche (SFB) programs at the university. The earliest one SFB-195 was started in 1992 and has a focus on electron localization. The present coordinator of SFB-195 is Prof. H. von Löhneysen, a professor in the Physics Department. Beginning in 1998 Prof. Dr. Manfred Kappes in the chemistry department will assume the role of coordinator. The focus of the program is on electron localization in macroscopic and microscopic systems, including clusters and cluster complexes. This is a multidisciplinary effort encompassing many research groups at Karlsruhe. Some of the groups participating in this program are those of

Prof. Dr. M. Kappes	Clusters, Physical Chemistry
Prof. D. Fenske	Semiconductor Cluster Complexes, Inorganic Chemistry
Prof. R. Ahlrichs	Theory
Prof. Freyland	Electron localization in metal salt systems
Prof. Hippler	Femtosecond spectroscopy of solvated electrons
Prof. H. von Löhneysen	UHV scanning probe techniques, Defect structures in Si
Prof. Schimmel	Fabrication and scanning probe techniques

The total effort is estimated at about 70-90 researchers, including postdoctoral and PhD students.

In addition to SFB-195, the Karlsruhe group has received approval for a second SFB commencing January 1998. This new SFB will obtain funds, initially ~ DM 2 million/year for three years, to pursue research on carbon materials. Again a strong multidisiplinary approach is evident, with groups from Physics, Chemistry and Engineering contributing. The goal of the new effort will be to understand carbon-fiber-reinforced materials. The approach will be multifaceted with studies of carbon deposition from the gas phase, chemical vapor infiltration and chemical synthesis of carbon structures including materials aspects of carbon nanotubes and fullerenes, gas phase kinetics and surface radical interaction on carbon surfaces to understand the fundamental growth mechanisms, and solid state physics of nanotubes and carbon materials for potential nanoelectronic properties. Some of the groups involved include those of Kappes, Fenske, Hippler, Hüttinger, Ahlrichs, and von Löhneysen at the University, and Rietschel at the FzK.

RESEARCH AND DEVELOPMENT HIGHLIGHTS

The WTEC team held individual discussions with Prof. Kappes, Prof. Fenske, and Prof. von Löhneysen, and visited the laboratories of Professors Kappes and von Löhneysen. It is clear from the discussions that nanoscale science is a high priority area in Karlsruhe, both at the university and at the FzK (Prof. Dr. H. Gleiter). As examples of efforts in this area, the research activities in the three groups we visited are summarized below.

Research in Prof. Kappes' Group

At the present time Prof. Dr. Kappes has a group of 12, consisting of 1 postdoc and 11 PhD students. The main focus of the research is to understand several fundamental physical, chemical, and electronic properties of metal and carbon (fullerene) clusters using spectroscopic and beam techniques. Efforts are focused on

1. photodissociation probes of mass selected transition metal clusters and derivatives
2. studies of charge separation and chemi-ionization processes in thermal energy collisions of alkali clusters with various molecules (e.g., O_{29}, C_{l29}, C_{60}...)
3. isolation and characterization of larger fullerenes, endohedral metallofullerenes, and fullerene derivatives

4. cluster ion-surface scattering including the determination of fragmentation, delayed ionization, and neutralization rates upon collisions to obtain activation energies
5. probes of cluster ion penetration into HOPG, including tailoring of surface morphologies by controlled etching of nanometer-sized impact defects

The laboratory is well equipped, having several molecular/ion beam systems, many different laser systems for spectroscopic and particle generation, ultrahigh vacuum (UHV) STM for surface studies, Ti-Sapphire laser-based Raman spectrometer, HPLCs for fullerene extraction, and purification, among other equipment.

In addition to its experimental effort, the Kappes group collaborates with the theory group of Prof. Ahlrichs. Each PhD student may be expected to spend about one-quarter of his/her time performing theoretical studies, perhaps calculating electronic spectra of fullerenes, alkali clusters or transition metal clusters, properties of isomeric structures, and/or model spectra, e.g., IR or Raman, for many of the new neutral and ionic species being studied.

Research in Prof. Fenske's Group

Prof. Fenske's group consists of about 15 PhD students and has historically focused on synthesis and structure/X-ray, primarily of new metal calcogenide molecular clusters. In addition to the effort on synthesis, more recent studies are now directed to probing the stability of the ligand-stabilized clusters, via ligand alteration and cluster size and composition. One goal is to synthesize molecular clusters of well-defined size and geometry in order to investigate quantum confinement in such species. Recent successes are in the area of copper selenium molecular clusters stabilized by the protective ligand field of $(PEt_2Ph)_x$. As an example, molecular clusters with cores of $Cu_{20}Se_{13}$, $Cu_{44}Se_{22}$, up to $Cu_{70}Se_{35}$, have been synthesized and characterized. The structures of the clusters smaller than $Cu_{70}Se_{35}$ are found to be spherical, whereas the structure of $Cu_{70}Se_{35}$ is pyramidal. The color of the material depends on the cluster's size. For nanoscale technology, the $Cu_{70}Se_{35}$ is found to be metastable. It decomposes under vacuum into smaller $Cu_{2x}Se_x$ clusters. When a sheet is coated with $Cu_{70}Se_{35}$, a nearly uniform coating of smaller clusters of $Cu_{2x}Se_x$ (quantum dots) is formed, thus creating a 2-D array of quantum dots. The next step—not a trivial one—will be to form interconnects. One thought is to use graphite surfaces using the beam techniques developed by Prof. Kappes. The semiconducting cluster complexes can then stick to the graphite surface at the defect site.

Research in Prof. von Löhneysen's Group

Prof. H. von Löhneysen in the Physics Department has a fairly large group of 20, with 5 postdocs and 15 PhD students. He has a well-funded operation and commented that funding in Germany may still be better than in the United States. However, he also felt that too much time has to be spent to get funding. He commented that the University of Karlsruhe strongly supports nanotechnology. He has strong interests and efforts in electron beam lithography, break junctions, metallic nanostructures, low temperature physics investigating nanostructures and thin films, metal insulator transitions, and magnetism/superconductivity. Research on break junctions is directed towards fabricating and characterizing nanometer structures with few atom contacts. Contacts are broken and then brought back together in a controlled fashion so that current voltage characteristics can be probed. A tour of his laboratories confirmed that support for equipment is certainly adequate, with strong capabilities in e-beam lithography, UHV STM, and 20 mK dilution refrigeration for low temperature physics studies.

Prof. H. von Löhneysen also outlined some of the other efforts in the Physics Department with some emphasis on nanoscale science. Prof. T. Schimmel uses STM treatments of surfaces to micromill and manipulate nanometer structures on surfaces, such as fabrication of small junctions. Prof. P. Wölfle carries out theoretical studies of phase coherence, and Prof. G. Scöhn is developing the theory for a single-electron current standard.

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BACKGROUND

The University of the Saarland has a strong focus on nanotechnology. Of particular interest are the following areas of investigation within the Department of Technical Physics:

1. Inert gas condensation processes to produce nanostructured powders
2. Determination of grain size distribution in nanomaterials from X-ray diffraction profile analysis
3. Nanocrystalline metals and oxides by reverse microemulsions
4. Nanocrystalline metals and oxides with pulsed electrodeposition
5. Characteristics of one-dimensional tunnel junction arrangements
6. Size control synthesis of $BaTiO_3$ by sol-gel hydrolysis
7. Nanocrystalline copper by pulsed electrodeposition
8. Muon diffusion in nanocrystalline copper
9. Growth kinetics of nanocrystalline CuTi
10. Implantation of ions on the surface initiation of reactions with metal alkoxides
11. Characterization of nanoparticle surfaces
12. Use of photoelectron microscopy for resolving surfaces
13. Ultrasonic energy for creating nanoparticles

RESEARCH AND DEVELOPMENT HIGHLIGHTS

The inert gas condensation method has been optimized by using an aerosol flow condenser. Characterization of product resulted in an 80% reduction in the primary particle mean diameter and 17% reduction in geometric standard deviation. Flow conditions also determined the primary particle diameter and the geometric standard deviation.

There is considerable investigation on the synthesis of nanocrystalline metal oxides and the formation of NH_4MnF_3 by microemulsion techniques. Size and distribution of the primary reverse micelles were determined by dynamic light scattering.

An analytical approach is now available for determining the Coulomb blockade and single electron tunneling phenomena for arbitrary tunnel junctions coupled in series.

Size controlled synthesis of nanocrystalline $BaTiO_3$ by a sol-gel type hydrolysis is currently under study in a microemulsion nanoreactor.

EQUIPMENT

- Small Angle Scattering
- NMR
- GC/MS
- ESCA
- TEM
- Neutron Diffraction
- SEM
- AFM
- Scanning Tunneling Microscopy
- ESCA Nanoscope
- Nanodensitometer
- X-Ray Diffraction
- Differential Thermal Analysis
- Dilatometry
- Differential Scanning Calorimetry
- Porosity measurements
- Magnetism/Vibrating Sample Magnetometer

CONCLUDING REMARKS

The synthesis and properties of nanocrystalline materials include researching metals, metal oxides and nanocomposites. Preparation methods are by inert gas condensation and ball milling. Specific properties of interest are catalytic, sensor, magnetic and mechanical attributes. Such work is providing increased understanding and capability in the investigation of nanoscale science and technology at the University of the Saarland.