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Materials Department annual progress report for 1993

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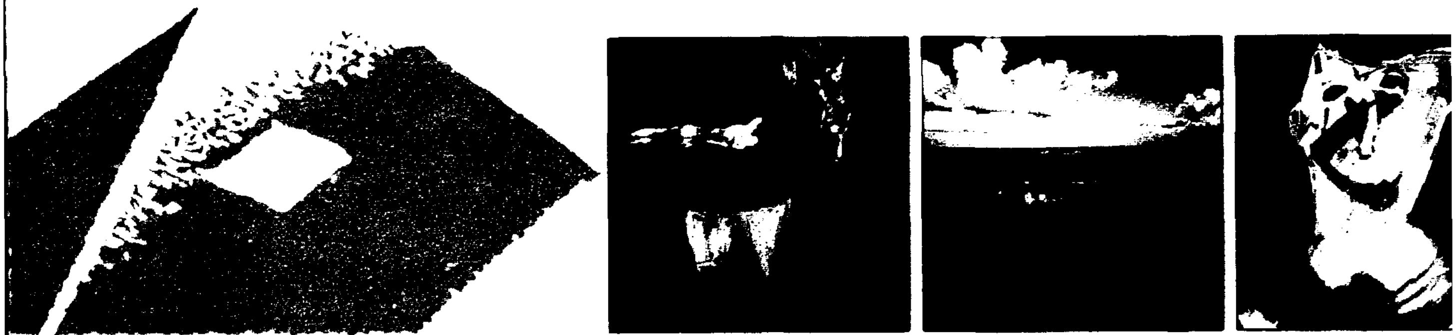

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Materials Department Annual Progress Report for 1993

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Abstract

Selected activities of the Materials Department at Risø National Laboratory during 1993 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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At the Materials Department, paintings, lithographs and sculptures decorate meeting rooms and offices as well as corridors, laboratory areas and work-shops. Photos of some of these works of art are to be found at various places within this report.

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Introduction

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1993 was an active year in which large programmes, which started in 1992, gained momentum. The same is true of the Engineering Science Centre for Structural Characterization and Modelling of Materials, in which graduate students have been hired, large equipment procured and new projects initiated. This applies also to the Solid Oxide Fuel Cells programmes, where project groups are now fully established and many technological processes have reached pilot plant scale. These two programmes encompass about 40 per cent of the total activities of the Materials Department.

In parallel with the start-up of the new programmes, a number of other activities are approaching completion. This applies in particular to the Danish Materials Technology Programme (MUP 1) which ends in the Spring 1994. The Department has been heavily engaged in this programme and related activities are being sought in a follow-up programme called MUP 2. An important aim is to obtain strong industrial involvement in research and development both technically and financially. To achieve this, most of the funding will be directed towards well-defined projects to be carried out in collaboration between Danish industry and the research institutes. Thus, in parallel with rounding off the MUP 1 activities of the Department, a number of proposals have been formulated for MUP 2. This has led to the formulation of a variety of research projects of high industrial relevance. A useful background in this work has been the scientific, technical and administrative experience in the Department gained through our involvement in many large national and international research and development programmes. This experience will no doubt be a strong asset in the years to come, especially in the light of the new research strategy recently set forward by the Ministry of Research and Technology to be summarized in the following.

Research Strategy

The research strategy of Risø and of the Materials Department is linked to the Danish national policy for research and development. In 1993 a new Government has formulated its policy and emphasized the following.

- **The results of research must find application in society with the aim of strengthening employment.**
- **Research administration must be carried out in such a way that it minimizes the burden on the individual researcher.**
- **Ethical and social effects of research and technological development must be taken into consideration.**

In addition to these issues, Government policy also emphasizes training of researchers and international collaboration especially within the framework of the European Union (EU).

The overall goals in the national strategy have, to a large extent, been included in a contract signed in December between the Ministry of Research and Technology and Risø National Laboratory. This contract, of four years duration, ensures that Risø's budget is to be exempt from annual cuts. In return, Risø has undertaken to complete a number of pre-defined research tasks (based on a strategic plan 'Risø 2000', streamline its administration and perform an enquiry among users in the beginning and at the end of the four year period. The contract also foresees an international evaluation of Risø in 1997.

For the Materials Department, a follow-up strategy requires that high level research must continue to be carried out as before. However, it is now more important than ever that this research contributes, and is seen to contribute, both to the advancement of knowledge and to industrial, social and economic advances in society. These aims are, to a large extent already fulfilled by the on-going and planned research projects; our impression from many international contacts is that the Materials Department's work is acknowledged as being in the forefront of materials research. However, existing links can be strengthened and new links should be created especially with Danish and European industry and with national and international teaching institutions. With this in mind a number of initiatives have been taken in 1993 and are reported in the following sections.

Resources

The income of the Materials Department has been satisfactory in 1993, due mainly to the number of large contracts obtained in 1992. These contracts have also allowed for investments in equipment of about 7 mio. DKK. including high temperature x-ray apparatus, equipment for image analysis and instruments for advanced materials testing.

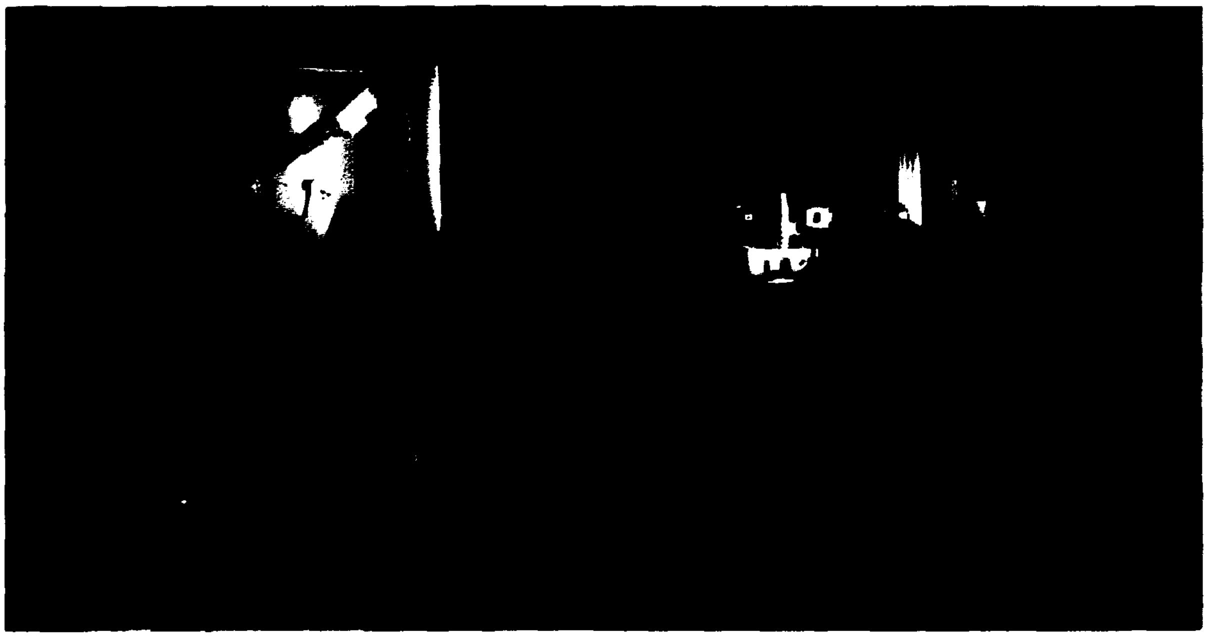
The present staff situation in which the Department has been able to maintain a moderate level of controlled growth by hiring young engineers, scientists and technicians, continues to be satisfactory. The influx of post-graduate students has increased markedly mainly due to the establishment of the Engineering Science Centre. Also, the number of post-doctoral scientists has increased. At present, more than a fourth of the academic staff is comprised of young scientists in temporary training positions. As a result, scientific and technical supervision is becoming very important both for the senior academic and the technical staff.

New programmes and new staff have put quite a pressure on the existing facilities. In order to provide more laboratory and office space it was planned to start refurbishment of the hot cell building in the beginning of 1994. However, due to the complex work of cleaning a highly active nuclear installation, the decommissioning of the hot cells has been delayed for half a year. This means that new laboratories will be ready in the Autumn of 1995, to house activities such as fuel cell research, ceramic technology and polymer composite technology. There will also be space for new educational activities directed towards schools, universities and industrial companies.

Organization

Research in the Materials Department is carried out within the three disciplines:

- Materials Science (theory and characterization)
- Materials Engineering (modelling and performance)
- Materials Technology (synthesis and processing)



The combination of these disciplines within the Department makes it possible to carry out work both in the development, characterization and modelling of materials and processes and on the application of materials in advanced products. Using industrial terminology, the work encompasses material design, process design and product design.

The work in the Department is organized in a number of projects each headed by a project leader. The project leaders are responsible both for the scientific quality and the administration of their projects. Due to the large number of projects, coordination is however, imperative. This is done in different ways. For larger projects or groups of related projects, management or coordination groups are established which combine scientific, technological and administrative expertise. Examples of such activities are the Fuel Cells Programme, the Engineering Science Centre and EU-contracts in which staff members are programme managers. 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, and for larger applications supplemented by staff experienced in administration. Research proposals are considered by internal groups established for each of the research disciplines of the Department supplemented with a group dealing with Energy Materials. The rôle of these groups is at present being expanded to include responsibility, through the chairman, for the follow-up of resources spent on all projects within each of the four groups. Such a regular follow-up of the research activities in the Department should ensure flexibility in the expenditure of resources, especially manpower, combined with an administrative control of income and expenditure.

Research

In this report the work in the Materials Department is described in three chapters, one for each of the research disciplines. Each chapter contains a general description of the work with emphasis on new experimental findings, models and novel techniques. The general research themes have been kept unchanged with one exception: work on polymer chemistry, which had been transferred to the Materials Department in 1990, from the Chemistry Department, has now been moved to the Solid State Physics Department.

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

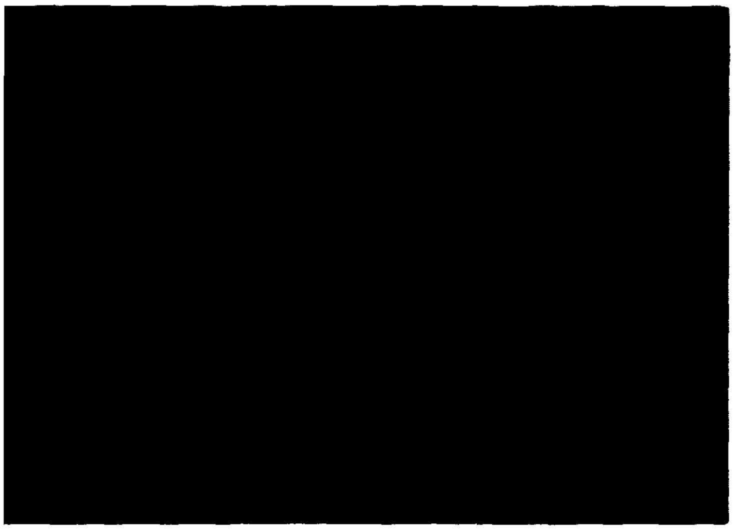
Industry related research

The Materials Department is an active partner in the Danish Programme on Materials Technology (MUP 1). This programme consists mainly of collaborative projects between industry, research institutes and universities organized within seven centres and a number of programmes. The Materials 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. 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 active collaboration and knowledge transfer between the participants. General information meetings have been held and the transfer of knowledge has been further extended to cover a large number of industries. The MUP 1 programme has to a large extent been successful and will be extended from 1994 with the start of MUP 2. In the new programme, basic research will be funded in centres and strategic and applied research will be funded in focused programmes. Financial contributions from industry will be made through an increase in their financial committment as the research becomes more applied and approaches production. The Department is planning to become actively involved in MUP 2 through participation in centres and programmes. The foreseen strengthening of links between the Department and industry will occur, to a certain extent, at the expense of the collaboration with universities and other research institutions established under MUP 1. This is mainly because of the limited resources of MUP 2 and the increased emphasis on industrial participation.

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 of small prototype modules and on longer range research in order to improve the efficiency, economy and versatility of fuel cells. One of the milestones in this programme is to achieve operation of small prototypes (0.5 - 1 kw) during 1996. This work is supported by the ELSAM electricity utility group and by the Ministry of Energy. The overall programme is managed by the Materials Department and participants are the two sponsors, Haldor Topsøe A/S and a number of research institutions.

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, The Force Institute, ELSAM and the Ministry of Energy also participate in this work which aims at increasing the elctrical production efficiency of the plant by raising the combustion temperature. A major problem is the lifetime of materials in a strongly corrosive and erosive environment at high temperature. Politically, waste and bio-mass combustion have been given a high priority in Denmark and as a result the Department has decided to strengthen its research in this field. A five year programme has therefore been formulated with the Technical University of Denmark and the Force Institute with the aim of creating a science and engineering base which can support manufacturers and users in Denmark and abroad. The first part of this programme will be supported by the Ministry of Energy and will include Tampere University of Technology, Finland where test facilities are available. Further support to this programme will come from the participation of the Department in 'COST 501, Work Package 13: Clean Combustion Technologies', with leading European industries as participants.

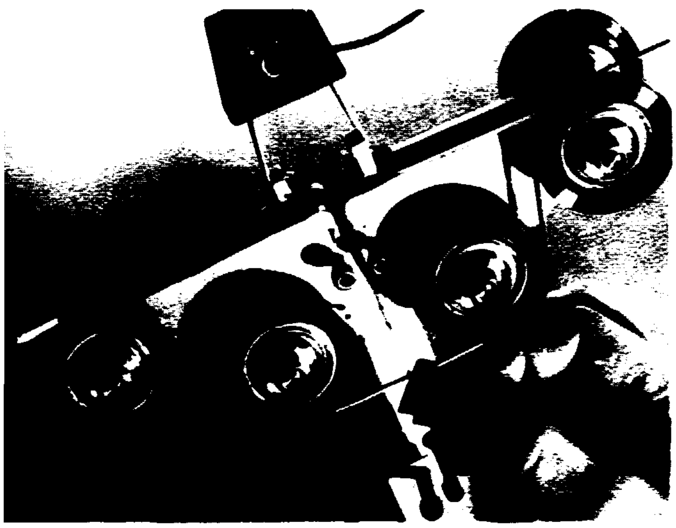
A continuing effort is the materials development, design and testing related to rotor blades for wind turbines. This also has a high priority within the Danish energy programme. The work of the Department in this area is part of a JOULE programme (EU) with the participation of research institutes and industries in many European countries, coordinated by the Materials Department.

Finally, within energy research, the Department is engaged in the European Fusion Technology Programme. The integration of part of the European work into ITER (International Thermonuclear Experimental Research) is at present ongoing. The work of the Department on irradiation effects in metals, especially copper and copper alloys, appears to be highly relevant to the initial design of ITER. The activities of the Department will therefore be continued in this area.

Basic research

The applied programmes described above are founded on long term 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 Materials 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 fabrication technologies in order that well characterized materials are available for the research.

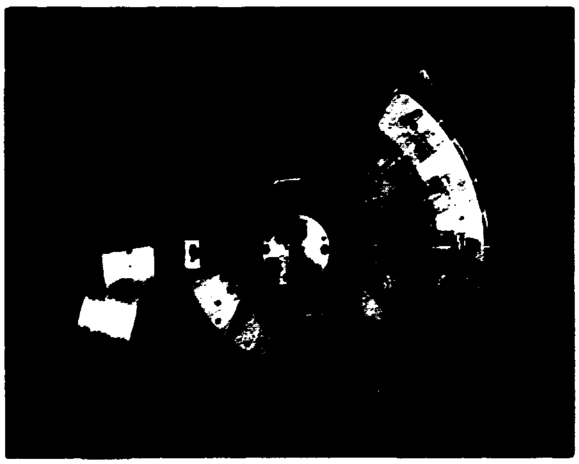
In 1993, research on structural characterization and modelling of materials has been structured in an Engineering Science Centre financed by The Danish Technical Research Council. This centre is to carry out long range research in combination with the training of post graduate students and post doctoral research fellows. The present projects in the centre are grouped under the following headings: (i) Plastic Deformation, Mechanics and Microstructure (ii) Residual Stresses (iii) Composite Materials (iv) Structure and



Mechanics of Polymers (v) Mechanical Properties of Layered Structures. Also, research training activities have started as five post-graduate students have joined the centre. The centre activities are organized in such a way that they are complementary to other activities of the Department on materials structures and modelling. Thereby, the research effort is strengthened as these activities can be financed from other sources including basic funding from the Department. In parallel to the research described above a number of activities are centred around the theme of Materials Processing, Properties and Modelling. The main research areas are numerical and experimental modelling of processes, for example deformation and annealing of materials. This area is both nationally and internationally in focus as it appears within reach to introduce quantitative observations of microstructure, texture, mechanical properties and residual stresses into general models of 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 considerable emphasis is placed on the development of various advanced experimental techniques. Major areas are the application of neutron scattering and 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 The Institute for Mathematical Modelling at The Technical University of Denmark and the Danish Technological Institute.
A highlight last year was the installation of an environmental scanning electron microscope. This new microscope is the first of its kind in Scandinavia and it allows for a number of studies which cannot be carried out in a conventional scanning electron microscope. A number of special materials combinations have already been examined and in-situ studies at elevated temperatures have been carried out. Equipment is being constructed which will allow in-situ mechanical testing of ceramic, polymer and composite specimens and layered structures as well as wear tests of ceramic samples.

A highlight this year has been the installation of new x-ray equipment allowing for structural analysis at high temperature; this being of special importance in the development of materials and components for solid oxide fuel cells. Other important areas which now can be explored is the use of x-rays for measuring residual stresses in thin layers and in multilayers as well as comparing x-ray and



neutron diffraction measurements as complementary techniques for the determination of residual stresses in materials and components.

Achievements

A number of scientific and technical achievements during the year may be mentioned.

In the area of Materials Science, modelling and characterization, studies on single crystals and polycrystals have confirmed a unified description which was proposed for the evolution of microstructures during deformation. Based on this description, texture simulations have shown improved agreement with experimental observations. Also within this area, a recrystallization model has been proposed taking into account nucleation and growth of grains of different crystallographic orientation. In irradiation defect studies, specifically designed experiments carried out in collaboration with Forschnungszentrum Jülich have shown agreement with predicted void swelling behaviour using the newly proposed production bias model.

In the area of Materials Engineering, finite element modelling (FEM) programmes have been implemented covering impact loading of a ceramic-polymer composite armour and a quality assurance (QA) system has been introduced for the manufacturing and testing of armour materials. In Materials Technology, very pure aluminium powder has been produced by atomization and then manufactured into metal matrix composites under clean conditions. In the field of joining, diffusion bonding has been developed for powder metallurgy parts and high strength joints have been made with good reproducibility. Within the field of Energy Materials, through the national programme on solid oxide fuel cells, ceramic interconnect parts have been developed with significantly improved properties especially regarding sinterability and chemical stability. One patent application has been filed on the use of vanadium for interconnect parts.

One member of staff has been elected Member of Academia Europaea. Four PhD projects were completed successfully in 1993.

Finally, all radioactive materials have been removed from the hot cell facility and the cells have been decontaminated. This work has been completed without exceeding the normal average personnel dose.

International Cooperation

The overall Danish research strategy emphasizes the need for international collaboration based on international quality of the national research. Within nearly all research areas of the Materials Department there is an active international cooperation. This is reflected by the increasing numbers of foreign students and guest scientists who have worked in the Department. Active international cooperation is also demonstrated by the many joint authorships of the scientific publications of the Department.

An important part of international collaboration is the participation in many programmes under the auspices of EU. Of special importance is the BRITE EURAM programmes where Departmental participation is in hot rolling of aluminium alloys, hot forging of metal matrix composites, damage tolerant design, engineering ceramics, and brazing of heat exchangers and fuel cells technology. Our activities in 6 BRITE EURAM programmes is supplemented by participation in three JOULE programmes on fuel cell materials and components and on the design and testing of rotor blades for wind turbines. Finally, the Materials Department, together with the Solid State Physics Department at Risø, participates in the EU Large Installations Plan (LIP) covering neutron scattering experiments at the DR-3 reactor carried out by researchers from other EU countries. In this programme the Department supervises experiments on crystallographic textures and on the determination of residual stresses.

New initiatives regarding EU-programmes have been sparse as the 3rd Framework

Programme is running out and the call for proposals under the 4th Framework Programme will not be advertised until the end of 1994. One new initiative has been the involvement of new partners from Eastern European countries and the Baltic region in running EU-programmes. The Department here has succeeded in establishing collaboration agreements financed by EU within two JOULE programmes, i.e. one on Solid Oxide Fuel Cell Materials where the new partners are research institutes in Slovenia, Latvia and Rumania and the other on rotor blades for wind turbines where the new partners are from Latvia and the Czech Republic.

Symposia and Workshops

An important activity in 1993 has been the organization of The Fourteenth Risø International Symposium on High

Temperature Electrochemical Behaviour of Fast Ion and Mixed Conductors. The Symposium followed the format of the Risø International Symposium series and was attended by about 100 scientists representing industry, research institutes and universities. Another activity has been to assist in the organization of two one-day meetings entitled 'Materials - Models - Technology' under the auspices of The Danish Academy of Technical Sciences. The meetings were of industrial interest and covered themes such as materials processing, tool design and manufacturing, materials properties and new experimental techniques for the examination of materials and components. The two meetings were held at Grundfos International A/S and at Risø National Laboratory, respectively, and the number of participants was around 70 each day.

Public Relations

PR activities have included media coverage of research and development and a very large number of visitors to the Department. Another PR activity is the continuation of a series of papers, in Danish, on important research and development items. Finally, plans have been prepared for the establishment of an exhibition area and an auditorium in the hot cell building. These facilities will allow for an expansion of the PR-activities when the refurbishment of the building has been completed in 1995.

Education

Educational activities are becoming an increasingly important part of the work of the Department. 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 their 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 EU. At present, about 20 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. An important objective of this programme is to offer courses for in-service training of employees in Danish industry. However, the economic climate in the industrial sector in 1993 has made this part of the programme less successful than expected.

A new initiative in 1993 was directed towards high schools to develop teaching material in the field of materials science and technology. This material is developed in a cooperation between the staff of the Materials Department and teachers of a nearby high school appointed to this task by the Union of Danish Chemistry Teachers. The teaching will consist of lectures at the school and laboratory experiments carried out in the Department which also will show 'materials at work'. By this initiative we hope to increase the pupils' awareness in the field of materials and also to inspire other research laboratories in the country to launch similar activities.

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 to the Department but the large number of guests has required a more focused effort than before.

Concluding Remarks

A number of important research programmes which were started in 1992 have come into full operation in 1993. Hiring of staff, procurement of equipment and organization of the research have therefore been of high priority. Consequently, less emphasis has been put on contract acquisition, with one exception however, namely the new Danish Programme on Materials Technology. The expected success rate of the applications to this programme cannot yet be estimated. In any event, the preparation of the very detailed proposals has strengthened the technical and scientific links with colleagues in Danish industry. Another positive result is a number of highly relevant proposals which may form a basis for participation by Danish partners in future European collaborations.

The many and diversified activities of the Materials Department reflect the problems and demands of society at large. In recent years, emphasis has been put on the environmental problems related to the production, use and recycling of materials. The Department has not yet looked at environmental problems through dedicated research projects. We are nonetheless currently involved in projects on light-weight materials, sensors, cleaner manufacturing and substitution of dangerous materials, all of which have a strong environmental aspect. Another demand from society concerns education, both of researchers and of employees in Danish industry. This demand has partly been fulfilled, but more can be done. It is therefore planned to increase the number of researchers undergoing training in the Department and to increase the number of courses for continuing education and in-service training. Also, a new initiative is being taken to provide lectures and practical experiments at high school level in materials science and technology at Risø. It is hoped that this will help in increasing the awareness in schools of science and technology in general and materials in particular.

Finally, a general political demand is that the results of research must be applicable and relevant to society. In the materials field this demand requires that there are particularly efficient channels of knowledge transfer between the science and engineering base and industry. This transfer has always been an integral part of the work of the Department through education activities, organization of meetings, symposia etc. Of equal importance is the direct knowledge transfer through collaboration with industry on specific projects. The large national and international programmes on materials research and development should ensure that such activities can be expanded in the coming years with the Materials Department as an active participant.

Materials Science - theory and characterization

2

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 Materials Department. Much of this basic research is carried out in close collaboration with colleagues from universities and government research laboratories around the world. The area was strengthened in 1993 in the Materials Department by the establishment of the 'Engineering Science Centre for Structural Characterization and Modelling of Materials'.

2.1 Plastic deformation with grain subdivision

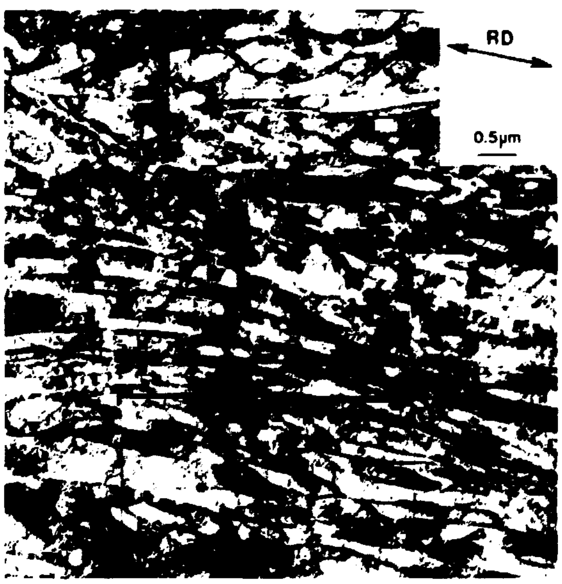
It has become increasingly clear in recent years that plastic deformation is not homogeneous within the grains. Deformation may occur through the development of 'organized structures' which, at a scale coarser than that of the cells or subgrains, repeat themselves more or less regularly throughout the grains. Many different types of organized structures have been, and are being, identified. Descriptions of the evolution of such structures are being developed.

The general concept of grain subdivision during plastic deformation has been investigated in collaboration with researchers from the University of Virginia and Sandia National Laboratories, USA and the Ecole des Mines de Saint Etienne, France. These studies are carried out along different lines; (i) effect of strain, strain rate, strain pattern and temperature on the microstructural evolution, (ii) experimental measurements and simulation of the texture evolution, (iii) qualitative and quantitative relationships between microstructure, texture and mechanical properties, (iv) comparison of single crystal and polycrystal behaviour.

Microstructural evolution from intermediate to large strain

High purity aluminium (99.996%) and high purity nickel (99.99%) have been deformed by cold rolling to reductions of between 37 and 98 per cent (von Mises effective strains of 0.5-4.5). The microstructures evolved with strain within a framework common to fcc polycrystals with medium and high stacking fault energies. This framework consists of a structural subdivision by higher angle boundaries (geometrically necessary boundaries) on one volume scale and at a

Thin foil from longitudinal section of 98% cold-rolled pure nickel. The rolling direction is marked RD. This large view covers several grains which are subdivided by dislocation configurations in the form of lamellar boundaries (LBs). (courtesy of D.A. Hughes)



smaller volume scale by lower angle cell boundaries (incidental boundaries) for all strain levels. The geometrically necessary boundaries take the form of dense dislocation walls (DDWs) and first generation microbands (MB1s) at low and intermediate strains and lamellar boundaries (LBs) at large strain. In addition to these microstructural features, dislocation structures are observed which show that extensive localized glide has taken place. This phenomenon is being studied in order to shed light on the factors which contribute to the transition to microstructural subdivision by new, intragranular, high angle grain boundaries. This type of microstructural subdivision begins at medium to large strains. Such high angle boundaries, created during deformation, are of major importance in subsequent recovery and recrystallization processes.

Deformation structures in single crystals

Different single crystal orientations of high purity aluminium (99.99%) have been deformed in channel die compression up to strains close to unity in order to correlate the dislocation structures in single and polycrystals with the slip system distributions. To obtain detailed information, the dislocation structures are characterized over a wide range of scales by optical microscopy, transmission electron microscopy and scanning electron microscopy with EBSP (electron back scattering patterns). Low energy dislocation matrix structures composed of cells, cell blocks, dense dislocation walls and first generation microbands are obtained in all orientations in agreement with the microstructure of rolled polycrystals. Two orientations (the S-orientation and the Cu-orientation) also develop narrow bands of localized glide associated with relatively high local misorientations. The S-orientation exhibits characteristic S-shaped band structures of first generation microbands, sheared on {111} planes, whereas the Cu-orientation forms non-crystallographic shear bands. The correlation observed between crystal orientation and the occurrence of localized glide forms a basis for further studies of this phenomenon.

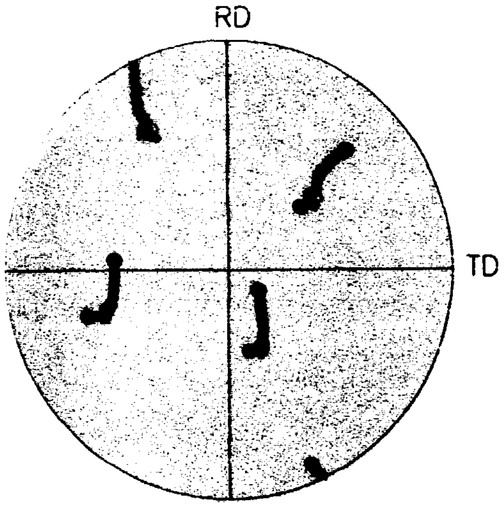
A Taylor-type model for grain subdivision

At the Materials Department, a simple quantitative continuum model has been formulated for plastic deformation with grain subdivision. The model considers grain subdivision by one family of parallel DDWs/MB1s into two families of cell blocks or bands. Strain continuity between the bands into which the grains are subdivided is maintained with relaxed constraints (with reference to the flat shape of the bands), while strain continuity between the grains is maintained with the full-constraint Taylor model (with reference to the fact that the model refers to low to moderate strains with approximately equiaxed grains). The average number of active slip systems in each band is found to be four, i.e. it is lower than the number of (at least) five found in the simple Taylor model. This reduction in the number of slip systems operating at any specific point in the material was actually predicted in the interpretation of the experimental observations.

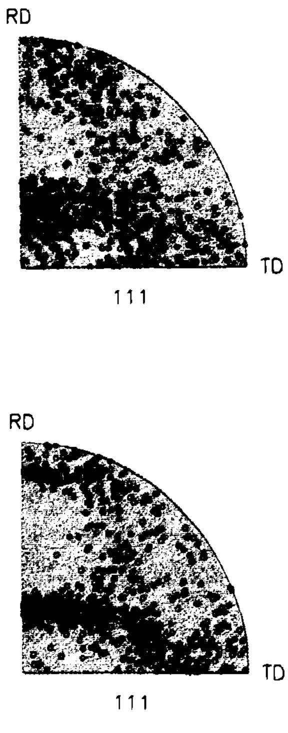
The model includes a special set of rules for the lattice rotations - which is necessary in the situation with two band families considered to deform with coupled deformation patterns. During deformation the lattices of the two band families rotate away from one another. The net result is a simulated texture which is far less sharp than that obtained by simple Taylor simulation without grain subdivision. This scattering of the texture is in agreement with experimental observations: it is well-known that the textures simulated with the simple Taylor model are far too sharp. The texture scattering was already foreseen as a result of grain subdivision in the interpretation of the experimental observations.

There are a number of possible variations in the continuum description of plastic deformation with grain subdivision as outlined above. These variations are not to be described in detail, but only to be listed: one may consider subdivision into more than two families of parallel bands, one may consider subdivision into crossing families of bands, one may consider grain subdivision without fulfilment of the Taylor condition for the individual grains, and one may consider subdivision into band families with orientations parallel to the rolling plane.

As for the original cause of grain subdivision, a continuum model like that presented here does not provide any suggestions: from an energetic point of view it makes no difference whether the grains subdivide or not. However, once subdivision has been initiated, continued deformation with differently deforming bands



The development in lattice orientation in a "typical" grain rolled to 50% reduction with grain subdivision as illustrated by the <111> poles in stereographic projection. The filled circles show the initial orientation, and the open circles show the final orientations of the two band families.



Simulated {111} pole figures for rolling to 50% reduction for 100 grains with subdivision into two band families (top) and for 200 grains with Taylor deformation without grain subdivision (bottom).

(with increasingly different lattice orientations) has an energetic advantage over homogeneous deformation - as reflected in a decrease in the overall Taylor m factor. It is a fair conclusion that a simple continuum model of the type presented here may provide a framework for the description of the development of plastic deformation with grain subdivision. Further theoretical input is required before the initiation of grain subdivision can be treated.

2.2 Micromechanical modelling

Micromechanical modelling based on a computational framework within the context of general continuum mechanics has been applied increasingly to predict the detailed behaviour of new materials at a microlevel. Application of the finite element method has provided insight into both local and overall inelastic behaviour of metal matrix composites (MMC).

Simulations of the development of the local behaviour in MMC's show the appearance of localized deformation zones in the aluminium matrix at the ends of the short ceramic fibres (whiskers). These highly strained regions are in close accordance with the appearance of highly dislocated regions at the whisker ends observed in transmission electron microscope (TEM) studies of deformed MMC's (research carried out in collaboration with the Engineering Department, University of Cambridge, UK). Details of the shapes of high dislocation density regions around fibre ends, as well as the occurrence of regions of low dislocation density around fibre mid-points, are highly similar in both the simulations and in the direct observations in the TEM. These comparisons have been carried out successfully for deformation levels of 3, 5 and 10%. The prediction of overall properties such as work hardening rates and flow stress also show good agreement. The present results, obtained for a dilute system containing 2 vol% of whiskers, are to be extended to 10 vol%, in which fibre/fibre interactions will be of significance. Generally speaking, the application of the well-established framework of continuum plasticity in the analysis of metal matrix composites seems to be an excellent tool for the study of these new materials in conjunction with experimental work, such as mechanical testing, neutron diffraction, and TEM studies.

2.3 Texture in hot deformed aluminium

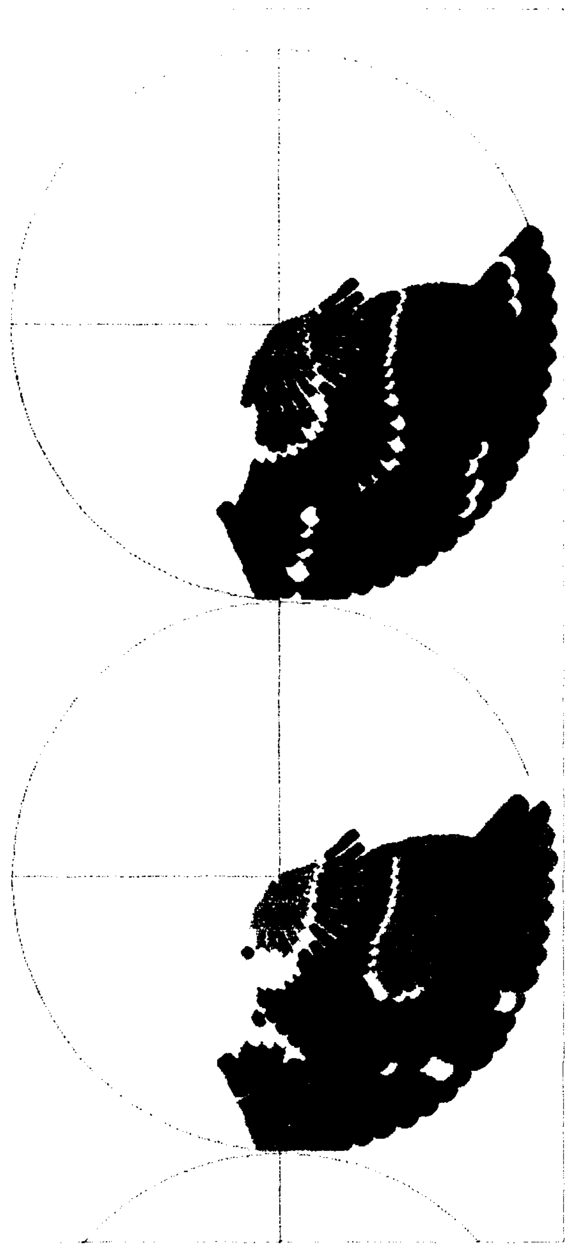
The crystallographic texture affects materials properties. For example, for flat rolled products the texture largely determines the formability and is a dominant source of anisotropy causing 'earring' in can production by deep-drawing.

One of the aims of a BRITE/EURAM project in this area is to study the effects of strain, strain rate and temperature, on the deformation and subsequent recrystallization textures in three aluminium alloys: Al-1050, Al + 1% Mn and Al + 1% Mg. Various modelling approaches are also used in an attempt to simulate the texture development. In the following, the Plane Strain Compression, PSC, deformation texture results will be summarized.

All three alloys have a fairly weak initial texture and develop a typical rolling type texture during PSC. With increasing strain, the strength of the rolling texture increases and the initial cube texture decreases. This result is expected and agrees with earlier findings. Of more interest is the effect of strain rate and temperature. For all three alloys, the strain rate seems to have no systematic effect on the texture development. This has the important consequence that the Zener-Hollomon parameter cannot be used to describe the deformation texture development.

The deformation temperature has a significant effect on the texture development. Higher temperatures generally lead to stronger and sharper rolling textures. The reason for this may relate to the development of heterogeneities in the deformation microstructure. After cold rolling, it is generally observed that the grains break up into cell blocks delineated by dense dislocation walls (DDW) or microbands (MB). Such a break up will influence the texture development. For the present Al + 1% Mn material, it was found by TEM investigations that the microstructure after deformation at 300°C contains significantly more DDW/MBs than at 500°C. These observations relate well to the texture results showing a weaker and more scattered texture at 300°C than at 500°C.

The deformation texture development was modelled using the Los Alamos Polycrystal Plasticity Simulation Code (LApp) and an FC/RC model which may include random stresses. Both models overpredict the development of the rolling components, i.e. at a given strain the simulated textures are stronger and sharper than those measured experimentally. The models predict the experimentally observed decrease in the cube component reasonably well. Better agreement between simulated and experimental rolling components is achieved by introducing an additional smearing of the simulated discrete orientation in the LApp simulation and by adding random stresses to the other type of modelling.



Texture determination from neutron diffraction. Hot-deformed aluminium investigated as part of a BRITE/EURAM project.

2.4 Cyclic plasticity and fatigue

Modelling of cyclic slip localization

Work on cyclic plasticity was concentrated on the phenomenon of cyclic localization of slip into persistent slip bands (PSBs), which are the observed sites of fatigue crack initiation. Cyclic slip localization is commonly described by the phenomenological two-phase model, in which a constant volume fraction of PSBs carries a finite amplitude of plastic macroshear e_{PSB}, while the embedding matrix merely undergoes reversible cyclic microshear. The two phase model is a useful partial model for small cumulative strains but it breaks down at large cumulative strains, where a slow but steady irreversible production of new PSBs is observed. The replacement of the two-phase model by a more realistic static-dynamic model for large cumulative strains has awaited the outcome of experimental checks of model predictions. However, independent experiments have recently confirmed the proportionality between stress and strain amplitudes in PSBs, which is predicted by two entirely different groups of models: Purely dynamic models predict a proportionality constant of 2π, subject to assumptions about mobile dislocations, such as blocking of dislocation production at both ends of the strain cycle or glide of dislocations in well defined groups. Direct quantitative observations of such dynamic features are difficult, for example because rearrangement or loss of mobile dislocations normally occurs during TEM thin foil preparation. A much simpler static model was therefore developed, which predicts that edge dipole arrays in PSBs (but not in the matrix) are destabilized at both ends of the strain cycle. This model predicts a different value of the proportionality constant, which is calculated from quantitative TEM observations of the static structure. The recent experimental confirmation of the stress-strain proportionality for PSBs was found to support the proportionality constant predicted by the static model.

Based on the experimentally justified static model the two-phase model was then developed into a static dynamic model, accounting for the crucial irreversibility of cyclic plasticity as well as its remarkable reversibility. The new model makes a number of microstructurally based predictions. For example, it shows that contrary to the two-phase model, the PSBs do not operate in a passive reversibly shearing matrix. Irreversible matrix hardening drives the continued formation of PSBs over cumulative shears of about 200. TEM based studies of the matrix as it varies with cumulative strain and strain amplitude are in progress. Only two general dynamical assumptions have been needed so far. They are (1) that the fatigue dislocation structures have no difficulty producing mobile dislocations and (2) that saturation of the stress amplitude is associated with an upper limit to the density of mobile screw dislocations. On this basis the model explains why slip is extremely uniform outside PSBs but non-uniform within PSBs, as a result of destabilization of the static critically stressed PSB structure. Thus, the model can provide a realistic physical basis for understanding the development of PSB surface roughness initiation and accumulation of cracks.

Low cycle fatigue in duplex steel

The fatigue of an austenitic-ferritic (α-γ) stainless steel was investigated by plastic strain controlled cyclic experiments and in-situ neutron diffraction monitoring of average strain-induced residual stresses in α-grains and γ-grains. It has recently been reported in the literature that the fatigue behaviour of duplex steel approaches that of the α-phase at high strain amplitude and that of the γ-phase at low strain amplitudes. This observation has been explained in terms of a composite model featuring hard α-grains and softer γ-grains. However, it has also been suggested that the addition of nitrogen preferentially increases the hardness of the γ-grains. At about 0.2 wt% nitrogen, the α-phase is expected to be the harder phase. Therefore, it is of interest to examine the effect of nitrogen on the phase coupling and fatigue of α-γ steel.

Centrifugally cast duplex steel of type DIN 1.4462 with a nitrogen content close to 0.15 wt% was selected for study. The published observations of composite behaviour were made for α-γ steel with only 0.07 wt% nitrogen. During monotonic loading to 500 MPa the average lattice strains measured by neutron diffraction were found to increase linearly to about 2×10^{-3} in both phases. In all experiments, after unloading, the γ-phase was left with a slight compressive lattice strain, but the effect was very small. This behaviour implies that the α-and γ-phases are of about the same hardness. The fatigue experiments were found to be consistent with the neutron diffraction data, there is no indication of a transition from α- to γ-controlled fatigue in the present duplex steel.

2.5 Internal stress in MMC's

A graphical model for the determination of thermally induced residual stresses in composite materials has been tested by neutron diffraction measurements. The measurements showed rather small internal stresses which were apparently independent of the volume fraction of the ceramic fibres in the metal matrix composite. These experimental observations were in agreement with the predictions of the model. Although these measured values were very small, they did provide some verification of the degree of accuracy of the model. The work is being continued in 1994 and extended to the study of the general internal stress development as a function of degree of plastic deformation.

Relaxation of internal stresses in Al/SiC have been analyzed using a new concept, denoted 'thermal strain relaxation loop', TSR-loop, which provides an overall view of the relaxation process. The concept will be used to construct TSR-loop maps for various composite systems.

2.6 Mathematical modelling of SOFC-stacks

A mathematical model describing the heat-, mass- and charge-transfer processes in a SOFC-stack can be a valuable tool in the process of designing the stacks, as well as for selecting proper conditions of operation. The transport processes are strongly coupled and it is impossible to predict the behaviour of the stack under various conditions without rigorous modelling.

Experimental studies of SOFC stacks are limited by the time involved in physically assembling the stacks. Ideas on improvement of the design are much more easily tried out by computer simulation. Furthermore, much insight into the problems can be gained through the modelling.

A full three dimensional model describing the heat-, mass- and charge-transfer processes in a SOFC stack has been developed. The model is based on a finite volume discretization of the governing partial differential equations. The model can be used for a number of different boundary conditions and allows a determination of the temperature- and current density distribution, as well as the distribution of the reactants and reaction products, over the stack.

The model is being used in simulation experiments aimed at optimizing the design of the interconnect plates. The interconnect plates connect the cells of the stack in electrical series, and gases are fed to and removed from the cells through channels cut in these plates. The design of the rib/channel-structure in the interconnect plates must take into account multiple and mutually competing requirements. Examples are, minimization of the overall ohmic resistance and maximization of the electrochemically active area. Computer simulations have shown that the customary choice of using channels of equal cross sections in the manufacture of interconnect plates is in fact not optimal. The stack performance of a cross-flow stack can be improved by varying the cross sectional area from channel to channel in a prescribed manner.

2.7 Deformation of polymer matrix composites

The response of polymer composites under long time loading is of important, both for the understanding of deformation mechanisms and from the more practical design aspect. In these analyses of deformation it is important to obtain a correct, and if possible, simple description of the behaviour.

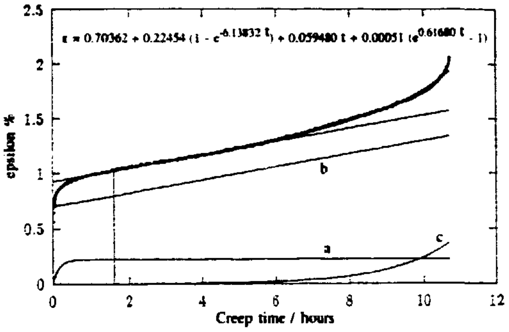
The long time response under constant load is creep, and an empirical equation has been established for the relation between strain and time:

$$\varepsilon = \varepsilon_0 + \Theta_1(1 - e^{-\Theta_2 t}) + \Theta_3 t + \Theta_4(e^{\Theta_5 t} - 1)$$

where ε is strain, t is time and Θ_n are empirical constants. The first term in the equation is the initial elastic strain, the second term is the visco-elastic creep, the third term is the linear creep, and the fourth term is the accelerating creep during accumulation of damage in the material. The Θ-parameters are found by iterative fitting of the equation to the experimental data of strains versus time. These Θ-parameters depend on stress, temperature and environment.

Extensive data have been collected for creep of composites of thermoplastic polymers with glass fibres. The polymers are polyphenylene-sulphide and polyethersylfone, respectively, and the glass content is 30 and 40 weight percent, respectively. The glass fibres are short and well oriented along the direction of injection moulding; test specimens are cut at various angles relative to the fibre direction. The stress levels are 20 to 120 MPa, the temperatures are 20 to 140 C, and the environment is water at pH-levels of 7 to 12.

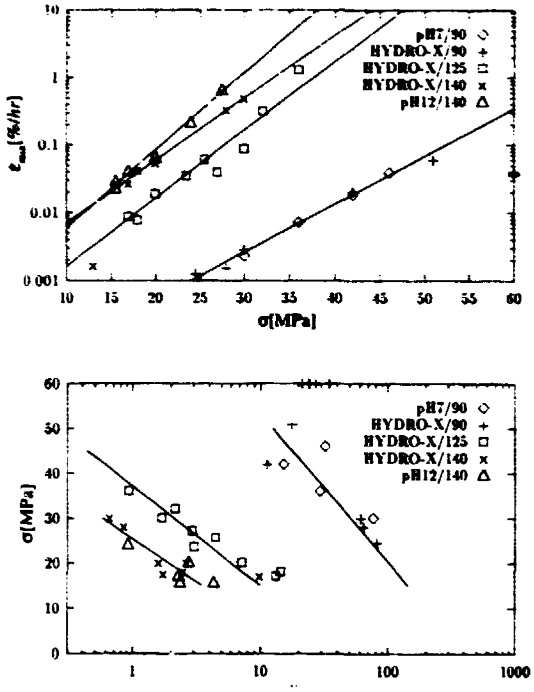
Creep curves of strain versus time are illustrated in the figure; the experimental data (marked by small squares) agrees very closely with the overall fitted equation. The individual terms of the equation are shown separately in the figure, and indicate the relatively fast decay of the visco-elastic term (a), the slope of the linear term (b) and the slow start-up of the damage of the accelerating term (c). The equation with the actual numerical values of the Θ-parameters is written in the figure.



Creep curves for thermoplastics with glass fibres. The individual elements, a) visco-elastic term, b) slope of the linear term and c) the gradually increasing and accelerating damage term are marked.

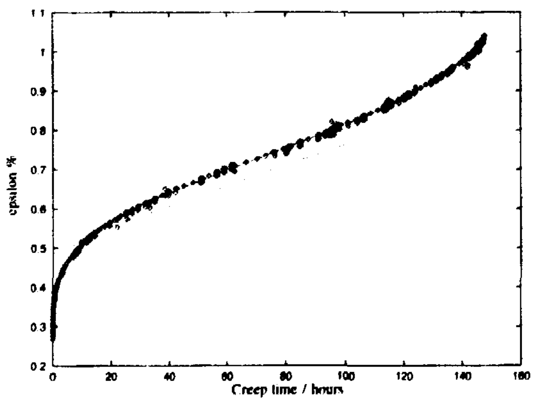
By differentiation, the time for the minimum creep rate and its value can be calculated: this minimum creep rate of 0.06039 is obtained from the figure as well as the corresponding time. This time is an effective time for the start of damage (as can be seen from the figure, where t_{min} is 1.58 hrs), whereafter the accelerating term gains significance.

An important relationship is the dependence of creep rate on stress; a plot of the minimum creep rate versus stress for several groups of experiments is also shown. The creep rate depends semi-logarithmically on the stress, at a given temperature and environment. Also, the time to minimum creep rate is shown as a function of stress, where a possible threshold limit for stress is indicated at 15 Mpa.



Minimum creep rate and time to minimum creep rate, plotted as a function of stress.

The Θ-parameters and their dependence on stress, temperature and environment have been analyzed and empirical relationships established. From these it is possible, for a given set of external conditions of stress, temperature and environment, to re-calculate the creep curve. This is illustrated by an example in the last figure, where the small diamonds mark experimental data, the long-dash curve is the individually fitted Θ-equation,



Experimental, Θ-parameter fitted, and re-calculated creep curves – see text.

and the short-dash curve is the re-calculated creep curve. It is clear that some difference occurs for the re-calculated curve; this is due to the scatter in fitting the Θ-parameters against the external conditions.

2.8 Irradiation damage

Defect accumulation during 2.5 MeV electron and fission neutron irradiations

(in collaboration with Forschungszentrum Jülich, Germany)

Recently, a new model called "Production Bias" has been proposed to explain the damage accumulation (e.g. void swelling) behaviour under cascade damage conditions. One of the major predictions of this model is that because of the asymmetry in the production of free and mobile vacancies and self-interstitial atoms (SIAs), the damage accumulation under cascade damage conditions (e.g. fission and fusion neutrons) would be substantially different from that under the conditions of single displacement production (e.g. 1 MeV electron and low energy light-ions). To test this prediction, pure copper specimens were irradiated with 2.5 MeV electrons and fission neutrons at 523 K with a damage rate of $\sim 10^{-8}$ dpa/s. Irradiations with electrons and neutrons were chosen because 2.5 MeV electrons produce displacement damage in the form of single vacancies and SIAs as Frenkel pairs whereas fission neutrons produce multidisplacement cascades and subcascades. Post-irradiation investigations of defect microstructures using electrical resistivity, transmission electron microscopy and positron annihilation techniques clearly demonstrate that the accumulation of defects during neutron irradiation is substantially increased compared to 2.5 MeV electron irradiations. This is in full accord with the prediction of production bias model. Furthermore, these results suggest that the production bias model is necessary to understand the swelling behaviour under cascade damage conditions.

A multi-model approach to defect production in high energy collision cascades

(in collaboration with Pacific Northwest Laboratory and University of California, Livermore, USA).

A multi-model approach is being employed to simulate defect production processes at the atomic scale. The model incorporates molecular dynamics (MD), binary collision approximation (BCA) calculations and stochastic annealing simulations. The central hypothesis is that the simple, fast computer codes capable of simulating large numbers of high energy cascades (e.g BCA codes) can be made to yield the correct configurations when their parameters are properly calibrated using the results of the more physically realistic, but much slower, MD simulations. The calibration procedure has been investigated using results of MD simulations of 25 keV cascades in copper. The configurations of point defects are extracted from MD cascade simulations at the end of the collision phase, thus providing information similar to that obtained with a binary collision model. The MD collisional phase defect configurations are used as input to the ALSOME annealing simulation code. The values of the ALSOME annealing quenching parameters are determined that provide the best fit to the post-quenching defect configurations of the MD simulations. A set of 100 cascades of 25 keV was produced in copper with the BCA code MARLOWE and run through the ALSOME quenching and short term annealing. The yields of total and freely migrating defects in these simulations were very similar to those obtained in the MD cascades after short term annealing.

2.9 Electroceramics

The majority of the ceramics research at the Materials Department is carried out in connection with the development of solid oxide fuel cell components. In addition, a wider spectrum of phenomena (and applications) of oxide ceramics are studied; basic studies of electronic and ionic transport in ceramics form the subject of PhD-projects; piezoelectric ceramics made by extrusion are under study in collaboration with a Danish firm; compact oxygen sensors based on yttrium-doped zirconia with a built-in reference chamber have been developed. The latter activity combines ceramic and electrochemical know-how with ceramic to metal bonding technology.

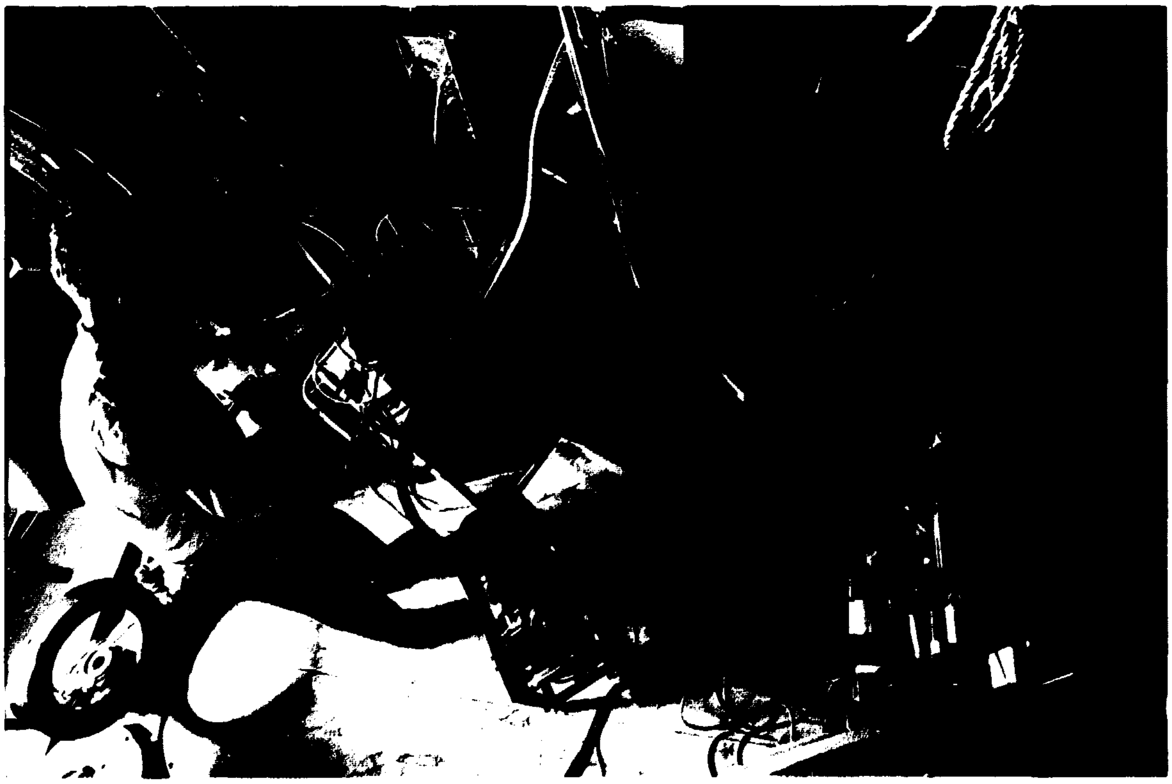
The Materials Department's basic activities within the field of electroceramics include the projects outlined below.

Rare earth oxides and ferrites are under investigation as components of solid oxide fuel cells and sensors. We are investigating electrical properties and structural phase transitions of these materials by means of electrical measurements and high temperature x-ray powder diffraction. This work is being carried out in co-operation with the foundation for Scientific and Industrial Research at the Norwegian Institute of Technology.

Thermoelectric power measurements on solid oxide fuel cell materials are determined in order to evaluate possible thermoelectric effects in the fuel cells. In addition, they also have a potential as a means of determining ionic and electronic transport properties and predict possible proton conductivity in bulk ceramics.

The search for new solid electrolytes and electrode materials for solid oxide fuel cell and sensor applications would be greatly facilitated by formulating criteria for predicting solids with high ionic mobilities. We are refining such criteria by systematic studies on a model system of perovskite-type oxides, a group of materials with very varied electrical properties. Their structures are investigated by neutron diffraction techniques, both using the DR-3 source at Risø and the pulsed neutron source at the Rutherford-Appleton Laboratory, UK. This study has revealed that crystallographic parameters and thermodynamic lattice energies can be correlated with the measured electric transport properties.

Thermoelectric power measurements (Seebeck effect) are carried out on solid oxide fuel cell ceramics. Photo taken at a busy time in the development of the project.



Experimental set-up for neutron diffraction in DR-3. Study of pervoskite-type oxides at varying temperatures and partial pressures of oxygen.

2.10 Microscopy

The ElectroScan™ environmental scanning microscope which was installed in December 1992 has been extensively used in a number of projects which can benefit from its special features. For instance, solid oxide fuel cells contain several insulating materials, and in the SOFC project it has therefore been of great advantage to be able to examine these materials without coating with conducting layers. This project has also benefited from the heating stage, which can operate at temperatures up to 1200°C. The heating stage has for instance been used in observation of melting and surface crystallization of silicon-free glasses used for sealing of cell stacks. Also, de-wetting of Ni-metal from a substrate of yttrium-stabilized zirconia was observed; such studies are of interest in predicting anode performance. In collaboration with the Department of Combustion, the heating stage has also been used to watch, in-situ, the processes of burning of coal particles. The deformation stage which was delivered with the microscope has been used to study the deformation of wood in compression in collaboration with the Building Materials Laboratory at the Technical University of Denmark. Only the strain can be measured with this deformation stage. Work has therefore begun to construct a new dynamic deformation stage in which stress and strain can be measured simultaneously. A stage to be used for bending test is also under construction.

The ElectroScan microscope was delivered with software and hardware for digital acquisition and storage of digital images. In August, a similar system was purchased for the JSM-840 scanning microscope. A slow scan CCD camera with a large dynamic range has been ordered for the JEOL-2000FX transmission microscope. This camera can be used both for diffraction patterns and for low intensity image. From early in 1994 there will thereby be easy access to digital image acquisition, processing, and storage at all these microscopes. Analysis of diffraction patterns and images will also be started in 1994 with the purchase of new image analysis equipment.

JEOL 2000FX transmission electron microscope. Used in a wide range of projects requiring microstructural, micro-crystallographic and micro-chemical information. To be equipped with a slow scan CCD camera in 1994, to allow digital image acquisition, processing and storage.

The software for automated electron back scattering pattern analysis has been extended with an algorithm that makes it possible to calculate a value for the pattern quality. The pattern quality depends on the crystal perfection. It can therefore be used to distinguish between recrystallized and non-recrystallized material in a partly recrystallized sample. The calibration procedure (i.e. determination of the pattern centre and the distance between source point and pattern centre) has also been fully automated. The automated procedure is based on the methods used earlier for manual calibration, where the parameters needed are calculated from a comparison between measured and simulated patterns.

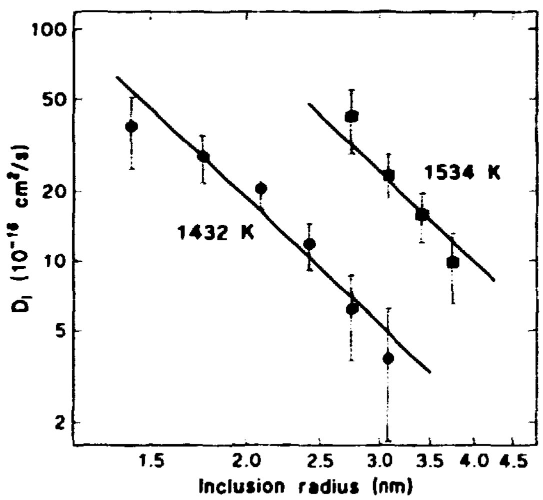
In collaboration with the Department of Materials and Metallurgy at the University of Cambridge, parallel electron energy loss spectroscopy (PEELS) has been used to study variations in manganese in yttria-doped zirconia. Variations in the PEELS spectra are being related to the manganese oxidation state/coordination.

At a meeting in April, arranged by the Materials Department, and at a meeting in November, arranged by the Danish Academy of the Technical Sciences, invited audiences from Danish industry were given an overview of the electron microscopy facilities at Risø. Special emphasis was given to new techniques such as environmental scanning electron microscopy and automated electron back scattering pattern analysis.

2.11 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 injected into a material are often trapped at vacancy type defects (single vacancies, vacancy clusters, vacancy-impurity clusters, porosities, gas bubbles, dislocations, surfaces, interfaces etc.). The annihilation characteristics (e.g. positron lifetime) change when the positrons become trapped. This change can be measured and contains information about those defects that trap the positrons.
An oven for in-situ measurements of positron lifetimes at temperatures up to 1000 C in atmospheres with a controlled oxygen partial pressure has been constructed. The oven will be used for studies of ceramic materials. In order to utilize the full potential of the oven it has been necessary to develop a new type of positron source which is stable against oxidation and against evaporation of the positron emitting isotope at high temperatures. Transmission electron microscope studies of the annealing behaviour of platinum, ion implanted with sodium (^{22}Na) have shown that some of the sodium forms inclusions which only diffuse very slowly in the platinum at high temperatures. It is therefore possible to prepare positron sources for use in the oven by ion implantation of platinum foils with the positron emitter ^{22}Na. Work has been carried out in a collaboration with the ISOLDE group at CERN to prepare such implantation.In order to gain information on the structure and annealing behaviour of vacancy like defects in ceramic materials, specimens of various metal

Size dependence of the diffusion coefficient for Na inclusions in Pt at two different temperatures. The inclusions were formed by implantation of 60 keV Na^+ ions into 70 nm thick Pt foil. Larger Na inclusions remain within the foil, even during long term annealing. This behaviour is utilized in a new type of ^{22}Na positron source for high temperature positron annihilation studies.



oxide crystals have been electron irradiated at low temperatures (80 K) in a collaboration with Stuttgart University. Isochronal annealing of the irradiated specimens has been carried out and positron lifetime measurements made after each annealing step. Various annealing stages (e.g. vacancy clustering) have been identified by these measurements.

Within the framework of the Centre of Powder Metallurgy and in collaboration with the Technical University of Denmark, the positron annihilation spectrometers have been used to study powders with nanometre sized grains, in particular Ni and FeCr powders. Adsorption of gases has been looked at, as well as the reduction of surface oxides on the particle surfaces and the subsequent sintering of the particles.

In collaboration with KfK Karlsruhe, Germany, annealing experiments have been carried out on Cu, He implanted at different temperatures and to different doses, to study the development of He bubbles and other microstructure. Also, in collaboration with Research Centre Jülich, Germany, the irradiation damage structure after electron and neutron irradiations of Cu at 250°C and its dose dependence has been investigated, in order to compare the structures in the framework of the "production bias model".

2.12 Texture measurements by neutron diffraction

The texture of bulk samples can be determined fully automatically by neutron diffraction. During the year, the texture work has covered studies such as:

i) texture in hot deformed aluminium. This is part of a BRITE/EURAM programme,

ii) recrystallization kinetics in aluminium, and

iii) effects of texture on flow stress anisotropy in aluminium.

About 3 weeks of the neutron texture beam-time were used by EU researchers supported by the 'Large Installations Plan'. One of these projects was an analysis of texture changes during phase transformations of low carbon steel, and required in-situ measurements over a period of about 3 days at temperatures in the range of 800-1100°C. In another project, results of neutron texture measurements of two-phase α/β-brass were compared to those obtained by x-ray diffraction. For the α-phase (80 vol%), excellent agreement was observed, whereas for the β-phase, large discrepancies were seen to exist. This is due to the fact that the absorption correction of the x-ray data is extremely complicated when there is a second phase present (as in the α/β-brass).

Finn Nielsen

Materials Engineering - modelling and performance

3

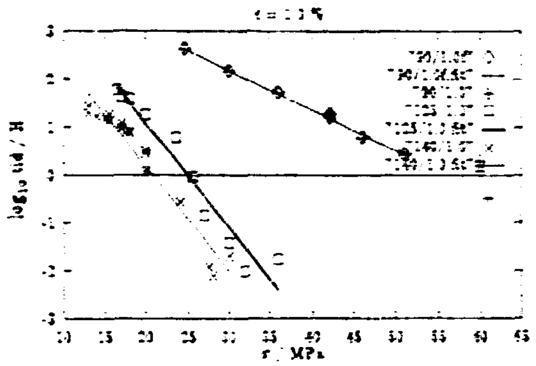
A thorough knowledge of the mechanical properties of engineering materials is essential for the design of advanced components and structures. Of special interest are polymer composite materials, metal matrix composites and engineering ceramics. The research activities in Materials Engineering are centred 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 and often directly related to improving the performance of a particular component or of a new material combination.

3.1 Design with polymeric composite materials

The design aspect for long time loading of polymeric composites is closely related to the description of the creep behaviour. In the design procedure it is of practical importance to have a simple, and still correct, description of the creep. This can be either analytical or as a set of diagrams. In some design tasks it is useful to have access to the full creep curve in terms of strain versus time, while in other cases the requirements are less stringent.

For the first case a useful empirical equation is presented in section 2.7. Here the designer can evaluate all stages of the creep, from the early visco-elastic deformation to the final stages where damage in the material makes the creep rate increase. This detailed knowledge may typically be relevant in design analysis of a rather complex component, where the loads on it are non-uniform from position to position, and where the loading is multiaxial (i.e. not uni-axial), so that the overall creep is composed of contributions where each region is at a different stage on its creep curve.

When only limited knowledge of the creep behaviour is needed, one procedure has been to estimate the time for a given (total) creep strain, and describe the relations to the stress, temperature and environment. This is illustrated by the time to reach a creep strain of 1%, and an example is shown in the figure with log time versus creep stress. Useful empirical relations are found for each temperature. The material is a polymeric composite of thermoplastic polyphenylene-sulphide with 30 wt% short glass fibres aligned along the loading direction. The described approach may typically be relevant for design cases with simple components under uniaxial loading, and with a design criterion of a maximum allowable strain.



For a given composite and known application, we may design by applying simple criteria, for example, the time to reach a creep strain of 1%.

3.2 Damage tolerance and repair of composites

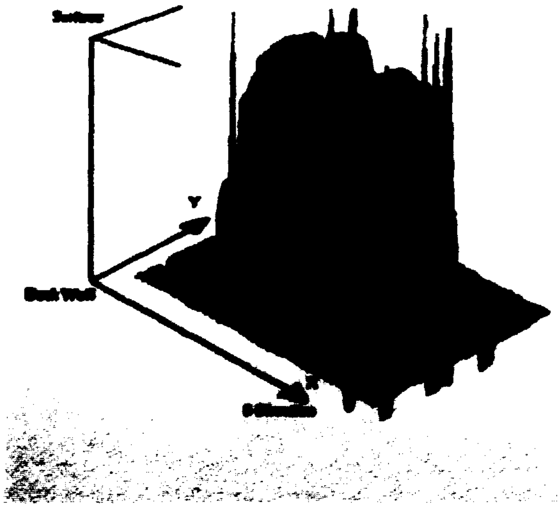
Polymer composite materials are widely used in engineering applications due to their high stiffness and strength, both for static loading and in fatigue loaded components. For many applications, material thicknesses of a few millimetres are sufficient to fulfil the functional requirements, and when sandwich designs are used, the skin layers with thickness less than 1 millimetre on each side of a foamed core material may be sufficient.

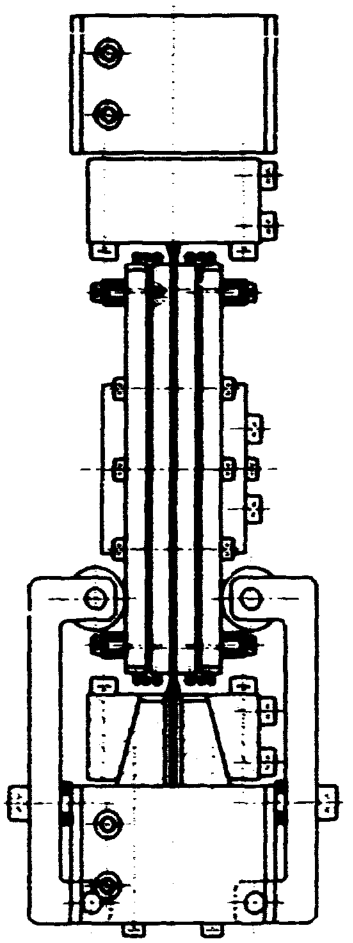
Solid panels or shells, as well as sandwich configurations, are designed and optimized for in-plane loading or for evenly distributed surface loads. The design does not specifically take in account concentrated loads or impacts and the structure may therefore be vulnerable to such loadings. Especially accidental impact loading cannot be avoided in general engineering applications, and it is therefore important to investigate the effect on the strength and stiffness properties of the material and to establish design rules which take property changes into account.

Within the framework of a BRITE-EURAM programme, a series of investigations has been carried out on the static and fatigue strength of carbon epoxy thin laminates and sandwich panels. The programme covers the detection and characterization of the damage (damage area or volume), the selection of suitable repair methods and the measurement of strength of the damaged panels and of the repaired panels.

Impact damage was introduced by impacting the test panels in an impact machine using a spherical indentor. Different impact energy levels (3-30 J) were applied, resulting in different damage zones. An example of the size of the damaged volume below and around the impact position is shown in the figure.

Impact damage in carbon epoxy laminates shown by results from ultrasonic testing.



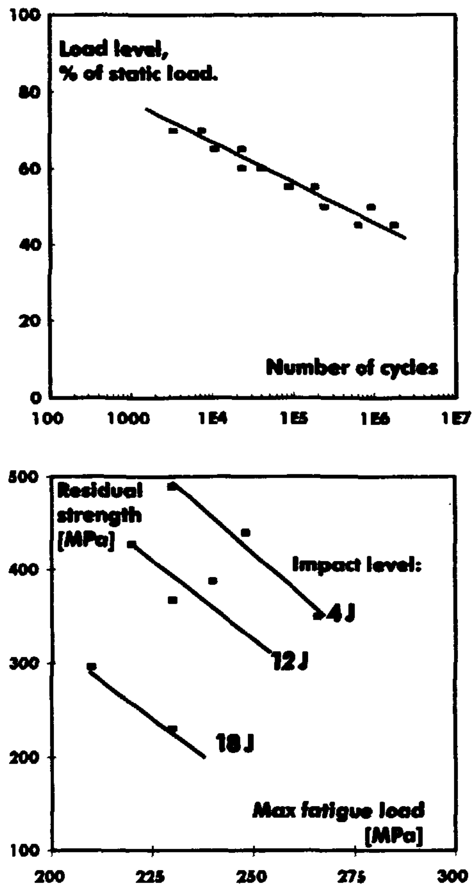


Anti-buckling device and gripping system for compression testing of large, thin laminate panels.

Compression loading is the critical loading when damage is involved, and as the test panels need to be quite large (200 x 200 mm), the panels are not structurally stable and will buckle during testing unless supported to avoid out-of plane deformation. An anti-buckling device was therefore designed for testing of panels of thickness 1-5 mm and for panel widths up to 200 mm. The design provides an accurate alignment of the mounted specimen while still allowing enough flexibility in order to be able to attach an array of strain gauges to the specimen.

Laminates with impact damage have been fatigue tested (compression-compression) until failure or until 10[illegible] cycles has been reached. The residual compressive strength has been measured for the panels surviving the fatigue test. Three different levels of impact damage were considered, ranging from barely visible damage to partial penetration. The residual compressive strength after fatigue loading was measured and it was seen that even after fatigue, the panels still maintain a highly acceptable load carrying capacity. This type of diagram can be used for the evaluation of a damaged structure and for reaching a decision on whether to repair or replace the damaged part.

Different repair methods for laminates are investigated in the programme, and only methods suitable for in-situ or in-the-field are considered. The method considered most reliable, because it initially provides the highest compressive strengths, is known as the Bonded External Pre-cured Patch (BEPP). The fatigue behaviour of laminates using this method have been investigated at Risø. The fatigue diagrams can be used to determine the maximum safe loads after repair of a component. The sandwich panels investigated in the programme consist of thin carbon/epoxy skin layers on a honeycomb core. Impacted specimens are tested in static compression after impact damage at three different impact levels: 3, 10 and 30 Joule. This corresponds to approximately circular damage areas with diameters of 25, 31 and 36 mm, the damage being barely visible, easily visible indentation and penetration of one skin layer, respectively. The static failure load for two different skin materials shows that the relative reduction in failure load is largest for the initially strongest panels, but that the strength for identical damage is still highest for the strongest material.



Fatigue diagram for repaired laminate panel (top), and residual strength after fatigue loading of impact damaged material (bottom).

3.3 Reinforced ceramic matrix composites

Ceramic matrix composites (CMCs) containing ceramic reinforcements can offer improved mechanical properties, especially fracture toughness, in comparison to the monolithic ceramic material. The reinforcing agents can be continuous fibres, single crystal whiskers, platelets or particles. Several toughening mechanisms have been identified, such as crack deflection, crack bridging, reinforcement pull-out and transformation toughening. The research carried out under this topic has been within the Centre for the Advanced Technical Ceramics within a project on processing science and technology. The overall project goal has been to produce oxide based ceramic materials with optimal mechanical properties (fracture toughness and strength), by incorporating dispersed ceramic reinforcements.

Various systems had been studied earlier using particles, or whiskers. Recent research has focused on ceramic platelets as reinforcement. Two such composite systems have been produced via the wet processing route, namely, alumina matrix - titanium boride, and yttria stabilized zirconia matrix - alumina. Pressure filtration technique has been developed, by which a green-body can be formed from the suspension state while part of the water is driven out. By this technique, the re-agglomeration of the ceramic powder or platelets during normal drying could be avoided, and the platelets could be given a certain degree of alignment.

Alumina-titanium boride

Alumina powder and titanium boride starting materials were co-dispersed in water with additives, a surfactant (ammonium carboxylate, Togosei A6114), and polyvinylpyrolidone. After ultrasonication and ball milling, the suspension was pressure filtrated. The filtered compacts were dried and cold isostatic pressed before they were sintered in a controlled atmosphere. Four-point bending was performed to measure the flexural strength. Indentation and four-point bend chevron method were used to estimate the fracture toughness. The effect of experimental parameters (including the platelet loading) on the sintered relative density was studied. The maximum flexural strength was 240 MPa, and the K_{IC} value was 4 $MPam^{1/2}$ for materials containing 5 wt% titanium boride platelets, having a relative density of ~95 %.

Zirconia-alumina

Research on this system has established a dispersive technique and sintering schedule for producing optimum mechanical properties. The effect of various parameters during pressure filtration on the platelet orientation was determined by SEM and neutron diffraction. A unique type of platelet orientation was found, herein termed 'eddy' texturing, which was affected mainly by the viscosity, and solid loading in the slurry. Strong platelet alignment was present parallel to all surfaces adjacent to walls in the filtration die. As a result of the microstructure, the composites exhibited an anisotropic K_{IC} behaviour which was dependent on the relative degree of alignment. A maximum value of 8.8 $MPam^{1/2}$ (versus 6.1 $MPam^{1/2}$ for the zirconia (TZ3Y) matrix) was measured by Vickers indentation on a surface sectioned from the centre of a 10 wt% platelet composite. The wear properties were determined by a micro-wear test which had been developed in another project within the Centre. The wear indices showed a small decrease for the composite in comparison to the matrix (2.4 mgs^{-1} versus 2.6 mgs^{-1}).

In-situ synthesis

Preliminary experiments have been carried out to investigate the possibility of producing Al_2O_3 - SiC composites by an in-situ process using chemical reactions between Al and SiO_2 in the presence of carbon. Promising results have been obtained and research is planned to study the reaction mechanisms, and to control the morphology of the SiC phase.

3.4 Mechanical properties of fusion materials

Effect of neutron irradiation on a molybdenum alloy

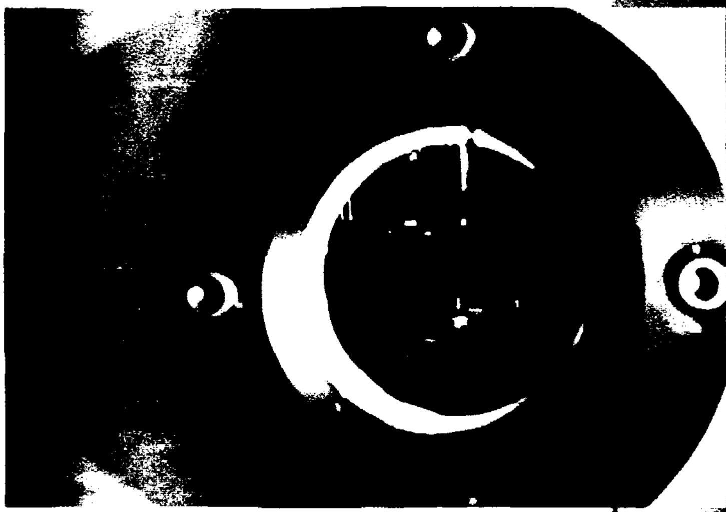
As a part of the European Fusion Technology programme, the effect of neutron irradiation on microstructural and mechanical property changes in two molybdenum alloys (TZM and Mo-5% Re) are being investigated. Tensile specimens of these alloys have been irradiated in DR-3 to a dose level of ~0.2 dpa (displacement per atom) at ~50, 100, 250, 350 and 450°C. A number of fatigue specimens of these alloys have been also irradiated to a dose level of ~0.2 dpa at ~50 and 450°C.

Microstructures of TZM and Mo-5% Re alloys irradiated (but undeformed) at ~50, 100, 250 and 350°C were investigated by transmission electron microscopy (TEM). For comparison, microstructures of the corresponding unirradiated specimens were also investigated. The dislocation line density in all specimens was found to be very low. The microstructure of irradiated specimens was dominated by a high density (0.4-1 x 10^{23} m^{-3}) of small (~3 nm diameter) loops. Clear indications of loop rafting were observed in TZM specimens irradiated at ~50°C. TZM specimens irradiated at 350°C were found to contain a high density (~4 x 10^{23} m^{-3}) of small (~0.75 nm diameter) voids.

Specimens of TZM and Mo-5% Re alloys were tensile tested at ~22 and 100°C both in the irradiated and unirradiated conditions. The results demonstrated that irradiation to a dose level of only ~0.2 dpa increases the strength of both alloys by a factor of almost two and reduces the ductility of these materials to practically zero. Fracture surfaces of TZM and Mo-5% Re specimens tensile tested at ~50 and 100°C in the irradiated and unirradiated conditions were examined by scanning electron microscopy. In most cases the fracture surface showed intergranular cleavage.

The main source of the irradiation-induced embrittlement appears to be the exhaustion of plastic deformability of the grains which contain a high density of irradiation induced dislocation loops. Deformation at grain boundaries occurs by sliding which cannot be accommodated by deformation in the irradiation hardened grains, giving rise to transgranular cleavage.

Tensile testing and HTLCF testing (high temperature, low-cycle fatigue) of irradiated specimens of copper and copper alloys for application in fusion reactor systems.



Fatigue behaviour of copper and CuAl25 specimens of two sizes

(in collaboration with University of Illinois Urbane, U.S.A. and Pacific Northwest Laboratory (Richland), U.S.A.)

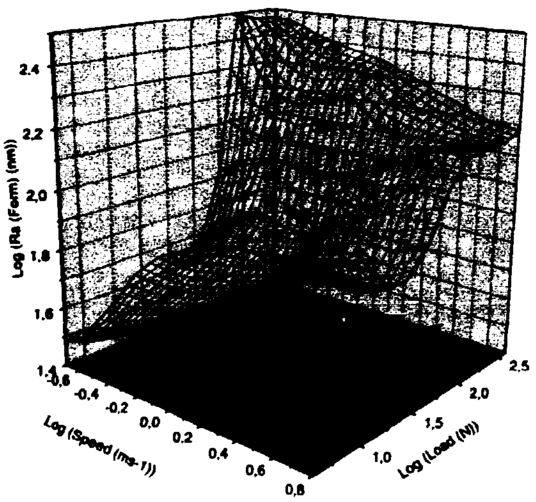
Copper and its alloys are attractive for application in fusion reactor systems for heat flux components because of their excellent thermal conductivity. The thermal and mechanical loading of such components will be cyclic in nature thus requiring an understanding of their fatigue behaviour. Oxygen-free high conductivity (OFHC) copper and CuAl25, an oxide dispersion-strengthened copper alloy, were fatigue tested under conditions of constant total strain amplitude at room temperature. Two specimen sizes were used for both materials in order to establish the effect of specimen size on fatigue response. ASTM standard size fatigue specimens with 6.35 mm gauge diameter were compared with subsize specimens of 3.1 mm gauge diameter. The subsize specimen has been developed at Risø for irradiation in DR-3 and EBR-II.

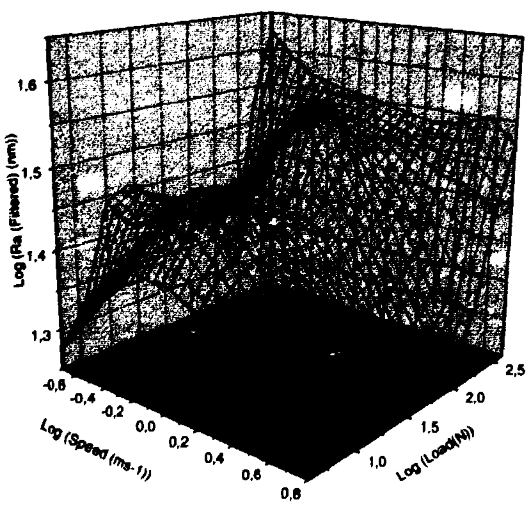
Tests were performed in strain control, fully reversed mode, with a frequency of 1 Hz. Strain ranges from 0.20 to 1.2% were used for CuAl25 and from 0.10 to 0.35% for OFHC copper. For both specimen sizes, the dispersion strengthened copper alloy shows superior fatigue performance compared to that of OFHC copper. Specimen size seems to have an influence on the failure lives of both materials. Fatigue life comparisons for both materials show that the larger (i.e. ASTM standard size) specimens have a longer lifetime at equivalent strain ranges than the subsize specimens.

3.5 Wear mapping of ceramics

Wear maps are a useful tool for showing how the incidence of wear mechanisms in ceramics and other materials varies with test load and speed. However they do not provide quantitative information about mechanisms studied using the roughness of worn surfaces as a parameter.

The examples shown in the figure are surface roughness maps for a tungsten carbide pin worn against a silicon carbide disc. These 3-dimensional representations show the variation of the average Ra of worn surfaces with both test load and speed. Surfaces produced in wear tests often have both a deformational and microwear component. The upper figure shows the data containing both these components. The lower graph shows the Ra with the deformation component removed. For surfaces of tungsten carbide, with only one wear mechanism operating, an excellent correlation has been found between the filtered profile and the wear rate. Multi-mechanism surfaces produce strong variations in roughness, as can be seen in the centre of the lower figure. This quantitative data is being used to compare wear map predictions with the wear found in industrial components.





Surface roughness maps for a tungsten carbide pin worn against a silicon carbide disc.

3.6 Residual stress determination

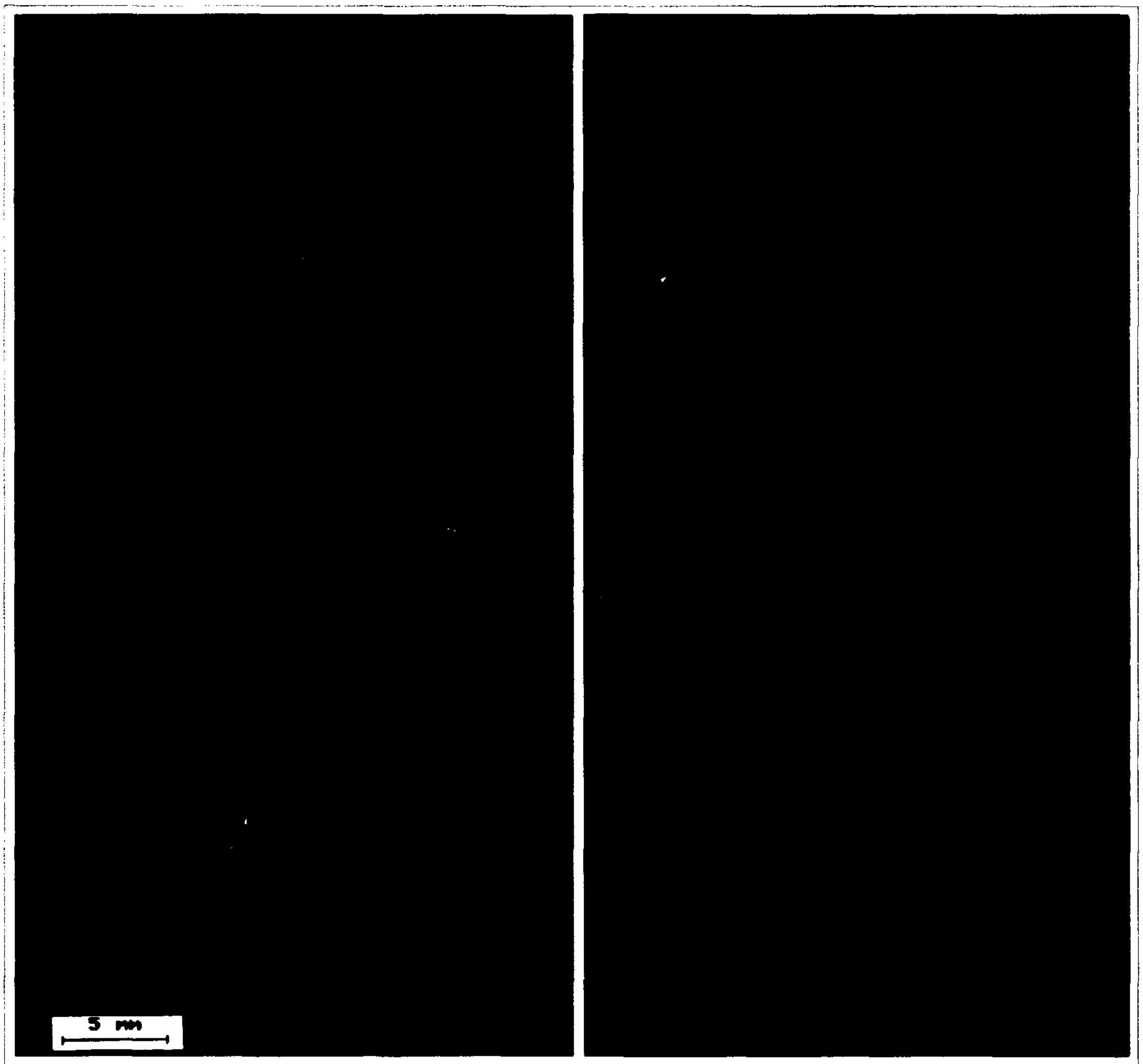
The reason for using the neutron diffraction technique for internal stress studies in Materials Engineering applications results from the high penetration power of neutrons. The diffraction technique can hence be used as a non-destructive probe for internal stresses inside engineering components - so 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 in weldments of offshore structures. The measurements are part of a PhD project focusing on fatigue predictions for offshore rigs. Differences in fatigue life under varying loading spectra are being investigated. As expected, it is found that residual stress levels are of great significance. Neutron diffraction measurements have shown that the residual stresses may be as high as 60 to 70% of the flow stress. These values are being used in theoretical predictions of fatigue lifetimes for comparison with the experimental observations.

3.7 Ultrasonic scanning of moulded ceramics

During 1993, a fast and accurate ultrasonic scanning system was used to characterize and test different advanced materials such as injection moulded ceramics, zirconia ceramics, carbon reinforced fibre composites (both as laminates and tubes) and fuel elements. In the following, work on injection moulded ceramics is used to illustrate the possibilities in ultrasonic scanning in finding defects and characterizing the homogeneity of the specimens. The scans were performed on the ceramic in the 'green' state and in the final sintered state. The specimens were made as part of a BRITE/EURAM project.

A large number of plates with different polymer binder contents and compositions have been examined using the pulse-echo technique combined with 'double through-transmission'. In the pulse-echo technique, echoes from defects such as pores and cracks within the material are measured. In double through transmission, the attenuation of the ultrasound is measured. This is done by measuring the echo reflected from the back



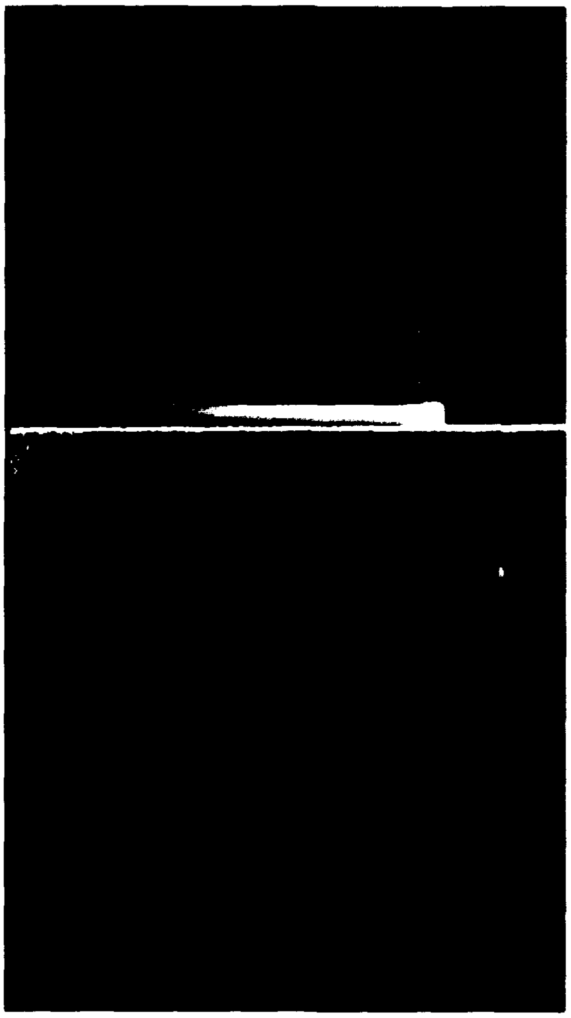
Ultrasonic scan of an injection moulded ceramic. The plate measures 80 mm x 80 mm and is 4 mm thick; scanning area, 50 mm x 32 mm with a resolution of 0.08 mm. The pulse-echo measurement in the right picture gives no indication of the two cracks which are clearly seen by double through-transmission in the left picture. On the other hand, some small defects are seen in the right picture which are not visible in the scan shown to the left. The two techniques are complementary to each other.

surface. The height of this back echo is dependent on the attenuation in the material and therefore also dependent on defects and the homogeneity of the material.

Measuring the echoes from the defects gives the best detectability of single defects. The back echo is normally also reduced but it depends on the type of defects. The figure shows that cracks perpendicular to the surface (in the same direction as the ultrasonic beam) can only be found by the double through transmission and not by the pulse-echo technique. This type of crack gives no reflected echo but the back echo is reduced. This plate was also examined after sintering and the cracks observed in the green state could still be found together with other defects arising from the sintering process.

3.8 Testing of fuel cell stacks

As part of the national Danish programme on Solid Oxide Fuel Cells (sections 4.2 and 4.3) a prototype test facility and test procedures have been established for assembling and performance analysis of small fuel cell stacks at temperatures up to 1250°C. The test facility, based upon a custom-built high temperature chamber furnace, includes instrumentation for changes in mechanical load on top of the stack



SOFC stack with 3 cells and 4 interconnect plates after assembling of stack components by sintering. Glass sealings and manifolds have not yet been mounted.

at high temperature, for electrical power output surveillance, for temperature and gas flow control and for continuous monitoring of stack integrity by acoustic emission.

Three small SOFC stacks were tested in 1993 at 1000°C over a maximum time span of 400h. The best result obtained was an internal stack resistance better than 1.7 $\Omega \cdot cm^2$/cell in a 3-cell stack. Although this value is as good as that of a grand average of results reported internationally from other SOFC groups, it still has to be improved to achieve commercially viable stacks. Detailed post-mortem analyses by scanning electron microscopy of each of the stacks were used to optimize material compositions for individual stack components and to improve stack assembling procedures.

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Materials Technology - synthesis and processing

4

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 Materials 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 pressure autoclave, and equipment for resin transfer moulding.

Three projects on manufacturing technology for thermoplastic composites with continuous fibres are being conducted: (i) filament winding, (ii) fibre pre-forms (fibre preforms are a pre-shaped fibre structure which has not yet been consolidated to the final component). Fibre pre-forms can be woven, knitted, braided or stitched together, (iii) film-stacking.

In the filament winding project, a new hot chamber filament winding technology has been developed and investigated. Basically, the technique consists of winding the materials (fibres and matrix) onto a mandrel at the process temperature. The incoming tape is preheated with hot air, and by applying a tensile stress to the tape, the consolidation is enhanced. The mandrel and the already-wound material are kept at the process temperature until the entire specimen has been wound. Then the mandrel and wound component are cooled, and the chamber

Hot chamber filament winding in which pre-heated tapes of fibres and matrix are wound onto a mandrel at the process temperature.

opened. The advantage of this filament winding technique is the potential of high winding speed. The disadvantage is that the technique will be limited to geodesic filament winding. The technique has been developed for hoop-wound tubes of carbon-fibre/PEEK material (APC-2 from ICI) and the most important process parameters (chamber temperature, preheat temperature and fibre tension) have been investigated.

Hot chamber filament winding has demonstrated that carbon-fibre/PEEK laminates of high quality can be fabricated. The best results are obtained with a chamber temperature of 345°C, a preheat temperature of 365°C, and a fibre tension of 64 MPa. Specimens produced using these optimized parameters have a porosity content of 0.3-1.1 vol%, an interlaminar shear strength of 93-97 MPa, internal stresses of 76-109 MPa, and acceptable matrix squeeze out.

In the pre-form project a new hybrid yarn of glass fibres and thermoplastic polyester (PET) fibres has been developed in collaboration with A/S Kaj Neckelmann, Silkeborg, Denmark. Glass fibres and PET are mingled together in a texturizing process to form a hybrid yarn. Fabrics are woven from the hybrid yarn, and laminates are fabricated by an autoclave consolidation of plies of the woven fabric. The technology is still under development, but very promising material properties have already been achieved (interlaminar shear strength above 70 MPa, and porosity content less than 1 vol%).

This technique has been developed in the film-stacking project and used to fabricate test panels of glass fibre/PP, aramid fibre/PET, and aramid fibre/PP. The technique consists simply of stacking layers of woven fibre fabrics and thermoplastic films of the matrix materials, followed by consolidating the stack into a laminate in a subsequent autoclave process. The laminates have been produced in thicknesses up to 32 mm and with three levels of controlled porosity content (2, 12 and 18 vol%). The different porosity content is achieved by varying the film thickness and the autoclave pressure.

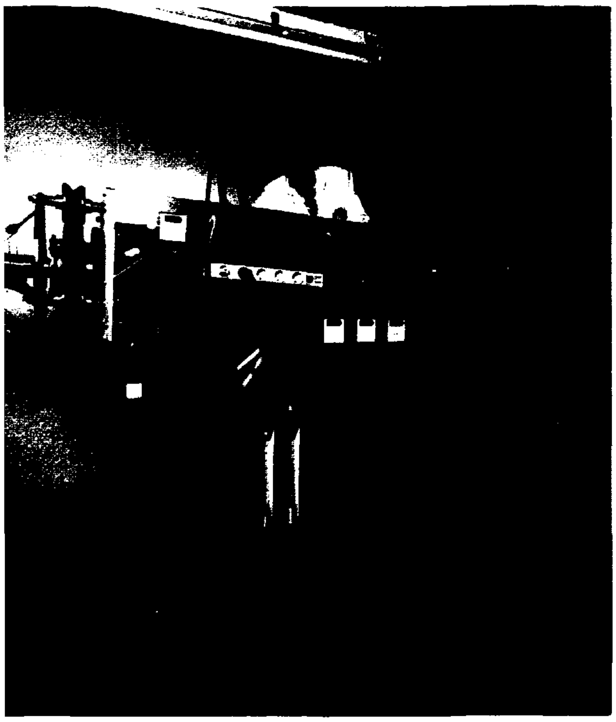
In another project on filament winding, a computer program (CADPATH) for design of filament wound structures has been further developed. CADPATH is an integrated computer tool that automates the process of designing, analyzing and manufacturing filament wound structures. For geodesic winding on surfaces of revolution, CADPATH calculates winding pattern, fibre path and local wall thickness for a selected fibre angle. Alternatively, CADPATH can determine fibre angles that will give uniform coverage of the structure. Strength analyses can be performed using classical laminate theory or finite element analysis. Finally, CADPATH can calculate the control data necessary for manufacturing the structure on a numerically controlled winding machine. The program integrates the CAD, CAE and CAM of filament winding. CADPATH is written in Fortran and utilizes the portable user-interface system Interacted. The CADPATH source code is available for user modification. The program was developed in collaboration with Alfa Laval Separation A/S.

Finally, our filament winding technique for the fabrication of high quality flat fatigue test specimens of angle-plied fibre reinforced polyester has been further developed. It is now possible to produce test specimens of hybrid laminates with a mixture of glass fibres and carbon fibres. The flat angle-ply laminates are produced by winding wetted fibres onto a steel plate mandrel. A symmetric laminate is laid down on both sides of the steel plate mandrel during one winding operation. After completion of the winding operation, the steel plate-mandrel with the wet laminates is pressed between two additional steel plates to obtain flat angle-ply laminates with uniform and pre-determined thicknesses.

Filament winding of flat, fatigue test specimens. Production of hybrid laminate specimens involves winding with seperate cords of glass fibres (white) and carbon fibres (black). The fibres are wetted with epoxy as they are wound onto the steel plate mandrel.

4.2 Building solid oxide fuel cells (SOFC)

Having established techniques for production of single solid oxide fuel cells with internal resistances in the range 0.3-0.4 $\Omega \cdot cm^2$, the national Danish SOFC programme has now moved emphasis to problems encountered with stacking of cells with ceramic bipolar plates and to problems anticipated with future operation of planar stacks.



Rotary tube furnace for the pyrolysis of perovskite precursors for electro-catalysts. Ceramic processing is a major part of the SOFC programme.

Major problems concerned with planar stacking are:

(i) establishment of low impedance electrical couplings between cell and neighbouring bipolar plates,

(ii) dimensional instability of bipolar plates during stack manufacture and during initial operation,

(iii) establishment of electrode seals and manifold seals which allow operation at elevated differential pressure,

(iv) the problem of matching the thermal expansion coefficients (TECs) well enough.

As many ceramics are electrical insulators, a large range of materials are available for the manifolds. A cheap spinel-based material has been tailored to match the TEC of the electrolyte. Material to be used for interconnect plates, however, needs good electric conductivity together with the environmental stability requirements. This limits the number of candidate materials severely. The main work performed on this subject was the optimization of properties of $LaCrO_3$ based interconnects by doping both on La- and Cr-sites. Also, a search for a suitable metallic interconnect material was initiated towards the end of 1993.

4.3 Sealing of SOFC stacks

Development of sealing materials is a significant materials development task in the national Danish SOFC programme. The current SOFC stack design requires seals to prevent mixing of fuel gas with air. Mixing will decrease the net electrical output of the stack due to fuel loss by combustion and will introduce unstable electrode environments with respect to temperature and oxygen pressure, thereby causing fast deterioration of the electrodes. The sealing glasses must possess a high chemical stability at SOFC working temperature (presently 1000°C) in a wide range of oxygen partial pressures, frequently in the presence of steam. In addition, the glass needs to be stable with respect to other SOFC stack components. The thermal expansion coefficient must match that of other stack components closely (approximately $105 \times 10^{-7} K^{-1}$) and volume changes caused by phase transitions in crystalline phases are unacceptable. For sealings of manifolds, high viscosity is desirable to maintain physical stability.

Work is now concentrated on phosphate and silica as glass formers. Borate was previously included as a glass former, but compositions based upon borate could not meet the chemical stability requirements. Glass properties are systematically optimized by addition of a variety of modifying and intermediate oxides. Key properties currently being optimized are stability in hydrogen and the tendency to crystallization. Manufactured glasses are initially characterized by;

(i) glass transition point (T_g), crystallization point (T_d), melting point (T_m) and TEC determined by differential thermal analysis (DTA) and dilatometry,

(ii) high temperature (1000°C) stability in relevant atmospheres with respect to other SOFC component materials – determined by contact to such components at high temperature in controlled atmospheres. Weight changes are recorded and samples are examined by optical and electron microscopy including element analysis,

(iii) influence upon electrochemical electrode performance determined by impedance spectroscopy of electrodes exposed to the glasses,

(iv) crystalline phases determined by x-ray diffraction.

This micrograph was obtained in the environmental SEM, during in-situ melting and solidification experiments. Surface crystallization is observed in a phosphate-based glass in which the growth morphology is spherulitic. When the growing spherulites meet on the glass surface, growth proceeds into the bulk material.

A substantial amount of work has been carried out to increase the PO_4-unit stability i phosphate glass structures by adding components which increase internal binding forces. Nevertheless, it has proven difficult to develop phosphate-based glasses that have a sufficiently high stability in hydrogen and at the same time do not crystallize. The main reason for the instability and the crystallization tendency is vaporization of phosphate from the glass structure, which causes surface nucleated crystallization. The actual crystalline phases are unstable in hydrogen.

As an alternative, glass compositions may be moved into regions giving stable crystalline phases with the required physical properties. This may be achieved through widening of the glass forming region by addition of glass forming oxides and metal oxides (intermediates). Experiments with addition of nucleating agents for such stable crystalline phases (glass-ceramic seals) are included in the present work.

4.4 Manufacture of MMC's

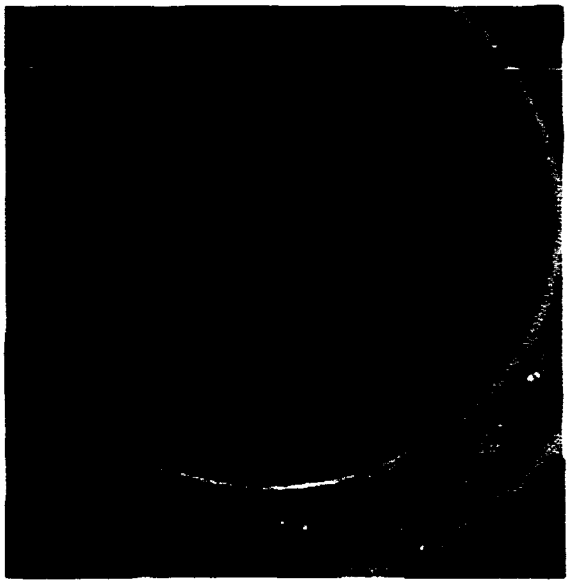
Aluminium composites manufactured by mixing aluminium powder and ceramic fibres/particles normally contain a small volume fraction (~1%) of alumina particles originating from the surface oxide layer of the aluminium powder. The presence of the small oxide particles has a significant effect on the thermomechanical behaviour of these composites. In order to isolate the effect of ceramic fibres/particles on deformation and thermal treatment, composites with a pure aluminium matrix are required.

The aluminium powder has been produced from an aluminium ingot of 99.999% purity, using the high-pressure gas atomizer. Special equipment and tools have been developed so that the transportation, storage of the powder and the powder metallurgy process is carried out in a controlled atmosphere. The oxidation of the powder during handling has been limited to a minimum level. Extruded rods of Al-SiC composite containing about 0.08 wt% alumina have been manufactured. As a model material, this type of composite has been used to study the effect of SiC whiskers on the dislocation structure during thermal cycling, and on the microstructure and texture development during cold-rolling and annealing.

4.5 Fine and ultra-fine powder metallurgy

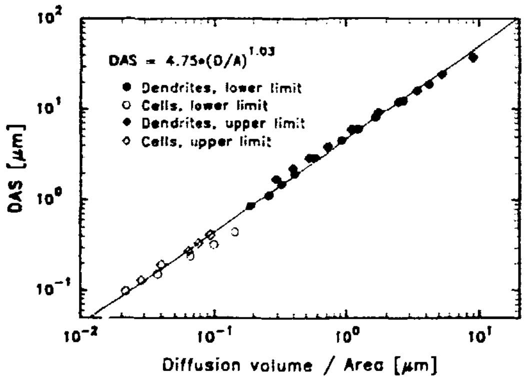
Atomization

Studies of the microstructure in atomized Cu6%Sn were continued. The dendrite arm spacing in particles of different sizes ranging from approximately 10 to 125 microns was determined by measuring concentration variations of Sn over cross-sections of particles and a linear relation between mean spacing and particle size was found. A solidification



The Risø gas-atomizer uses induction melting in an inert and clean atmosphere.

model developed at Oxford University, UK, was used to describe the relation between solidification parameters and microstructure. The model was extended to three dimensions,



Dendrite arm spacing (DAS) plotted against solidification time for Cu6%Sn samples.

covering the full length of the solidification zone. The modified model predicts a linear relation between dendrite arm spacing and diffusion volume/area of the solid-liquid interface as shown in the figure. The linear dependence is identical for cells and dendrites, indicating that the formation of cells and dendrites is controlled by the same diffusion mechanism. Other results obtained by the model compare very well with experiments.

Much effort has been put into the production of iron-based alloys in the Risø atomizer during 1993. The jump from the lower melting point alloys, like Cu-Sn in the processing temperature range 1000-1200°C, to the iron-based alloys with processing temperatures in the range 1400-1650°C involved a number of difficulties with respect to melting techniques, refractories and design of melt-valve and outlet. These difficulties have been overcome and a reliable high temperature melting system is now available.

A series of stainless steel powders (316L) was produced with He, Ar, and N_2 as the atomization gas. The obtained particle size distributions were wide and had a larger median size than seen for Cu-Sn alloys, probably due to a different surface tension. Atomization with He resulted in smaller median sizes than Ar and N_2 (199 micron as compared to 225 micron).

In collaboration with the Technical University of Denmark a rapidly solidified sintering additive to facilitate liquid phase sintering of 316 stainless steel was produced. The additive was admixed in small quantities (about 1%) and caused a transient liquid phase formation during sintering. The corrosion properties can be improved by optimization of the additive composition.

Ultra-fine powders

(in collaboration with the Institute of Physics, the Technical University of Denmark)

While studying ultra-fine, passivated structures of Fe-Ni, Fe-Cr and pure Ni by microgravimetry it was observed that the Fe-Ni samples are partially reduced, and the Ni samples completely reduced by only moderate heating in inert gas. These observations have been quantified during 1993. Pure Ni samples were passivated by controlled oxidation in air and examined by electron microscopy and x-ray diffraction. The phases were metallic Ni and NiO and the particle size was approximately 10 nm.

4.6 Materials for incineration of biomass and waste

Biomass and municipal solid waste (MSW) have become a well-established source of energy, utilized for heat and power production through incineration. However, the efficiency of electricity production by incineration plants is still rather low, as problems are experienced regarding high temperature corrosion of the superheater, when superheating steam for electric power production. To overcome these problems, a programme has been launched to study the corrosion mechanisms in incineration plants, including high temperature materials for power production plants which use biomass as fuel. In this programme, materials will be tested both under laboratory conditions, where synthetic flue gasses will be the media, and in operating plants, where the actual environment is created. The corrosion mechanisms will be established, and the rate of corrosion modelled on the basis of both laboratory and field tests, in order to estimate lifetimes of the materials. The materials to be tested will be both commercially available alloys and newly developed materials with improved resistance to high temperature corrosion.

A range of materials has been tested in different plants (biomass-fired and MSW-fired) at temperatures between 475°C and 575°C using cooled probes. The preliminary results show a high rate of corrosion in the initial state, up to 2 mm/1000 h for the least resistant materials. The effect of different alloying elements on the corrosion mechanisms and rate, is being examined. Future work will include exposure for prolonged periods of time, in order to provide estimates of critical component lifetimes.

4.7 Manufacture of LEU fuel elements

The fabrication of low enriched uranium (LEU) fuel elements was continued in 1993, and a total of more than 230 LEU elements have now been fabricated in the Materials Department. In all, approximately 140 LEU elements have been irradiated in DR-3 since the changeover from highly enriched uranium (HEU) fuel elements started in 1988. All LEU elements have performed successfully.

The effort to further improve the ultrasonic scanning method, which was developed in order to provide automatic measurement of cladding thicknesses on LEU fuel plates, was continued with promising results.

Also, the marketing effort to sell LEU fuel elements to research reactor customers outside Denmark was continued.

4.8 Gelcasting – a new ceramic forming method

In the Danish Centre of Advanced Ceramics the development of a new casting technique has been progressing. Gelcasting is a near-net-shape technique based on a synthesis of ideas from both traditional ceramic processing and polymer chemistry and is investigated with the intention of fabricating complex, thick walled ceramics.

Before casting, a slurry is prepared. The slurry consists of a dense suspension of ceramic powders in a solvent of water and organic monomer. After the addition of catalyst and initiator, the slurry is poured into the mould where it polymerizes. The organic monomers form a macromolecular network that holds the ceramic particles together in the desired shape. After a short period of time (5-10 min) the 'green' body can be removed from the mould. The dried shapes have a relatively high strength and can be machined in the green state. Because of the special forming technique, the system only requires the addition of 4 wt% of binder which facilitates easy binder burnout.

At the current stage of development of the technique, gelcasting of thinner objects (plates, bars etc.) can be accomplished without problems. However, since one of the main attractions of the process is the ability to cast thick, complex shapes, the hip joint ball was chosen as a suitable component to evaluate the method. In this type of specimen, the thicker sections of the part gave rise to drying problems. These problems were partly solved by employing coarser powders, and a number of apparently crack-free balls were cast.

Just recently, a controlled humidity chamber has been acquired, in which it is possible to control the drying conditions more precisely. Initial controlled drying experiments have provided encouraging results. Although only a limited number of experiments has been performed so far, a remarkable reduction in the percentage of rejected components has been obtained.

4.9 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 in the Materials Department are

A new method for forming ceramics is gelcasting in which machinable 'green' bodies are formed by polymerization of an organic binder in the mould. Subsequent sintering of the ceramic burns out the binder. The photo shows cast hip joints.

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 BRITE EURAM programme and through direct industrial contracts.

Brazing of stainless steels and superalloys

The work on a new, 45 month, BRITE/EURAM programme on the development of brazed novel plate heat exchangers was started this year together with partners from Denmark, Sweden and Germany. The main Risø tasks in this project are to develop test specimens and test methods to establish the influence of various phases found in the filler metal on mechanical properties and corrosion resistance of the brazed joints. The investigations include a variety of base metals in combination with both commercial and experimental filler metals.

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 project was completed during this year, and the results and experience from the experimental work has been transferred to the industrial partners.

The equipment for diffusion bonding has been utilized for producing large bonded test specimens for fatigue testing by another partner in the project. The diffusion bonding of the joints was carried out at the optimized parameters established earlier in the project. Although good tensile properties were found for the bonded parts, fatigue testing results had a very large scatter. Such scatter suggests that voids and porosities at the interface of the joints could well be regions of crack initiation even though a good tensile strength was found.

The brazing process was tested under industrial conditions by brazing P/M-parts in sintering furnaces at the P/M-manufacturers. Variations in geometry, brazing filler metal and brazing cycle were repeated from laboratory work made earlier in vacuum furnaces. The protective atmospheres in the industrial furnaces (one furnace with cracked ammonia and one furnace with hydrogen) were however, found not to be dry enough to completely reproduce the laboratory results obtained with the vacuum furnace. This is due to partial oxidation of the filler metals during the brazing cycle. It was shown that brazing with NiCrSi- and NiMnSi based filler metals requires a dew point below -60°C. This is considerably lower than normally used for sintering iron-based P/M-parts, but in the region of that for sintering stainless steels.

Joining of ceramics

In the Danish Centre for Advanced Technical Ceramics, the joining programme was continued on brazing and solid state diffusion bonding. Having finished the investigations of the influence on the four-point bend strength for brazed joints in alumina and zirconia from various brazing parameters, new investigations were carried out on the mixed oxide ceramic TZ320A. An active AgCuTi filler metal was used and four-point bend strengths found to be vary from 260 MPa, using a 0.15 brazed gap, to 375 MPa for a 0.05 mm gap. Only a minor influence of brazing temperature varied between 850 and 950°C was observed.

Finally, an ion beam thinning, cross-sectioning technique has been developed to examine and analyze metal-ceramic brazed joints in the analytical TEM. The very narrow zone (about 10 nm wide) in which the TiO reaction layer on partially stabilized zirconia is wetted by the active AgCuTi-based filler metal has been characterized. A considerable drop in yttria concentration of the yttria-doped zirconia was observed in a very narrow zone very close to the TiO reaction layer.

Contract work

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. The growing interest shown by Danish industry for joining ceramics to metals has resulted in major development activities using the active solder alloys as well as an active high temperature filler metal.

[illegible]

Finances 1993

5

The activities of the Department are supported by a combination of direct government funding, focused project funds from national, EU and international programmes and fully commercial 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 149). Inflation from 1992 to 1993 was negligible (~1.5%); the Danish kroner was weakened with respect to the US dollar (minus 8.5%) over this period.

The Materials Department continues to grow, with a total increase in income of 14% in 1993 compared to 1992. Reductions in direct government funding, which had been the norm over the five year period up to 1991/92, are no longer significant. The apparent cut in basic funding to the Materials Department of approx. 8% compared to 1992 is a direct consequence of the transfer of the Polymer group (4 scientists and 3 technical staff) to the Solid State Physics Department.

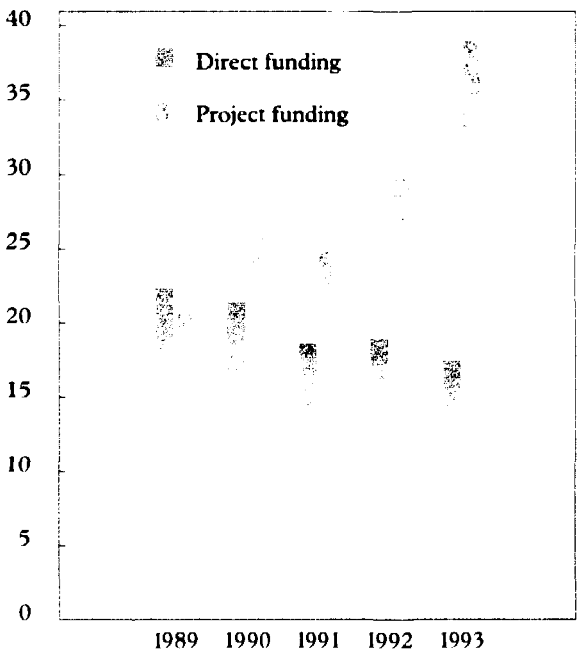
In 1993, project activities were again increased, with a growth in project funding of 28% compared to 1992. This is primarily due to the formation of the 'Engineering Science Centre for Structural Characterization and Modelling of Materials', which receives DKK 7.5 million per annum from the Danish Council for Scientific Research. There was also a sizable increase in the solid oxide fuel cell (SOFC) programme. Altogether, almost 70% of the total income to the Materials Department now comes from sources other than direct government grant.

Expenditure on salaries increased by a mere 4% in 1993, reflecting the policy of controlled growth. The number of academic and technical staff has increased only conservatively over the past five years. At the same time, the number of PhD students, post docs and guest scientists has more than doubled.

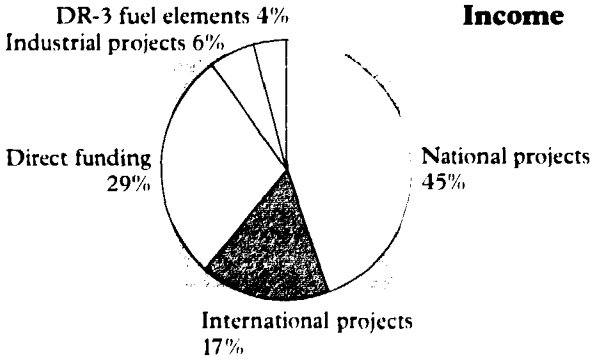
Income

	DKK 1000	USD 1000
Direct funding (Ministry of Energy)	17 500	2 600
Project funding	39 000	5 800
	56 500	8 400

Income



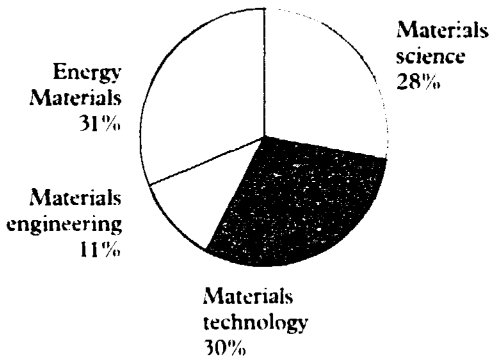
Income



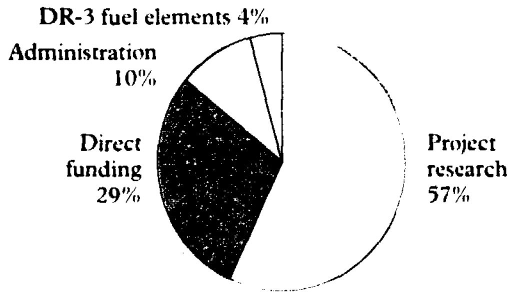
Expenditure

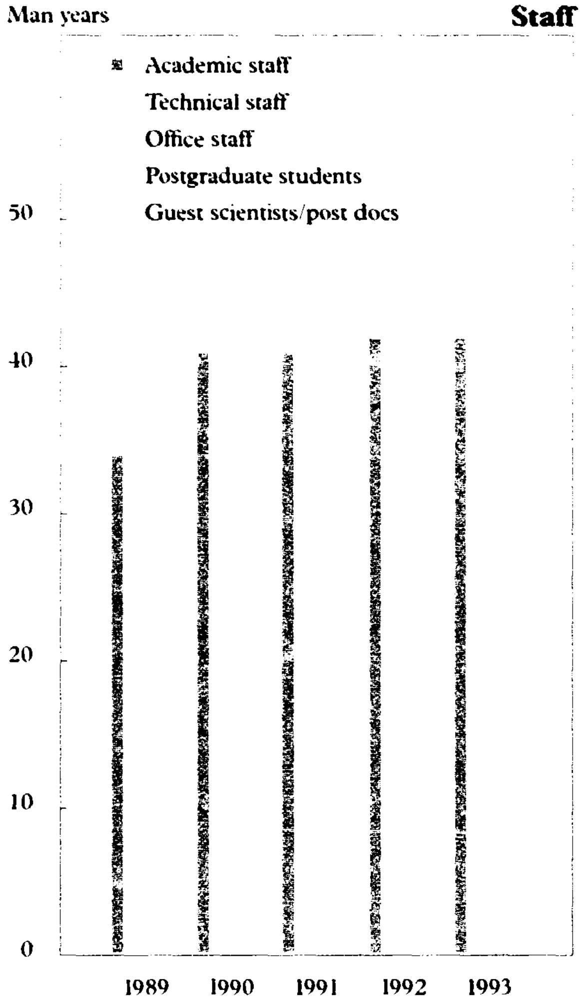
	DKK 1000	USD 1000
Salaries	30 000	4 460
Operating expenses	12 000	1 790
Equipment	5 900	880
Administrative charge	5 300	790
Total	53 200	7 920

Research areas



Manpower





Staff of the Department

6

In 1993, 23 staff members left and 22 new members joined (*) the Department.

0) Transferred to the Department of Solid State Physics April 1.

Head of Department
Niels Hansen

Scientific staff
Adolph, Eivind
Almdal, Kristoffer 0)
Andersen, Svend Ib
Bagger, Carsten
Bentzen, Janet J.
Berg, Rolf 0)
Bilde-Sørensen, Jørgen B.
Borring, Jan
Borum, Kaj K.
Brøndsted, Povl
Carlsen, Hans
Christensen, Jørgen
Debel, Christian P.
Eldrup, Morten
Gormsen, Steffen
Gotthjælp, Klaus
Gundtoft, Hans Erik
Hendriksen, Peter Vang *
Henneso, Erik until 31 Oct
Horsewell, Andy
Hvilsted, Søren 0)
Johansen, Bjørn S.
Juhl, Mette
Juul Jensen, Dorte
Kindl, Bruno
Kjølby, Sif *
Knudsen, Per
Larsen, Peter Halvor
Leffers, Torben
Lilholt, Hans
Linderoth, Søren
Lindegaard, Thomas until 31 May
Liu, Yi-Lin
Lorentzen, Torben
Lystrup, Aage S.
Løgstrup Andersen, Tom
Mogensen, Mogens B.
Pedersen, Allan Schrøder
Pedersen, Ole Bøcker
Pedersen, Walther Batsberg 0)
Poulsen, Finn Willy
Primdahl, Søren *
Rasmussen, Karen W.
Singh, Bachu N.
Sørensen, Ole Toft
Toft, Palle
Toftegaard, Helmuth L.
Østergård, Maria J.L.

Postgraduate students
Ahlgren, Erik O.
Bjerregård, Henrik *
Christoffersen, Henrik *
Krieger Lassen, Niels C.
Pickup, Chris*
Poulsen, Jes R.
Ranløv, Jens
Rasmussen, Torben Valdbjørn*
Tiedje, Niels
Winther, Lars 0)

Post docs
Carter, J. David
Clausen, Charlotte
Godfrey, Andy *
Johannesson, Birgir
Jørgensen, Ole until 30 Sep
Li, Wen-Yu
Liu, Qing *
Richter, Helmuth *
Rosen, Gady until 28 Feb
Sørensen, Bent F.
Sørensen, Niels Jakob

Guest scientists
Alcock, Jeffrey R.
Bonanos, Nicholas *
Cai, Shidong *
Dirnfeld, Shraga F. *
Jerdal, Lennart O. until 26 Nov
Jiang, Zhen-Jian until 26 Mar
Shen, Xian until 15 July
Teng, Y. Harry *
Yao, Jianlong until 28 Feb
Vulfson, Yurij

Consultants
Domanus, Josef C.
Nilsson, Tage M.
Waagepetersen, Gaston

Technical staff
Adrian, Frank
Andersen, Axel B.
Arnsberg, Carsten *
Aukdal, Jørgen A.
Bindner, Jørgen *
Borchsenius, Jens F.S.
Brejnholt, Niels until 31 Dec *
Bülow Christensen, Carl J.
Bønke Nielsen, Anne 0)
Christensen, Peter Mørch
Christensen, Svend E.
Dreves Nielsen, Poul
Eschricht, Lars *
Frederiksen, Henning
Gravesen, Niels Nørregaard
Hansen, Jørgen Lang
Hansen, Lotte 0)
Hansen, Niels J.
Hersbøll, Bent
Hubert, Lene 0)
Jensen, Finn E.
Jensen, Knud
Jensen, Palle V.
Jepsen, Ole Kjær until 30 Apr
Jespersen, John
Kjøller, John
Kjær, Anne-Mette Heie
Klitholm, Cliver
Larsen, Arne
Larsen, Bent
Larsen, Birgit N. *
Larsen, Jan
Larsen, Kjeld J.C.
Lindbo, Jørgen
Mikkelsen, Claus
Nielsen, Birgitte
Nielsen, Ove
Nielsen, Palle H.
Nielsen, Peter until 30 Apr
Nilsson, Helmer
Nørgaard, Jesper
Olesen, Preben B.
Olsen, Benny F.
Olsen, Henning
Olsen, Ole
Olsson, Jens O.
Paulsen, Henrik
Pedersen, Børge E.
Pedersen, Knud E.
Pedersen, Niels Jørgen
Petersen, Klaus Bo until 10 May *
Robl, Steen
Sandsted, Kjeld
Strauss, Torben R.
Sørensen, Erling
Aagesen, Sven

Office staff
Christensen, Helle Baastrup until 31 Mar
Dreves Nielsen, Elsa
Hoffmann Nielsen, Lis
Lauritsen, Grethe Wengel
Mortensen, Jytte *
Sindholt, Birgitte
Sørensen, Eva M.
Thomsen, Ann

Apprentices
Andersen, Morten
Forné, Anders
Foss, Heidi E. *
Hammer, Christian
Hansen, Martin until 31 Aug
Nielsen, Jens Cordt
Ploug, Martin
Wolfe, Thomas *
Ørgaard, Henrik

Departmental activities 7

7.1 Participation in committees

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

Danish committees

Technical assessors, DANAK (Dansk Akkreditering). Copenhagen.
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.
Janet J. Bentzen

Editorial Board of 'Keramisk Information'
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 and Jens Olsson (Staff Representatives)

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 the Engineering Science Centre at Risø for Structural Characterization and Modelling of Materials.
Niels Hansen, Dorte Juul Jensen and Torben Leffers

The Danish Ministry of Industry and Coordination, Reference Group for the BRITE EURAM programme. Copenhagen.
Niels Hansen and Ole Toft Sørensen

The Danish Electrical Research Institute (DELRI). 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 Bio-technology 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

Committee for the Danish Environment Framework Programme: Integrated environmental and occupational health assessment of new materials in the Danish Materials Technology Development Programme.
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

Editorial Board of 'Advanced Composites Letters'.
Povl Brøndsted

Programme Committee of the EU Working Group 'Hot Laboratories and Remote Handling'. Brussels, Belgium.
Hans Carlsen

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

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

Evaluation Group for the Joint Research Centre's Institute for Transuranium Elements, Karlsruhe.
Niels Hansen (Chairman)

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

Editorial Board of 'Revue de Métallurgie'.
Niels Hansen

Editorial Board of 'Monographs in Materials Science'.
Niels Hansen

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

The Advisory Committee for the International Conference on 'Low Energy Dislocation Structures (LEDS IV)'. Winnipeg, Canada.
Niels Hansen and Torben Leffers

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

Editorial Board of 'Plasticity'.
Torben Leffers

Editorial Board of 'Textures and Microstructures'.
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

Scientific Committee and Coordinating Committee of the 'Seventh European Conference on Composite Materials (ECCM-7)'. Bordeaux, France.
Hans Lilholt

Editorial Board of 'Advanced Composite Materials'.
Hans Lilholt

Editorial Board of 'Composites Science and Technology'.
Hans Lilholt

Editorial Board of 'Advanced Composites Letters'.
Hans Lilholt

Editorial Board of 'Polymers and Polymer Composites'.
Hans Lilholt

Editorial Board of 'Applied Composite Materials'.
Hans Lilholt

The Nomination Committee for the Outstanding Achievement Award of the High Temperature Materials Division of the Electrochemical Society, Penningston, USA.
Mogens Mogensen

Extended Group for Evaluation of BRITE EURAM proposals, Spring 1993, Brussels, Belgium.
Tage M. Nilsson

Editorial Board of 'Powder Metallurgy'
Allan Schrøder Pedersen

The Fuel Cell Committee under the Nordic Energy Research Programme. Oslo, Norway.
Finn W. Poulsen

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

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

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

Organizing Committee of the 'Seventeenth International ASTM Symposium on Effects of Radiation on Materials'. Sun Valley, USA.
Bachu N. Singh

International Organizing Committee of the International Workshop on 'An Assessment of Fundamental Aspects of Radiation Damage Production and Accumulation in Metals and Alloys'. Obninsk, Russia.
Bachu N. Singh

Organizing Committee for the 'Symposium on 20 years of Progress in Fusion Materials Research, Ispra, Italy.
Bachu N. Singh

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

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

7.2 Education and training

Many of the staff members of the Materials Department are actively involved in education and training in materials science as university external lecturers and examiners. Also, research on projects by undergraduate and postgraduate students and the research of post doctoral fellows was carried out under the supervision of Materials Department staff members.

External lectures

Janet J. Bentzen
'Zirconia Ceramics'. Course in Advanced Technical Ceramics for students at Aarhus University. Risø National Laboratory, Roskilde.

Jørgen Bilde-Sørensen
'Electron Microscopy'. Course for Materials Science students from Aarhus University. Risø National Laboratory, Denmark.

Niels Hansen
'Materials Science'. The Danish Academy of Engineering. Lyngby, Denmark.

Søren Linderoth
'Studies of Oxide Reduction and Gas Adsorption by using Positron Annihilation Spectroscopy'. NATO Advanced Study Institute on Nanophase Materials: Synthesis, Properties and Applications. Crete, Greece.

Aage Lystrup, B.S. Johansen, H.E. Gundtoft and K. Borum
'Fabrication and Control of Advanced Fibre Composites'. Continuing Education in Materials Technology. Risø.

Ole Toft Sørensen
'Solid State Chemistry: Defect Chemistry'. The Technical University of Denmark. Lyngby, Denmark.

External examiners

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

Christian P. Debel
BSc examiner, Copenhagen Technical College, Copenhagen.
Morten Eldrup
MSc examiner, Aarhus University, Aarhus.

Niels Hansen
PhD examiner, The Technical University of Denmark, Lyngby.

Andy Horsewell
MSc examiner, University of Copenhagen, Copenhagen.

Torben Leffers
MSc examiner, The Technical University of Denmark, Lyngby.

Hans Lilholt
MSc examiner, The Technical University of Denmark, Lyngby.

Hans Lilholt
PhD examiner, The Technical University of Denmark, Lyngby.

Hans Lilholt
MSc examiner, The University of Aalborg, Aalborg.

Mogens Mogensen
MSc examiner, The Technical University of Denmark, Lyngby.

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

Ole Toft Sørensen
MSc examiner, The University of Aalborg, Aalborg.

Postgraduate (PhD) projects

PhD projects successfully completed during 1993

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

Ole Jørgensen
'Damage in Laminates'.
The Technical University of Denmark.
Supervisors: Svend Ib Andersen, Povl Brøndsted and Hans Lilholt

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

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

PhD projects in progress during 1993

*) supervised at other laboratories.

Erik O. Ahlgren
'Thermoelectric Effects in Solid Oxide Fuel Cells'.
The Technical University of Denmark. Lyngby.
Supervisor: Finn W. Poulsen

Henrik Bjerregaard
'Flexible Forging of Metal Matrix Composites'.
The Technical University of Denmark.
Supervisor: Hans Lilholt

Franz Bødker *)
'Properties of Amorphous and Crystalline Fine Particles'.
The Technical University of Denmark. Lyngby.
Supervisor: Søren Linderoth

Henrik Christoffersen
'Development of Microstructure in Copper during Plastic Deformation'.
The University of Copenhagen, Copenhagen.
Supervisor: Torben Leffers

Jan Behrendt Ibsø *)
'Fatigue Life of Offshore Steel Structures under Stochastic Loadings'.
The Technical University of Denmark. Lyngby.
Supervisor: Povl Brøndsted

Niels Christian Krieger Lassen
'Automatic Determination of Crystallographic Orientations by Scanning Electron Microscopy'.
The Technical University of Denmark. Lyngby.
Supervisor: Dorte Juul Jensen

Michael Stanley Pedersen *)
'Supermagnetic Nanostructures'.
The Technical University of Denmark. Lyngby.
Supervisor: Søren Linderoth

Chris Pickup
'Internal Stress in Layered Structures'.
University of Cambridge, Canbridge, UK.
Supervisor: Andy Horsewell

Jes R. Poulsen
'Investigations of Defects in Ceramic Materials using Positron Annihilation Techniques'.
The University of Copenhagen.
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.
Supervisors: Finn W. Poulsen and Mogens Mogensen

Torben Valdbjørn Rasmussen
'Time Dependent Interface Parameters in Concrete Based Composite Materials'.
The Technical University of Denmark. Lyngby.
Supervisor: Povl Brøndsted

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

Undergraduate projects

Projects carried out as a part of undergraduate training

Daniel Ashford
'Microstructure and Texture Analysis of Cold-Rolled Al-SiCw Fabricated with High Purity Aluminium by Powder Metallurgy'.
Brunel University, Uxbridge, UK.
Supervisor: Yi-Lin Liu

Peter Bloch
'Texture Formation in Superconducting Bi-2233/Ag Bands'.
The University of Copenhagen, Copenhagen.
Supervisor: Torben Leffers

Philippe Brethe
'Measurements of R-curve in Ce-TZP Ceramics by Stable Crack Growth'.
Ecole Nationale Supérieure de Céramique Industrielle, Limoges, France.
Supervisors: Bent F. Sørensen and Ole Toft Sørensen

Michael Jakobsen
'Characterization of Zirconia Ceramics'.
The Danish Academy of Engineering. Lyngby.
Supervisor: Janet J. Bentzen

Aleksander Kiersz
'Hot Chamber Filament Winding with APC-2 Prepreg Tape'.
The Danish Academy of Engineering, Lyngby.
Supervisor: Tom Løgstrup Andersen

Steen Arnfred Nielsen
'Calculation of Materials Constants with Ultrasound methods'.
The Technical University of Denmark, Lyngby.
Supervisor: Hans Erik Gundtoft

Henrik Olsen
'Production and Shaping of SOFC Cathode Materials'.
The Technical University of Denmark, Lyngby.
Supervisor: Carsten Bagger
Jens Thomsen
'Hot Chamber Filament Winding with APC-2 Prepreg Tape'.
The Danish Academy of Engineering, Lyngby.
Supervisor: Tom Løgstrup Andersen

Nils Thorup
'The Cathode Process in Solid Oxide Fuel Cells'.
Odense University, Odense.
Supervisor: Mogens Mogensen

7.3 Visiting scientists

Staff members on assignment abroad during 1993

Bruno Kindl
Queen's University, Kingston, Canada. 30 Sept-20 Oct

Niels Hansen
University of Virginia, Charlottesville, USA. 13-27 Feb, 25-28 Nov

Bent F. Sørensen
University of Michigan, Ann Arbor, USA. 1 Nov-31 Dec

Guest scientists at the Materials Department during 1993

Dr. Henrik Albertsen
TZN, Unterlürs, Germany. 15-18 March

Dr. Jens H. Andreasen
Aalborg University Centre, Aalborg, Denmark. 1 Jan-1 Apr

Dr. John H. Beynon
18 July-13 Aug

Dr. Claire Barlow
University of Cambridge, UK. 23-26 July and 30 Aug-17 Sept

Dr. N. Brännström
Linköpings Tekniska Högskola, Linköping, Sweden. 19-20 June

Dr. John H. Evans
Royal Holloway College, London, UK. 8-20 July

Professor Julian Driver
Ecole des Mines, St. Etienne, France. 13 Apr-6 May

Dr. Gavin M. Hood
Atomic Energy Authority of Canada, Chalk River, Canada. 23-27 Oct

Dr. Darcy A. Hughes
Sandia National Laboratory, Livermore, USA. 2-27 Aug

Dr. Michael T. Hutchings
AEA Industrial Technology, Harwell, UK. 17-31 Jan

Dr. Gro Ostensen Lauvstad
The Technical University of Norway, Trondheim, Norway. 4-25 Jan

Dr. Truls Norby
University of Oslo, Norway.

Dr. J. Osborne
Loughborough University, UK. 28 June-4 July

Dr. Philip Prangnell
The University of Manchester and UMIST, UK. 22 Feb-7 March

Dr. Valerie Randle
University College, Swansea, UK. 17-21 Jan and 2-27 Aug

Professor Brian Ralph
Brunel University, Uxbridge, UK. 27-30 July

Professor David Shores
University of Minnesota, Minneapolis, USA. 6-17 Sept

Dr. S. Skolianos
Aristotelian University of Thessaloniki, Greece. 8-14 Nov

Dr. Roy Vandermeer
Naval Research Laboratory, Washington, USA. 16 Aug-10 Sept

Dr. Luca Viviani
CESNEF Politechnico Milano, Italy. 13-24 Sept

Dr. Philip J. Withers
University of Cambridge, UK. 22-28 Feb

Undergraduates from abroad at the Department during 1993

Terry Downes
University of Cambridge, UK. 22 Feb-7 March

Raymond Gill
Brunel University, Uxbridge, UK. 19 Apr-17 Sept

Michael B. Hall
University of Cambridge, UK. 28 June-3 Sept

Annette Kendall
University of Cambridge, UK. 28 June-3 Sept

Rafael Schouwenaars
Katholieke Universitet Leuven, Belgium. 23 March-4 April and 14-22 Nov

7.4 Colloquia

Visitors and guests are encouraged to hold colloquia on their current research activities. Members of staff, guest scientists and students are encouraged to attend and participate in this form of general information transfer activity.

'The development of research in glasses at Sheffield University', Friday 22 January
Dr. Michael Cable
University of Sheffield, Sheffield, UK

'Saturation of radiation strengthening in copper irradiated with high energy ions', Thursday 4 February
Professor G. Szenes
Eötvös University,Budapest, Hungary

'Interlaminar fracture toughness of fibre-reinforced plastics' (in Danish), Tuesday 16 marts
Dr. Henrik Albertsen
DLR, Cologne, Germany

'Thin films and interfaces studied by x-ray scattering', Thursday 18 March
Dr. Robert Feidenhans'l
Department of Solid State Physics, Risø, Roskilde, Denmark

'Novel side-chain liquid crystalline polyester architecture for optical storage', Thursday 15 April
Søren Hvilsted
Department of Solid State Physics, Risø, Roskilde, Denmark

'Which slip systems in hot rolled aluminium ?' Wednesday 5 May
Professor Julian Driver
Ecole des Mines de Saint Etienne, St Etienne, France

'Development of monolithic solid oxide fuel cells and dense ceramic membranes (for natural gas utilization) at Argonne national laboratory' Monday 14 June
Dr. U. (Balu) Balachandran
Argonne National Laboratory, Michigan, USA

'Microstructure, texture and isotropy in ceramics' Tuesday 22 June
Dr. Keith J. Bowman
Purdue University, Indiana, USA

'Interfaces in Al-SiC particulate composites' Thursday 24 June
Dr. Y. Harry Teng
Queen's University, Ontario, Canada

'Rolling texture transition in fcc metals and alloys and Formation of cube texture in cold rolled copper' Thursday 8 July
Dr. C.S. Lee
University of Birmingham, Birmingham, UK

'On measuring the elastic and damping constants of orthotropic sheet materials' Monday 26 July
Dr. Jim Woodhouse
University of Cambridge, Cambridge, UK

'Damage in composite materials: a synergistic micromechanics and continuum damage mechanics approach' Wednesday 25 August
Ramesh Talreja
Georgia Institute of Technology, Georgia, USA

'Non-stoichiometric transition metal carbides - crystal structure, order-disorder phase transformations and properties' Thursday 16 September
Dr. Andrei A. Rempel
Russian Academy of Sciences, Ekaterinburg, Russia

'The status of SOFC R&D at Mitsubishi heavy industries in Nagasaki' Monday 20 September
Dr. Akihiro Yamashita
Nagasaki R & D Centre, Nagasaki, Japan

'What on earth is the National Environmental Research Institute ? (in Danish) Thursday 23 September
Dr. Lars Carlsen
National Environmental Research Institute, Roskilde, Denmark

'From cradle to grave' (in Danish) Thursday 23 September
Dr. Karl Kjeldgaard
National Environmental Research Institute, Roskilde, Denmark

'Hardening of metals under cyclic non-proportional loadings' Wednesday 20 October
Dr. Ahmed Benallal
Laboratoire de Mécanique et Technologie, Cachan, France

'Defects and diffusion in zirconium - 1993' Monday 25 October
Dr. Gavin M. Hood
Atomic Energy of Canada Limited, Chalk River, Canada

'Current status of SOFC development at Osaka Gas' Thursday 18 November
Dr. Masamichi Ippommatsu
Osaka Gas, Osaka, Japan

'Quantitative x-ray radiography and analysis' (in Danish) Wednesday 24 November
Dr. Jørgen Thyge Rheinländer
Technical University of Denmark, Lyngby, Denmark

'Sheet metal forming simulation using explicit finite element method' Monday 13 December
Dr. Karl Brian Nielsen
Aalborg University, Aalborg, Denmark

7.5 Social activities

The Materials 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 those interested in sports, the 5 km 'Risø 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 and summer groups of rowers on the fjord. There is an annual ten-pin bowling tournament organized by MAK.

Members of the Materials Department are also active participants in hobby activities, organized through evening classes at Risø, including stone masonry, piano classes and language training. There is a Risø art society which sponsors regular exhibitions in the foyer of the Risø canteen and loans out pictures to members' offices and to departments. There are also regular group trips to the theatre, opera and ballet 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.

Publications

8

International publications

1. Aernoudt, E.; Houtte, P. Van; *Leffers, T.*, Deformation and textures of metals at large strain. In: Materials science and technology. A comprehensive treatment. Vol. 6: Plastic deformation and fracture materials. Cahn, R.W.; Haasen, P.; Kramer, E.J. (eds.), (VCH, Weinheim, 1993) p. 89-136

2. *Ahlgren, E.; Poulsen, F.W.*, Review of thermoelectric power of doped chromites and manganites. In: High temperature electrochemical behaviour of fast ion and mixed conductors. 14. Risø international symposium on materials science, Risø (DK), 6-10 Sep 1993. Poulsen, F.W.; Bentzen, J.J.; Jacobsen, T.; Skou, E.; Østergård, M.J.L. (eds.), (Risø National Laboratory, Roskilde, 1993) p. 193-200

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11. Barlow, C.Y.; *Hansen, N.; Liu, Y.L.*, A microstructural investigation of deformation in fine SiC particulate- and whisker-reinforced aluminium-matrix composites. J. Microsc. (1993) v. 169 p. 225-232

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Risø National Laboratory is a broad-based research organization with primary research activities in energy, the environment and in materials. There is a total of 893 employes.

The research programmes include basic studies of materials structure and properties, structural mechanics and materials testing, and processing techniques for polymer composites, powder metallurgical products and engineering ceramics. Advanced characterization techniques used in the Department are electron microscopy, positron annihilation, neutron diffraction, small angle neutron scattering and mechanical testing.

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