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Abstract Selected activities of the Materials Department at Risø National Laboratory during 1992 are described. The work is presented in three chapters: *Materials Science*, *Materials Engineering* and *Materials Technology*. A survey is given of the Department's participation in international collaboration and of its activities within education and training. Furthermore, the main figures outlining the funding and expenditure of the Department are given. Lists of staff members, visiting scientists, publications, lectures and poster presentations are included.

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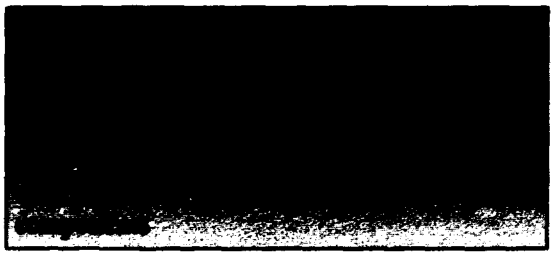
Introduction

The objective of the Department's research is to develop, characterize and model materials and material technologies in order to advance the efficient and safe use of materials. The research is carried out within the three disciplines, materials science, materials engineering and materials technology. The work of the Department is directed towards the energy, industry and research sectors in Denmark and abroad. Educational and knowledge transfer activities are integral parts of the work of the Department.

This objective was fulfilled in 1992, as will be shown in the present progress report. That this objective also can be met in the future is illustrated in the following by a number of important research contracts received in 1992:

- Within the energy field, the Danish utility group ELSAM has decided to initiate a programme on solid oxide fuel cells (SOFC) which has a high potential for the production of electricity and heat with high efficiency and low atmospheric pollution. As part of this programme, ELSAM and the Ministry of Energy will finance a Danish programme with the aim of developing a 0.5 - 1 kw SOFC prototype within the period 1993 - 1996. This programme will be managed by the Department, with participation from industry and other research institutes. Also within the energy field the Department has received three contracts under the EC programme JOULE. The titles of these programmes are given in the table.
- Within the industry field, three new programmes are being initiated within the framework of the EC programme BRITE-EURAM. The titles of these programmes are given in the table. Also, an important programme has been started together with a Danish firm under the auspices of the European programme EUCLID. In this programme, composite materials will be developed for protection against explosions and weapons' projectiles.
- Within the field of basic research the Department has received funding from the Danish Technical Research Council for the establishment of a centre for research on materials structures and materials models. The materials of interest will be metals, ceramics, polymers and composites, characterization will be concentrated on microstructure, texture and mechanical properties. This centre, which will be funded for a period of five years, will have a scientific staff of about 15, including 10 post graduate students and post doctoral scientists. That this centre is to be established within the Department is seen as acknowledging the high academic standard attained in this area.

The research and development work covered by these contracts fits well into the general framework of the Department and the volume and length of the contracts ensures that longer term planning is possible. 1992 has therefore been a year in which the foundations of the Department have been significantly strengthened.



Resources

The income structure of the Department is continuously changing as a direct consequence of Government policy. This policy aims at replacing direct funding of research institutions like Risø with funding for focused national and international programmes in which interested parties compete for the funds. The Department has been quite successful in this competitive environment. Thus the project funding continues to grow. However, as most of the project funding only covers direct expenses, it is becoming increasingly difficult to finance the Department's own and mainly basic research, procurement of equipment and the building and refurbishing of laboratories and offices. In this situation, money saved in previous years has now been spent and it has therefore been of great support that the Velux Foundation of 1981 granted the Department 2 Mill. DKK, allowing us to buy an environmental scanning electron microscope (ESEM). This microscope is the first of its type in Denmark and it ensures that the Department can be at the forefront in the microstructural characterization of metals, ceramics and polymers.

The present staff position, in which the Department has been able to maintain controlled growth by hiring young engineers and scientists, continues to be satisfactory. Through this expansion it has been possible to maintain a satisfactory age profile for the academic personnel. The inflow of young post-doctoral scientists has increased in 1992 and will continue to increase. This is due to the emphasis both nationally and within the EC on the training of researchers. Such researchers typically spend one or two years in the Department carrying out a research project.

With the aim of strengthening the scientific and educational profile of the Department a number of the senior staff have been evaluated by peer committee. As a result of this evaluation, 21 of the researchers in the Department have been promoted to senior scientists. Four scientists were already qualified which brings the total number to 25. One of the qualifications of a senior scientist is that he or she is deemed able to supervise graduate students. Should funding for research education be increased, the Department is therefore well prepared to expand this type of activity.

The general modernization of the laboratories and offices in the Department was completed in 1992. The activities of the Department have in recent years expanded to a degree that laboratory and office space were becoming cramped. It was therefore decided at Risø to use the hot cell building, which is being decommissioned, for an expansion of the Department. In this building it is planned to house activities such as fuel cells and ceramic technology, polymer research and polymer composite technology. There will also be space for an expansion of educational activities. Planning will take place in 1993 with the aim of starting refurbishing of the building at the beginning of 1994 and completion at the end of 1994.

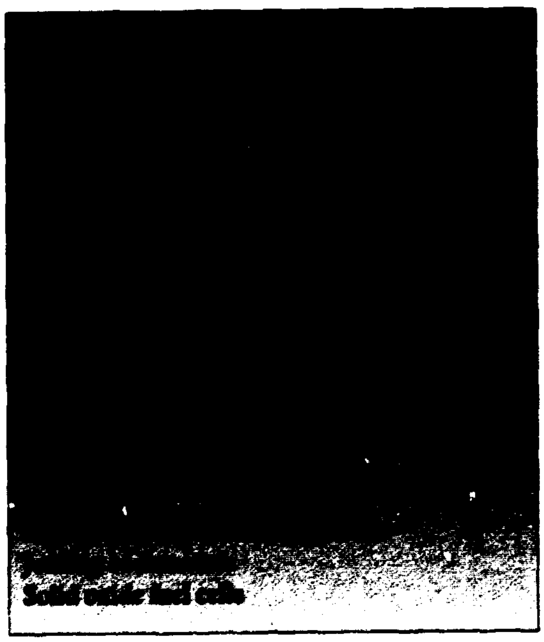
Organization

Research and development in the Materials Department is carried out within the three disciplines: Materials Science, Materials Engineering and Materials Technology. The combination of these disciplines within the Materials Department makes it possible to carry out work both on the development, characterization and modelling of materials and

The general chemistry and metallography laboratories have been refurbished in 1992. Efficient, safer and more pleasant working conditions were achieved by combining new laboratory utilities and work surfaces with the original solid wood cupboards and drawers.

on the application of materials in advanced products. Using industrial terminology, the work encompasses materials design, process design and product design.

The work in the Department is organized in a number of programmes each headed by a programme leader. For the larger programmes, management groups are established combining scientific, technological and administrative expertise. An important part of the work is to formulate new projects both for internal and external research funding. This is done by the researchers individually or in groups which, for large external applications, are supplemented by staff experienced in management. Research proposals are considered by advisory groups established for each of the research disciplines of the Department. One main task of these groups is to assess which proposals should be financed through the Department's own funds. A second task is to give guidelines for future research, especially for projects and programmes to be proposed for external funding.



Research

In this report the work in the Department is described in three chapters, one for each of the research disciplines. Each chapter contains a general description of the work but concentrates on results, for example new experimental findings, models and novel techniques.

Research programmes and projects related to industry and energy are summarized below. Finally, there is a subsection on basic research.

Industry Related Research

The Department is an active partner in the Danish programme on Materials Technology (MUP). This programme consists mainly of collaborative projects between industry, research institutes and universities organized within seven centres and a number of programmes. The Department participates in three centres, namely the Centre for Powder Metallurgy, the Centre for Advanced Technical Ceramics and the Centre for Polymer Composites. The administration and coordination of the two first mentioned centres is placed within the Department, which also coordinates two programmes, one on polymers with controlled structure and properties and another on modelling of mechanical properties of metals. Both in the centres and in the programmes most of the tasks are those given a high priority by the industrial partners. This has led to an active collaboration and a fruitful reorientation of the applied research within the research institutes and the universities. These programmes have, to a large extent, been successful, as confirmed by an international evaluation of the centres carried out in 1991. However, part of the programme has also been criticized for putting too little emphasis on focused, long range research. This criticism is not surprising as it illustrates the genuine difficulty in combining an industrial approach, aiming at an efficient solution of complex process and product materials problems, with a scientific approach, aimed at an in-depth understanding of materials. In a planned continuation of the present MUP programme, this problem is addressed by proposing that longer range basic research is carried out within centres at research institutes and universities. These institutions should also participate in applied programmes together with Danish industry and it is foreseen that the technical and financial contribution of the industry partners shall increase as the research and development approaches practical application.

Industry related research in the Department includes a number of projects carried out on a proprietary basis. Such work relates to design, materials testing, non-destructive testing and failure analysis. Furthermore, the Department undertakes work as an industrial subcontractor in areas where expertise has been built up, for example manufacturing of components in polymer composites, processing of ceramics, and dip brazing of electronic parts.

Energy Related Research

The primary research activities within this area are related to the development of solid oxide fuel cells (SOFC). This work focuses on the building and testing of small prototypes and, in parallel, on longer range research in order to improve the efficiency, economy and versatility of the fuel cells. This has been strongly supported by the decision in December 1992 by the ELSAM utility group to initiate a large focused programme on SOFC covering both research and development of small prototypes (0.5 - 1 kw) in Denmark as well as testing of large prototypes abroad. The work in Denmark will be carried out as a continuation of a national programme (1990 - 1992) managed by the Department and with the participation of utilities, industry, research institutes and the Ministry of Energy. The management of the new programme (1993 - 96) is also placed in the Department and it is planned that staff from ELSAM and from Haldor Topsøe A/S will work together with the project group in the Department for shorter or longer periods. This organization will ensure that research and development is carried out in an efficient way thereby supporting and accelerating the industrial development of the SOFC technology underway at Haldor Topsøe A/S.

Energy research also covers a programme together with a Danish industrial firm on advanced materials for waste and bio-fuel combustion. The Combustion Department at Risø, FORCE Institutes, ELSAM and The Ministry of Energy also participate in this work which aims at increasing the efficiency of the plant by raising the combustion temperature. A key problem is the lifetime of high temperature materials in a strongly corrosive environment.

A continuing effort is materials development, design and testing related to rotor blades for wind mills which have a high priority within the Danish energy programme. The Department's work in this area is carried out as part of a JOULE programme with the participation of research institutes and industries in many European countries, coordinated by the Department.

Finally, within energy research, the Department is engaged in the European Fusion Technology Programme. The integration of this programme into the ITER (International Thermonuclear Experimental Reactor) project is at present under preparation. The outcome of this integration will determine the future activities in the Department in this area.

Basic Research

The applied programmes described above are founded on long range programmes covering modelling of materials properties and behaviour as well as characterization of structures and properties. The materials investigated are metals, ceramics and polymers. Composite research has become a major activity in recent years. The

Department is active in programmes on metal matrix composites, polymer composites and ceramic matrix composites. The study of these systems is often made difficult due to the lack of high quality, reproducible test specimens. The approach in the Department has therefore been to develop manufacturing technologies in order that well characterized materials are available for the research.

In 1992, research on materials structures and materials modelling was given a high priority by the Danish Technical Research Council. As a consequence it was decided that the Council would finance a centre within these areas to do long range research in combination with the training of young scientists. The Department applied for this centre and was successful. A centre in the Department is therefore now being planned for the period 1993 to 1997 within a financial framework of DKK 7.5 million per year. It is foreseen that five scientists and three other staff members will work directly within this centre and that the number of young researchers under training will be about 10. The research activities of the centre will be based on present expertise but several new activities will be started, for example polymer structures and mechanics, low cycle fatigue and microstructure and properties of layered structures. A high priority is also given to studies of internal stresses in single phase metals, alloys and composites.

The research on deformation and annealing of materials in combination with the development of advanced characterization and modelling techniques has given results of both scientific and industrial relevance. This area is internationally in focus as it appears within reach to introduce quantitative observations of microstructure, texture, mechanical properties and internal stresses into general models on the mechanical behaviour of materials. This approach is used in the Department in a number of programmes directed towards practical applications, for example cold forming of steel, hot rolling of aluminium alloys and hot forging of aluminium matrix composites.

It follows from the type of research carried out in the Department that emphasis is put on the development of various advanced experimental techniques. Major areas are the application of neutron scattering and of electron diffraction. In the area of electron diffraction, automatic techniques for crystallographic orientation measurements are being developed, especially within the area of electron back scattering patterns (EBSP) and Kikuchi pattern analysis. Digital image analysis is applied both routinely and in research, where the Department collaborates with IMSOR at The Danish Technical University.

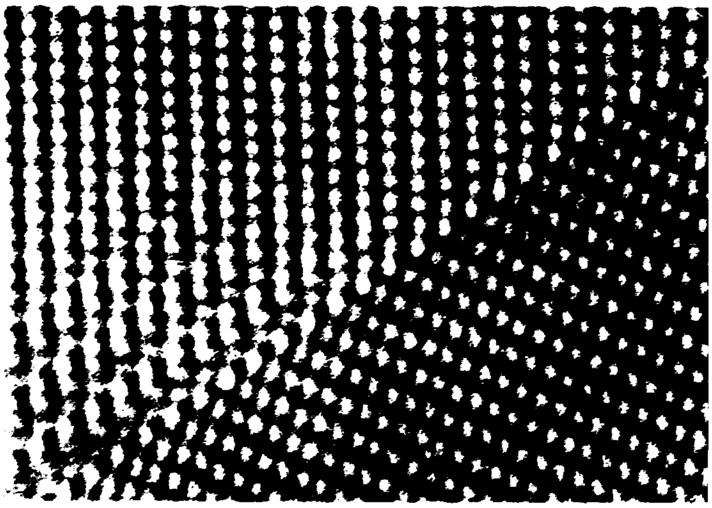
A highlight of the year is the installation of a new environmental scanning electron microscope (ESEM). This acquisition was made possible by a large donation from the Velux Foundation of 1981. This new microscope is the first of its kind in Denmark and it allows for a number of studies which cannot be carried out in a standard scanning electron microscope. In the ESEM the pressure in the test chamber can be as high as 50 torr allowing the examination of water containing specimens, and electrical insulators such as ceramics and polymers. The microscope is equipped with a heating table allowing in situ studies up to 1400°C. This microscope will become an important tool in the Department's research programmes and it will attract customers from other research institutes and from industry.

Achievements

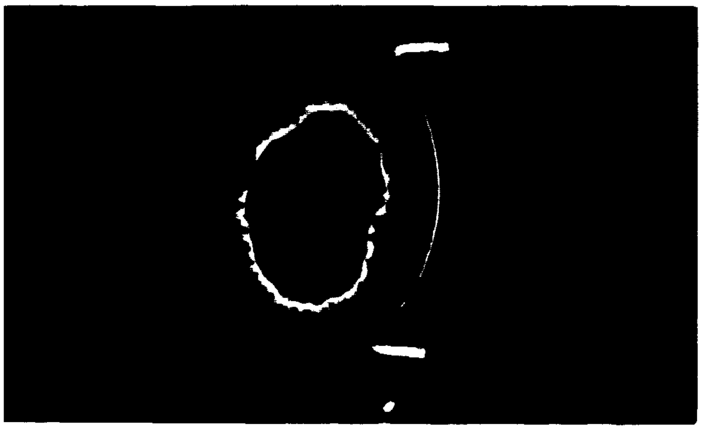
A number of scientific and technical achievements made in the course of the year may be mentioned.

In the area of materials modelling and characterization a new model for basal twinning in sapphire was developed and corroborated by HREM of the twin boundary structure; a unified description has been proposed for the evolution

HREM shows stacking of cations at a basal twin boundary in sapphire to be CBABC. This structure is predicted by a new model for basal twinning.



of deformation microstructures of materials; orientation dependent grain growth during recrystallization has been shown using the electron back scattering (EBSP) technique. Also within this area, a viewpoint set has been organized for Scripta Metallurgica et Materialia on »Microstructure and Flow Stress of Cell Forming Materials«. In the area of metal matrix composites, the evolution of internal stresses during high temperature creep has been studied on-line by neutron diffraction. In polymer research, a synthesis method was developed for liquid crystal polyesters with optoelectrical properties having a potential for digital and holographic data storage.



In irradiation defect studies, a production bias model was shown to be successful in accounting for damage accumulating in metals during particle-irradiation.

In materials engineering and testing, an efficient and economical fly wheel for energy storage has been designed, and techniques for fatigue testing of elastomers have been developed. Within non-destructive evaluation, ultrasonic scanning techniques have been developed for testing of larger scale engineering components; as an example can be mentioned the use of this technique for defect and dimensional control of fuel plates for low enriched uranium fuel elements.

In materials technology a technique has been developed for the manufacture of parts of glass-fibre reinforced thermoplastics from a cloth woven from the two types of fibres. Also within composites, a light and mobile ramp for handicapped people using wheel chairs has been designed and manufactured by two engineering students from Haslev Teknikum. For this project the students have received the annual prize for mechanical engineering from the two Danish Engineering Societies. In powder metallurgy, very pure aluminium powder has been rapidly solidified and consolidated by compaction and extrusion. Within the national programme on solid oxide fuel cells a three-cell stack with internal manifolding has been operated successfully for about 500 hours. Within the area of materials development, cells have been developed with a very low area specific resistance of 0.25 - 0.40 ohm cm².

Three patent applications have been filed on (i) fatigue resistant aluminium alloys (EURAM-project), (ii) a light and efficient fly wheel, (iii) materials for solid oxide fuel cells. One European patent application on metal matrix composites has been granted.

One of the staff members has publicly defended a thesis »Cyclic Plasticity of Metals« thereby fulfilling the requirement of the Technical University of Denmark for the degree of Doctor Technices. Three graduate students have received the Ph.D. degree.

Finally, the second phase of the decommissioning of the hot cell facility has been completed without exceeding the normal average personnel dose.

International Cooperation

Two years ago, a new initiative was launched to engage the Department more actively in the formulation and organization of new international projects with the special objective of increasing the involvement of Danish industry in European Community sponsored research. This initiative was supported by own funds and with a grant from the Danish START programme. A senior consultant was put in charge of this activity and formulated in 1991/92, together with senior scientists from the Department, a number of proposals for the EC programme BRITE/EURAM and JOULE. This initiative, together with others, resulted in 11 proposals for the two programmes. The Department succeeded well; six of the proposals were graded A in the EC evaluation. The titles of these proposals are given in the table, page 5. Of these proposals, three have the Department as *prime proposer*. The objective of involving Danish industry was also successful; there is participation by Danish firms in four of the six proposals accepted by the EC. This participation allows the Department to provide a bridge between international and national materials research.

The participation in BRITE EURAM and in JOULE is supplemented by other European activities. For example, our activity on materials for combustion plants is integrated in the COST 501 programme, and the research on neutron diffraction measurements of texture and elastic strain forms a basis for participation together with the Physics Department at Risø in EC's Large Installation Plan. This programme covers neutron scattering experiments at the DR3 reactor carried out by researchers from other EC countries. Finally, the Department participates together with a Danish firm in a project within the frame of the Independent European Programme Group (IEPG)'s EUCLID programme.

Symposia and Workshops

An important activity during 1992 has been the organization of the thirteenth Risø International Symposium on Materials Science, »Modelling of Plastic Deformation and Its Engineering Applications«. This symposium followed the format of the Risø International Symposia series and was arranged in collaboration with the Danish Materials Technology Programme. The number of participants was around 100. Another activity has been the organization of the fifth International Workshop within the field of radiation damage. This workshop was held in Switzerland and arranged in collaboration with PSI, Switzerland. The title of this workshop was »Time Dependency of Radiation Damage Accumulation« and it was attended by about 40 invited scientists. The Department also participated in the arrangement of a conference in Zeltingen, Germany on »Fundamentals of Recrystallization« together with the German institutes »Institut für Allgemeine Metallkunde und Metallphysik, RWTH Aachen« and »Institut für Metallphysik der Universität Göttingen«. This conference was attended by about 40 invited scientists from Europe, U.S.A. and Japan. The Department also arranged a two day workshop on materials research within the area »High Temperature Electrode Materials« sponsored by the Fuel Cell Committee under the Energy Research Programme and the Council of the Nordic Ministries. This workshop was attended by 30 scientists. Finally, meetings were arranged at Risø in connection with the Danish Programme on Materials Technology.

Public Relations

Public relation activities have included participation in major exhibitions in Denmark, viz. The Tech Trans Center 92 in Herning as well as Teknovision and the National Museum in Copenhagen, where the Department concentrated on programmes within the areas of polymer composites, polymers and solid oxide fuel cells. The PR activities have also included media coverage of research and development. The three subjects which attracted most interest this year were solid oxide fuel cells, the result of the industry-research laboratory collaboration within the Danish Materials Technology Programme and the research with the University of Copenhagen on synthesis and modelling of molecules which bind very efficiently to DNA having potentials as therapeutic agents.

Education

Educational activities are becoming an increasing part of the Department's work. Many staff members act as external lecturers and examiners at undergraduate and post-graduate levels. Also, undergraduates and graduates from engineering schools in Denmark and abroad have carried out experimental work and received academic training in the Department, supervised by staff members.

The number of graduate students and post graduates has increased significantly in recent years due to new funds both nationally and within the EC. At present, about 15 young researchers are studying in the Department. In addition, the Department also teaches apprentices in mechanics and electronics.

Educational activities also include participation in a comprehensive course for technicians and engineers on materials properties, processing, testing and product design. This course, sponsored by the Danish Ministry of Education (Law No. 271) is carried out in a collaborative effort between research institutes, universities and technical schools in Denmark. An important objective of this programme is to offer courses for in-service training of employees in Danish industry. However, the economical climate in the industrial sector in 1992 has made this part of the programme less successful than expected.

The many educational and training activities involve a large group of guests staying in the Department. Therefore, the professional activities have to be supplemented with social activities. This is especially important for the foreign students. Such activities are not new for the Department but the large number of guests required a more focussed effort than before.

Concluding Remarks

The important research contracts received in 1992 shows the strong position held by the Department within Danish materials research and development. That a number of the contracts have been won in international competition shows that the research is of international quality. These achievements have confirmed the conclusions reached by the international evaluations in 1991. The achievements have also confirmed the viability of the research strategy to combine in the Department the disciplines materials science, materials engineering and materials technology and to combine basic research with applied research. This has allowed complex and large research projects to be initiated and made it possible to extend such projects into the semi-industrial phase. An example is the development of the solid oxide fuel cell technology. More generally, the combination of disciplines has facilitated the often difficult transfer of basic research results to practical applications.

The change in the income structure of the Department from direct to indirect funding supplied by sources outside Risø has to a great extent been accommodated especially due to the large and competent effort of the members of the Department both in acquiring new contracts and in carrying out the research. In last year's progress report it was mentioned that a major drawback of this change in income structure was reduced possibilities of carrying out long range research. This year there is cause to be somewhat more optimistic as funding for basic research will be increased in Denmark in the coming year. So we feel confident that such research can be carried out also in the future at Risø. An example of such research is our work on materials structures and materials models which in the coming years will be partly funded as a centre by the Danish Technical Research Council.

The many activities of the Department reflect the problems and demands of society at large. Such problems and demands have always existed but in recent years new ones have been added. More emphasis is put on education and environmental problems are becoming a major issue. In education, the Department has significantly increased its activities by almost doubling in recent years the number of post graduate students and post doctoral scientists and a continued increase is planned. Concerning environmental problems, related for example to the production, use and recycling of materials, the Department has started to collaborate with specialized research institutes in Denmark in order to introduce considerations on safe and clean technology into the research programmes. However, we have never looked specially at environmental problems through dedicated research projects. It is foreseen that such projects will come and the expertise of the staff and the facilities available will ensure that the Department also can contribute in this important area.

2

Materials Science

Efforts to improve the inherent properties of materials are based on our ability to characterize, understand and finally to modify microstructure. The research in this area, although of a *fundamental nature*, is often initiated in response to specific technological and engineering demands for new and improved materials. The research themes in Materials Science are therefore closely related to the applied programmes within the Department. Much of this basic research is carried out in close collaboration with colleagues from universities and government research laboratories around the world.

2.1 Modelling within Materials Science

Materials Science deals with very complex problems for which it is normally not possible to formulate quantitative theories in the strict sense of ordinary physics. Therefore, one has to make do with models. A good model describes the essential features of a certain process or a certain phenomenon in the simplest possible way. Thus simplicity, not complexity, is a virtue. But of course one has to accept that even »the simplest possible way« may be rather complex - increasingly much so with increasing demands on the level of details that the model is requested to reflect.

Some processes can only be understood by considering the behaviour of the individual atoms, and such processes must be modelled at the atomic level. However, most of the relevant processes in materials take place at such a coarse (even though still microscopic) scale that modelling at the atomic level is out of the question. For instance, the growth of one single recrystallized grain involves many trillions of atoms, which even the biggest computers cannot keep track of. And, as to be described later, a model for the growth of recrystallized grains must include a large number of grains.

The process of working with a model may be seen in three stages: (i) the formulation stage, (ii) the testing stage, and (iii) the application stage. Ideally one may say that at stage (iii) the model has left the realm of science and become an engineering model. However, the present trend is to apply the models in engineering (stage (iii)) while they are still being investigated at stages (i) and (ii) - with the justification that a model, even at a preliminary stage, is better than no model. One should notice that engineering application of a model does not necessarily imply quantitative implementation. Qualitative use of the insight gained during the work with the model may be equally important.

The sections to follow will overview some of the modelling activities within basic materials science and the way in which they cover the range of scales from the atomic scale to the scale of the layers in laminated fibre composites, i.e. a scale range of a factor of $\sim 10^6$. Most of the models in this review will be described in more detail later in the text.

The Atomic Scale

The fundamental process in radiation damage is the displacement of the individual atoms when hit by high energy particles. This may be modelled with molecular dynamics (MD), and this type of modelling is done in collaboration with Pacific North West Laboratory, Richland, USA. The experimental reference is damage with cascade formation, in particular the radiation damage in fusion devices. The problem with MD models is that they are very expensive in computer time, and therefore they can only deal with an insufficient number of atoms. Alternative models based on the binary collision approximation (BCA) can deal with bigger events (displacement cascades of higher energy). Therefore, the aim of the MD modelling is to calibrate the cruder BCA models.

The Scale of Defect Clusters

The macroscopic effects of radiation damage are related to the clusters of defects, their stability and their interaction with the population of vacancies and interstitials. In collaboration with Atomic Energy of Canada Ltd., Pinawa a production bias model has been suggested. The model couples to the phenomena at the atomic scale described above, but the migration of the defects is described with continuum rate theory (and not as migration in the atomic lattice). This jump in scale provides a model which refers directly to the technologically relevant properties of materials exposed to radiation damage.

Another way of modelling clustered defects (in a displacement cascade) has been pursued in collaboration with Paul Scherrer Institute, Villigen, Switzerland: the cascade is considered as a superheated liquid droplet. The cooling of these droplets near metal surfaces has been compared with cooling in the bulk.

Dislocation Scale

In collaboration with Case Western Reserve University, Cleveland, USA a new model has been developed for basal twinning in α-Al_2O_3. Basal twinning is an important deformation mode at temperatures below approximately 1000°C. The model is based on the concept that the cation layers are staggered to an extent that makes it is reasonable to assume that the cations during slip follow the oxygen layers in which they are partly embedded. In this way the atomic movements in the basal plane can be explained physically by the movement of a single dislocation.

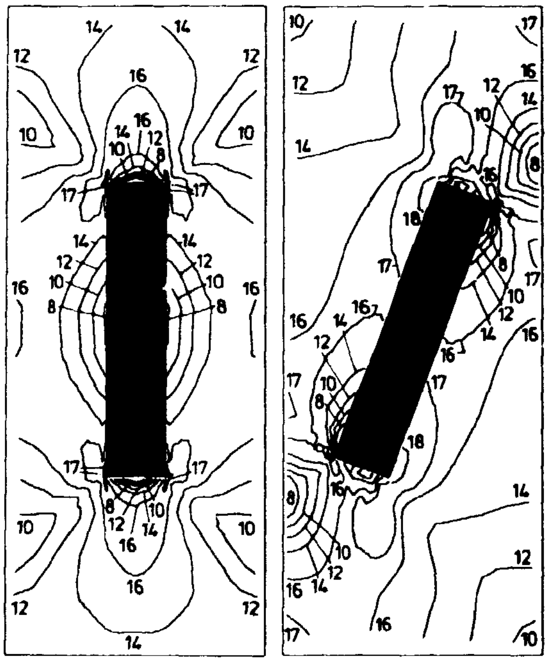
Fibre Scale

The micromechanical behaviour of composite materials with metallic and ceramic matrices and the relationship to the macromechanical properties is modelled with different models (in collaboration with University of Cambridge, UK, Brown University, USA and the Technical University of Denmark). The experimental references are unidirectional, cyclic and creep deformation in metal matrix composites and fatigue damage in ceramic matrix composites.

For some applications, models derived from an Eshelby approach provide attractive, relatively simple descriptions of the deformation behaviour - a description which includes the dislocation behaviour. These models may actually be extended to deal with single phase metals considered as pseudo composites consisting of dislocation-dense hard regions in a softer matrix.

For other applications where more details of the local behaviour round the fibres are needed, the finite element method (FEM) is the obvious modelling tool. For instance, the effect of fibre geometry and fibre distribution in metal matrix composites and the effect of fibre/matrix contact in ceramic matrix composites are modelled with the FEM technique.

Stress-distribution in a fibre-reinforced MMC with parallel and misaligned fibres (left/right respectively). The orientation of the misaligned fibres is not stable as the MMC undergoes straining and a strain induced rotation of the fibres is predicted.

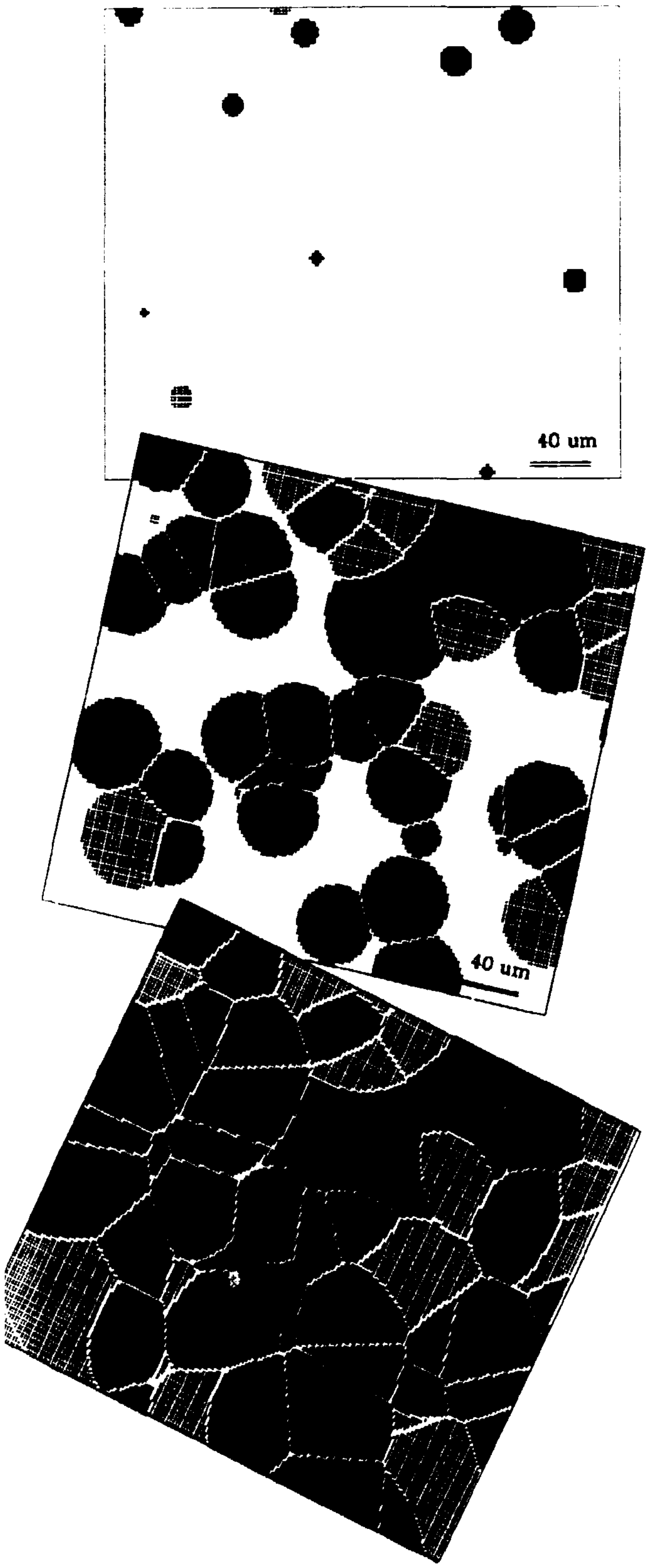


Grain Scale

The formation of recrystallized grains and the associated recrystallization texture is investigated with a model which considers the nucleation and growth of grains with different crystallographic orientations (with different nucleation and growth rates). The output is grain morphology and texture at any stage of the recrystallization process.

A conceptual model for the subdivision of the grains in deformed metals into »cell blocks« with different deformation patterns is investigated in collaboration with University of Virginia, Charlottesville, USA, Sandia National Laboratory, Livermore, USA and the Danish Academy of Engineering. A computer model for plastic deformation with subdivision of the

Recrystallized grains with three different orientations (as indicated with different shadings of grey) grow into the deformed matrix with different rates in a model experiment. The three frames represent 6%, 50% and 100% recrystallization.



grains into cell blocks is being developed. The model maintains strain and stress continuity between the cell blocks and strain continuity between the grains. A »conical slip model« has been formulated for the mechanical anisotropy introduced by the macroscopically aligned dislocation walls/microbands between the cell blocks. The agreement, or occasionally lack of agreement, between experimental deformation textures and deformation textures simulated with simple deformation models (without grain subdivision) is also under investigation.

Laminate Scale

Laminated structures with different fibre orientations in the different layers are of great importance in the technology of polymer composites. The typical thickness of the laminate layers is some tenths of a millimetre. In many connections, elastic properties of laminates may be dealt with by plate theory modified to account for the heterogeneity of the layers. However, in cases with high stresses perpendicular to the plane of the plate, for instance caused by indentation, the concept of plate theory is inadequate. For such cases a new type of FEM modelling has been developed at Risø. The model considers the case of rotational symmetry so that the requirement on computer power is reduced as compared with the case of a full three-dimensional model.

2.2 Micromechanical Modelling

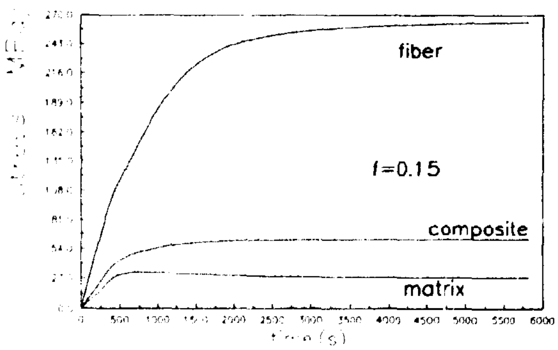
The finite element method (FEM) has been increasingly used to model the micromechanical behaviour of materials. Particularly with composites, the inherent microstructural inhomogeneities may be modelled in order to get insight into how deformation and failure processes might occur. The results of such models are compared with experimentally obtained properties, data and microstructural observations.

Micromechanical Analysis of MMC's

Micromechanical analysis for characteristic volume elements are an important tool for determining the mechanical properties of composite materials. Often such analyses are carried out numerically, taking full account of fibre geometries and material properties. The computational requirement for the analyses of elastic-viscoplastic behaviour of composites are often quite large and involve the use of supercomputers. Cell models containing a single fibre are representative of materials containing uniformly distributed parallel or misaligned fibres, while cell models containing several fibres can account for the effect of clustering of the inclusions, or almost random distributions.

Shown in the figure is the development of the volume average true stress in the different phases of a metal matrix composite in the loading direction. The stress development is obtained by a numerical simulation of a slow hot tensile test. Volume average stresses which are easily computed with the finite element method (FEM) are of considerable interest, because such average stress components can be measured experimentally by the use of recently developed in-situ neutron diffraction set-up for internal stress determination at elevated temperatures.

Computer simulation of the development of volume average stresses in an MMC undergoing a slow hot tensile test. Considerable load transfer from the weak metal matrix to the strong ceramic fibres persists after the overall stress in the composite has reached an almost constant value. Further work has shown that the amount of load transfer is related to the degree of uniformity of the dispersion of inclusions.



Analysis of the effect of reinforcement shape on the viscoplastic behaviour of MMC's at elevated temperatures shows that the presence of corners on the inclusions has a marked influence on the behaviour of MMC's and very large differences exist between metal matrix composites with spherical and cylindrical inclusions, respectively. Studies of MMC's with misaligned short fibres showed that misalignment leads to a reduction in the creep resistance together with a strain induced rotation of the short fibres towards the more stable parallel position.

Stress Analysis of Laminated Composites

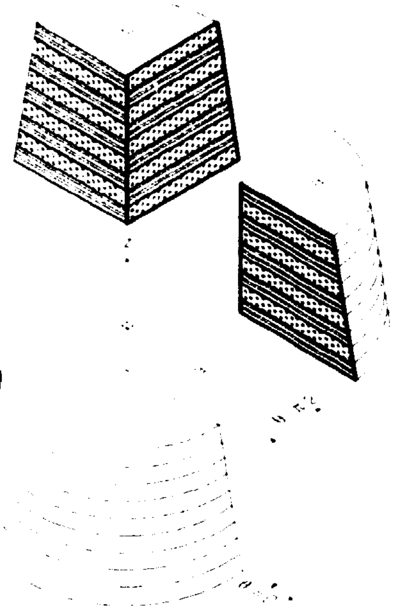
Laminated fibre-composites are usually used to build up plate or shell components. Accurate prediction of the bending and stretching response of the laminates has been a challenge for engineers since the early sixties. The generalization of the classical Kirchhoff theory to account for layerwise heterogeneity was established and is now denoted the Classical Laminate Theory. However, the shear flexibility of typical laminate materials magnifies the influence of the transverse shear strains. Therefore, for laminated fibre-composites, the limitations of the classical plate theory are met at an earlier stage than for isotropic materials. Consequently the development of higher order plate theories has become a major activity, and today several higher order theories and their finite element formulations are available in the literature.

The plate theory concept is, however, inadequate for determining the stress state in regions where high stresses normal to the plane of the plate are present. Such is the case in problems involving indentation, where a full 3D field solution is required in the region below the indentor. The use of 3D finite elements requires at least one layer of elements for each lamina orientation. Hence, the computer power available restricts the number of fibre orientations that may be treated in the analyses. In order to model the stresses in thick laminates involving many fibre orientations, a new methodology has been developed at Risø. The method treats a rotational symmetric volume in which the field variables are Fourier expanded in the circumferential direction. An example of such a volume is depicted in the figure, where the inner part of a cross-ply laminate is subjected to the analysis. The contact between a rigid spherical indentor at the upper surface is modelled and equilibrium is established. The distribution of principal tensile stresses normal to the fibre direction in orthogonal sections is used to predict position, angulation and sequence of matrix cracking.

Micromechanical Simulation of Fatigue Hysteresis in CMC's

Ceramics reinforced with continuous aligned fibres are damage tolerant and can survive many thousands cycles of mechanical loadings, although the material can be damaged by multiple matrix cracking and fibre/matrix

Sectioning the inner volume of a cross-ply laminate. Of particular interest are the radial sections $\Theta=0$ and $\Theta=90$ from which indentation damage initiates.



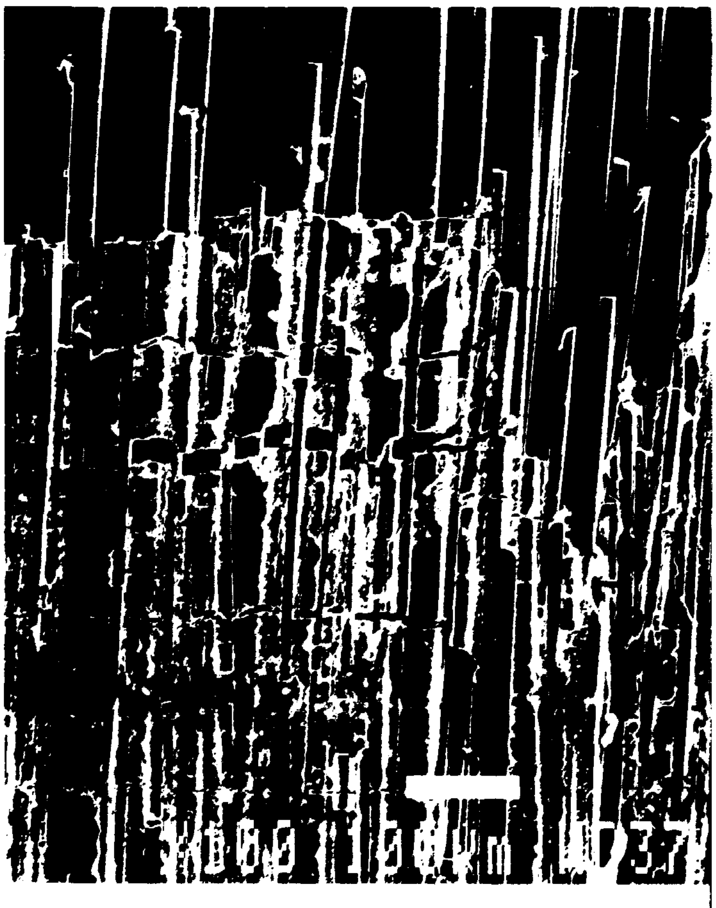
debonding. In order to get insight into how the mechanical fatigue damage evolves, it is necessary to develop models relating the microstructural mechanisms to overall response. By comparing experimental and calculated macroscopical behaviour, such as the shape of hysteresis loops, models can be used as a means of extracting interfacial sliding properties.

In this study, the overall material response was simulated by the finite element method of a representative volume cell, and fibre/matrix sliding was modelled by Coulomb friction at the contact surfaces. As the sliding state is history dependent, the problem was solved in steps.

In the simulations, the properties of the fibre and the interface were varied, as these were the parameters known with the least accuracy. The magnitude of hysteresis was strongly affected by the change of these material parameters. When the composite was strained above a characteristic value (dependent on the thermal mismatch), the fibre/matrix contact ceased, due to the Poisson's contraction of the fibre. Studies of the state of sliding at various load levels revealed that, as long as sticking friction existed at some part of the interface, the stress-strain response was non-linear. When slippage took place along the entire interface, the stress-strain response was linear. In this situation the tangential stiffness was lower than that which would be expected if the fibre alone were to carry the load increment. This was due to the fact that, as the applied load was increased, the frictional stress transfer decreased due to the fibre Poisson's effect, causing the matrix to unload, and transfering more load onto the fibre.

2.3 Scale-Effects in Cu/W

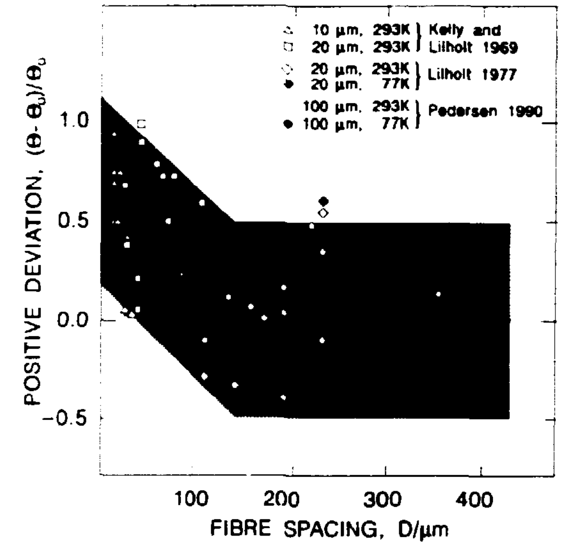
Modelling of the plasticity of composite materials is often based on the assumption that the plastic deformation behaviour of the matrix material is unaffected by the presence of the reinforcement. This approach provides useful descriptions of many aspects of the overall plasticity of the composites; but it has the limitation that it cannot by itself account for any effects of the scale of reinforcement. It is therefore interesting that an extensive study of the cyclic plasticity of the Cu/W model system has revealed a marked scale-dependence of the



A ceramic reinforced with aligned fibres displays various damage mechanisms up to failure, such as multiple matrix cracking, fibre/matrix debonding and sliding, with final failure by fibre pull-out.

work hardening rate θ in the fibre direction. The effect is illustrated in the figure, where $(\theta - \theta_0)/\theta_0$ is the positive deviation of θ from the value predicted on the basis of a matrix unaffected by the presence of the fibres. Within the experimental scatter, the positive deviation is seen to depend upon the fibre spacing.

At small fibre spacings, the axial work hardening rate of Cu with continuous W fibres deviates strongly from that predicted for an ideal plastic matrix.



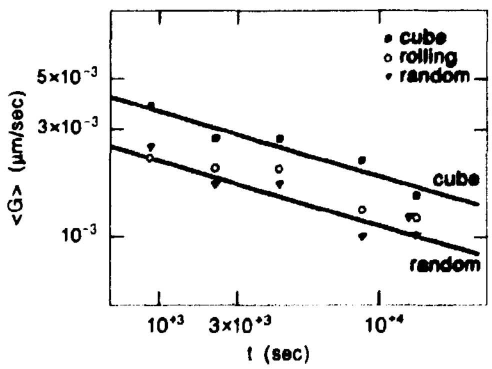
The scale-dependent positive deviation shows that a complete theory of the plasticity of metal matrix composites cannot be based purely upon phenomenological continuum plasticity, although accurate numerical analyses of non-uniform flow patterns are important ingredients for such a theory. In order to account for scale effects, it is necessary to include crystal dislocations in the description, and this involves a distinction between long- range glide of dislocations past many fibres and short-range glide in the vicinity of the individual fibres. Models based on long-range glide predict the maximum scale-independent positive deviation: this reflects the frictional effect of fluctuating local stresses at the fibres. The scale-dependence requires an additional description of relaxation of internal stresses, which is likely to occur by short-range glide. Continuum plasticity may be interpreted as averaging out the dislocations in a crystalline element at a local point, and hence a partial account of stress relaxation is possible in terms of continuum plasticity. Nevertheless, the observed scale-dependence at small fibre spacings requires complementary dislocation mechanisms and it seems possible that these are related to the composite's capacity for storage of prismatic dislocation loops between neighbouring fibres.

2.4 Effects of Orientations on Recrystallization

During recrystallization, grains of different orientations may grow at different rates. This will significantly affect the recrystallization texture and grain size distribution, and therefore, also the properties of the recrystallized material. This has been studied for heavily cold rolled aluminium. The textures of the bulk materials were determined by neutron diffraction. This technique was also used to measure the recrystallization kinetics. With the electron back scattering technique, in the scanning electron microscope, the crystallographic orientation of individual nuclei/grains as well as their size and »free«, unimpinged surface area was determined. This was done for a series of partly recrystallized samples. Based on this data, the average growth rate can be calculated as a function of annealing time and grain orientation.

It was found that the recrystallized grains could be classified into three orientations: cube, retained rolling, and random (of all other orientations). For grains of all three orientations the average growth rate decreases with annealing time, as $G = kt^{-\alpha}$, where α equals 0.3 and k is a constant, which is different for each of the different orientations (see figure). This decrease in growth rate with annealing time may be ascribed to recovery which occurs simultaneously with the recrystallization and/or to an inhomogeneous distribution of the stored energy in the deformed matrix.

Study of recrystallization grain growth kinetics for heavily cold rolled aluminium. The growth rate of individual grains is related to crystal orientation.



It was furthermore found that for the entire annealing period, grains of cube orientation grow, on average, twice as fast as grains of random and rolling orientation (see figure). This means that although there are only relatively few cube nuclei, they will dominate the recrystallization texture, and the grain size distribution becomes wide. Modelling shows that the maximum average grain size in this case is 4, which should be compared to a value of 2.5 if all grains had grown at the same rate. Experiments confirm the modelling result.

2.5 Models for Creep of Polymer Matrix Composites

In the analysis of the long term properties of polymer matrix composites and the establishment of lifetime predictions, it is of theoretical, as well as practical importance, to obtain a correct and, if possible, simple description of the creep behaviour.

Analyses have been made of the mechanical aspects of creep as well as of the microstructural aspects of deformation and damage developed during creep. The analytical descriptions serve to rationalize the experimental observations, to predict creep under different creep conditions, like stress and temperature, and to predict the lifetime of materials and components under external loadings, like mechanical, thermal and chemical influences. A number of relationships have been found to be useful in the general description of creep behaviour and related aspects of polymer matrix composites. These relationships will be described and they are used in an integrated analysis of creep.

The stress-strain relation for the polymeric matrix itself is well simulated by the trigonometric equation

$$\sigma = K \times \sin\left(\frac{\pi}{2} \times \frac{\varepsilon}{\varepsilon_u}\right)$$

where

$$\sigma_{max} = K = \frac{2}{\pi} \cdot E \cdot \varepsilon_u$$

and the initial slope $d\sigma/d\varepsilon$ is equal to the modulus, E.

The temperature effect on strength and stiffness is well described by an empirical relation of the form

$$X = X_o \left(1 - \frac{T}{T_g}\right)^{\alpha}$$

where T_g is the glass-transition temperature of the polymer and X_0 and α are constants, of which X_0 may be interpreted as the strength or stiffness at T = 0 K. For stiffness, the relation is written

$$E = E_o \left(1 - \frac{T}{T_g}\right)^{\alpha_E}$$

and for maximum strength:

$$\tau_{max} = \tau_{max} \left(1 - \frac{T}{T_g}\right)^{\alpha_\sigma}$$

These may be combined via the above equation to give a relation for strain ε_u at σ_{max}:

$$\varepsilon_u = \frac{\pi}{2} \times \frac{\tau_{max}}{E}$$

$$= \frac{\pi}{2} \times \frac{\sigma_{max_o}}{E_o}\left(1 - \frac{\tau}{\tau_g}\right)^{\alpha_\sigma - \alpha_E}$$

The creep curve, which is the relation between creep strain ε and time t, can be analyzed in combination with different models.

An important aspect of creep in polymers and in polymeric composites is the significant part of visco-elastic creep which occurs: this is in contrast to metals where visco-elastic deformation is normally insignificant relative to creep by plastic deformation (by dislocation mechanisms). A simple mechanistic model is based on springs and dash-pots in combination, both series and parallel; a simple and often sufficient model is a spring and dash-pot in parallel, and this part-model is combined further with a spring and a dash-pot in series. The mathematical equation is

$$\varepsilon = \frac{\sigma}{E_1} + \frac{\sigma}{E_2}\left(1 - \exp\left(-\frac{E_2}{\eta_2} \times t\right)\right) + \frac{\sigma}{\eta_3} \times t$$

where E_1 and E_n are elastic moduli of the springs, and η_2 and η_3 are the frictions of the dash-pots.

This relation is fully analogous to an equation which has been presented in the literature as the empirical, so-called, θ-concept:

$$\varepsilon = \varepsilon_{elast} + \theta_1(1 - \exp(-\theta_2 \times t)) + \theta_3 \times t$$

The second term in both equations represents the visco-elastic creep, and the asymptotic strain at infinite creep-times is

$$\varepsilon\ (t = \infty) = \theta_1 = \frac{\sigma}{E_2}$$

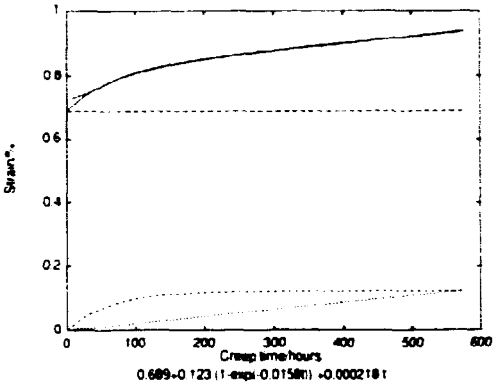
The creep rate is derived from the creep-strain vs. time relation:

$$\dot{\varepsilon} = \frac{d\varepsilon}{dt}$$

$$= \frac{\sigma}{\eta_2}\exp\left(-\frac{E_2}{\eta_2} \times t\right) + \frac{\sigma}{\eta_3}$$

$$= \theta_1\,\theta_2\exp(-\theta_2 \times t) + \theta_3$$

Experimental creep curve for a thermoplastic (PPS) containing 30% short, aligned glass-fibres, together with the best fitted curve according to the θ-equation. The three dotted lines show the separate terms of the equation.



It is clear that the creep rate is not constant and is decreasing from the initial value at t = 0:

$$\dot{\varepsilon}(t = 0) = \frac{\sigma}{\eta_2} = \theta_1\,\theta_2$$

to a (constant) value

$$\dot{\varepsilon}\ (t = \infty) = \frac{\sigma}{\eta_3} = \theta_3$$

which is only reached at infinite creep times. In practice nearly constant creep rate is reached within the practical creep times in experiments, and thus also for components in use.

The effect of external conditions, like stress and temperature, on the creep behaviour is obtained only indirectly via their effects on the parameters of the model E_1, E_2, η_2 and η_3, as well as θ_1, θ_2 and θ_3. Therefore a second, empirical step in the analysis is required to plot the model-parameters, versus the stress, temperature and other external parameters.

At present, microstructural characteristics like volume fraction of fibres, fibre length and fibre orientation are not included directly in the modelling, but their effect can be established by empirical plotting in relation to the model-parameters.

The above creep equations for strain vs. time are well suited for automatic computer-based analysis and fitting to establish the parameters. As an example, the experimental creep curve for a thermoplastic PPS (poly-phenylene-sulphide) with 30 weight per cent of short, aligned glass-fibres is shown in the figure, together with the best fitted curve according to the θ-equation. Furthermore, as shown by the dotted lines, the three terms of the equation are shown separately. The equation with the numerical parameters is the following:

$$\varepsilon = 0.689 + 0.123(1 - \exp(-0.0158 \times t)) + 0.000218 \times t$$

where strain is in per cent and time in hours.

It is clearly seen that the three terms represent different aspects of the creep deformation and, in particular, that the visco-elastic term demonstrates a (rather fast) decreasing contribution to the total creep rate, so that the asymptotic nature of the visco-elastic creep is illustrated.

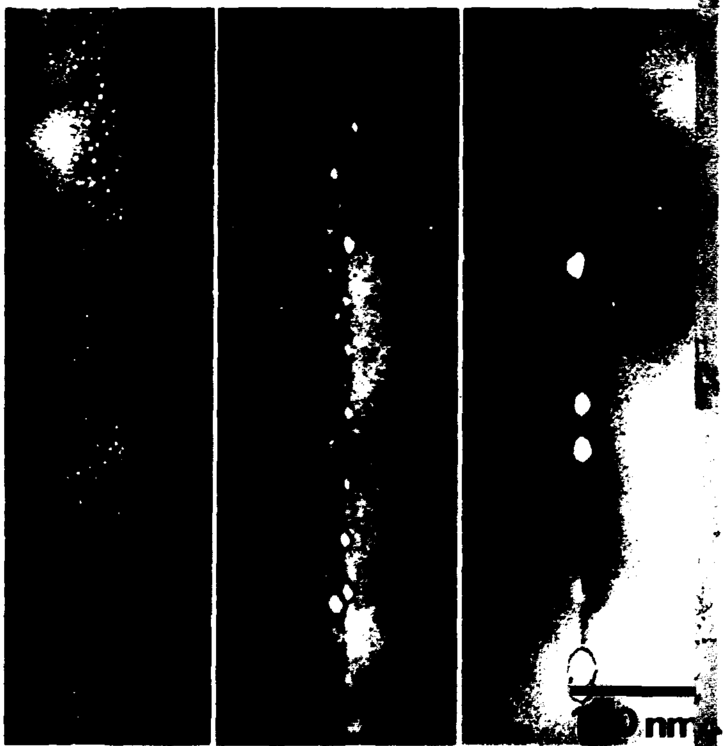
2.6 Irradiation Defects - Fusion Materials

Displacement Damage and Helium Effects in Copper and Copper Alloys

The investigation of effects of displacement damage and helium in copper and copper alloys was continued. Specimens of pure copper and a dispersion strengthened Cu-Al_2O_3 alloy irradiated with 600 MeV protons at 250 and 400°C to a dose level of about 0.5 dpa (displacement per atom) were investigated by transmission electron microscopy (TEM). The irradiation with 600 MeV protons produces concurrently multi-displacement cascades and helium atoms at a high rate (about 125 appm per dpa). The TEM investigation of these irradiated specimens showed a complete absence of cavity formation in pure Cu as well as Cu-Al_2O_3 at the irradiation temperature of 250°C. At 400°C, cavities were observed in pure Cu but not in Cu-Al_2O_3 alloy. The lack of cavity formation in copper at 250°C during 600 MeV proton irradiation is very unusual and may be a result of the high helium generation rate.

In order to evaluate the effect of helium generation rate, pure copper specimens were implanted with helium at different rates at temperatures in the range 405 - 510°C. The implanted specimens were investigated by transmission electron microscopy and positron annihilation techniques. Both temperature and implantation rate dependencies of bubble density in the grain interiors, helium flux to, and bubble density at, grain boundaries were determined. Although the flux of helium atoms reaching the boundaries remains very similar, the bubble density at grain boundaries varies enormously from boundary to boundary. The fact that there is such a large variation in the nucleation and growth behaviour of helium bubbles from boundary to boundary is a matter of concern. If, for instance, the large bubbles were to act as crack nucleation sites or the high density of small bubbles was to embrittle the grain boundaries, then at a very low helium generation rate and at a relatively low level of helium concentration fracture may start at one of the

TEM of helium bubbles at grain boundaries in copper showing large variations from boundary to boundary.



boundaries. Thus, from the point of view of materials performance, it is not only the generation rate of helium which is important but the structure of the grain boundaries (and texture of the material) may also play an important role in controlling the lifetime of a structural component in service.

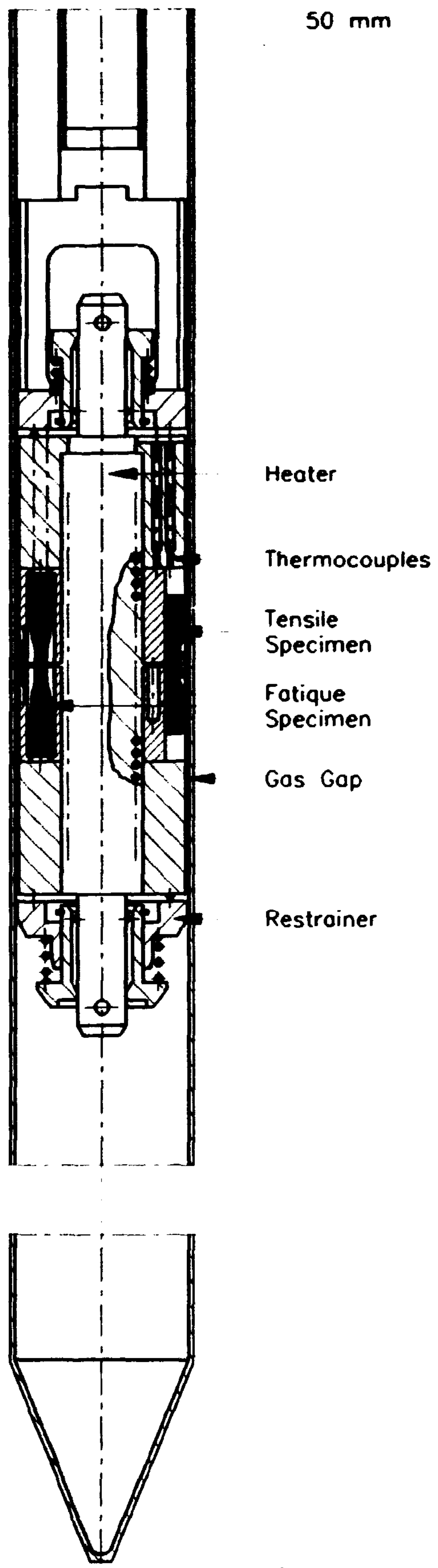
Effect of Neutron Irradiation on Molybdenum Alloys

Molybdenum alloys are being evaluated for their possible use in the heat-sink component of the divertor in the next European Torus (NET) and International Thermonuclear Experimental Reactor (ITER). As a part of the European Fusion Technology programme, an investigation has been initiated to determine the effect of neutron irradiation on microstructural and mechanical property changes in Mo-alloys (i.e. TZM and Mo-5% Re). Tensile specimens have been irradiated in DR-3 at ~50, 100 and 250°C to a dose level of ~0.2 dpa (displacements per atom). Irradiations at ~50 and 100°C were carried out in newly designed capsules. The irradiation at 250°C was carried out in the new temperature-controlled rig in which the temperature of the magazine containing specimens is measured and controlled continuously.

A number of unirradiated specimens of TZM and Mo-5% Re alloys have been tensile tested at room temperature. In order to investigate the effect of 5% Re addition on the ductility of Mo-5% Re alloy, a single crystal of this alloy has been bought. Tensile specimens of the Mo-5% Re alloy single crystal are being prepared for irradiation.

Production Bias, Glide of Interstitial Clusters and Void Swelling

(in collaboration with Harwell Laboratory, England, and Forschungszentrum Jülich, Germany)
In order to test the validity of the »production bias« model, the evolution of void swelling in the transient regime has been calculated and compared with experimental results on fully annealed, pure copper. The general trends of the calculations were found to be in good accord with the experimental results. A quantitative rationalization of the experimental results made



Temperature-controlled irradiation rig for DR-3.

it necessary to assume, however, that the small clusters produced directly in the cascades are mobile and that about 15% of them escape to sinks other than voids. Recently, the validity of this assumption has been investigated by calculating the ranges for the one-dimensional glide of small interstitial clusters/loops. It has been shown that in pure and annealed metals the one-dimensional glide of interstitial clusters to dislocations and grain boundaries does, indeed, represent a very efficient mechanism for the removal of interstitial clusters. In pure annealed materials with low dislocation densities, grains with diameters up to 100 μm, for example, would be virtually transparent for gliding interstitial clusters at the beginning of irradiation. This would remain true during the initial stages of the microstructural evolution when cavity nucleation takes place. The removal of interstitials in the form of clusters yields an excess of vacancies which drives the nucleation and growth of voids. Further consequences of the one-dimensional glide of interstitial clusters and the production bias are being investigated.

Modelling of the Primary Damage State in Multidisplacement Cascades

(in collaboration with Pacific Northwest Laboratory, Richland, USA)
Quantitative information on defect production in cascades in copper obtained from molecular dynamics (MD) simulations has been compared to defect production information determined with a model based on the binary collision approximation (BCA). The total numbers of residual defects, the freely migrating defects, and the sizes of immobile clusters compare very well, especially when the end-point conditions of the two simulations are taken into account. Thus, it appears that a BCA-based model, properly calibrated with information from MD simulations, can give a correct description of defect production in displacement cascades of any energy. A strategy has been suggested for integrating the details of the cascade quenching phase determined by MD into a BCA-based model that is practical for simulating cascades of much higher energies than MD alone can achieve.

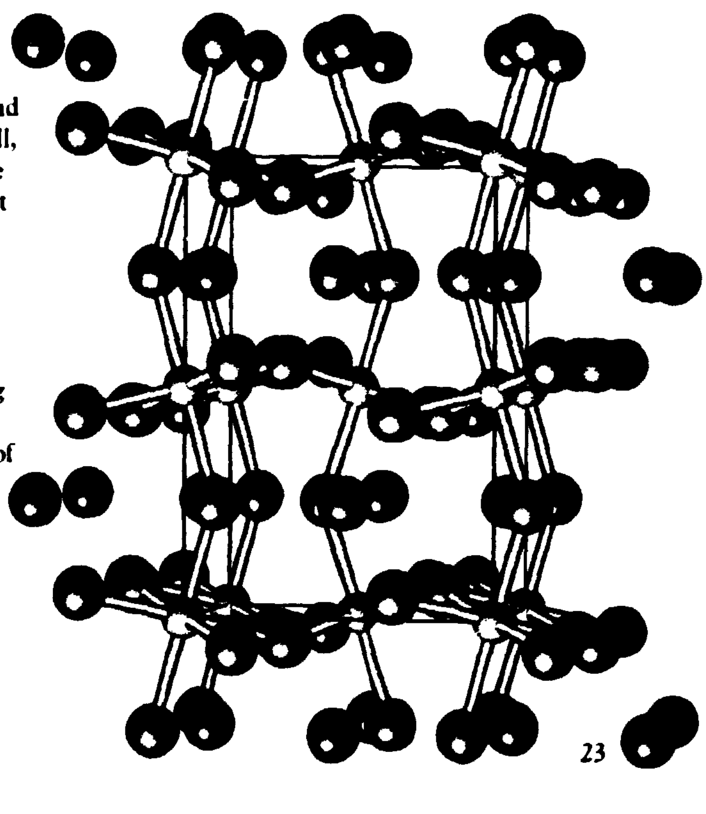
2.7 Solid Electrolytes - New Materials and Methods

Certain ceramic materials allow high diffusion rates of ions through the bulk of the grains as well as across grain boundaries. These materials are called solid electrolytes and have numerous applications as electrolyte membranes in oxygen sensors, gas purification systems, solid oxide fuel cells and steam electrolysers.

The technologically important solid electrolytes are those enabling diffusion of lithium-, sodium-, oxygen- and hydrogen ions. Materials, which have both high ionic and electronic conductivity are candidate materials for electrodes, catalysts and semi-permeable membranes for gas separation. The fundamental properties of this class of compounds are studied with a range of electrical-, thermomechanical-, and structural methods. Modelling studies are also performed in order to be able to predict the existence and conductivity of new materials. The input for such model calculations is thermodynamic and crystallographic data.

The work is both experimentally and theoretically addressed to the study of materials with fluorite-, perovskite-, ferrite-, and pyrochlore structures. Two Ph.D.-studies in this

Crystal structure of the protonic conductor $SrCeO_3$. Strontium atoms are coloured blue, cerium atoms green and oxygen atoms are red.



field are being carried out. Ionic conductivity, thermoelectric power (Seebeck coefficient) and crystal structure have been determined. An algorithm for calculating the concentration of electronic and ionic defect concentrations in complex oxides has been developed; in this routine nine equations (some of which being non-linear) are solved simultaneously. Collaboration between the Department and Materials Research Centre, Oslo and Technical University of Norway, Trondheim has been established. Part of this collaboration has involved the exchange of scientists over extended periods.

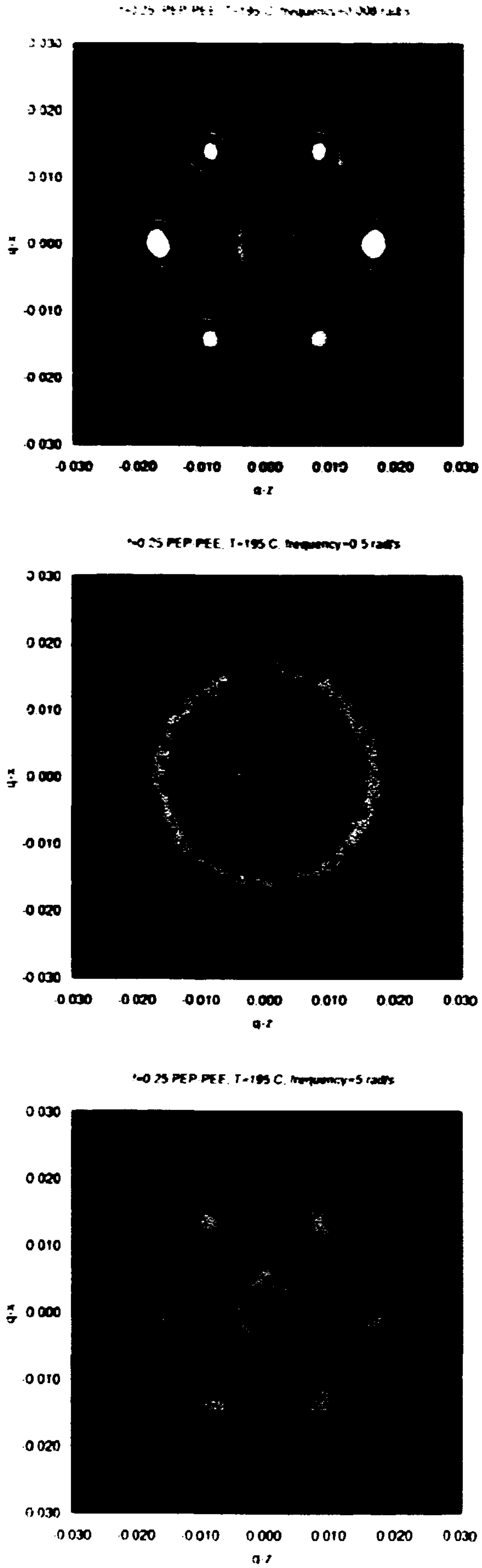
Equipment for the synthesis of block copolymers. The reaction vessel is shown in close-up.

2.8 Block Copolymers

In the continued work on the phase behaviour of block copolymers close to the order-disorder transition, a study has been initiated on the dynamics of deformation of ordered block copolymers. A block copolymer consists of two different kinds of polymer chains linked by a chemical bond. The macroscopic phase separation often observed in polymer mixtures is precluded. Instead, local segregation occurs on a length scale dictated by the size of a single block copolymer molecule. Above the order-disorder transition temperature, the long-range is lost and the systems behave in a way similar to polymer melts. This fact is utilized in applications of block copolymers. Processing is performed at temperatures above the order-disorder transition temperature whereas application conditions are at temperatures below this transition temperature. The locally segregated block copolymers fill space through the formation of different microstructures. On a short length scale - < 10Å - these materials are amorphous. However, at a larger length scale - ~100Å - one discovers a periodic arrangement of different amorphous domains analogous to the placement of atoms in a crystal lattice.

In a particular PEP-PEE diblock copolymer, (poly(ethylene-propylene)-*block*-poly(ethylethylene) with molar mass, $M_n = 1.00 \times 10^5$ g mol, two such ordered structures are observed. One ordered structure, stable below 175°C, consists of rods of the minor component - PEP - packed on a hexagonal lattice in a matrix of the major component - PEE. At temperatures above 175°C, the minor phase forms spherical domains instead of rod-like domains. These spherical domains pack on a body centred cubic (BCC) lattice. Above 217°C, the order-disorder transition temperature, the long-range order is destroyed.

The existence of the BCC structure has interesting consequences for the mechanical properties of the material. In linear polymers with sufficiently high molar mass a so-called rubbery plateau is manifested in the dynamical mechanical properties. Above a certain frequency, the elastic dynamical mechanical modulus, G' is of the order of MPa and is



SANS intensity data obtained from a PEP-PEE material that forms a BCC ordered state. Shear applied at various frequencies in a plane perpendicular to the neutron beam.

practically independent of frequency, ω. At low frequencies, the material behaves like a liquid and $G' \sim \omega^2$. In the BCC structure, a second plateau is seen at lower frequencies than the rubbery plateau. In this plateau, which is three orders of magnitude wide in frequency, the modulus is between 1 and 10 kPa.

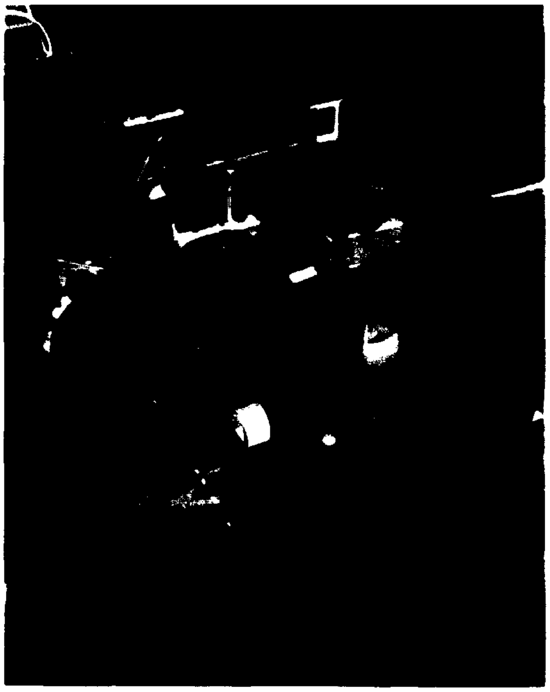
The response of the BCC structure to large strain (~100%) oscillatory shear deformation has been studied by small angle neutron scattering (SANS). Such a shear field can change the normally polycrystalline material into a near-perfect single crystal, with the (111) planes of the BCC structure perpendicular to the shear gradient. However, if this single crystal is subjected to oscillatory large strain shear deformation, at a frequency in the middle of the secondary plateau, the crystalline order is basically destroyed. At even higher frequency, the single crystalline long range order reappears.

This work is conducted in collaboration with Department of Chemical Engineering and Materials Science, University of Minnesota and The Department of Solid State Physics, Risø.

2.9 New Polyester for Optical Information Storage

Novel side-chain liquid crystalline polyesters have been synthesized which show great promise as erasable media for optical information storage. The chosen strategy used in the synthesis of these polymers allows great flexibility in the molecular architecture of the polyesters. There are three adjustable design parameters: (i) The spacing of the side-chains which respond to laser light. This side-chain spacing can be altered over an extended range by altering the length of the dicarboxylic ester precursor. (ii) The length of the flexible spacer, the actual linker between the photoactive azo group in the side-chain and the main-chain. (iii) The molecular mass of the polyesters can be adjusted in the actual polymerization step. These novel polyesters are synthesized in the long lasting collaboration with CNR, Chemical Engineering Department, University of Pisa, Italy.

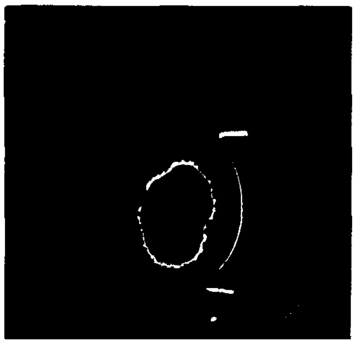
The optical information storage potential is being investigated in a newly established collaboration with the Optics and Fluid Dynamics Department, Risø. It has been shown possible to record holograms with a resolution greater than 5000 lines/mm on unoriented films of these polyesters with an argon ion laser



Experimental set-up for two beam holography. A blue-green (argon ion) laser beam is split into two by a polarization beam splitter and recombined on the photosensitive polyester film.

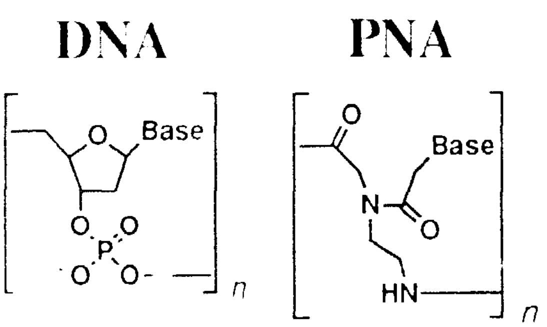
recording energy of about 1 J/cm². Diffraction energies greater than 30% have been achieved using polarizing recording of holograms. Holograms recorded 10 months ago show no sign of decay in the diffraction efficiency. However, holograms can be simply erased by heating to approx. 80°C. The films are then available for re-recording. In polyesters employing medium long dicarboxylic acid precursors, it is possible to observe novel biphotonic processes. Gratings generated through the interference of an argon ion laser beam are fixed in the materials by the light from a red laser (He-Ne). These holograms have a typical efficiency of about 15% with a characteristic size of 1 mm corresponding to the diameter of the red laser beam. One interesting aspect of the recording is that one could wait for several minutes between the exposures of the argon ion laser beam and the red laser beam. These polyester materials are expected to have a great potential as holographic interconnects in an optical neural network.

The clear, circular area in this side-chain liquid crystalline polyester film is caused by exposure to the argon ion laser beam.



2.10 DNA with a Polyamide Backbone

Working in collaboration with the H.C. Ørsted Institute and the Panum Institute, we have recently participated in the construction of peptide nucleic acids (called PNA), i.e., analogues of DNA in which the familiar nucleobases of DNA are attached to a polyamide backbone rather than to a sugar-phosphate backbone. The backbone of PNA is structurally homomorphous with that of DNA but chemically it is totally different. PNA oligomers, substituted with thymine nucleobases, were found to bind very efficiently and sequence-specifically to their DNA complements. It was also observed that PNA



recognizes its complement in double-stranded DNA, namely by strand displacement. In conclusion, a backbone has been discovered, for the first time, which can replace the entire backbone of DNA, with the resulting molecule retaining the base-specific hybridization properties. These results were published last year in »Science« and an editorial comment was brought in »Nature«. In 1992, further studies have demonstrated that a second nucleobase, cytosine, can be incorporated into PNA, and that it does recognize its complementary nucleobase, guanine, in DNA. Two communications have been published in the »Journal of the American Chemical Society«, the premier journal of chemistry, and one article has been accepted for publication in the »Proceedings of the National Academy of Sciences USA«. Editorial comments have appeared in several journals including »Angewandte Chemie« and »Chemical and Engineering News«. An agreement on contractual work has been arranged with the newly formed company PNA Diagnostics A/S.

2.11 Electron Microscopy

An ElectroScan™ environmental scanning electron microscope (ESEM) was installed in the Department in December. The purchase was made possible by generous support from the »Velux Foundation of 1981«. An ESEM differs from a conventional scanning microscope in that it can operate with a pressure of up to 50 Torr in the specimen chamber. A conventional SEM typically works at a pressure of 10^{-5} to 10^{-6} Torr. An important advantage of the relatively high pressure in the ESEM is that it is possible to examine electrically insulating materials without covering them with a conducting material. The pressure of saturated water vapour is approximately 20 Torr at ambient temperature. It is therefore also possible to examine materials containing water, like, for example, cement or biological materials. The microscope is equipped with a number of attachments: a high-temperature stage that can work at temperatures up to 1400°C; equipment for in-situ deformation; and a microinjector and micromanipulator. The microscope is further equipped with an X-ray spectrometer that can detect boron and heavier elements and with hardware and software for image analysis. A system has been installed in collaboration with Jydsk Telefon, Aarhus, which allows images from the ESEM to be transmitted via the ISDN telephone net. This gives the possibility for customers connected to the ISDN net, to follow an examination of a specimen in the ESEM on a personal computer screen in their own office.

Micrograph of wood taken on the ESEM, operating at 20 keV. No conductive coating was applied to the specimen, charging by the beam being prevented by ~10 Torr of air in the specimen chamber.

The ElectroScan™ environmental scanning electron microscope (ESEM). The purchase was made possible by a generous grant from the »Velux Foundation of 1981«.

Microstructural characterization has been carried out for a number of external customers. Electron microscopical work has also been done for the Physics Department, the Department of Nuclear Safety and the Department of Combustion at Risø.

A collaboration agreement in the field of electron microscopy and image analysis between the Materials Department at Risø, the Department of Chemistry at the Danish Technological Institute and the Institute of Mathematical Statistics and Operational Research at the Technical University of Denmark was signed in January. The purpose of the agreement is provide customers with a joint offer of techniques in the field of microstructural characterization and to coordinate the research efforts and the procurement of equipment in the participating departments.

Electron Back Scattering Patterns (EBSP) and Kikuchi Patterns

To correlate microstructure and texture studies, it is often important to be able to determine the crystallographic orientation of selected local regions in a sample. Since this often has to be done for a large number of regions, such microtexture techniques have to be fast and easy to use. This has been the goal for the development of two such microtexture techniques, the electron back scattering pattern (EBSP) and the Kikuchi pattern technique.

With the EBSP technique in the scanning electron microscope, it is possible to determine the local orientation of regions of about 1 μm in size. This technique is now fully automated. The patterns are indexed using a modified Hough transform procedure, and the sample is moved with respect to the electron beam by computer controlled step motors. The operator can choose how the sample is moved: either by preselecting points in the microstructure at which the orientation has to be determined, or by defining a line or a mesh along which the orientations are to be measured. The reliability of the procedure was checked by comparing the automatic indexing with repeated manual indexing. In all cases the result of the automatic procedure was within the scatter of the manual results. Furthermore, the procedure is fast; it typically takes less than 15 seconds to index a pattern.

With a Kikuchi pattern analysis technique in the transmission electron microscope, local orientations of areas down to ~50 nm can be determined. In general, microtexture studies on this scale require a simultaneous inspection of the microstructure by the operator. Computerized sample movement is therefore not necessary in this case. The indexing is done by selecting and marking three bands in the Kikuchi pattern by changing the voltage to the detector coils. This procedure is easy to use, it requires no crystallographic knowledge, and it is fairly fast; typically the indexing takes about a minute.

During the year, the EBSP technique has mainly been used for studies of growth rates during recrystallization of heavily deformed aluminium and copper, and for studies of the texture development in Al-SiC metal matrix composites. Detailed investigations of the distribution of orientations within grains has been carried out for deformed and for recovered aluminium using the Kikuchi pattern technique.

2.12 Non-Microscopical Characterization

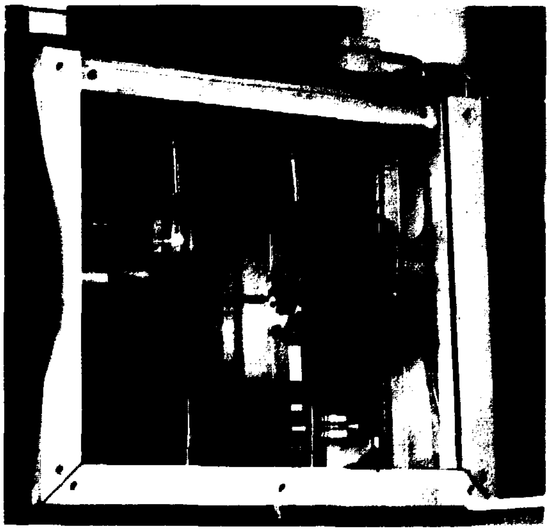
Optical and electron microscopy is used extensively in the Department to characterize the microstructure of materials. As such, the spatial variation in structural elements of the materials are documented and these variations related to materials properties. Non-microscopical techniques tend not to reveal point to point differences in the material, but provide average bulk information about changes in the material in response to external factors. Often, the data obtained from non- microscopical techniques may be directly applied in the developing materials models.

Internal Stress Determination by Neutron Diffraction

The neutron diffraction technique for internal stress determination finds a great variety of applications in Materials Science. Primarily because it offers the ability to monitor internal deformations in selected parts of a microstructure. In comparison with the traditional testing techniques, where an external device like a clip-gauge or a strain gauge is used to give information on the overall macroscopic deformation, then the selective character of the diffraction technique offers a unique opportunity to study deformation processes in much greater detail.

In 1992 the focus has been on studies of metal/ceramic composites, primarily the common aluminium/silicon carbide system (Al/SiC). In such a system the diffraction technique provides the ability to study the internal deformation characteristics. A number of investigations have been performed using a small stress-rig device, whereby uniaxial test samples can be loaded in-situ, while lattice deformations are studied by neutron diffraction. One specific investigation was related to the modelling of creep in MMC's as described earlier. Here elevated temperature creep experiments were performed in-situ using the neutron diffraction technique to monitor the evolution of the lattice strains in the composite.

The same set-up has been used for a series of hot-tension experiments also on the Al/SiC system. Hot-tension experiments can be used to evaluate the effect of internal stresses on the composite deformation characteristics. The interpretation is however complicated by the fact that relaxation is an on-going process throughout the hot-tension experiment. In order to investigate this effect, the hot-tension experiments have been supplemented by studies of the relaxation behaviour of the composites. This was done using a small furnace originally developed and used for texture investigations. Small samples of the composite materials have been taken through specified thermal loading paths, while the relaxation was monitored by neutron diffraction by studying the development of lattice strains.



Neutron diffraction stress-rig. New grips, fast load-control, semi-sealed high temperature chamber and on-line data acquisition were added in 1992.

Another type of a composite-like system of interest is Duplex steel. Duplex materials consist of a ferritic and an austenitic phase, and in studying the deformation behaviour of these materials the neutron diffraction technique offers the unique opportunity to study how these two phases deform individually. Using the stress-rig, small uniaxial samples have been loaded in tension and the diffraction technique has been used to monitor the deformation behaviour of the two phases.

The neutron diffraction technique is primarily used as an elastic strain monitoring device by studying diffraction peak shifts. However, the peak shape also contains information on the plastic deformation at a microscopical level. By carefully monitoring the peak shape at selected levels of uniaxial plastic deformation, the neutron diffraction technique has been used to study plastic deformation in test samples of copper, nickel and iron.

Texture Measurements by Neutron Diffraction

The texture work has, during the year, covered studies such as: i) the texture development during hot deformation of aluminium. This is part of a Brite/Euram programme, ii) the effects of different heating rates and annealing temperatures on the development of recrystallization textures in heavily deformed aluminium and iii) comparison of results for recrystallization kinetics in copper determined by neutron texture measurements and by stored energy measurements.

About three weeks of the neutron texture beam time have been used for the »Large Installation Programme«, in which researchers from EC are invited to apply for beam-time, and after an evaluation of the proposals, to come and perform the measurements. In total, seven scientists and students performed neutron texture measurements under this programme. The project covered investigations of preferred orientations in cold-extruded steel rods, texture and mechanical damage of highly deformed aluminium alloys, textures in two phase fcc/bcc materials and texture analysis during phase transformations of low carbon steel.

Positron Annihilation

The positron annihilation equipment consists of three lifetime spectrometers, a Doppler broadening and an angular correlation spectrometer. They are used for studies of defect behaviour in various materials. Positrons which are injected into a material are often attracted to vacancy type defects (e.g. single vacancies, vacancy clusters, vacancy-impurity clusters, gas bubbles, dislocations, surfaces, interfaces etc.). Positron annihilation techniques may therefore detect the presence and characteristics of such defects.

Work on ultrafine powders (mainly Ni and Mo) has been concentrated on studies of the reduction of surface oxide in hydrogen and of possible decomposition in inert gases and vacuum, as well as on the question of adsorption of gases on particle surfaces. Reduction of the oxide was found to be required for subsequent sintering of the particles to take place. During sintering, defects develop or become incorporated into the growing particles. This work is part of a collaboration with the Technical University of Denmark within the framework of the Centre of Powder Metallurgy, where the experimental results form part of the basis for the efforts to establish models for the sintering processes in both nano- and micro-sized particles.

In a continued collaboration with KfK Karlsruhe, Germany a series of measurements has been carried out on helium bubbles generated in copper implanted with He at different temperatures and to different doses in order to obtain information on bubble size and concentration and on density of He in the bubbles.

3

Materials Engineering

A thorough knowledge of the mechanical properties of engineering materials is essential for the design of advanced components and structures. Of special interest are composite materials for wind mills, helicopters and lightweight pressure vessels as well as engineering ceramics. The research activities in Materials Engineering are centred around structural mechanics analysis of destructive and non-destructive materials testing procedures. A considerable number of the projects are carried out in close collaboration with Danish and European industrial partners.

3.1 Internal Stress by Neutron Diffraction

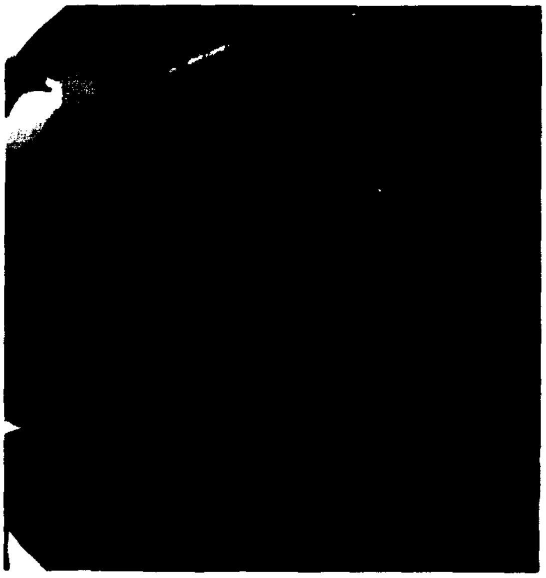
The reason for using the neutron diffraction technique for internal stress studies in Materials Engineering is different from that of the applications in Materials Science. Although the experimental principles resemble one another, the uniqueness of the neutron diffraction technique as a tool in Materials Engineering lies in the high penetration power of neutrons rather than in its ability to monitor deformation in selected parts of the microstructure. The diffraction technique can hence be used as a non-destructive probe for internal stresses inside engineering components; so relatively deep inside that no other technique can provide the same depth profiling capability. Typically the technique is used to study the spatial distribution of internal strains and stresses in components.

One example is a recent investigation of the internal stresses and strains in a shrink-fit ring assembly. The neutron diffraction technique was used as a non-destructive evaluation technique to verify the shrink-fit pressure between the rings. Other recent applications are studies of internal stresses in welded steel assemblies, and in forged steel components for the automotive industry.

3.2 Design of Wing Blades in Composite Materials

The use of wind energy requires wind turbines for the collection of this energy. At present, wind turbines are generally of the horizontal axle design with rotors of three, and in some cases two wing blades. The dimensions of the wind turbines are slowly increasing, and at present the large wind turbines have rotors of diameters 50 - 60 m, corresponding to a maximum generator power of up to 2 MW.

These long wing blades are all of the cantilever design with a root fitting mounted on the hub of the rotor. During operation of the wind turbine, the rotor and its blades are under load both from the wind-forces and from gravity, such that for larger wing blades the importance of gravity loads increases relative to the wind loads. During rotation, the blades are loaded in an oscillating mode, i.e. fatigue, both in tension and in compression (due to wind-loads) and in tension-compression (due to gravity-loads). With rotational speeds of 20 - 30 revolutions per minute, the total number of fatigue load cycles over a 20 year lifetime is more than 100 million



The hub of a wind turbine rotor.

cycles. The materials for the wing blades therefore need to be selected from materials with low density (to reduce gravity loads) and with good long term fatigue strength. Such materials are fibre reinforced polymers, and the practical materials of use in to-day's wind turbines are glass-fibre-polyester and glass-fibre-epoxy composites.

Extensive studies are in progress to evaluate the fatigue behaviour of such composites, both on a national scale and on a European scale. The aim is to understand the materials during fatigue loading and to establish design rules and standards. The results of an extensive European programme (under the EC-JOULE- programme in renewable energies) have led to information and understanding on glass/polyester, glass/epoxy and wood/epoxy materials subjected to a wide range of external conditions, like mechanical (fatigue) loading, humidity, impact damage, as well as methods to record the state of internal damage of the materials during loading.

In relation to materials selection and design, extensive fatigue measurements have been performed on glass/polyester materials. Data up to 10 million cycles is established; the long lifetimes are achieved at low loads, and it is likely that a fatigue limit is established so that at very low loads the lifetime will be infinite. The data for tension fatigue and for compression fatigue are very similar, indicating that earlier observations of low compression fatigue data may not be correct. It is also established that a relatively large fraction of fibres arranged in the loading direction ensures good fatigue properties, and off-axis fibre orientations, up to 45° - 60°, will reduce the fatigue strength significantly.

During the experimental tests of composites the change in stiffness of the material is recorded, and shows a slow and steady drop in the stiffness during the fatigue lifetime. This observation forms the basis for the establishment of design rules, where a certain stiffness reduction, e.g. 5%, may be taken as a design criterion. This philosophy will be in contrast to typical metals and alloys where loads or cycles to complete failure of the material is taken as a design criterion. To further assist the designer, diagrams are worked out to show the effect on fatigue life of the different load combinations, e.g. tension fatigue and tension-compression fatigue.

The loading by a simulated wind-load spectrum has been tested, and shows that the fatigue performance is very similar to that expected from testing with constant amplitude loading. The monitoring of the material's state of damage has been attempted with recordings of the material-temperature, which increases during the fatigue life, and of the hysteresis-loop-area of individual fatigue cycles, which also increases. Such recordings show a close correlation with the change, i.e. reduction, in material stiffness during the fatigue life, and thus constitute potential methods of monitoring the state of damage in the composite materials. Studies on glass/epoxy composites show a fatigue behaviour which is very similar to that of glass/polyester, and thus confirms only a small, if any, difference of these two composite types in fatigue. Supplementary fatigue testing of glass/epoxy after impact from hailstones and under wet conditions has demonstrated that the impact has very little effect on fatigue performance, while the wet conditions reduce the fatigue performance, relative to testing under dry conditions.

Studies of wood under fatigue loading show rather similar behaviour for a range of wood species including both hard-wood and soft-wood. The most significant parameter is the density of the wood, with higher fatigue strength at the higher density. It is normally necessary to assemble wood-sections from smaller parts by gluing; a study of different types shows some, but rather small, differences in fatigue performance.

As a link between materials performance and full scale wing blades, a number of wing-sections and wing-root-assemblies have been tested in fatigue. This has demonstrated excellent overall performance of such part-components, and also allowed identification of weak points in the design, by monitoring of the material state of damage during fatigue loading.

3.3 Local Damage Effects in Polymer Composite Panels

An understanding of the resistance against local damage in polymer composite materials is important for the application of this class of materials in mechanical engineering in general and of special importance within the transport industry. Research on this subject is carried out in the Department within the framework of internationally funded programs with an emphasis on manufacturing defects and local impact damage in laminate panels and in

Ultrasonic scan of impact region in a 2 mm thick, 17 layer carbon composite. This thickness plot shows that the damage, which is almost invisible from the impact side, contains large delaminations below the surface.

sandwich panels. The manufacturing defects may be either porousness or localized delaminations. Impact damage is localized damage which may have been initiated by, for example, dropped tools, impact of foreign objects etc. In the investigation, emphasis is given to practical methods for the detection and description of the defects and to a comparison of the load carrying capacity of the defective or damaged material to that of the undamaged material.

A number of carbon/epoxy laminate panels, measuring 20×200 m^2 and 2.5 mm thick (17 plies) have been investigated. In this case, the panels contained simulated manufacturing defects of different sizes in the form of delaminations. The panels' structural behaviour for compressive loading, the most severe loading for this kind of defect, was established by mechanical testing. The tests showed that the static load-carrying capacity was not influenced by the defects to any large extent, but that for the larger delamination areas, local buckling was observed at the defect area.

3.4 Data Acquisition and Control for Mechanical Testing

Advanced mechanical testing entails the increasing use of automatic data acquisition and control equipment. The laboratory for mechanical testing has implemented this approach with the appropriate use of analogue to digital conversion (A/D) computer boards, communication interfaces and software. All the boards and interfaces are of the plug-in type designed for a PC. The laboratory uses a variety of data acquisition and control software, both commercially available and written in-house. This flexibility of both software and hardware makes it easier to pre-design test runs on new materials, based on initial modelling data.

The hardware and software that are in use enable amongst other things the following:

Acquiring Data: Data acquisition from many types of inputs such as analog , digital, thermocouple, counter, frequency, strain gauge along with acquisition and control via A/D boards or RS-232/IEEE- 488 interfaces.

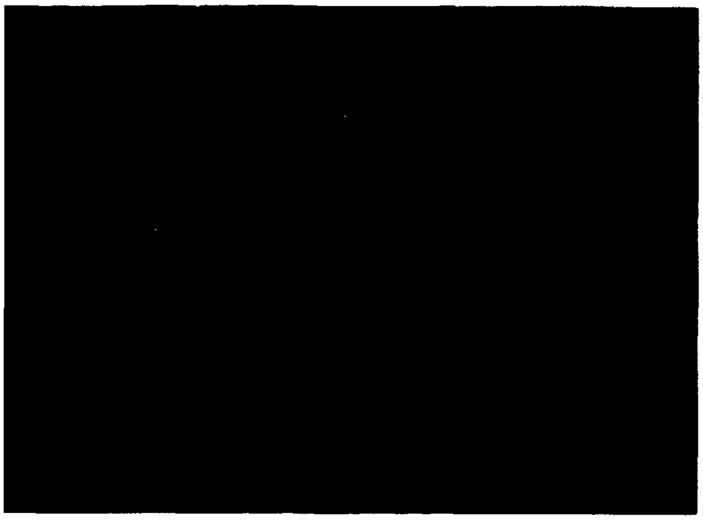
Real-Time Display: Real time calculations, allowing the display of information which is more meaningful to the operator than a simple voltage plot, such as load-strain curves, hysteresis plots, stiffness analysis over a period of time, temperature curves etc.

Post-Acquisition Analysis: The saving of test data in a variety of file formats, which allows post data processing in a large number of external programs, such as spreadsheets, database, maths programs etc.

Data Acquisition and Control in Elastomeric Testing: A good example of advanced data acquisition and control is our recently established Elastomer Testing Programme. In this case the acquisition of data, and the control of test run parameters are achieved via IEEE-488 communication interfaces, and with the use of a specially designed elastomer software package.

In a typical elastomeric test, an elastomer specimen is exposed to various amplitudes, cycling frequencies and different temperatures within the same test run. The run is performed in accordance with a pre-defined set-up that continuously changes the amplitude and frequency at a specified temperature. The software inputs the pre-defined test data which enables the test machine to feed back the actual run data to the computer. If the run-amplitude is within the pre-set amplitude error, the results are calculated and shown on a monitor as a real time display curve. The calculated test data from this section of the test set-up are stored in the computers random access memory.

If the achieved run-amplitude is not within the pre-set amplitude error, the software will automatically tune the run-amplitude into the right size by gradually increasing the amplitude input signal to the test machine. When the test run is finished all the data is stored on a disk in an ASCII file format. The ASCII file can then be conveniently ported to another system for analysis. The photograph shows the elastomeric set up in the laboratory.



Wear in silicon carbide has been produced by shallow surface indentation and subsequent lateral cracking. The much softer alumina has undergone greater surface penetration and the removal of material by plastic flow, leaving micro-ploughing tracks on the surface. Silicon carbides have a wear resistance of more than three times that of alumina.

3.5 Abrasive Ceramics

The Centre for Advanced Technical Ceramics has an ongoing programme investigating wear resistance and wear mechanics. A current feature of this programme is the investigation of resistance to three-body wear, i.e. wear produced by abrasive particles trapped between two counter faces. To investigate material resistance to abrasion, a variety of ceramic materials have been tested against a silicon carbide slurry under standardized conditions. This has produced a ranking of materials, which can be used as a general guide to material selection. Of particular interest, however, is the ability to predict the wear properties of materials from known parameters. This involves an understanding of the mechanisms of material removal from the surface. This approach has been followed via both scanning electron microscopy and profilometry.

Wear in silicon carbide has been produced by shallow surface indentation and subsequent lateral cracking. The much softer alumina has undergone greater surface penetration and the removal of material by plastic flow, leaving micro-ploughing tracks on the surface.

3.6 Fracture of Ceramic Matrix Composites

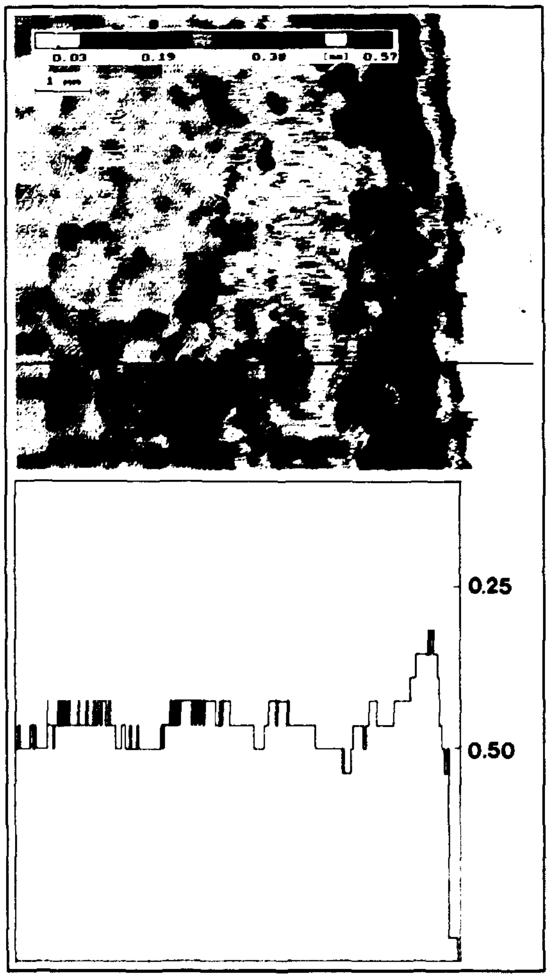
Ceramic matrix composites are usually divided into two classes: »damage-tolerant« composites and »crack-sensitive« composites. The class of damage-tolerant materials, such as composites with continuous fibres, is characterized by having non-linear constitutive laws, due to the evolution of damage. Thus, it is the evolution and interplay of various damage mechanisms that is important and requires further understanding. Work in the Department concerns the study of the transition between notch sensitivity and insensitivity as well as the modelling of mechanical fatigue mechanisms.

The second class of ceramic composite materials is the class of crack-sensitive composites, and in this area the aim is to develop materials with higher fracture toughness. For the same design stress this will allow a larger (critical) flaw size, which may then be detectable by non-destructive testing. Another means of improving the material reliability is to develop composites with rising fracture resistance with increasing crack length (R-curve behaviour). For both cases, toughness can be imparted by energy absorbing mechanisms, such as crack bridging, pull-out, phase transformation, etc. These mechanisms are best exploited in composites, such as ceramics reinforced with short fibres or platelets. A relationship between the R-curve behaviour and strength variability has been derived. Methods for measuring R-curve behaviour by the incremental growth of cracks are being developed.

3.7 Ultrasonic Testing of Low Enriched Uranium (LEU) Fuel Plates

Fuel elements for material testing reactors are produced in the Materials Department. This element is a plate fuel type. Each plate is made as a sandwich construction with an aluminum frame and cover plates encapsulating a uranium core. Recently the fuel type has been changed from high enriched uranium to low enriched. Now the core is made of a uranium silicide powder mixed with aluminum powder using powder metallurgy techniques, instead of the previous one which used a uranium-aluminum alloy core manufactured by casting. Two inspection and control tasks in this new type of fuel plate have been solved using precision ultrasonic scanning. One is non destructive measurement of the thickness of the aluminum cover plate over the core material, and the other is detection of blisters between the single layers in the fuel plate. Using our scanning system, millions of single measurements can be made in a scanning pattern. The highest resolution in the pattern is 10 μm in both directions. The results of ultrasonic scanning may now be presented as colour plots where the different distances or echo heights have different colours. The ultrasonic time of flight measurement of the distance from the plate surface to the uranium core is of special interest because it offers the possibility for non destructive evaluation of the thickness of the cover plate. The current practice is an acceptance or rejection of a fuel plate production batch based on a single microscopic cross section from a single plate. By using ultrasonic scanning, the examination can be made non destructively, and cross sections can be made everywhere. Furthermore a graphic colour presentation of the thicknesses all over the plate can be made.

Ultrasonic distance plot of a part of an LEU fuel plate end with different colours showing variations in cladding thickness. The exact thickness may be measured along any single scanning line, as shown. Coverplates over the fuel are normally thinner near the core end (»dog-boning«). These measurements may be used in production control to ensure that coverplates are thicker than a pre-set minimum.

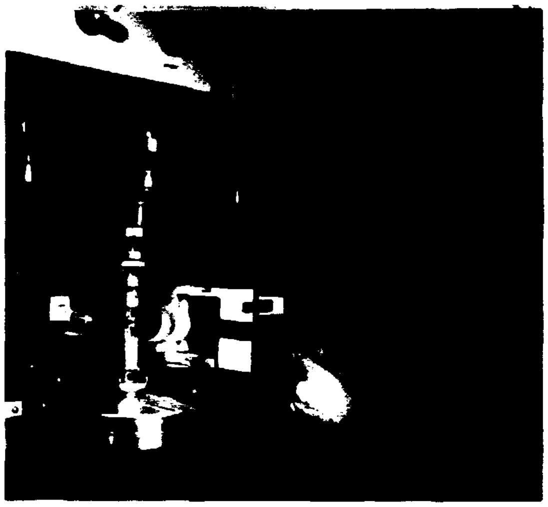


Measuring peak echo values instead of distances, the blisters (non bonded areas between cover plate and frame) outside the core area as well as blisters in the cover plate itself over the core can be found and documented in colour plots. Using ultrasonic scanning should result in improved quality documentation. This may reflect the demands in the specifications.

3.8 Polymer Rheology

The polymer group is equipped with rheological instruments that allow measurements of viscosity and dynamical mechanical properties in shear deformation. The instrumentation covers a wide range of shear rates and frequencies. It is possible to perform measurements on soft systems with viscosities similar to water as well as solid systems such as polymer melts or networks in the glassy state. The instruments are mechanical spectrometers operating in rotational geometries such as plate and plate, cone and plate and torsion of rectangular specimens. The temperature can be controlled between - 150°C and 350°C with a possibility of using inert atmosphere environment. It is also possible to characterize the development of mechanical properties as a function of time in curing systems.

This instrumentation has been central to the involvement in the two projects »Polymeric Systems with Controlled Structure and Characteristics« and »Materials Technology in the Danish Rubber Industry« in the framework of the Danish Materials Technology Development Programme. In the project »Materials Technology in the Danish Rubber Industry« the low strain dynamical mechanical properties of a series of carbon black filled rubbers has been characterized as a function of temperature and a highly non-linear behaviour of rubbers at very low strains has been identified. In the project »Polymeric Systems with Controlled Structure and Characteristics« one focus has been



Rheometrics RMS800 mechanical spectrometer. The sample being tested is a chewing gum.

the potential of dynamical mechanical analysis to characterize polymeric systems with a potential application as a pressure sensitive adhesive.

3.9 Film-Supported Solid-Phase Synthesis

The technique of solid-phase synthesis employs an insoluble solid matrix on which to build bio-polymers such as peptides. In collaboration with Nobel Laureate Bruce Merrifield and his colleagues at The Rockefeller University, USA, we have developed a new type of solid matrix PEPS made up of a polystyrene-grafted polyethylene film which provides a number of advantages in the practical performance of peptide synthesis. A US patent on the PEPS material is expected to be issued in 1993. The original work was published in the Journal of the American Chemical Society in 1989. Because of its hydrophobic nature, PEPS is not wetted by aqueous solutions and, therefore, it is not well suited for diagnostic testing, affinity purification etc. To overcome this limitation, studies in part as a graduate program, are now well under way to construct a hydrophillic version of PEPS. Contractual work employing the PEPS material has been carried out for Bay Development Corp.

4

Materials Technology

The manufacture of advanced materials components often requires new processing, fabrication and joining techniques. Pilot plant studies of the production of fibre reinforced polymer composites, fine-powder metallurgical components and thin ceramic layered structures demand the construction of specialized equipment. This research and development also provides test specimens of new advanced materials for other programmes of the Department. The research activities in Materials Technology involve the manufacture of components of polymer matrix composites, engineering ceramics, prototype solid oxide fuel cells and fine-powder metals. Brazing and bonding techniques are being applied to a variety of these materials. The research programmes are carried out partly within the three Danish centres, Advanced Technical Ceramics, Powder Metallurgy and Polymer Composites, and partly in collaboration with other Danish and European research organizations and industrial partners.

4.1 Manufacturing Processes for Advanced Composites

The involvement of the Materials Department in manufacturing of continuous fibre reinforced plastics serves mainly three purposes: a) the study of the fundamental principles of filament winding, autoclave processing, and resin transfer moulding, b) fabrication of test specimens, and c) development of prototype components. The processing equipment consists of a computer-controlled filament winding machine, a hot-air high-temperature high-pressure autoclave, and equipment for resin transfer moulding.

Two projects on manufacturing technology for high temperature thermoplastic composites with continuous fibres are being conducted. The first project is on filament winding and the second one is on fibre pre-forms. (Fibre pre-forms are a pre-shaped fibre structure which has not yet been consolidated to the final structural component. Fibre pre-forms can be woven, knitted, braided or stitched together). The purpose of the projects is to examine the possibilities, limitations, and the characteristics of a fabrication technique where the material (fibre + matrix) is either wound onto a mandrel at high temperature inside an oven or placed in a mould at high temperature in the form of a fibre pre-form, and consolidated by a subsequent autoclave process.

In the filament winding projects a new technique for fabrication of components of thermoplastic fibre composites is under development. Basically, the technique consists of winding the materials (fibres and matrix) onto the mandrel at high temperature inside an oven. The oven is electrically heated to the process temperature, and the material in the form of a thermoplastic prepreg is put into the oven through a pre-heating zone, to ensure that the material reaches the process temperature before it is wound onto the mandrel. The first material being investigated is carbon fibre/PEEK.

In the pre-form project the process technology for carbon fibre/PEEK has been established both for the material in a prepreg form (APC-2 from ICI) and in a post impregnated form (postpreg) (Filmix™ from Heltra). Two autoclave processes with different pressure cycles have been investigated. One with, and one without, the consolidation-pressure applied during the heating of the material to the process temperature. The influence of adding more matrix (PEEK-foil) to the Filmix™ material has also been investigated. The quality of the Filmix™ laminates (porosity content) has been determined as a function of the consolidation pressure at 3, 7, 14 and 20 bar for the three experiments.

An APC-2 laminate has been consolidated at a pressure of 3 bar, and the porosity content determined. The consolidation pressure is the most important of the investigated process parameters. By changing the consolidation pressure from 3 to 20 bar the porosity content in the Filmix™ laminates drops from 13 to 1 vol-%.

High temperature, high pressure autoclave for fabrication of composite components. The car door panels were fabricated from a hybrid cloth woven from thermoplastic and structural fibres.

Some improvement can also be obtained by pressurizing the lay-up during heating to the process temperature. The improvement is obtained if the pressure is so high, that it can prevent the stretched matrix fibres in the Filmix™ material from shrinking. If the matrix fibres shrink, it will result in disarranged carbon fibres and folds in the lay up which is undesirable, because it leads to a poor material quality. The experiments have shown that a pressure of at least 7 bar is necessary if shrinkage of the matrix fibres should be avoided. If the consolidation pressure is 20 bar the advantage of pressurizing the lay up during heating disappears because the higher pressure is able to »smooth out« the folds and disarrange the carbon fibres.

The matrix content in the composite adding some PEEK-foil to the Filmix™ laminates and material has also an important influence on the material quality. By adding a PEEK-foil to the Filmix™ laminates and changing the matrix content from 40 vol-% to 50 vol-%, the desired consolidation pressure to produce a laminate with a porosity content of less than 2 vol-% is dropped from 15 bar to 6.5 bar.

Comparison of process conditions for laminates fabricated from woven Filmix™ fabric and APC-2 tape, shows that it requires less pressure to produce high quality laminates from the APC-2 tape. An APC-2 laminate with 60 vol-% of carbon fibre consolidated at 3 bar has a porosity content of 0.5 vol-%. Laminates of Filmix™ material need 20 bar of consolidation pressure to achieve high quality. A Filmix™ laminate with 57 vol-% carbon fibre content was fabricated with a porosity content of 0.9 vol-%, and a carbon fibre content of 52 vol-% leads to a Filmix™ laminate with a porosity content of 0.6 vol-%.

4.2 Polymer Antioxidants

Long grafted chains of a polymer are formed during irradiation of a trunk polymer in the presence of a vinyl monomer. The polymerisation is initiated by free radicals generated in the trunk polymer. The PEPS film material used for peptide synthesis is produced following this route with polyethylene (PE) as the trunk polymer and styrene (S) as the vinyl monomer.

Substitution of the vinyl monomer with antioxidants belonging to the class of hindered phenols leads to a new type of polymer bound antioxidant. Antioxidants are added to most hydrocarbon polymers in order to avoid degradation at their processing temperatures due to thermal and thermo-oxidative processes. Throughout long-term applications, antioxidants prevent oxidation and embrittlement of the polymer.

Polymer bound antioxidants have intrinsic advantages over «free» antioxidants, such as (i) better matching of the solubility in the host polymer and (ii) lower migration rates both at room temperature and at higher temperatures.

The first indication of the formation of a polymer bound antioxidant was seen after irradiation of polyethylene containing Santonox R. Even after exhaustive extraction of irradiated films, UV absorption was still detected at 280 nm. This absorption originates from the substituted benzene ring present in Santonox.

Unequivocal proof of the formation of a covalent bond between model hydrocarbons and hindered phenols at irradiation doses as low as 25 kGy was obtained from size exclusion chromatograms of irradiated mixtures using UV detection at 280 nm. The extent of immobilisation was found to be highest in highly amorphous hydrocarbons. Future work will include studies of bond structure and the antioxidant behaviour of hindered phenols bound to hydrocarbon polymers with different morphologies.

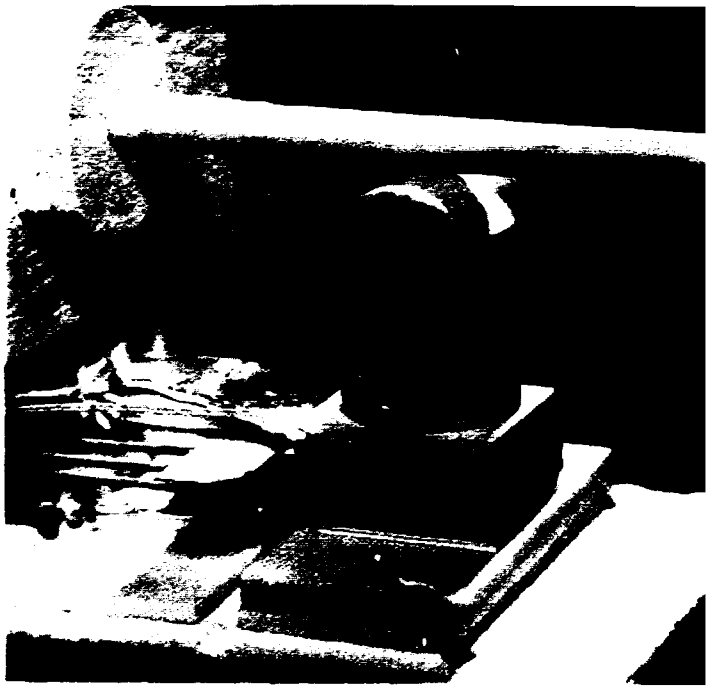
4.3 Stacked Solid Oxide Fuel Cells

The Danish solid oxide fuel cell (SOFC) programmes are carried out in collaboration with the Chemical Institute of Odense University, the Physical Chemical Institute and the Chemical Laboratory A of the Danish Technical University and two industrial partners, Innovision A/S and Haldor Topsoe A/S. Recently the Institute of Chemical Engineering of the Danish Technical University was included in the group.

A three year programme (DK-SOFC 1990-92), aiming at provision of material and design criteria for SOFC stacks has just been completed. The programme was sponsored by the Danish Energy Research Programme, the two groups of electricity utilities ELKRAFT and ELSAM and by the project participants. Within the framework of the programme, know-how was established for:

- Synthesis of ceramic materials not commercially available for stack components
- Shaping of electrolyte foils by tape casting and of electrodes by spray painting (4×4 and 8×8 cm)
- Formulation of material compositions for electrodes. This has resulted in single solid oxide fuel cells with area specific internal resistances lower than any reported in the literature for similar materials
- Shaping of ceramic plates with gas feed channels for serial connectors between cells
- Formulation, fabrication and testing of glasses of potential interest as sealing materials
- Assembling and manifolding of SOFC stacks.

The programme included a large effort on characterization of materials with respect to chemical, electrical, electrochemical and mechanical properties necessary to achieve the above mentioned results. Parallel work on the identification of new materials of potential interest for SOFC resulted in a patent application on ceria-based anode material suitable for direct conversion of methane.



The first Danish SOFC stack, consisting of 3 small cells. The stack delivered 3 watts continuously over a total of 400 hours of operation at 1000°C.

Concurrent national programmes with the same group of participants and sponsors aim at establishing computer models for SOFC stacks and search for SOFC materials to be used with lower temperatures than the present 1000°C. The modelling programme, which also includes acquisition of necessary experimental data, is required for optimizing the SOFC-stack design and for scale-up of stack dimensions to obtain power yields of interest to consumers. New materials with lower operating temperatures (below 850°C) may enable use of metallic construction materials to some extent, may increase efficiency and reduce the overall cost of fabrication and possibly open new application areas as, for example, chemical reactors.

A 4-year continuation (1993 - 96) of the DK-SOFC programme was agreed upon between the Danish Energy Research Programme, ELSAM and the aforementioned partners in late 1992. A major outcome of this programme will be the manufacture of a SOFC stack with an electrical output in the range 0.5 - 1 kW to be tested in 1995.

Participation, SOFC related, R&D programmes supplement the above activities. Subjects covered by such participation were in 1992:

- Tape casting of zirconia (NORDTAPE project, sponsored by the Nordic Industry Fund).
- Development and characterization of SOFC stack materials (in the EC's JOULE I and Brite/Euram programmes, led by ABB).
- Modelling and Evaluation of Advanced Solid Oxide Fuel Cells (IEA collaboration programmes on R&D of advanced fuel cells).
- SOFC evaluation (Phase II of a study sponsored by the electric utility group ELSAM).

Recently, two EC, JOULE II projects were approved to start in 1993. One project, led by the Department, will focus on the development of new SOFC electrode materials to be used in the temperature range 600 - 850°C. The other project, led by British Gas, attempts to solve co-firing problems for electrolyte/interconnect materials in a promising SOFC stack design by ICI.

4.4 Ceramic and Glass Processing

Activities related to the Department's solid oxide fuel cell programmes have necessitated research and development of ceramic and glass processing techniques. Methods have been developed for the production of chemically complex ceramic powders, for shaping of thin fuel cell electrolyte foils by tape casting and for deposition of fuel cell electrodes on sintered electrolyte foils by spray painting. Emphasis is presently put on shaping of complex ceramic components to be used for electrical connection of single solid oxide fuel cells in series (interconnect plates for stacking) and on development of glasses to be used as sealing material for SOFC.

Shaping and Interconnect Plates

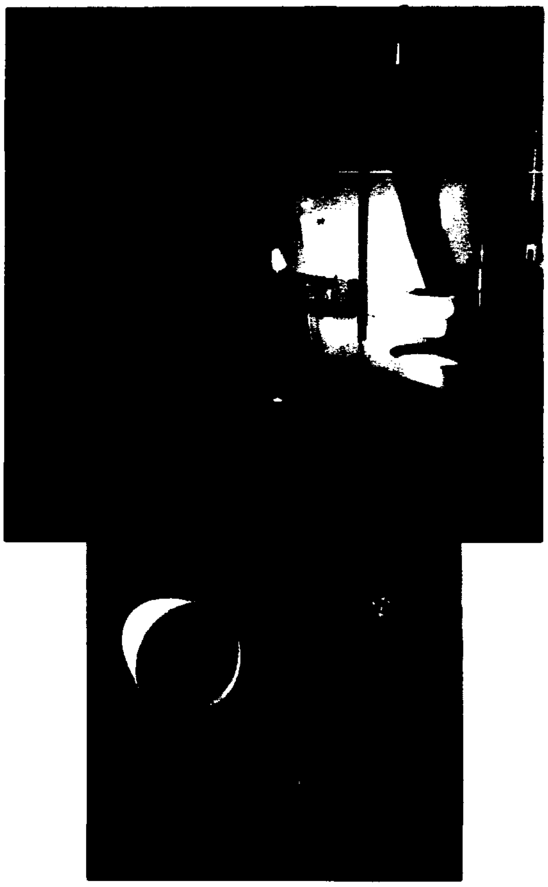
A solid oxide fuel cell stack is a layered structure with cells sandwiched between interconnect plates. Feed of fuel gas and of air to each of the two electrode areas of a cell is accomplished through gas channels in the interconnect, the design of which, therefore, may become quite complex. Interconnect plates are presently made from calcium-lanthanum chromite, one of the few electrically conducting ceramics, which are stable at 1000°C, both in air as well as in wet hydrogen. In cooperation with Danish partners in the solid oxide fuel cell programme, shaping experiments have been initiated via two main routes, warm pressing and injection moulding. Satisfactory plates of 45x45x5 mm have been shaped by warm pressing followed by sintering at 1300 - 1350°C. Shaping experiments with organic binder systems suitable for injection moulding (polystyrene) are close to completion.

Glass for SOFC Sealing

The current Danish SOFC design requires a number of sealings of the ceramic components to prevent mixing of gases. A sealant with glass structure is preferred, because the seal becomes viscous beyond the glass transition point, T_g, and hence will be able to yield according to the varying strain situation of the fuel cell stack. Additionally, glass performance may be tailored through continuous variation of component concentrations.

The sealing glass requires a high chemical stability at normal operating temperature (1000°C) in the pO_2 range $0.2 - 10^{-18}$ atm., in steam and in contact with the ceramic SOFC components. The thermal expansion coefficient below T_g should match that of the ceramic SOFC components closely (~10^{-5}/K and volume changes caused by phase transitions in crystalline phases below T_g cannot be accepted.

Test glasses are made by melting in a specially designed furnace, where the crucible may be transferred from the hot zone (1500 - 1600°C) to



Casting of glass for SOFC sealing onto a graphite plate. Glass wires and blocks are tested for stability at SOFC relevant temperatures and atmospheres.

room temperature in a few seconds for subsequent casting of the glass on graphite plates. Casting produces thin wires as well as blocks. These are used for characterization of fundamental glass parameters and for SOFC relevant performance tests.

A large number of borate and phosphate glasses have been made with combinations of oxides of Mg, Ca, Sr, Ba, Co, Ni, Zn, Al, La, Nb, Cr, Zr, Mo, Ti and Mn as stabilizing elements. Glasses with the required chemical stability have been identified and shown to produce leak tight seals at 1000°C. The best candidate has been used successfully in a working cell stack over a period of 400 h. Work is progressing on reduction of the tendency to surface crystallization.

4.5 Fine and Ultra-fine Powder Metallurgy

Atomization

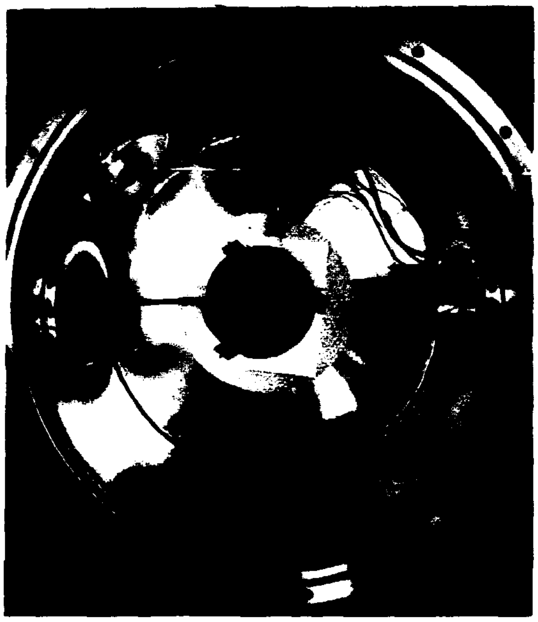
The Risø atomizer has been used for the production of powders of tin, copper alloys and pure aluminium during 1992. The experiments on copper alloys were designed to identify basic relations between the experimental parameters used and the resulting powder in terms of mean particle size and size distribution. One parameter, which strongly effects the powder characteristics, is the melt flow rate through the feeding tube and this, in turn, depends on the pressure in the feeding tube during atomization. The relation between the position of the gas nozzle relative to the feeding tube orifice and the obtained feeding tube pressure for the gas supply pressure has been determined. It was found that the feeding tube pressure is extremely sensitive to the positioning of the gas nozzle and therefore the setting up must be done with care and precision.

Gas velocities in the spraying plume were measured for some of the nozzle systems used in the atomizer. The measuring technique used was double exposure by two laser pulses on one film and measurement of displacements of small and light particles, which were assumed to have the speed of the gas. The results showed that gas speeds are in the neighbourhood of 200 m/sec whereas video recordings of the atomization process show, that droplet speeds may be at least one order of magnitude lower.

Ultrafine Powders

The work on ultrafine powders is carried out in collaboration with the Laboratory of Applied Physics (now the Institute of Physics) at the Technical University of Denmark.

Pilot plant production of metal powders in the inert gas atomizer. Powders of tin, copper alloys and pure aluminium were produced during 1992.



Detail of the crucible in the inert gas atomizer. Metal is melted by induction heating and then atomized by spraying into high pressure gas. Spherical particles with sizes in the range 10-250 microns are formed.

The materials studied were alloys of Fe-Ni, Fe-Cr and pure Ni with particle sizes around 5-10 nm. The measuring techniques were positron annihilation, ultra-micro-gravimetry, Differential Thermal Analysis and Differential Scanning Calorimetry. In particular, attention has been focused on the reduction of surface oxide on the powder grains. In a hydrogen gas the reduction takes place at about 450K in a $Fe_{0.5}Ni_{0.5}$ powder as compared to about 360K for a Ni powder. For the Ni case this gives rise to a weight loss of 15-20% and an endothermic response consistent with the reduction of almost totally oxidized particles (a mixture of NiO and Ni_2O_3, as is also indicated by TEM). An interesting observation is that a similar weight loss, but with no thermal response, can be observed when heating the Ni-oxide particles in an inert gas to about 525K. The origin of this effect will be pursued further.

4.6 Manufacture of Homogeneous MMCs

In the past few years, the Materials Department has put a great deal of effort into basic studies of MMCs in collaboration with the Technical University of Denmark, University of Cambridge, UK, Queen's University, Canada, and Hydro Aluminium, Norway.

For the primary manufacturing process, the powder metallurgy route has been optimized with respect to microstructure and properties. A sol-gel technique has been introduced, which allows Al-SiC MMCs with homogeneous microstructure and with modified interfaces to be made. As a result, mechanical properties and thermal stability have been improved. The microstructural and textural evolution during the secondary manufacturing process, such as hot extrusion, cold rolling and annealing has been studied by TEM, EBSP technique and neutron diffraction. Basic information on the deformation zone microstructure near SiC whiskers/particles, on the nucleation and growth has been obtained. Furthermore, the work-hardening of the Al-SiC MMCs has been studied both experimentally and theoretically. As compared to the unreinforced Al materials, the MMCs show an enhanced work- hardening rate during straining. Using tensile testing in combination with TEM and neutron diffraction, an analysis of the various strengthening contributions has been performed. There are contributions from grains and subgrains, from dislocations generated during cooling and deformation, and from dispersion strengthening and long range internal stresses. The variable constraint model (VCM), modifying the Eshelby approach, has been used to provide a theoretical basis for the prediction of the elastic, thermoelastic and plastic behaviour of short fibre MMCs. The model can be adapted to describe both plastic and diffusional relaxation effects. which are particularly important for Al MMCs. Also the creep behaviour of Al-SiC MMCs has been modelled.

On the basis of the research involvement as described above, the Materials Department was able to form a Brite/Euram project aiming at industrial applications of MMCs: a project on »Forging of Aluminium Metal Matrix Composites for Automotive Components«. This project is carried out in collaboration with Alcan International, Centro Ricerche FIAT SpA, Stampal SpA, Italy, United Engineering Steel, UK, University of Cambridge and University of Manchester, UK.

4.7 Fabrication of LEU Fuel Elements

Fabrication of LEU fuel elements was continued in 1992. 44 elements were delivered to the Danish DR3 reactor. In total nearly 200 LEU elements have been made since production of this type started in 1988.

In September, Riso National Laboratory hosted the 15th International Meeting on Reduced Enrichment for Research and Test Reactors. The meeting, which was held in Roskilde, was arranged in cooperation with IAEA in Vienna, Austria, and the RERTR-Programme Group at Argonne, Illinois, USA. At the meeting, the Materials Department presented the paper »Quality Assurance and Ultrasonic Inspection Studies in LEU Fuel Element Fabrication«, where a new NDT method for measuring cladding thicknesses was presented. The nearly 100 participants also visited the Department, where the fuel fabrication facility, non-destructive testing equipment and other areas of the laboratory could be seen.

Acid bath cleaning and etching of aluminium sheeting components during one stage of the production of low enriched uranium (LEU) fuel elements for the DR-3 reactor.

The efforts to sell LEU fuel elements to research reactors outside Denmark were continued. This will involve production of elements with different design and fabrication techniques than is used for the DR3 reactor. The market research has confirmed that production of such elements will contribute in a satisfactory way to the activities and finances of the Department.

4.8 Joining of Advanced Materials

An important part of the development of advanced materials is to study how such materials can be joined. The major joining projects are carried out, and partly sponsored, as integral parts of the Danish Materials Development Programme - within the Centres of Powder Metallurgy and Advanced Technical Ceramics, the EC BRITE/EURAM programmes and industry.

The BRITE/EURAM project on the development of improved new NiCr-based brazing alloys was finished and a new BRITE/EURAM contract on development of brazed novel heat exchangers was signed at the end of the year.

Brazing of Stainless Steels and Superalloys

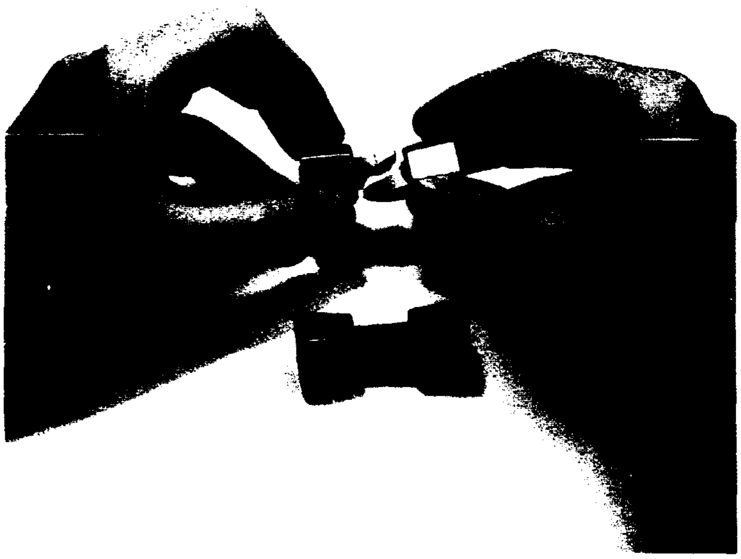
The four-year BRITE/EURAM research project on development of improved NiCr-based brazing filler metals was finished during the year. Newly developed and optimized filler metals have shown brazed joints with drastically improved corrosion resistance in 3% HNO_3 at 75°C and with slightly improved ductility for both narrow and wide brazed gaps.

Fracture mechanics methods have been introduced to analyze joints of stainless steels brazed with commercial and modified nickel-based filler metals. The fracture mechanics approach has given valuable information about the different phases in the joints, and it is possible to closely relate crack propagation to specific phases, precipitates and interfaces. A boron-rich filler metal such as the NB 150 shows considerable precipitation of borides in the centre line of the gap, and the brittleness of these borides enhances crack growth in the centerline. The boron- free filler metal NB30 has no precipitates in the gap, and the filler metal in the gap is so crack resistant that the crack propagates in the base material.

Joining Powder Metallurgical Materials

In the Danish Centre for Powder Metallurgy, work has been carried out on joining P/M-parts by diffusion bonding and brazing.

The primary diffusion bonding parameters (bonding time, temperature and pressure) have been optimized so as to obtain a maximum tensile strength in the joints, and at the same time avoid large deformations of the parts during



Diffusion bonding parameters for powder metallurgical components have been optimized. Tensile and torsional properties of the joints are often superior to those of the bonded parts.

bonding. Also, surface roughness and macro geometry have a great influence, as asperities have to be coalesced to establish full bonding. When bonding using these optimized parameters, plates of pure iron powder in the coined state show tensile strengths of 160 ± 89 MPa. However, plates bonded in the lapped state show tensile strengths of 122 ± 40 MPa and as-sintered plates have tensile strengths of only 114 ± 16 MPa. The differences in scatter and especially the large scatter for the coined plates is closely related to the surface conditions.

In brazing P/M-parts, different means have been used to overcome the problems of liquid filler metal infiltrating the bulk of parts to be joined and thereby draining the gap. Large amounts of filler metal alone will not secure a filled gap unless a full penetration of the part is accepted. However, by carefully controlling the brazing temperature to between 0 and 25°C above the liquidus temperature, a complete filling of the gap can be achieved. At this temperature, certain filler metals react with the base material, and the filler metal readily solidifies isothermally in the pores. The amount of filler metal relates closely to the interactions between the base material and the filler metal, and also to the surface quality of the parts. For example, as-sintered surfaces with a huge amount of open porosities lead to the need for more filler material. These parameters have been optimized for the materials and geometries investigated in the project.

Joining of Ceramics

In the Danish Centre for Advanced Technical Ceramics, the joining programme was continued on brazing and solid state diffusion bonding.

The earlier developed diffusion joining equipment, was proven to give reliable, reproducible, thermoshock resistant and leak tight joints in 10/6 mm tubes of zirconia and alumina with an interlayer of platinum foil. Although this process has not yet been finally optimized, leak-tight joints have been obtained.

Having finished the investigations of the influence of the four point bend strength for brazed alumina from various brazing parameters and pretreatments of the surfaces, corresponding investigations have been started on partly stabilized zirconia and SiC. Preliminary investigations have shown, that a combination of the neutron diffraction technique and the finite element method can be used to estimate at which temperature the residual stress build-up starts.

Contract work was continued on industrial applications of dip brazing of aluminium alloys as well as vacuum brazing of stainless steels, superalloys, ceramics and ceramic/metal.

5

Finances

The activities of the Department are supported by a combination of direct government funding, focused project funds from national, EC and international programmes and payments from industrial contracts.

The numbers given in the tables are in units of 1000 Danish kroner. The equivalent amount in US dollars is also shown (DKK 1000 equals USD 163). Inflation from 1991 to 1992 was approx. 2.5 per cent; the Danish kroner was marginally strengthened against the US dollar (+1.1%) over the period.

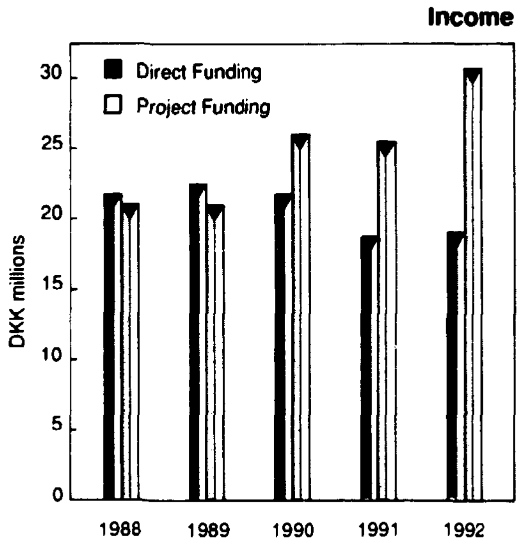
The major reductions in direct government funding which have occurred over the last five years have now slowed significantly (minus 0.9% compared to 1991, inflation adjusted). Direct funding currently amounts to 38% of the total income to the Department compared to approx. 50% five years ago. Fortunately, the Department has been able to accommodate to this change in policy by successful efforts to increase the proportion of project funding. In 1992 project activities were again increased, resulting in inflation adjusted growth in the total income by 8.3% over that of 1991.

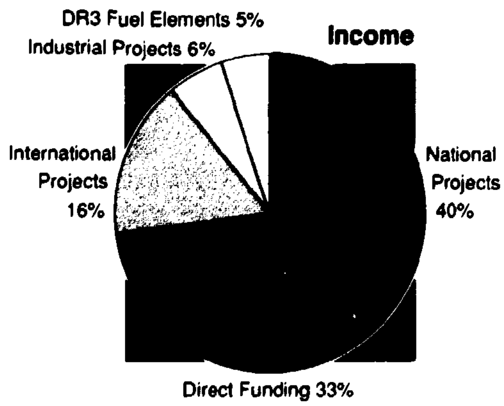
The increase in income to the Department in 1992 has allowed a major refurbishment and small expansion of laboratory facilities, and office space. These improvements operating expenses and the purchase of new equipment constituted 32% of the total expenditure in 1992; operating expenses and equipment were 26% of the total in 1991.

The total expenditure on salaries was almost unchanged with respect to 1991 with 49% of the funds available for salaries now being directly related to project programmes, compared to 44% the previous year.

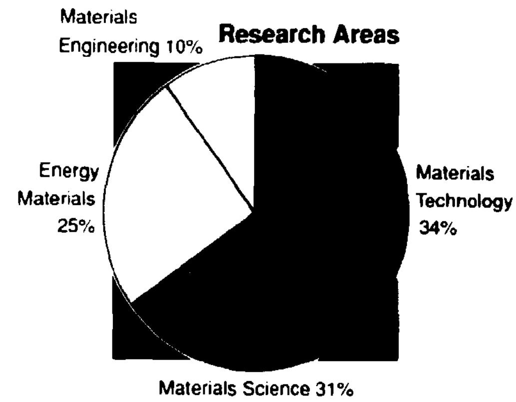
The number of academic and technical staff has increased only conservatively over the past five years. At the same time, the numbers of Ph.D. students and of post docs and guest scientists have more than doubled.

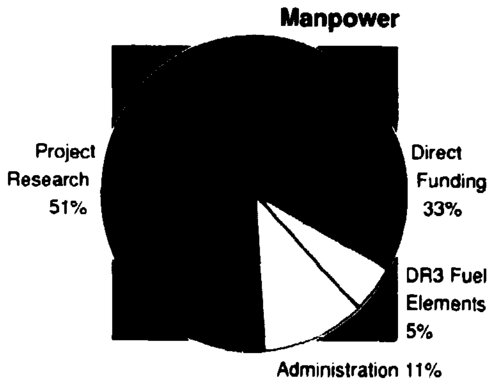
Income	[redacted]	USD 1000
Direct funding (Ministry of Energy)	[redacted]	[redacted]
Project funding	30600	[redacted]
	49600	[redacted]

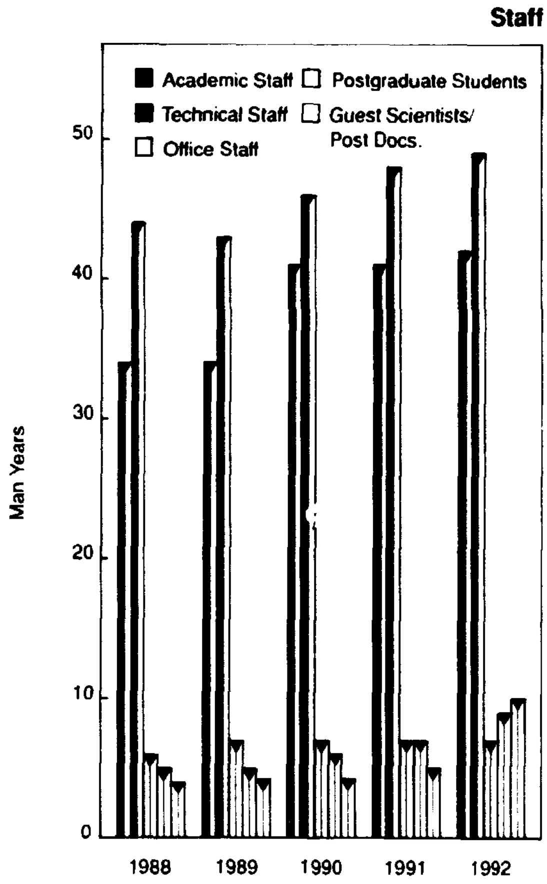




Expenditure	[redacted]	Funds	[redacted] Funds		[redacted] tal	
	[redacted]	USD 1000	[redacted]	USD 1000	[redacted]	USD 1000
Salaries	[redacted]	2418	[redacted]	2304	[redacted]	4722
Operating expenses	2600	425	7200	1176	9800	1601
Equipment	1000	[redacted]	4400	[redacted]	5400	[redacted]
Administrative charge	-	[redacted]	4174	[redacted]	4174	[redacted]
Total	18400	[redacted]	29874	[redacted]	48274	[redacted]







6

Staff

In 1992, 18 staff members left the Department and 25 new members joined (*) the Department.

Head of Department
Niels Hansen

Scientific staff
Adolph, Eivind
Almdal, Kristoffer
Andersen, Svend Ib
Bagger, Carsten
Bentzen, Janet J.
Berg, Rolf
Bilde-Sørensen, Jørgen B.
Borring, Jan
Borum, Kaj K.
Brøndsted, Povl
Carlsen, Hans
Christensen, Jørgen
Christensen, Lone, until 31 Aug.
Debel, Christian P.
Eldrup, Morten
Gormsen, Steffen*
Gundtoft, Hans Erik
Hennesø, Erik*
Horsewell, Andy
Hvilsted, Søren
Johansen, Bjørn S.
Juhl, Mette
Juul Jensen, Dorte
Knudsen, Per
Larsen, Peter*
Leffers, Torben
Lilholt, Hans
Lindegaard, Thomas
Linderoth, Søren*
Liu, Yi Lin
Lorentzen, Torben
Lystrup, Aage S.
Løgstrup Andersen, Tom
Mogensen, Mogens B.
Pedersen, Allan Schrøder
Pedersen, Ole Bøcker
Pedersen, Walther Batsberg
Poulsen, Finn Willy
Rasmussen, Karen*
Rørbo, Kaj, deceased 20 Oct.
Singh, Bachu N.
Sørensen, Ole Toft
Toft, Palle
Toftegaard, Helmuth L.

Postgraduate Students
Ahlgren, Erik
Jørgensen, Ole
Krieger Lassen, Niels C.
Møller, Carsten V., until 30 Apr.
Poulsen, Jes R.
Ranløv, Jens
Sørensen, Niels Jakob
Tiedje, Niels
Tricker, David
Winther, Lars*

Post docs
Clausen, Charlotte
Sørensen, Bent
Østergård, Maria J.L.

Guest Scientists
Alcock, Jeffrey R.
Carter, J. David*
Gertsen-Briand, H., until 31 Aug.*
Holt, Arve, until 30 Jun.*
Hughes, Darcy, until 27 Jul.*
Jiang, Zhen-Jian
Johannesson, Birgir*
Li, Wen-Yu*
Rosen, Gady
Saoucha, A., until 5 Nov.*
Yao, Jianlong*
Vandemeer, Ray A., until 1 Nov.*
Vulfson, Yurij, until 3 Nov.*
Warner, Dean, until 30 Apr.
Warner, Kathryn, until 30 Apr.

Consultants
Domanus, Josef
Kindl, Bruno
Nilsson, Tage M.
Waagepetersen, Gaston

Technical Staff
Adrian, Frank
Andersen, Axel B.
Andersen, Kaj K., until 29 Feb.
Aukdal, Jørgen A.
Borchsenius, Jens F.S.
Bülow Christensen, Carl J.
Bønke Nielsen, Anne
Christensen, Peter
Christensen, Svend E.
Dreves Nielsen, Poul
Frederiksen, Henning
Gravesen, Niels
Hansen, Jørgen*
Hansen, Lotte
Hansen, Niels J.
Hersbøll, Bent
Hubert, Lene
Jensen, Finn E.
Jensen, Knud
Jensen, Palle
Jepsen, Ole
Jespersen, John
Kjøller, John
Kjær, Anne-Mette Heie*
Klitholm, Cliver
Kristensen, Tonny, until 31 Mar.*
Larsen, Arne*
Larsen, Bent
Larsen, Jan
Larsen, Kjeld J.C.
Lindbo, Jørgen
Mikkelsen, Claus
Nielsen, Ove
Nielsen, Palle H.
Nielsen, Peter
Nilsson, Helmer
Nørgaard, Jesper
Olesen, Preben B.
Olsen, Benny F.
Olsen, Henning
Olsen, Ole
Olsson, Jens
Paulsen, Henrik
Pedersen, Børge E.
Pedersen, Knud E.
Pedersen, Niels Jørgen
Petersen, Jan H.A., until 31 Dec.
Robl, Steen
Sandsted, Kjeld
Strauss, Torben R.
Sørensen, Erling
Thomsen, Leif F., until 30 Apr.
Aagesen, Sven

Office Staff
Christensen, Helle Baastrup
Dreves Nielsen, Elsa
Hoffmann Nielsen, Lis
Lauritsen, Grethe
Sindholt, Birgitte
Sørensen, Eva
Thomsen, Ann

Apprentices
Andersen, Morten*
Christensen, Kim, until 25 Sep.
Enghave, Christian, until 15 Jan.
Forné, Anders
Hammer, Christian*
Hansen, Martin
Nielsen, Jens
Olsen, Morten, until 31 Oct.
Ploug, Martin*
Westergaard, Kenneth, until 30 Apr.
Ørgaard, Henrik*

7

Department Activities

Participation in Committees

The Department is widely represented in scientific, educational and technical Committees within Denmark and abroad. Such participation provides a base for international collaborative research activities and information exchange.

Danish Committees

Technical Assessors, The Danish Institute of Fundamental Metrology. Lyngby.
Eivind Adolph and Niels Hansen.

The Steering Committee for the Danish Solid Oxide Fuel Cell Programme. Risø.
Carsten Bagger, Niels Hansen, Per Knudsen and Mogens Mogensen.

The Executive Committee of the Danish Society for Materials Research and Testing. Copenhagen.
Janet J. Bentzen.

The Executive Committee of the Danish Metallurgical Society. Lyngby.
Povl Brøndsted.

The Board of Governors of Risø National Laboratory. Roskilde.
Morten Eldrup (Staff Representative).

The Danish Ministry of Industry, Reference Group for the BRITE/EURAM programme. Copenhagen.
Niels Hansen.

The Danish Ministry of Energy, Advisory Group for Fuel Cells. Copenhagen.
Niels Hansen.

The Steering Committee for the Centre for Advanced Technical Ceramics. Copenhagen.
Niels Hansen.

The Advisory Committee for Materials Testing. The Danish Technological Institute. Aarhus.
Niels Hansen.

The Governing Board of The Danish Society for Polymer Technology. Copenhagen.
Søren Hvilsted (Chairman).

The Danish Electrical Research Institute (DEFU). Lyngby.
Per Knudsen.

The Research Committee of the Danish Society of Chemical, Civil, Electrical and Mechanical Engineers. Copenhagen.
Torben Leffers (Chairman).

The Danish Technical Research Council. Copenhagen.
Torben Leffers (Vice Chairman).

The Joint Committee for Biotechnology of the Danish Research Councils. Copenhagen.
Torben Leffers.

The Steering Committee for the Centre for Polymer Composites. Copenhagen.
Hans Lilholt.

The Advisory Committee for Continuing Education in Materials Technology. Copenhagen.
Aage Lystrup.

The Executive Committee of the Danish Electrochemical Society. Copenhagen.
Mogens Mogensen.

The Advisory Board of »Materials Technology in the Danish Rubber Industry«. Materials Technology Development Programme.
Walther Batsberg Pedersen.

Steering Committee for the Danish Environmental Framework Programme. Copenhagen.
Ole Toft Sørensen.

International Committees

Permanent Committee for Stress Analysis. Lisbon, Portugal.
Svend Ib Andersen.

Editorial Board of »Journal of Strain Analysis«.
Svend Ib Andersen.

The European Structural Integrity Society. Delft, The Netherlands.
Svend Ib Andersen, Povl Brøndsted and Christian Debel.

Council of the European Peptide Society.
Rolf H. Berg.

The International Jury on The Josef Rudinger Memorial Award.
Rolf H. Berg.

The International Jury on The Leonidas Zervas Award.
Rolf H. Berg.

Programme Committee of the EC Working Group »Hot Laboratories and Remote Handling«. Brussels, Belgium.
Hans Carlsen.

International Institute of Welding. Subcommission »Brazing and Soldering«. Paris, France.
Jørgen Christensen (Chairman).

Euratom Neutron Radiography Working Group. Subgroup »Practical Neutron Radiography«. Petten, The Netherlands.
J.C. Domanus (Chairman).

The International Advisory Committee on International Conferences on Positron Annihilation.
Morten Eldrup.

The Halden Programme Group, OECD.
Niels Hansen.

The COST 501 Management Committee on Materials for Energy Conversion Using Fossil Fuels. Brussels, Belgium.
Niels Hansen.

The Fusion Technology Steering Committee (FTSC-I). EC, Brussels, Belgium.
Niels Hansen.

Editorial Board of »Revue de Métallurgie«.
Niels Hansen.

Editorial Board of »Monographs in Materials Science«.
Niels Hansen.

Expert Group on Structural Materials, Fusion Technology Programme. EC, Brussels, Belgium.
Niels Hansen (Chairman) and Bachu N. Singh.

International Advisory Board for the 4th European Polymer Federation Symposium on Polymeric Materials. Baden-Baden, Germany.
Søren Hvilsted.

European Polymer Federation. Mainz, Germany.
Søren Hvilsted.

Steering Committee for Nordic Tape Casting Project. Oslo, Norway.
Per Knudsen.

Editorial Advisory Board of »Plasticity«.
Torben Leffers.

Editorial Advisory Board of »Textures and Microstructures«.
Torben Leffers.

The International Scientific Committee for the Asia-Pacific Symposium on »Advances in Engineering Plasticity and its Application«. Hong Kong.
Torben Leffers.

The International Scientific Committee for the »Fourth International Symposium on Plasticity and Its Current Applications«. Baltimore, USA.
Torben Leffers.

International Committee for Composite Materials. Philadelphia, USA.
Hans Lilholt.

European Association for Composite Materials. Standing Committee. Bordeaux, France.
Hans Lilholt.

International Evaluation of Latvian Science, Panel 2: Mechanics, Engineering and Energetics. Copenhagen and Riga.
Hans Lilholt.

Scientific Committee and Coordinating Committee of 6th European Conference on Composite Materials (ECCM-6). Bordeaux, France.
Hans Lilholt.

Editorial Board. »Advanced Composite Materials«.
Hans Lilholt.

Editorial Board. »Composites Science and Technology«.
Hans Lilholt.

Editorial Board. »Advanced Composites Letters«.
Hans Lilholt.

Editorial Board. »Polymers and Polymer Composites«.
Hans Lilholt.

The Fuel Cell Committee under the Nordic Energy Research Programme. Oslo, Norway.
Mogens Mogensen and Finn Willy Poulsen.

Powder Metallurgy Group. Institute of Materials. London, UK.
Allan Schrøder Pedersen.

The Governing Board of The International Society for Solid State Ionics.
Finn Willy Poulsen.

IEA Working Group on SOFC-Practices. Lausanne, Switzerland.
Finn Willy Poulsen.

The Working Party on Nuclear Corrosion (under The European Federation of Corrosion).
Kaj Rørbo.

Organizing Committee for the Sixth International Conference on Fusion Reactor Materials. Stresa, Italy.
Bachu N. Singh.

Organizing Committee of the 16th International ASTM Symposium on Effects of Radiation on Materials. Denver, USA.
Bachu N. Singh.

Nordic Society for Thermal Analysis and Calorimetry. Risø, Denmark.
Ole Toft Sørensen (Chairman).

Nomination Committee of International Confederation for Thermal Analysis and Calorimetry. Hatfield, UK.
Ole Toft Sørensen.

Education and Training

Many of the Department's staff members are actively involved in education and training in materials science as university external lecturers and examiners. Further, research on projects by undergraduate, postgraduate and postdoctoral students was carried out under the supervision of Department staff members.

External Lectures

Kristoffer Almdal.
»Rheology of Synthetic Polymers«. The Danish Research Academy.

Kristoffer Almdal.
Course on »Methods in Soft Materials Science«. University of Roskilde, Denmark.

Janet J. Bentzen.
»Zirconia Ceramics«. Materials Technology Course (Law 271). The Danish Technological Institute. Taastrup, Denmark.

Janet J. Bentzen.
»An Example of Chemical Engineering«. Basic Technical Course for Chemical Engineering Students. The Technical University of Denmark. Lyngby, Denmark.

Christian P. Debel.
Understanding Materials-Engineering Ceramics: Mechanical Properties. The Danish Technological Institute. Taastrup, Denmark.

Niels Hansen and Kaj Rørbo.
»Materials Science«. The Danish Academy of Engineering. Lyngby, Denmark.

Andy Horsewell.
»Microstructure and Properties of Orthodontic Materials«. Danish Ministry of Health, Continuing Education for Orthodontists. Århus, Denmark.

Søren Hvilsted.
»FTIR Spectroscopy on Polymeric Samples - Selected Examples«. Ph.D. Course in Soft Materials Science. Roskilde University. Roskilde, Denmark.

Hans Lilholt.
»Course on Polymers and Fibres«. Continuing Education in Materials Technology. Copenhagen and Roskilde, Denmark.

Mogens Mogensen.
Training of Teknovision Guides. Risø, Denmark.

Walther Batsberg Pedersen.
»Advanced Course on Separation Techniques«. Copenhagen, Denmark.

Walther Batsberg Pedersen.
»Advanced Course on Liquid Chromatography«. Chromatography Discussion Group. Copenhagen, Denmark.

Finn Willy Poulsen.
»The van der Pauw Method«. ITEK, NTH, Norway and Senter for Materialeforskning. Oslo, Norway.

Finn Willy Poulsen.
»Simulation of Defect Chemistry in $LaMnO_{3+\delta}$«. ITEK, NTH. Norway.

Ole Toft Sørensen.
»Solid State Chemistry«. The Technical University of Denmark. Lyngby, Denmark.

Ole Toft Sørensen.
»Technical Ceramics«. The Danish Technological Institute. Taastrup, Denmark.

Bent F. Sørensen.
»Characteristics of Design«. Materials Technology Course (Law 271). The Danish Technological Institute. Taastrup, Denmark.

External Examiners

Svend Ib Andersen.
M.Sc. examiner, The Technical University of Denmark. Lyngby, Denmark.

Christian P. Debel.
B.Sc. examiner. Københavns Teknikum. Copenhagen, Denmark.

Niels Hansen.
Ph.D. examiner. The Technical University of Denmark. Lyngby, Denmark.

Andy Horsewell.
M.Sc. examiner. The University of Copenhagen, Denmark.

Andy Horsewell.
Ph.D. examiner. EPFL. Lausanne, Switzerland.

Søren Hvilsted.
M.Sc. examiner, The Technical University of Denmark. Lyngby, Denmark.

Søren Hvilsted.
Ph.D. examiner. The Technical University of Denmark. Lyngby, Denmark.

Torben Leffers.
M.Sc. examiner. The Technical University of Denmark. Lyngby, Denmark.

Hans Lilholt.
M.Sc. examiner. The Technical University of Denmark. Lyngby, Denmark.

Hans Lilholt.
M.Sc. examiner. The University of Aalborg. Denmark.

Hans Lilholt.
Ph.D. examiner. The Univeristy of Aalborg. Denmark.

Hans Lilholt.
Ph.D. examiner. The Technical University of Denmark. Lyngby, Denmark.

Mogens Mogensen.
Ph.D. examiner. The Technical University of Denmark. Lyngby, Denmark.

Allan Schrøder Pedersen.
Ph.D. examiner. The University of Copenhagen. Denmark.

Walther Batsberg Pedersen.
M.Sc. examiner. The Technical University of Denmark. Lyngby, Denmark.

Finn Willy Poulsen.
M.Sc. examiner. The Technical University of Denmark. Lyngby, Denmark.

Ole Toft Sørensen.
M.Sc. examiner. The Technical University of Denmark. Lyngby, Denmark.

Ole Toft Sørensen.
M.Sc. examiner. The University of Aalborg. Denmark.

Ole Toft Sørensen.
Dr.Scient. examiner. The Technical University of Oslo. Norway.

Postgraduate (Ph.D.) Projects

The following Ph.D. projects have been finished during 1992:

Charlotte Clausen.
»Characterization of Interfaces in Ceramics by Electron Microscopy«. *Supervisor: Jørgen B. Bilde-Sørensen.*

Bent F. Sørensen.
»Damage Mechanisms in Ceramic Matrix Fiber Composites«. *Supervisors: Ole Toft Sørensen and Svend Ib Andersen.*

Henrik Albertsen.
»The Influence of Fibre Surface Treatment on Interlaminar Fracture Toughness of CFRP«. *Supervisor: Svend Ib Andersen.*

The following Ph.D. projects were in progress during 1992:

Erik O. Ahlgren.
»Thermoelectric Effecs in Solid Oxide Fuel Cells«. The Technical University of Denmark. Lyngby, Denmark.
Supervisor: Finn Willy Poulsen.

Jens H. Andreasen.
»Micromechanics and Transformation Toughening of Ceramics«. Aalborg University. Denmark.
Supervisor: Ole Toft Sørensen.

Franz Bødker.
»Properties of Amorphous and Crystalline Fine Particles«. The Technical University of Denmark. Lyngby, Denmark.
Supervisor: Søren Linderoth.

Ole Jørgensen.
»Damage in Laminates«. The Technical University of Denmark/ The Danish Research Academy. Denmark.
Supervisors: Sven Ib Andersen, Povl Brøndsted and Hans Lilholt.

Martin Heide Jørgensen.
»Fatigue of Ceramic Materials Under Cyclic Loading«. The University of Aalborg. Denmark.
Supervisor: Ole Toft Sørensen.

Niels Christian Krieger Lassen.
»Automatic Determination of Crystallographic Orientations by Scanning Electron Microscopy«. The Technical University of Denmark/The Danish Research Academy. Lyngby, Denmark.
Supervisor: Dorte Juul Jensen.

Christine Papadakis.
»Experimental Studies of Complex, Structured Fluids with Special Emphasis on Phase Behaviour and Mechanical Properties of Block Copolymer«. The University of Roskilde. Roskilde, Denmark.
Supervisor: Kristoffer Almdal.

Jes R. Poulsen.
»Investigations of Defects in Ceramic Materials using Positron Annihilation Techniques«. The Univeristy of Copenhagen/The Danish Research Academy. Denmark.
Supervisors: Janet J. Bentzen, Morten Eldrup and Andy Horsewell.

Jens Ranløv.
»New Electrode- and Electrolyte Materials for High Temperature Solid Oxide Fuel Cells«. The Technical University of Denmark. Lyngby, Denmark.
Supervisors: Finn Willy Poulsen and Mogens Mogensen.

Niels Jacob Sørensen.
»Thermomechanical Properties of Metal Matrix Composites at High Strains and Temperatures«. The Technical University of Denmark/The Danish Research Academy. Denmark.
Supervisors: Niels Hansen, Dorte Juul Jensen and Hans Lilholt.

Niels Tiedje.
»Gas-Atomized Metal Powder«. The Technical University of Denmark. Lyngby, Denmark.
Supervisor: Allan Schrøder Pedersen.

David Tricker.
»The Electron Microscopical and Electrical Characterization of Boundaries in Solid Oxide Fuel Cell Materials«. University of Cambridge, U.K.
Supervisor: Jørgen B. Bilde-Sørensen.

Lars Winther.
»Hydrophilic Support for Chemical Solid Phase Synthesis«. The Technical University of Denmark. Lyngby, Denmark.
Supervisors: Kristoffer Almdal and Rolf H. Berg.

Undergraduate Projects

Eric Papin.
»Ceramic Nano Composites«. École Nationale Supérieure de Céramique Industrielle. Limoges, France.
Supervisors: O. Toft Sørensen and Jørgen B. Bilde-Sørensen.

Visiting Scientists and Students

Assignments Abroad

Jørgen B. Bilde-Sørensen (visiting professor)
Department of Materials Science and Engineering. Case Western Reserve University. Cleveland, Ohio, USA. 15 Sept-5 Dec.

Povl Brøndsted (visiting scientist)
Materials Department. University of California Santa Barbara, California, USA. 1 Jan-30 June.

Niels Hansen (visiting scientist)
University of Virginia, Charlottesville, USA. 10-21 Feb, 9-13 Nov.

Finn Willy Poulsen (visiting professor)
The Norwegian Institute of Technology. Trondheim, Norway. 1 Jan-30 Sept.

Bachu N. Singh (visiting scientist)
Los Alamos National Laboratory. Los Alamos, New Mexico, USA. 29 June-3 July

Bachu N. Singh (visiting scientist)
Paul Scherrer Institute. Würenlingen, Switzerland. 15-19 Aug.

Visiting Scientists

Dr. G. Albertini.
University of Ancona, Italy. 9-20 March.

Dr. Claire Barlow.
University of Cambridge, UK. 30 Aug-18 Sept.

Prof. Frank S. Bates.
University of Minnesota, USA. 25 June-7 July.

Dr. Bill Bergman.
Royal Institute of Technology, Stockholm, Sweden. 1-28 Feb.

Prof. Giovanni Caglioti.
CESNEF, Italy, 14-15 May.

Dr. David Carter.
University of Missouri-Rolla, USA. 15 Sept-31 Dec.

Dr. Monica Ceretti.
Laboratoire Leon Brillouin, France. 10-17 May.

Dr. F. Cernuschi.
ENEL, Milano, Italy. 9-20 March.

Dr. Olaf Engler.
Technische Hochschule, Aachen, Germany. 17-19 May.

Dr. Howard L. Heinisch.
Battelle Pacific Northwest Laboratory, USA. 30 Aug-5 Sept.

Dr. Darcy A. Hughes.
Sandia National Laboratory, USA. 29 June-25 July.

Dr. M.T. Hutchings.
AEA Industrial Technology, UK. 1-14 May, 14-20 July.

Dr. Kevin M. Knowles.
University of Cambridge, UK. 1-14 May.

Dr. P.C. McKeighan.
University of Bristol, UK. 3-11 Sept.

Dr. J. Osborne.
Loughborough University, UK. 30 Sept-4 Oct.

Prof. Brian Ralph.
Brunel University. The University of West London, U.K. 2-6 July.

Dr. Abdelbaki Saoucha.
Haut Commissariat à la Recherche. Centre de Development des Techniques Nucléaires, Algiers, Algeria. 5 Oct-6 Nov.

Dr. D.J. Smith.
University of Bristol, UK. 3-11 Sept.

Dr. G.M. Swallowe.
Loughborough University, UK. 30 Sept-4 Oct.

Dr. R.I. Todd.
University of Oxford, UK. 27-31 Jan.

Dr. Helmuth Trinkaus.
Forschungszentrum Julich, Germany. 11-15 Jan.

Prof. Roy Vandermeer.
Naval Research Laboratory, Washington, USA. 1-29 Oct.

Yurij Vaitson.
University of Riga, Lithuania. 1 Aug-31 Oct.

Clive T. Walther.
European Institute for Transuranium Elements. Karlsruhe, Germany. 3-7 Feb, 2-6 March, 5-10 April.

Dr. Phil. J. Withers.
University of Cambridge, UK. 1-14 May.

Dr. Erik Woldt.
Technische Universitat Braunschweig, Germany. 7-9 Oct.

Dr. Chung H. Woo.
Atomic Energy of Canada Ltd., Canada. 4-10 Oct.

Jianglong Yao.
University of Science and Technology of China. P.R. China. 1 Sept-31 Dec.

Dr. Steven J. Zinkle.
Oak Ridge National Laboratory, USA. 14-18 March and 22-28 Oct.

Visiting Students

Asger Abrahamsen.
The Technical University of Denmark. 1 May-31 Aug.

Catherine Davey.
Brunel University, The University of West London. UK. 6 April-25 Oct.

M.E. Fitzpatrick.
University of Cambridge, UK. 1-14 May.

Nina M. Fussing.
University of Cambridge, UK. 1 July-15 Sept.

Mrs. Giovannie.
University of Ancona, Italy. 9-20 March.

Arve Holt.
Oslo University, Norway. 3 months.

Zhenjian Jian.
Shandong Mechanical Engineering Institute, China. 1 Jan - 31 Dec.

Christian Kulinna.
University of Essen, Germany. 15-19 June and 9-14 November.

Jeffrey H. Rosedale.
University of Minnesota, USA. 25 June-7 July.

Rafael Schouwenaars.
Katholieke Universiteit Leuven, Belgium. 27-31 Jan.

Mark Schulz.
University of Minnesota, USA. 25 June-7 July.

Johnny Stewart.
University of Cambridge, UK. 22 June-24 Aug.

Social Events

The department's social committee, MAK, keeps track of special birthdays, anniversaries, weddings etc. and organizes annual events including a Christmas party and a Summer picnic. The traditional Christmas party takes place in Roskilde and for the Summer picnic, a scenic location, like the beaches of northern Zealand or the forests to the north of Copenhagen is chosen.

For thpose interested in sports, the 5 km 'Riso Run' takes place every year with a large number of participants from the Department. We are also well represented in Risø badminton, pétanque and football. There is an active group in orienteering. This year MAK introduced an annual bowling tournament.

Hobby activities, organized through evening classes at Risø, include stone masonry, piano classes and language training. There are also regular trips to the theatre and opera, as well as lectures at Risø by celebrities and artists.

These departmental social activities promote interactions between the regular staff and the increasing numbers of new graduate students, post docs and visiting scientists, many of them from abroad. Furthermore, the various cultural activities of Roskilde and Copenhagen are easily accessible and offer lots of opportunities for visitors to participate in cultural events.

8

Published Work

International Publications

1. **Ahlgren, E.**, Thermopower of yttria stabilized zirconia. In: 1. Nordic symposium on materials for high temperature fuel cells. Materials for high temperature fuel cells, Oslo (NO), 9-10 Dec 1991. Bergman, B. (ed.), (Royal Institute of Technology, Stockholm, 1992) p. 147-148.

2. **Alcock, J.R.; Toft Sorensen, O.**, Analysis of abrasive wear mechanisms in ceramics by indentation techniques. Tribologia (1992) v. 11 (no.2) p. 57-65.

3. **Almdal, K.**; Bates, F.S.; Mortensen, K., Order, disorder, and fluctuation effects in an asymmetric poly(ethylene-propylene)-poly(ethylethylene) diblock copolymer. J. Chem. Phys. (1992) v. 96 p. 9122-9132.

4. **Almdal, K.**; Koppi, K.A.; Bates, F.S.; Mortensen, K., Multiple ordered phases in a block copolymer melt. Macromolecules (1992) v. 25 p. 1743-1751.

5. **Almdal, K.**; Mortensen, K.; Koppi, K.A.; Bates, F.S., On the origin of complex phase behaviour in block copolymer melts. In: Proceedings of the Joint Nordic Spring meeting '92. Extended abstracts. 3. Nordic conference on surface science; 6. Nordic symposium on computer simulation; 3. Nordic symposium on superconductivity, Nyborg Strand (DK), 7-10 May 1992. Lindgård, P.-A. (ed.), Riso- R-628(EN) (1992) p. 297.

6. **Almdal, K.**; Mortensen, K.; Koppi, K.A.; Bates, F.S., On the origin of complex phase behaviour in block copolymer melts. In: 4th European Polymer Federation symposium on polymeric materials. Programme. Abstracts. 4. European Polymer Federation symposium on polymeric materials, Baden-Baden (DE), 27 Sep - 2 Oct 1992. (EPF-Secretary, Baden-Baden, 1992) Paper O24.

7. **Andersen, S.I.; Bilde-Sorensen, J.B.; Hansen, N.; Juul Jensen, D.; Leffers, T.; Lilholt, H.; Lorentzen, T.; Pedersen, O.B.**; Ralph, B. (eds.), Modelling of plastic deformation and its engineering applications. 13. Riso international symposium on materials science, Riso (DK), 7-11 Sep 1992. (Riso National Laboratory, Roskilde, 1992) 548 p.

8. **Andersen, S.I.**; Nielsen, K., Thermally induced residual stresses and failure initiation in angle ply laminates. In: Developments in the science and technology of composite materials. 5. European conference on composite materials. ECCM 5, Bordeaux (FR), 7-10 Apr 1992. Bunsell, A.R.; Jamet, J.F.; Massiah, A. (eds.), (European Association for Composite Materials, Bordeaux, 1992) p. 359-364.

9. **Bagger, C.**, Manufacture of thin ceramic layers for high temperature fuel cells. In: 1. Nordic symposium on materials for high temperature fuel cells. Materials for high temperature fuel cells, Oslo (NO), 9-10 Dec 1991. Bergman, B. (ed.), (Royal Institute of Technology, Stockholm, 1992) p. 59-66.

10. **Bagger, C.**, Improved production methods for YSZ electrolyte and Ni-YSZ anode for SOFC. In: Fuel Cell. Program and abstracts. 1992 Fuel cell seminar, Tucson, AZ (US), 29 Nov - 2 Dec 1992. (Courtesy Associates Inc., Washington, DC, 1992) p. 241-244.

11. Balcells, L.; Tholence, J.L.; **Linderoth, S.**; Barbara, B.; Tejada, J., Quantum tunneling of magnetization in metallic FeC ferrofluids. Z. Phys. B (1992) v. 89 p. 209-212.

12. Barbara, B.; Paulsen, C.; Sampaio, L.C.; Uehara, M.; Fruchard, F.; Tholence, J.L.; Marchand, A.; Tejada, J.; **Linderoth, S.**, Observation of quantum tunneling of the magnetization vector in small particles with or without domain walls. In: Studies of magnetic properties of fine particles and their relevance to materials science. Dormann, J.L.; Fiorani, D. (eds.), (Elsevier Science Publishers, Amsterdam, 1992) p. 235-242.

13. Bay, B.; **Hansen, N.**; Kuhlmann-Wilsdorf, D., Microstructural evolution in rolled aluminium. Mater. Sci. Eng. A (1992) v. 158 p. 139-146.

14. Bay, B.; **Hansen, N.**; Hughes, D.A.; Kuhlmann-Wilsdorf, D., Overview no. 96: Evolution of F.C.C. deformation structures in polyslip. Acta Metall. Mater. (1992) v. 40 p. 205-219.

15. **Bellon, O.**, Dilatometric sintering studies of zirconia toughened ceramics. (Riso National Laboratory. Materials Department, Roskilde, 1991) vp.

16. Bentzon, M.D.; **Linderoth, S.**; Pedersen, A.S.; Madsen, M.B., Reduction of surface layer oxides on synthesized ultra-fine Fe-Ni particles. Solid State Phenom. (1992) v. 25 26 p. 187-196.

17. **Berg, R.H.**; Nielsen, P.E.; Buchardt, O.; Egholm, M., Peptide nucleic acids (PNA), a new strategy toward the rational design of DNA mimics. In: European peptide symposium. Program and abstracts. 22. European peptide symposium, Interlaken (CH), 13-19 Sep 1992. (University of Bern, Bern, 1992) L27.

18. **Brondsted, P.; Adrian, F.; Olsson, J.**, Tensile and fatigue properties of RS Al-alloys at room temperature and at elevated temperatures. (Riso National Laboratory. Materials Department, Roskilde, 1992) vp.

19. Bunsch, A.; **Juul Jensen, D.**, Recrystallization and texture transformation kinetics in isothermaly annealed cold rolled copper. Arch. Metall. (1992) v. 37 (no.2) p. 157-173.

20. Bodker, F.; Morup, S.; Oxborrow, C.A.; **Linderoth, S.**; Madsen, M.B.; Niemantsverdriet, J.W., Mossbauer studies of ultrafine iron-containing particles on a carbon support. J. Phys. Condens. Matter (1992) v. 4 p. 6555-6568.

21. **Carlsen, H.**, Decommissioning of the Riso Hot Cell facility. 3. Periodic report covering July 1 to December 31, 1991. EC programme on decommissioning of nuclear installations, 1989-1993. 3. Meeting of the working group on section C, Augsburg (DE), 23 Jun 1992. Riso-Hot-Decom-P-3 (1992) 7 p.

22. **Carlsen, H.**, Decommissioning of the Riso Hot Cell facility. 4. Periodic report covering January 1 to June 30, 1992. EC programme on decommissioning of nuclear installations, 1989-1993. 4. Meeting of the working group on section C, Krefeld (DE), 22-23 Oct 1992. Riso-Hot-Decom-P-4 (1992) 15 p.

23. **Christensen, J.; Gotthjaelp, K.**, Diffusion bonded joints in alumina and ps- zirconias with excellent thermo-shock and mechanical properties. In: Hart- und Hochtemperaturloten und Diffusionsschweissen '92. Vortrage und Posterbeitrage. 3. Internationale kolloquium, Aachen (DE), 24-26 Nov 1992. (DVS-Verlag, Dusseldorf, 1992) (DVS-Berichte band 148) p. 187-189.

24. **Clausen, C.**, Electron microscopical characterisation of interfaces in SOFC materials. Riso-R-626(EN) (1992) 96 p.

25. **Clausen, C.; Bentzen, J.J.; Bilde-Sorensen, J.B.**, Characterization of the interface YSZ-CeO$_2$ by electron microscopy. In: 1. Nordic symposium on materials for high temperature fuel cells. Materials for high temperature fuel cells, Oslo (NO), 9-10 Dec 1991. Bergman, B. (ed.), (Royal Institute of Technology, Stockholm, 1992) p. 143-144.

26. Clausen, C.; **Bilde-Sørensen, J.B.**, Observation of voltage contrast at grain boundaries in YSZ. Micron Microsc. Acta (1992) v. 23 p. 157-158.

27. **Domanus, J.C.**, International neutron radiography newsletter no. 21. Efforts to control image quality in neutron radiography. Brit. J. Non-Destr. Test. (1992) v. 34 p. 235-238.

28. **Domanus, J.C. (ed.)**, Practical neutron radiography. (Kluwer Academic Publishers, Dordrecht, 1992) (EUR-14424) 269 p.

29. Egholm, M.; Buchardt, O.; Nielsen, P.E.; **Berg, R.H.**, Peptide nucleic acids (PNA) - Oligonucleotide analogues with an achiral peptide backbone. J. Am. Chem. Soc. (1992) v. 114 p. 1895-1897.

30. Egholm, M.; Buchardt, O.; Nielsen, P.E.; **Berg, R.H.**, Peptide nucleic acids (PNA): A novel approach to sequence-selective recognition of double-stranded DNA. In: Innovation and perspectives in solid phase synthesis. Peptides, polypeptides and oligonucleotides. Collected papers. 2. International symposium on innovation and perspectives in solid phase synthesis. Canterbury (GB), 27-31 Aug 1991. Epton, R. (ed.). (Intercept Limited, Andover, 1992) p. 325-328.

31. Egholm, M.; Nielsen, P.E.; Buchardt, O.; **Berg, R.H.**, Recognition of guanine and adenine in DNA by cytosine and thymine containing peptide nucleic acids (PNA). J. Am. Chem. Soc. (1992) v. 114 p. 9677-9678.

32. Egsgaard, H.; Larsen, E.; **Batsberg Pedersen, W.**; Carlsen, L., Analysis of antioxidants in polymer material by a strategy employing tandem mass spectrometry and liquid chromatography. Trends Anal. Chem. (1992) v. 11 p. 164-168.

33. Ehlert Nielsen, F.; **Sejer Johansen, B.**, Kieldsteen, B., Geometry of filament winding and computer-aided design. (Centre for Polymer Composites, Danish Technological Institute, Taastrup, 1992) 21 p.

34. **Eldrup, M.**, Positron studies of gases and gas bubbles in metals. In: Positron Annihilation. Part 1. 9. International conference on positron annihilation. Szombathely (HU), 26-31 Aug 1991. Kajcsos, Z.; Szeles, C. (eds.). (Trans Tech Publications, Aedermannsdorf, 1992) (Materials Science Forum, 105-110) p. 229-248.

35. **Eldrup, M.**; Hood, G.M.; **Pedersen, N.J.**; Schultz, R.J., A positron lifetime study of defect recovery in electron irradiated zircaloy-2 and Zr-2.5 Nb. In: Positron Annihilation. Part 2. 9. International conference on positron annihilation. Szombathely (HU), 26-31 Aug 1991. Kajcsos, Z.; Szeles, C. (eds.). (Trans Tech Publications, Aedermannsdorf, 1992) (Materials Science Forum, 105-110) p. 997-1000.

36. **Eldrup, M.**; **Pedersen, N.J.**; **Larsen, B.**; Bentzon, M.D.; **Linderoth, S.**, The reduction of surface oxide on ultra-fine FeNi particles. In: Positron Annihilation. Part 3. 9. International conference on positron annihilation. Szombathely (HU), 26-31 Aug 1991. Kajcsos, Z.; Szeles, C. (eds.). (Trans Tech Publications, Aedermannsdorf, 1992) (Materials Science Forum, 105-110) p. 1553-1556.

37. Gehlsen, M.D.; **Almdal, K.**; Bates, F.S., Order-disorder transition: Diblock versus triblock copolymers. Macromolecules (1992) v. 25 p. 939-943.

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Selected activities of the Materials Department at Risø National Laboratory during 1992 are described. The work is presented in three chapters: *Materials Science*, *Materials Engineering* and *Materials Technology*. A survey is given of the Department's participation in international collaboration and of its activities within education and training. Furthermore, the main figures outlining the funding and expenditure of the Department are given. Lists of staff members, visiting scientists, publications, lectures and poster presentations are included.

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Risø National Laboratory is a broad-based research organization with primary research activities in energy, the environment and in materials.

The research programmes in the Materials Department include basic studies of materials structure and properties, structural mechanics and materials testing, and processing techniques for polymers, polymer composites, powder metallurgical products and engineering ceramics. Research into materials and technologies for the energy sector includes programmes on solid oxide fuel cells. Advanced characterization techniques used in the Department are electron microscopy, positron annihilation, neutron diffraction, small angle neutron scattering, Fourier-transform infra red spectroscopy and mechanical testing.

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Within the Danish Programme of Materials Technology, the Materials Department participates in the Centres of Polymer Composites, Advanced Technical Ceramics and Powder Metallurgy.

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