# Materials Research Department annual report 1999

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*Publication date:*
2000

*Document Version*
Publisher's PDF, also known as Version of record

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# Materials Research Department
## Annual Report 1999



Risø National Laboratory
April 2000

Risø-R-1155 (EN)

**Front cover:**
Materials microstructures, test specimens and schematics of graphs characterising macroscopic material properties.

**Back cover:**
New materials help the industry to develop new, more efficient products, such as lighter cars, larger wind turbines (photo: LM Glasfiber A/S) and superconductor cables (photo: NST A/S).

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# Materials Research Department

# Annual Report 1999

Published by
Materials Research Department

Risø National Laboratory
Roskilde · Denmark

April 2000

**Abstract**

Selected activities of the Materials Research Department at Risø National Laboratory during 1999 are described. The scientific work is presented in three chapters: Materials Science, Materials Engineering and Materials Technology. A survey is given of the Department's participation in collaboration with national and international industries and research institutions and of its activities within education and training. Furthermore, the main figures outlining the funding and expenditures of the Department are given. Lists of staff members, visiting scientists, publications and other Department activities are included.

Risø-R-1155(EN)

Published by the Materials Research Department, Risø National Laboratory, April 2000

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ISBN 87-550-2644-3
ISBN 87-550-2645-1 (Internet)
ISSN 0106-2840
ISSN 1397-8071

# Contents

# Introduction

**Materials Research Department 1999**

10 years development is reflected in the two solid oxide fuel cells. The small one was developed in 1990 - 1992 for operation at 1000 °C. The large one was developed in 1998 - 1999 for operation at 750 - 850 °C, and it is the basis for continuation of the national SOFC programme. The continuation includes upscaling through establishment of a pre-pilot-scale fabrication plant.

The research and development in the Materials Research Department has as its main objective to improve the performance of products and materials of the future. The Department receives input through our many national and international projects and by participating in conferences, educational activities etc. To widen these contacts an advisory committee to the Department has been established in 1999. This committee represents industry and academia in Denmark and abroad. It will advise the Department on planning, prioritisation and implementation of its research tasks. This committee will also make recommendations concerning how to maximise the impact of the Department's research in the industrial and educational sectors.

1999 has been yet another successful year due to a number of scientific and technological achievements, but also due to new large projects to be carried out in 2000 and thereafter, including new projects within our solid oxide fuel cell programme and 5 projects within EUs 5th Framework Programme. The estimated annual funding from these and other projects will bring the income of the Department to above 40 million Danish Kroner (DKK) per year. In the foreseeable future the Department can therefore run its activities with the surplus which is needed for investments.

## Achievements

**New techniques.** A 3D X-ray diffraction microscope was commissioned at ESRF in France. A number of experiments by Risø staff have already been performed. *In-situ* studies of nucleation and growth during recrystallisation of aluminium were carried out and phase transformations in high temperature superconductors were studied. With this instrument, it was also, for the first time, possible to determine the crystallographic orientation of a grain in the interior of a polycrystal and to trace the outline of the grain.

**New technology.** New technologies in the solid oxide fuel cell (SOFC) programme led to the construction of cell stacks composed of a cheap metallic interconnect, a thin electrolyte on an anode support and a graded cathode. Following a new patented process, a single cell stack test has shown power densities as high as 0.6 W $cm^{-2}$ at 850 °C and 0.4 W $cm^{-2}$ at 750 °C. These and other results showed that economical manufacturing of SOFCs is within reach. As a result the SOFC programme will be expanded in the coming years and a pre-pilot plant will be build in the Department in collaboration with Haldor Topsøe A/S, a Danish research-based company. In another research area, spray forming of metals was advanced also to include composites. A boron steel with the addition of 14 percent hard aluminium oxide has been spray formed and a material with a large increase in wear resistance and only a small loss in strength and ductility was obtained.

**New models.** Atomistic modelling of the cross-slip process (in collaboration with the Department of Physics, the Technical University of Denmark (DTU)) was expanded to include jogged dislocations. This modification brings the model into better accord with the physical realities. There is now a good agreement between calculated and experimentally determined values of the activation energy for this process. A new stereological technique was developed to analyse the crystallographic orientation of planar dislocation boundaries in deformed metals. A correlation between the dislocation structure and the active slip systems can thereby be deduced. This correlation

The advisory committee of the Department (left to right): John A. Kilner, Allan Schrøder Pedersen, Ricky Ricks, Niels Hansen, Günter Gottstein, Otto B. Christensen, Brian Ralph, Torben Lorentzen, Dorte Juul Jensen and Jens Olsson. Not present are Jens Als Nielsen, Hans Jørgen Pedersen, and Henrik Thorning.

**Advisory Committee**

**Industry and academia:**
Otto B. Christensen, Managing Director, Denmark (Chairman)
Günter Gottstein, Professor Dr. rer. nat., RWTH Aachen, Germany
John A. Kilner, Professor of Materials Science, Imperial College of Science, UK
Jens Als Nielsen, Professor dr. phil., Niels Bohr Institutet, Denmark (Vice chairman)
Hans Jørgen Pedersen, Vice President, Corporate Ventures, Danfoss A/S, Denmark
Brian Ralph, Professor, United Kingdom
Ricky Ricks, Dr., Director of Materials Research, Alcan Corp. Ltd., United Kingdom
Henrik Thorning, Managing Director, Fiberline Composites A/S, Denmark (Vice chairman)

**From the Materials Research Department:**
Niels Hansen, Head of Department
Dorte Juul Jensen, Deputy Head of Department
Torben Lorentzen, Senior Scientist (staff representative)
Jens Olsson, Laboratory Technician (staff representative)
Allan Schrøder Pedersen, Deputy Head of Department

The main role of the Advisory Committee is to advise the head of the Department on all matters concerned with the planning, prioritisation and implementation of its research tasks. Additionally, the committee will make recommendations about how to maximise the impact of this research activity in the industrial and educational sectors.
The Advisory Committee consists of 8 - 10 external members appointed by the head of the Department for an initial period of 3 years. The members who are personally appointed may be re-appointed.

Each member of the Advisory Committee is free to make personal recommendations to the head of the Department as well as contributing to the advice and recommendations of the Advisory Committee. The committee's chairperson and two deputies are selected by the committee amongst its members.

The Advisory Committee is required to meet at least twice each year. The Autumn meeting will have as its main agenda the discussion of the annual plan of the Department, whilst the Spring meeting will consider the more general trends in the development of the Department.

The head of the Department, his deputies, and staff representatives will participate in the meetings of the Advisory Committee. Furthermore, if required, other staff members may participate. The Department functions as a secretariat for the Advisory Committee. Thus, it organises the meetings, submits agenda and prepares the minutes of the meetings.

Members of the Advisory Committee are free to suggest items for the meeting agendas.

will be used in future modelling of the evolution of structure, texture and strength during plastic deformation.

**Nanoscale materials.** The first step in the amorphous-to-nanocrystalline transformation in Fe-rich ribbons was investigated by a highly sensitive differential scanning calorimetry technique. Results show that Cu-atom clusters act as nucleation sites. In collaboration with Alcan Aluminium Ltd., Canada, very thin, commercial aluminium foils were analysed by transmission electron microscopy. The submicron-scale deformation structure has been quantified as a basis for studies of the relationship between processing, structure and properties of the foil.

**Newsletters.** Many of our industrial customers have expressed the wish that we inform about our work and our achievements on a regular basis. This will be done in the form of newsletters to be published 3 times a year. Since Summer 1999 we have published two newsletters, which have been well received.

**Symposium.** The 20th Risø International Symposium on Materials Science with the title 'Deformation-Induced Microstructures: Analysis and Relation to Properties' was held in September 1999 as a continuation of this well-established series.

**New Programmes under EUs 5th Framework Programme:**

- Joining Dissimilar Materials and Composites by Friction Stir Welding (JOIN-DMC).
- Welding of Air Frames by Friction Stir Welding (WAFS).
- Improvement of Service Life and Reliability of Cold Forging Tools with Respect to Fatigue Damage due to Cyclic Plasticity (COLT).
- Scale up of Multi-Functional Solid Oxide Fuel Cells to Multi-Tens of Kilowatt Levels (MF-SOFC).
- Development and Test of Materials for Ceramic Membranes to be used in Production of Syngas (HTAS-membran).

**Challenges**

- A major challenge in the next 3 years is to reach the ambitious goals for the SOFC programme. Among these goals are (i) an improvement in the efficiency and durability of the cells and stacks, (ii) the building of a pre-pilot plant and (iii) a cost reduction. This research and develoment will be carried out in close collaboration with Haldor Topsøe A/S which will also be responsible for the marketing of technology and products. This heavy involvement of industry illustrates the natural shift in objective as a public supported energy programme matures into its industrial phase.

- Another challenge is to re-establish the close collaboration with the Danish electricity utilities which have been through major changes due to the opening of the European energy market. As part of these changes a new system has been introduced for funding of research and development. This funding, named Public Service Obligation (PSO), is aimed at energy production, which is friendly to the environment, i.e. clean and efficient. The work will be paid by the consumers and the results will be open to the public. The Department has already obtained support for one large project related to the development of fuel cells. However, other themes may also be of interest to the utilities, for example the effect of damage on residual life of rotor blades for wind turbines, high temperature materials for combustion plants, high temperature superconductors, hydrogen technology and energy storage in general.

With the introduction of PSO funding the energy-related materials research will in the foreseeable future have three main sources of income, PSO (the Danish utilities), EFP (The Ministry of Environment and Energy) and 5th Framework Programme (EU). This gives the Department a unique opportunity to formulate sizeable research programmes of both national and international interest.

• It is a permanent challenge to establish closer links with industry in order to accelerate the transfer of our knowledge into practise. In 1999 we have established quite large joint projects with five Danish firms in a variety of areas. Three of these co-operations also encompass a joint participation in projects under the 5th Framework Programme. We plan to double this number in 2000 – 2001 and thereby keep the Department in the technological forefront through this international collaboration. The relationship between the Department and Danish industry is also strengthened through summer jobs offered to university students. Through their vacation period the students work on joint projects between the Department and industrial companies, thereby getting practical experience on relevant materials problems.

• A final challenge is to maintain the basic research in the Department at an international level. External funding for this research has mainly been through the Engineering Science Centre. The funding through this centre is decreasing annually towards its end in 2002. New funding is therefore required, particularly when considering that the direct government funding to the Department in general is dwindling. One new approach, already met with some success, is to convince our public sponsors, especially in the energy field, that funding of long term research is a must if high quality strategic research and development shall continue. Another approach is to do part of the long term research in collaboration with Danish universities. We thereby also fulfil a national goal that education in materials science and technology be strengthened in order to produce more and better graduates. A good example is our collaboration with the Physics Department at DTU which includes both research and teaching.

Many new programmes will be the backbone of the Department in the next 3 – 5 years. These programmes are very diverse ranging from polymer composites for transport to high temperature electroceramics for electricity power production. A common feature, however, is the close collaboration between scientists, engineers and technologists in the materials field. Another common feature is the use of advanced models and experimental characterisation techniques from nanoscale to macroscale. The interdisciplinary approach enables the Department to handle the large complicated projects, which are becoming increasingly important, both in basic and applied research. It also enables the Department to be in the forefront in important research fields. This position increases visibility and credibility and it attracts students and young scientists. It is, therefore, with great optimism that we start our work in the new century.

The mechanic apprentices Casper Hammershøj Olsen and Jesper Nilsson were awarded a bronze and a silver medal, respectively, for their finishing test piece of work. The awards were handed over at a ceremony by the master of guild, Arne Hammer (centre).

**Prizes**

In 1999 several members of the Department have been recognised for their work:

| | |
|---|---|
| The Jubilee Prize of Danish Welding Society | Jørgen T. Rheinländer |
| Statoil Prize 1999 | Dorte Juul Jensen |
| Member of the Academy for the Technical Sciences (ATV) | Dorte Juul Jensen |
| The Glass Sellers Award in Science and Technology 1999 | Peter Halvor Larsen |
| Fosters Research Prize, University of Sheffield | Peter Halvor Larsen |
| The Medal of the Blacksmith's Guild | Jesper Nilsson<br>Casper Hammershøj Olsen |

# Materials Science

**- theory and characterisation**

*This chapter covers the research activities within three programmes: (i) Materials Models and Materials Structures, (ii) Local Structure and Properties and (iii) Irradiation Damage, Defects and Fusion Materials. The focus is on the basic understanding of material properties (predominantly mechanical properties) in terms of the structure of the materials. At the same time all projects have a clear relation to potential applications. Furthermore, the methodology (experimental and theoretical) developed within the three programmes finds applications in other programmes aiming more directly at technological applications.*

**Highlights are as follows:**

i incorporation of results from atomic-scale modelling of the annihilation of screw-dislocation dipoles by cross-slip in the theory for cyclic plasticity,

ii *in-situ* neutron diffraction investigation of stress/strain loops during cyclic deformation of stainless steel in terms of the lattice strains in different populations of grains with specific crystallographic orientation,

iii establishment of relations between macroscopic bands and local lattice rotation in single crystals deformed in tension,

iv successful implementation of the 3D X-ray diffraction (3DXRD) microscope at European Synchrotron Radiation Facility (ESRF),

v non-destructive characterisation of grain structure in 3D by the 3D X-ray diffraction microscope,

vi determination of the order of reaction kinetics as a function of the frequency of directional changes of one-dimensionally diffusing clusters of self-interstitial atoms,

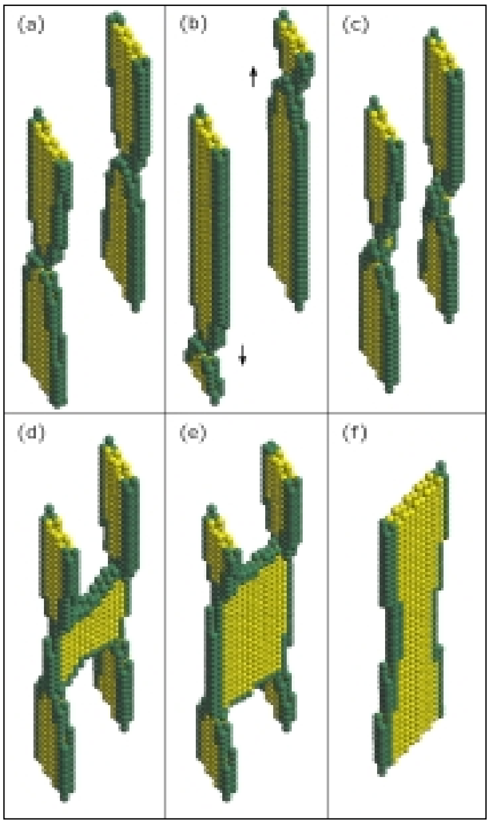
vii identification of micro-voids in the low activation F82H steel irradiated even at 50 °C to a dose level of only ~0.2 displacement per atom.

## Materials Models and Materials Structures

*This programme focuses on the development of micromechanical models for the mechanical behaviour of single- and multiphase materials, including the complex microstructures formed during deformation. The models cover the whole range of length scales from the atomic scale to the macroscopic scale. Collaboration with Department of Physics at the Technical University of Denmark (DTU) on the modelling of dislocation dynamics at the atomic scale is part of the activities. At the other end of the scale are measurements of residual stresses in large components by neutron diffraction, which is closely linked to polycrystal models at the mesoscopic scale.*

In 1999 the 20th Risø International Symposium on Materials Science with the title 'Deformation-Induced Microstructures: Analysis and Relations to Properties' was organised. The symposium successfully fulfilled its aim, namely to demonstrate the latest progress in the understanding and the experimental investigation of deformation-induced microstructures.

The Engineering Science Centre for Structural Characterisation and Modelling of Materials (IVC), financed jointly by the Danish Technical Research Council and Risø National Laboratory, is managed within the programme. The centre consists of a core and a shell. So far, the core was mainly positioned within this programme whereas the shell has covered a wider range of activities. The centre is




Selected configurations from the annihilation of a screw dislocation dipole of height 15 {111} planes with jogs in a face centred cubic metal. Atoms in the perfect atomic lattice are 'invisible', while 'green' and 'yellow' atoms are part of Shockley partial dislocations and stacking faults, respectively.

**Project Funded Research: Materials Science**

| Project type / **Project name** | Co-participants |
|---|---|
| Danish Technical Research Council (STVF). Engineering Science Centre (IVC) / **Structural Characterisation and Modelling of Materials** | |
| The Energy Research Programme of The Danish Ministry of Environment and Energy (EFP) / **DK Superconductors** | • NST A/S, Denmark<br>• NKT Cables A/S, Denmark<br>• NKT Research Centre A/S, Denmark<br>• Dept. of Appl. Eng. Design and Production, DTU, Denmark<br>• Dept. of Electric Power Eng., DTU, Denmark<br>• Research Assoc. of Danish Electric Utilities |
| Danish Natural Science Research Council / **Danish Centre for X-ray Synchrotron Radiation (DanSync)** | • Novo Nordisk A/S, Denmark<br>• CISMI, Symbion, Denmark<br>• Condensed Matter Physics and Chemistry Department, Risø<br>• Danish Space Research Institute, Denmark<br>• Department of Physics, DTU, Denmark<br>• H. C. Ørsted Laboratory, KU, Denmark<br>• Haldor Topsøe A/S, Denmark<br>• Department of Chemistry, AU, Denmark<br>• Institute of Chemistry, KU, Denmark<br>• Royal Danish School of Pharmacy, Denmark<br>• Department of Chemistry, DTU, Denmark |
| Training and Mobility of Researchers (TMR) / **Neutron Diffraction** | • Condensed Matter Physics and Chemistry Department, Risø<br>• DR3, Risø, Denmark |
| BRITE-EURAM / **Improvement of Quality and Productivity for Rolled and Extruded Aluminium Products through Microstructure and Texture Modelling (REAP)** | • Hydro Aluminium a.s., Norway<br>• Pechiney Recherche, France<br>• Gränges AB, Sweden<br>• Norwegian University of Science and Technology, NTNU, Norway<br>• Ecole des Mines de Saint-Etienne, France<br>• Swedish Institute for Metal Research, Sweden<br>• Norwegian Institute of Technology, SINTEF, Norway |
| BRITE-EURAM / **Residual Stress Standard using Neutron Diffraction (RESTAND)** | • Rolls-Royce - Gas Turbines, UK<br>• Volkswagen AG, Germany<br>• British Aerospace - Airbus, UK<br>• Sintech Keramik, Germany<br>• Schunk Kohlenstofftechnik, Germany<br>• AEA Technology, UK<br>• Rutherford Appleton Laboratory, UK<br>• Hahn-Meitner Institut, Germany<br>• Institute Laue Langevin, France<br>• NFL (Studsvik), Sweden<br>• Joint Research Centre Petten, The Netherlands<br>• University of Salford, UK<br>• Imperial College, UK<br>• University of Cambridge, UK |
| BRITE-EURAM · Thematic Network / **Training Industry in Neutron Strain Scanning (TRAINSS)** | • Daimler-Chrysler AG, Germany<br>• CRF, Italy<br>• FLAMATEL, Italy<br>• ISQ, Portugal<br>• NEL, UK<br>• Nuova, Italy<br>• PSA, France<br>• SNCF, France<br>• Technatom, Spain<br>• Rutherford Appleton Laboratory, UK<br>• University of Manchester, UK<br>• The Open University, UK<br>• Upsala University, Sweden<br>• University of Ancona, Italy<br>• Université Catholique de Louvain, Belgium<br>• Kiel University, Germany<br>• ENSAM, France<br>• Laboratoire Léon Brillouin, France |

*(continues on the next page)*

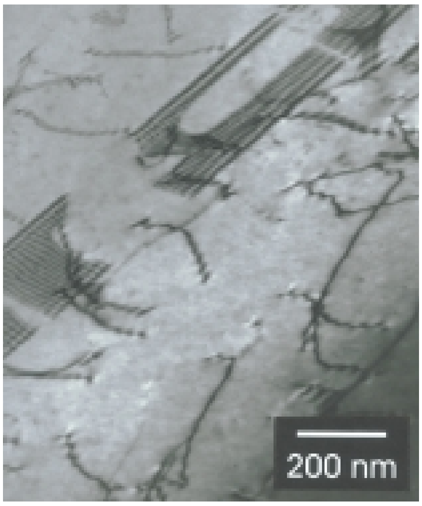
now in its second and last 5-year period, during which the activities should be ‘anchored’ so that they can continue after the last centre period. In this connection some of the activities are being transferred to other programmes, but the centre of gravity of the core is still within this programme.

### Atomistic modelling of the annihilation of jogged screw dislocations

The annihilation of pairs of opposite screw dislocations gliding on parallel atomic planes is an important recovery mechanism when face-centred cubic materials deform plastically at low temperatures where the available thermal activation is insufficient for dislocation climb. Annihilation of screw dislocations is therefore expected to set an upper limit to the dislocation density in low-temperature plastic deformation phenomena like cyclic saturation and stage III in monotonic deformation.

However, numerous jogs are produced on dislocation lines during plastic deformation as a result of dislocation intersections. A dissociated dislocation may tend to be constricted at a jog and this might considerably decrease the activation energy for cross-slip. Thus the jog would become a strongly preferred site for the onset of screw dipole annihilation. Its presence also increases the ‘annihilation distance’, the maximum distance between slip planes over which spontaneous annihilation occurs.

In collaboration with the Department of Physics at DTU the effect of jogs on the annihilation of screw dislocations in copper was examined for screw dipoles of four different heights. The jogs were chosen to lie in the cross-slip plane and the heights correspond to 4, 6, 10 and 15 {111} planes of spacing 0.218 nm. A stan-




TEM micrograph of polycrystalline Cu-30 % Zn cyclically strained into the primary hardening stage at a plastic strain amplitude of 3.4 x $10^{-3}$. An array of narrow edge dipoles contains 'holes', which are interpreted to form by destabilisation of edge dipoles and subsequent annihilation by cross-slip of dislocations in screw configurations.

dard energy minimisation procedure showed that the minimum stable dipole height increases from 4 to about 11 {111} planes, when jogs are introduced.

The 'nudged elastic band' (NEB) method was used to determine the minimum energy path through configuration space between the initial state with a jogged screw dipole of height 15 {111} planes and the final state, i.e. a perfect crystal. The NEB method shows that the energy barrier against annihilation is associated with successive reductions in dipole height by jog migration along the dislocations. When the height of about 11 {111} planes is reached, a spontaneous annihilation via cross-slip follows.

### Atomistic dislocation modelling of fatigue damage

Atomistic modelling is expected to make strong impacts on dislocation theories of fatigue by establishing the length scales of the dislocation microstructures responsible for damage production. These new possibilities were explored by combining results of mechanical testing, microstructural characterisation and continuum elastic dislocation modelling with the results of the atomistic dislocation modelling of cross-slip processes.

Cyclic saturation is a stage of continued formation of persistent slip bands (PSBs), which are sites of intense fatigue damage and crack nucleation. Thus the damage production is associated with a characteristic microstructure of narrow primary edge dipoles arranged in dense walls. A 'static-dynamic' dislocation model shows that although the walls are extremely hard, the stresses on suitably shaped and oriented walls in PSBs are high enough to destabilise even the narrowest edge dipoles approaching the peak stresses during cyclic saturation. Edge dipole destabilisation may spread and produce intense slip lines (ISL). However, the spreading requires an efficient process of dislocation annihilation.

Based on atomistic modelling of cross-slip activated annihilation of screw dislocations it was found that experimental activation energies for cyclic saturation in Cu at room temperature equals that of the annihilation of jog-free screw dislocation dipoles of about 2 nm height. Such nanoscale annihilation of screw dislocations in a PSB implies a concentrated plastic shear of about 26 %, which exceeds the average plastic shear of a PSB by an order of magnitude.

On the other hand, the atomistically based prediction is remarkably close to the average plastic shear amplitude of about 23 % measured for ISLs forming mainly inside PSBs. The analysis is supported by its prediction of the average length a screw dislocation glides before it meets an opposite screw dislocation within a slip plane distance of 2 nm and hence annihilates. The average glide length, predicted from the local dislocation density of the walls, turns out to be roughly the observed finite length of the edge dipoles. Thus, it appears that microstructures and fatigue damage at the micrometer scale can be coupled to dislocation annihilation at the atomic scale.

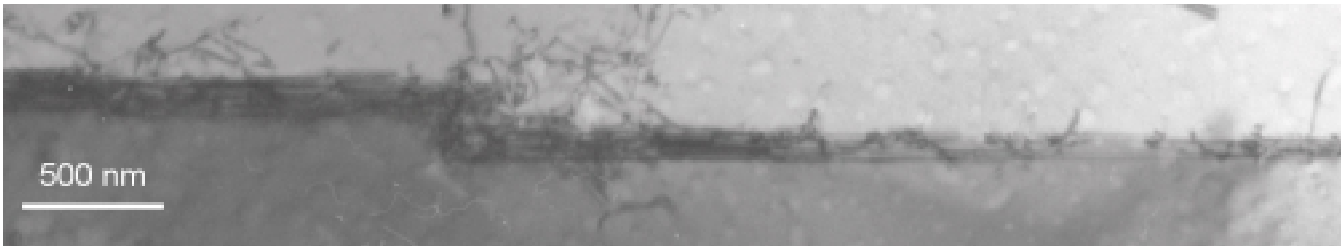
### Fatigue-induced microstructures in brass

Systematic transmission electron microscope investigations were carried out on polycrystalline Cu-30 % Zn cyclically deformed at constant plastic strain amplitudes. Characteristic dislocation microstructures associated with the successive stages of primary hardening, softening and secondary hardening were revealed.

Primary cyclic hardening is supposed to reflect a statistical distribution of short

**Project Funded Research: Materials Science (continued)**

| Project type / **Project name** | Co-participants |
|---|---|
| EU-Fusion Technology Programme (EU-FTP)<br>Next Step Technology. International Thermonuclear Experimental Reactor R & D · (ITER) /<br>**Copper and Copper Alloys Irradiation Testing for First Wall and Divertor, Effect of Irradiation on Mechanical Properties of Titanium Alloys** | • EU-sponsored, in collaboration with:<br>- Pacific Northwest National Laboratory, USA<br>- University of Illinois, USA<br>- VTT Manufacturing Technology, Finland |
| EU-FTP · Underlying Technology<br>**Effects of Irradiation on Physical and Mechanical Properites of Metals and Alloys** | • EU-sponsored, in collaboration with:<br>- Oak Ridge National Laboratory, USA<br>- London University, UK<br>- Research Centre Jülich, Germany<br>- Inst. of Physics and Power Engr., Russia<br>- National Research Institute for Metals, Japan<br>- Pacific Northwest National Laboratory, USA |
| EU-FTP · Long Term Materials Programme /<br>**Effects of Irradiation on Deformation Behaviour of Iron and Low Activation Steels** | • EU-sponsored, in collaboration with:<br>- CRRP, Switzerland<br>- Research Centre Jülich, Germany<br>- AEA Technology, UK<br>- UCLA, USA |




TEM micrograph of a grain boundary in Cu-2wt%Ni deformed in diffusional creep. Lattice dislocations are emitted at a step in the boundary. In general, there is an increased number of lattice dislocations in the immediate vicinity of the boundaries.

range ordered clusters. The softer regions of the distribution harden by cross-slip induced formation of increasingly narrow edge dipole arrays. Consequently, the sites of cyclic plasticity continually shift to harder regions of the material. Cyclic softening sets in when the stress amplitude is sufficiently high to trigger the formation of intense slip bands (ISBs). They form to accommodate the plastic deformation and give rise to pronounced strain localisation. In order to operate they must be continuously softened by a high rate of annihilation of mobile dislocations. Atomistic modelling suggests that this is a cross-slip process occurring in configurations of narrow screw dipoles. These dipoles are likely to be produced by destabilisation of the narrow edge dipole arrays formed during primary hardening.

Apart from consuming the edge dipole arrays the active ISBs are supposed to destroy the remaining short-range order, and collectively this produces the observed softening. The ISBs end their active phase as ‘hard’ bands of dense dislocation debris, which force the strain localisation to shift to other sites. Thus more and more ISBs form to accommodate the continued plastic deformation, and the polycrystal is gradually filled up with dislocation debris, which eventually induce the secondary hardening.

Despite the planar slip mode of Cu-30 % Zn the present investigations confirm the general view that cross-slip is a key process in the formation of metastable static structures of edge dipole configurations. Independently of their mesoscale morphology, which depends upon the degree of slip planarity, these structures destabilise at a given stress amplitude and give rise to (intense/persistent) slip bands, which are the sites of fatigue damage in the Cu-Zn system.

## Grain boundaries in metals subjected to diffusional creep

In earlier work the behaviour of individual boundaries during diffusional creep was examined by observing the deformation of the boundaries on the surface of a grid-covered sample. It was shown that the boundaries deform by a combination of grain boundary sliding, grain boundary migration and deposition or removal of material at the boundary by climb of grain boundary dislocations.

In order to further the understanding of the deformation mechanism, a study of individual boundaries by transmission electron microscopy was initiated. Many of the boundaries - probably the active ones - had a rather special and characteristic appearance being surrounded by a substantial number of lattice dislocations. It was also observed that emission of lattice dislocations had taken place at steps in the boundaries. The interior of the grains generally contained only few lattice dislocations, whereas an enhanced dislocation density was observed near some triple junctions.

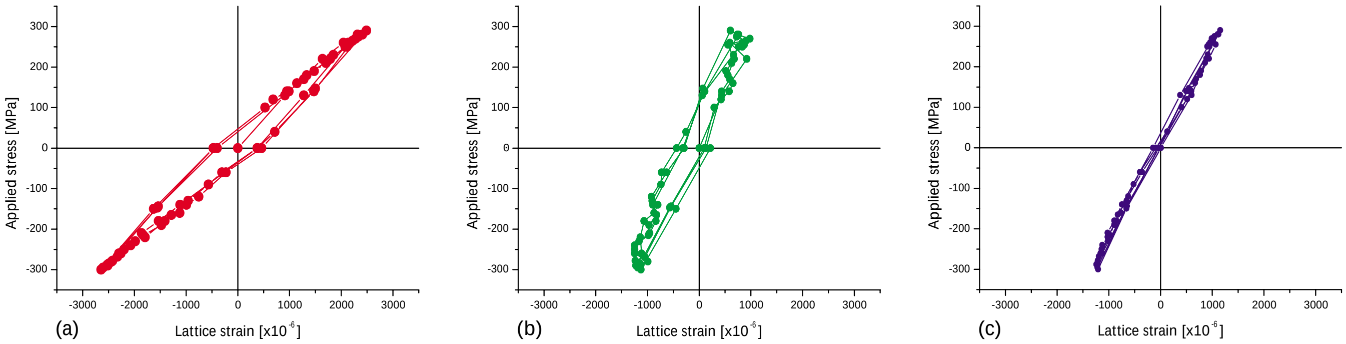
## Fibre textures simulated with a modified Sachs model

Traditionally, Sachs-type models have been left out of consideration for the modelling of fibre texture in face-centred cubic materials because they were assumed to produce a <211> fibre texture instead of the experimentally observed dual <100>/<111> fibre texture. However, this assumption was based on the questionable use of a lattice-rotation rule, which refers to the lattice rotation in single-crystal tensile experiments (‘Schmid tension analysis’). When one uses the standard rule in solid mechanics as an alternative rule for the lattice rotation (‘mathematical analysis’) the result is different – particularly so when the ‘mathematical analysis’ is used together with the ‘modified Sachs model’ for polycrystal deformation.

The modified Sachs model is a polycrystal model, which attempts to include the interaction of grains with different crystallographic orientations in a stochastic manner. It is a ‘pseudo n-site model’ and not a single-slip model. Combined with ‘mathematical analysis’ the modified Sachs model produces a dual fibre texture with a strong <111> component and a weaker <100> component in perfect agreement with experimental observations. Obviously this supports the status of the modified Sachs model as a viable alternative to the Taylor-based models for plastic deformation of polycrystals.

## Anisotropy in lattice strain evolution during monotonic and cyclic loading

The deformation mechanisms of polycrystalline materials are studied both experimentally and theoretically. In recent years, neutron diffraction has been utilised as a non-destructive probe for characterising the orientation specific elastic/plastic deformation, by measuring the evolution of elastic lattice strains under controlled external loading. Monotonic uniaxial loading experiments revealed highly non-linear lattice strain evolution, resulting in the development of a self-equilibrating state of residual intergranular lattice strains




Stress/strain loops in terms of the elastic lattice strain vs. the applied stress for three representative crystallographic reflections spanning from the elastically stiffest <111> direction to the most compliant <200> direction of the cubic crystallites. The elastic stiffness along the <331> direction is approximately 90 % of that along <111>. (a) (200), (b) (331), and (c) (111) reflections.

(leaving some grain orientations in tension and some in compression after unloading). These microstructure-related features of the aggregate deformation were modelled using a self-consistent modelling scheme, which proved to capture the dominating features of the deformation pattern.

These investigations were now extended to cyclic loading. Using a so-called time-of-flight technique, the elastic lattice deformation was monitored in the loading and the transverse direction of a uniaxially loaded stainless steel specimen. The specimen was cycled through 8 cycles within fixed total-strain limits of ±0.4 %, accumulating more than 9 % plastic deformation. Neutron diffraction patterns were measured *in-situ* rendering measures of the lattice deformation in various grain orientations simultaneously. In the preliminary analysis we consider 6 hkl-reflections: (111), (200), (220), (311), (331) and (420).

The analysis of the diffraction data shows stress/strain loops with shapes strongly depending on grain orientation. The most striking features of these loops are the variations in width and lattice strain amplitude. While the (200)-orientation shows an open loop with relative large strain amplitude, the (111)-orientation shows an essentially linear behaviour and a smaller amplitude. The non-linearities and hysteresis behaviour are consequences of load partitioning between families of grains with different elastic/plastic behaviour. Another striking feature is the rapid saturation of the residual intergranular lattice strains. While the hysteresis loops clearly show the residual strain level (at zero load) alternating between tension and compression, there is no apparent accumulation of residual strains with the accumulation of macroscopic plastic strain. Relaxation prevents the residual intergranular stresses and strains from growing beyond a certain level.

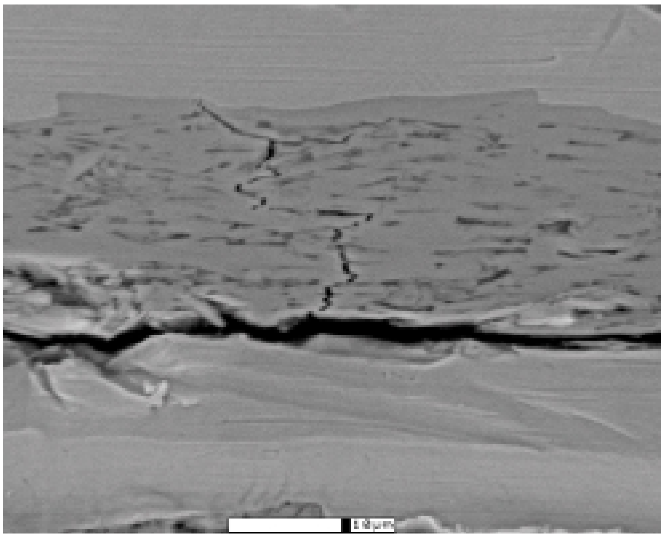
### Standardisation and industrial use of neutron diffraction for residual stress determination

Industrial interest in the utilisation of neutron diffraction for residual stress determination is growing rapidly. One of the reasons is a collaborative effort known as the VAMAS-initiative on the standardisation of the technique. Within the Versaille Project on Advanced Materials and Standards (VAMAS), the Department is participating in a technical working group preparing a code-of-practice, which will form the basis of an international standard. Through a series of round robin investigations a draft-standard document have been generated providing guidance on the 'best practice' in experimentation and data analysis. The main round robin investigation, covering a shrink-fit ring/plug assembly, has been completed as a separate report showing very good correspondence between measurements made at 15 facilities world-wide. The draft-standard will be forwarded to ASTM in mid 2000, and will likely be published as a so-called Technology Trend Assessment-document in the process towards an approval as an official standard.

Along with this standardisation initiative, two other EU-projects are further promoting the standardisation process and the approval by industry. An ongoing EU-sponsored project supports the VAMAS-initiative by round robin investigations made in collaboration with European industries. The industrial interest is further promoted through an EU-sponsored Thematic Network with the aim to train members of European industries in performing residual stress determination by neutron diffraction.

### *In-situ* observations during mechanical testing in ESEM: industrial problems

A number of test rigs can be used for mechanical testing inside the environmental scanning electron microscope (ESEM) of the Department. The advantage of performing mechanical tests in the ESEM is that while the macroscale mechanical response is measured, the underlying microscale damage mechanisms can be observed directly. Fixtures are available for tensile, bending, compression and controlled crack growth experiments (double cantilever beam specimens loaded by pure bending moments). Applied load and deformation can be measured. Acoustic emission can also be detected during experiments in the ESEM. One example of *in-situ* observations is splitting of unidirectional fibre composites (which are used e.g. in boats and trains, rotor blades in wind turbines or sport equipment). Since not all fibres are perfectly aligned, crack bridging occurs by cross-over bridging. The sequence of damage evolution



Micrograph of a transverse crack in a superconductor filament subjected to uniaxial tension in an ESEM. The crack path through the brittle ceramic layer is not straight, indicating weak grain boundaries. Near the ceramic/silver interface the crack bifurcates, resulting in splitting cracks parallel with the interface.

was clearly identifiable as initiation of a new crack at another plane followed by an increase in split length and fibre failures, which finally cause the bridging ligament to fail. Fibre bridging is beneficial in the sense that it increases the fracture resistance significantly.

Another example is uniaxial tensile testing of a superconductor tape consisting of ceramic filaments enclosed in a silver sleeve. In this case, transverse cracks initiate in the superconductor ceramic filaments. The cracks stop near the ceramic/metal interface by splitting. With increasing applied strain more cracks appear. The simultaneous increase in split length causes the effective non-superconducting volume to increase significantly at each transverse crack. This is clearly undesirable from the viewpoint of the application.

### Electron microscopy for external customers

The electron microscopes available in the Department comprise a conventional scanning electron microscope (SEM) equipped with electron back-scattering diffraction (EBSD) attachment and an energy-dispersive X-ray spectrometer (EDS), an environmental SEM equipped with EDS, heating and cooling stages and fixtures for *in-situ* deformation, a low-vacuum SEM, and two transmission electron microscopes (TEMs), one equipped with EDS and an electron energy loss spectrometer (EELS). Besides, the Department has a comprehensive selection of equipment for sample preparation.

A number of samples was examined by electron microscopy for external customers. Among the tasks can be mentioned:

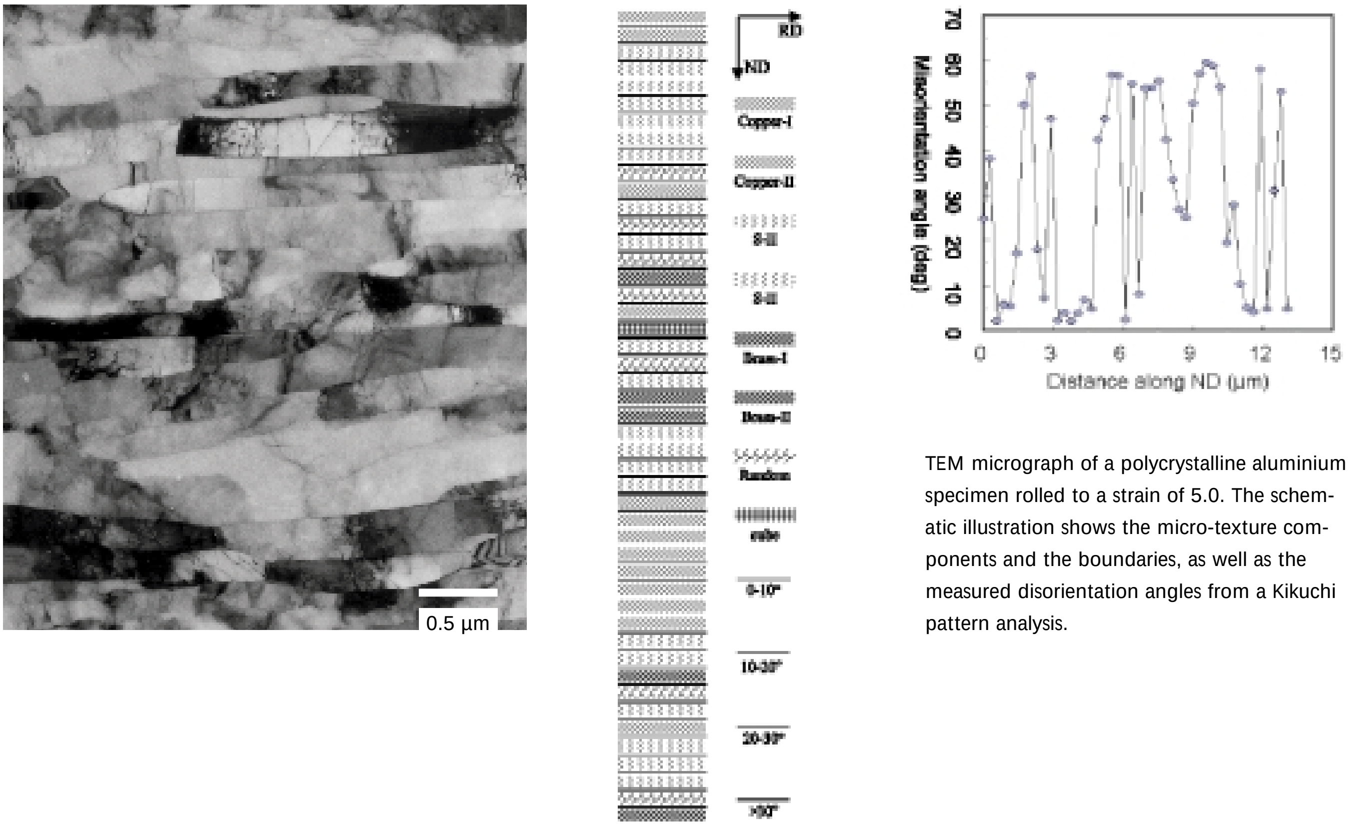
- elemental analysis of various paints and lacquers by EDS in SEM
- elemental analysis of various metallic tools by EDS in SEM
- determination of powder morphology by SEM
- examination of adhesive bonds in metals by EDS and SEM
- examination of combustion products from fluid beds by EDS and SEM
- determination of elemental distribution at metallic interfaces by EDS in TEM
- determination of local texture by EBSD

## Local Structure and Properties

*Understanding of the development of local structure and texture during mechanical and thermal treatments as well as understanding of relations between structure and properties are the main aims of this programme. These goals are approached via detailed quantitative experimental characterisations and modelling based on the experimental data. The modelling is thus typically on a micrometer scale intermediate between atomistic and coarse scale finite element modelling. Standard techniques are often not sufficient to give the necessary experimental data. Development of new methods or even new techniques is therefore a focal theme within the programme to enable quantification of important parameters as well as local processes occurring during the mechanical and thermal treatments. In 1999 the modelling work was focussed on the development of disorientations across deformation induced dislocation boundaries, on the selection of active slip systems in tensile deformation and on the relations between local lattice rotations and the macroscopic bands occurring when single crystals are deformed in tension. Most of the technique development effort was devoted to the implementation of the 3D X-ray diffraction (3DXRD) microscope. The instrument was used for in-situ characterisation in small selected volumes in the bulk of various samples during mechanical deformation and during annealing. Another major effort was the development of a procedure for automatic identification of recrystallisation nuclei in deformation microstructures from electron back-scattering patterns.*

### Crystallographic and macroscopic orientation of planar dislocation boundaries

Planar dislocation boundaries are an important structural feature in deformation structures. In a transmission electron microscope (TEM) planar dislocation boundaries can be revealed by their traces. It has been observed that planar dislocation boundaries have preferred macroscopic orientation, i.e. preferred inclination with respect to the principal axes of deformation. The boundary planes are in general close to the macroscopic planes of highest shear stress in the sample. Planar dislocation boundaries have also been found to have a preferred orientation in the crystallographic lattice. They lie on planes




TEM micrograph of a polycrystalline aluminium specimen rolled to a strain of 5.0. The schematic illustration shows the micro-texture components and the boundaries, as well as the measured disorientation angles from a Kikuchi pattern analysis.

with certain crystallographic indices. The relative importance of the preferred macroscopic and crystallographic orientations was examined for an understanding of the origin of the preferred boundary orientation.

Macroscopic and crystallographic orientations were analysed for planar dislocation boundaries observed in tensile deformed aluminium. In tension the most stressed planes are inclined 45° to the tensile axis and are tangents to a cone around this axis. If the boundaries would be observed on the macroscopically most stressed planes only, the planar dislocation boundaries in different grains could not be expected to have any preferred crystallographic orientation. However, detailed analysis showed that planar dislocation boundaries have a strong preference to form on certain crystallographic planes, which depend on the crystallographic orientation of the grains. The crystallographic boundary planes are distributed around, but do not coincide with the most stressed macroscopic planes. A weaker macroscopic preference is present compared with the crystallographic preference. The strong correlation between crystallographic boundary planes and grain orientation indicates that the boundary orientation is closely linked to the active slip systems.

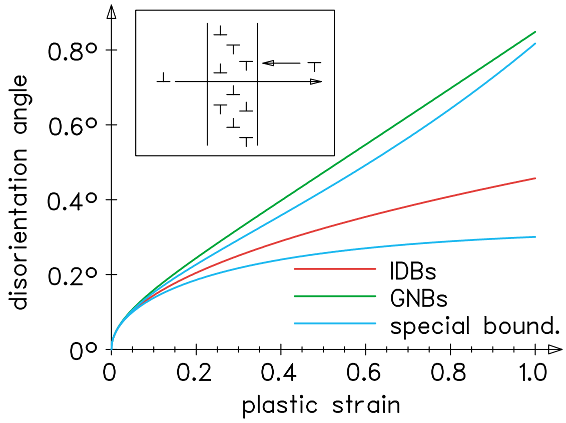
**Large strain deformation of commercial pure aluminium**

During cold-rolling the microstructure of polycrystalline face-centred cubic metals of medium to high stacking fault energy evolves from a cell block structure delineated by dislocation boundaries forming an angle of about 40° with the rolling plane to a cell block structure delineated by lamellar boundaries parallel to the rolling plane. This structural evolution must be characterised in order to further the understanding of the mechanical and thermal behaviour of cold-worked metals and alloys. The microstructural evolution at low and medium strains has been investigated extensively whereas large strain structures are much less investigated.

In collaboration with Alcan Aluminium Ltd. commercial purity aluminium (AA1200) was rolled to different reductions up to a maximum strain of 6.0. The microstructural evolution was followed by TEM and the local crystallography was determined by Kikuchi pattern analysis in TEM. Well-developed lamellar structures were found at a strain of about 2.0 and above. The spacing between lamellar boundaries decreases and the disorientation angle across the boundaries increases with increasing strain. No saturation was observed either in spacing or in disorientation angle. The large strain structure consists predominantly of small volume elements of ideal rolling texture components (Copper, S and Brass) separated by high angle boundaries. These volume elements are further subdivided by medium and low angle dislocation boundaries.

**Prediction of subdivision in single crystals**

The systematic investigation during previous years of single crystals deformed by rolling revealed that the macroscopic domain pattern in rolled crystals reflects constraints imposed by the rolling operation. To explore macroscopic domain patterns developed under deformation conditions with less constraint, aluminium single




Model predictions for the evolution of disorientation angle with plastic strain of a single slip system for different types of dislocation boundaries and constant boundary spacing. The insert shows the partial trapping of dislocations passing a boundary.

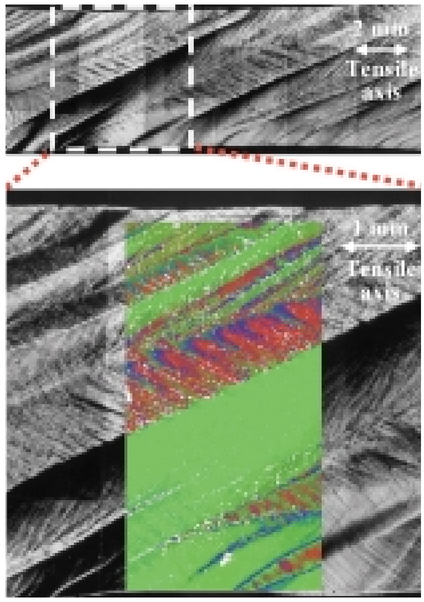
crystals deformed in tension were investigated in collaboration with the University of Tokushima in Japan. The close matching of features between the optical and crystal orientation images reveals that the prominent domain structure visible on the surface of the crystal reflects a set of crystal orientation domains. Detailed analyses of such orientation images have uncovered an answer to a half-century-old question: why do macroscopic domain structures, visible by eye, appear in tensile tested single crystals? Transforming the crystal orientation pattern into a shear amplitude pattern unveils the strain heterogeneity pattern. The strain heterogeneity pattern was then related to constraints imposed on the sample by the testing machine.

The analysis revealed that an explanation of single crystal deformation has to consider the specimen grip constraints, which engender the orientation domain pattern. Thus, single crystals deformed in tension, like their rolled companions, exhibit a macroscopic crystal orientation domain structure that reflects constraint by the deformation processing machinery. Local crystal orientation measurements reveal a large influence of the deformation process on material behaviour during tests that form the foundation of our understanding of single crystal deformation processes.

**Prediction of disorientations across dislocation boundaries**

During plastic deformation orientation differences arise across dislocation boundaries. Two types of boundaries can be distinguished with respect to their formation: incidental dislocation boundaries (IDBs) which are assumed to be a result of statistical mutual trapping of dislocations and planar geometrically necessary boundaries (GNBs) with a different activity of slip systems on each side of the boundary. Disorientations across the dislocation boundaries evolve from trapping of mobile dislocations passing the boundary, if an excess of dislocations of one sign of the Burgers vector is accumulated. Several reasons for excess dislocations occur depending on the boundary type. Different cases were modelled in terms of stochastic equations and the resulting mean disorientation angles as well as their distribution functions were predicted:

Disorientations across IDBs arise from statistical fluctuations in the dislocation fluxes from the two adjacent sides. The pure statistical accumulation of excess dislocations leads (for constant boundary spacing) to a square root dependence of the mean disorientation angle on plastic strain. An additional deterministic contribution exists for GNBs from the different activity of the slip systems on either side of the boundary leading to a stronger increase of the mean disorientation angle. This diversity in the behaviour of both



Aluminium single crystal strained 30 % in tension. The upper portion shows a reflected light image and the lower portion shows an expanded view of the deformed crystal with a superposed crystal orientation image. The macroscopic domain pattern visible by eye corresponds closely to the underlying crystal orientation pattern.

types of boundaries explains the experimentally observed differences between IDBs and GNBs in the dependence of the disorientation angle on plastic deformation. For special boundaries there might be a further contribution to the evolution of disorientations due to misfit dislocations deposited at the boundary as soon as a small disorientation angle arises.

**Hot deformation of commercial aluminium alloys**

A BRITE-EURAM project on the improvement of quality and productivity for rolled and extruded aluminium products through microstructural and textural modelling was completed. The work in the Department was concerned mainly with characterisation of hot deformation microstructures and textures as well as the subsequent recrystallisation kinetics, microstructures, and textures. It was found that at low strains the hot deformed microstructures resemble those after cold deformation whereas at high strains a more equiaxed subgrain structure is developed.

Over a wide range of temperatures and strain rates similar deformation textures are formed in commercial aluminium (alloy AA3104) deformed by plane strain compression. The weakest textures developed at low values of Zener-Hollomon parameters. With an increase in Zener-Hollomon parameters, there was a consistent increase in the strength of the resultant deformation texture. Except for the high temperature/high strain rate specimens, all deformed specimens produced textures upon annealing with a weaker version of the deformation texture and a cube volume fraction indistinct from that of a random texture. The lack of a distinct recrystallisation texture may be attributed to an excess of nucleation sites.

The experimental data have been used in industrial models for the deformation and recrystallisation processes developed by partners in the project.

### Commissioning of a 3D X-ray microscope

The Department has finished the commissioning of a 3D X-ray diffraction (3DXRD) microscope at the European Synchrotron Radiation Facility (ESRF) in Grenoble. The microscope is dedicated to local structure characterisation within millimetre thick specimens. Parameters such as strain, grain orientation, grain volume, and grain morphology can be investigated *in-situ* during annealing or deformation processes. The instrument is based on focusing of monochromatic hard X-rays (50 - 100 keV) and the use of two-dimensional detectors. The size of the gauge volume is variable with a minimum of 5 x 5 x 50 $\mu m^3$. A small furnace and a 25 kN stress-rig is available. External users can for beamtime via the ESRF review committees.

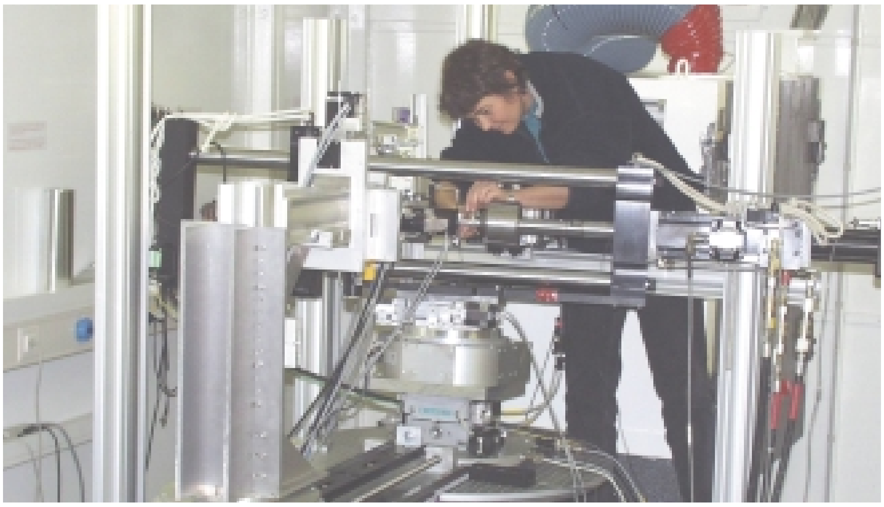
Several experiments have demonstrated the prospect for studies of the individual grains. Identified by their distinct orientations, grains of size 1 $\mu m^3$ can be observed. As an example, tapes produced at Nordic Superconductor Technologies A/S (NST) and containing a $Bi_2Sr_2CaCu_2O_x$ (Bi-2212) rich powder within a Ag sheet were annealed on-line. Unique information on the transformation of the grains to the superconducting phase $Bi_2Sr_2Ca_2Cu_3O_x$ (Bi-2223) was obtained.

A feasibility test of new concept was performed. Using a conical slit and a two-dimensional detector, the local strain and texture within an embedded volume are determined simultaneously. The conical slit has 20 µm openings positioned to match the Debye-Scherrer cones characteristic for the X-ray diffraction pattern of the material in question. The sample was scanned in the direction of the beam, acquiring images at each position. For each acquisition X-ray tracing was performed to associate parts of strain profiles with points of origin in the sample. The full strain profiles, and thereby the average strains, were found as a function of position by reconstruction. This procedure has the advantage that the slit can be positioned at a distance of 10 cm from the sample, providing space for sample surroundings.

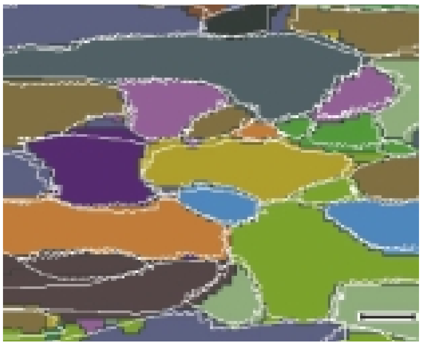
### Three dimensional maps of polycrystalline materials

The 3D X-ray microscope allows three-dimensional mapping of embedded grains within thick samples by a special tracking technique. As a validation test a surface on an aluminium sample was investigated with both the tracking technique electron back-scattering diffraction (EBSD). The variation in the position of the grain boundaries determined by the two techniques is at the worst 100 µm. Maps of the grain boundary structure determined by the tracking technique will thus be of sufficient quality for many applications. The grain orientations determined by the two techniques were found to reproduce within the combined alignment errors of the set-ups. The total data acquisition time for the synchrotron data was not more than two minutes.

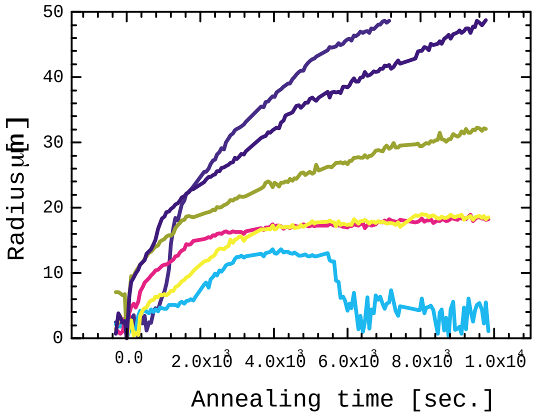
The tracking technique is based on a monochromatic high energy X-ray (50-80 keV) beam and a two-dimensional detector. The incoming beam focused into a line defines a layer within the sample. All grains in this layer that fulfil the Bragg condition give rise to diffraction spots on the detector. The diffraction spots are the projected image of the corresponding diffracting grains in the observed layer. The position of each diffraction spot is measured and the intensity-weighted centre-of-mass (CM) of each diffraction spot is computed. This procedure is re-



Changing of a sample in the 3DXRD microscope with a horizontal tensile stage mounted. The X-ray beam originates from the synchrotron source behind and is diffracted in the sample to be registered by the two-dimensional detector which can be translated along the direction of the beam on the black granite arm.



Grain structure of aluminium for validation of 3DXRD tracking algoritm. The black lines indicate the grain boundaries as determined by EBSD, and the white lines indicate the grain boundaries resulting from 3DXRD and X-ray tracking. The colours indicate different grain orientations measured by EBSD. The scale bar at the bottom is 400 µm.




The 3DXRD diffraction microscope allows *in-situ* studies of the growth of individual nuclei during annealing. The growth of 6 nuclei during annealing of 90 % cold-rolled 99.5 % pure aluminium reveals several features of the recrystallisation process that could not have been seen by conventional techniques.

peated at several detector distances and linear fits through corresponding CM points extrapolate to the CM of the diffracting grains. To obtain the cross-sectional grain shape, the periphery of the diffraction spot (determined by a fixed intensity threshold) in the image acquired at the closest distance to the sample is projected into the illuminated sample plane.

By rotating the sample around an axis, perpendicular to the illuminated plane, all grains will come to fulfil the Bragg condition. Thus, a complete map of all grain boundaries and grain orientations in the plane was produced. A three-dimensional map can then be obtained simply by translating the sample along the rotation axis and repeating the procedure for several layers.

**Recrystallisation kinetics characterised by the 3D X-ray microscope**

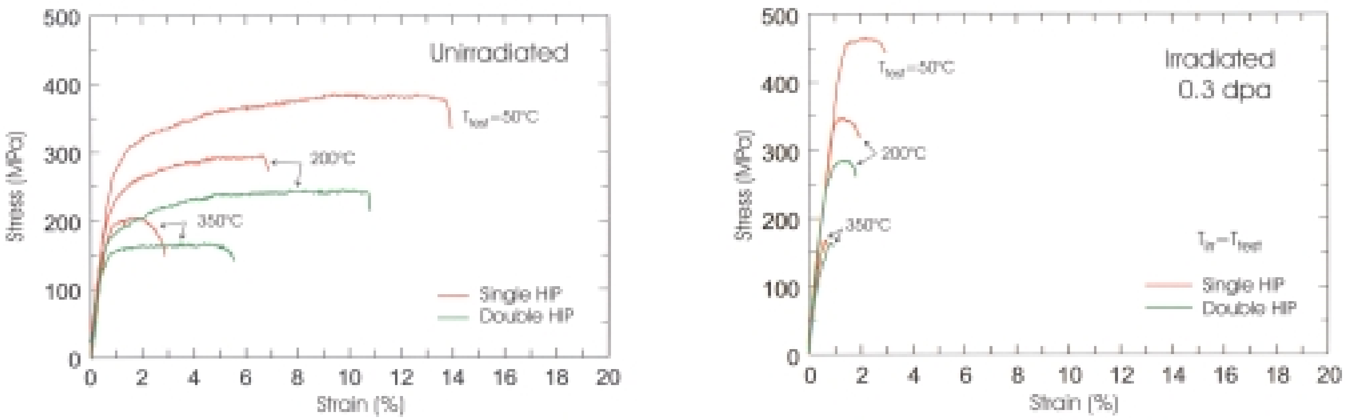
Recrystallisation is a thermally activated process reducing the energy stored in a plastically deformed metal or alloy. Recrystallisation is generally separated into two distinct processes: nucleation and growth, which may occur consecutively or simultaneously. During nucleation almost defect free nuclei form in the deformed microstructure. During growth these nuclei grow by grain boundary migration until they impinge upon each other.

A plethora of models exists to simulate recrystallisation ranging from purely geometrical or numerical models to fundamental analytical models. A common characteristic of the models is their focus on the average behaviour of nuclei. Hence, one or several of the following assumptions are generally applied: randomly distributed nucleation sites, instantaneous or constant nucleation rates, and a growth rate identical for all nuclei. The main reason for these assumptions is the lack of experimental data for the behaviour of individual nuclei.

The 3D X-ray microscope provides a technique for *in-situ* studies of the nucleation and growth of individual nuclei during recrystallisation of metals. A suitable intrinsic gauge volume is defined by focusing the incoming beam and by performing ray tracing on the diffracted beams, using a two-dimensional detector. By this procedure, the orientation and volume of several hundred nuclei can be determined simultaneously during annealing. The first results relate to 1 mm thick 90 % cold-rolled 99.5 % pure aluminium. The threshold for observation of nuclei in this case is a radius of 1 µm. In contrast to model assumptions the variations in onset of nucleation and growth rate are substantial. Direct evidence was found for an incubation process before the onset of nucleation and some nuclei were observed to disappear abruptly at an early stage.

**Optimisation of superconducting tapes**

The Department participates in a Danish programme for use of high-temperature superconductors in the electric power sector. Superconducting cables for power transport are to be based on tapes containing the compound $Bi_2Sr_2Ca_2Cu_3O_x$ (Bi-2223). The tapes were produced at NST A/S by filling a pre-cursor powder into silver tubes and processing the tubes by a series of rolling and annealing steps. The processing controls the phase purity, grain alignment and grain connectivity between the Bi-2223 grains – three factors that in turn determine the superconducting properties. The aim of the Department in close collaboration with the Department of Condensed Matter Physics and Chemistry at Risø National Laboratory is to optimise the annealing process with respect to the critical current density. Recently, the structural bottlenecks for the critical current density were identified. The critical parameters are the density of the ceramic and the thickness of the individual filaments in the tapes. In that context it is encouraging that the addition of small amounts of $CaF_2$ was found to improve the diffusion and sintering process and thereby increase the density substantially by 10 %.




Tensile stress-strain curves for joint specimens of Cu-$Al_2O_3$ with 316 (LN) stainless steel (joined by hot isostatic pressing) tested before and after irradiation at different temperatures. Note that double HIPing reduces the strength and irradiation with neutrons reduces the uniform elongation significantly.

## Irradiation Damage, Defects and Fusion Materials

*The activities within the Association Euratom - Risø National Laboratory are divided into three parts: the 'Next Step', the 'Long Term' and the 'Underlying European Fusion Technology Programme'.*

The investigations carried out in the field of the 'Next Step Technology' constitute a part of the European contribution to the 'International Fusion Technology Programme' aiming at design and construction of the International Thermonuclear Experimental Reactor (ITER). The activities in the Department in this area were concentrated on the effect of irradiation on copper and copper alloys, which possess the high thermal conductivity needed in components such as the first wall and divertors. Similar invesigations were carried out on titanium alloys.

The 'Long Term Technology Programme' covers activities dealing with the problems related to design and construction of the DEMO reactor (a demonstration fusion reactor). At the Department, irradiation effects on iron and low activation steels, which are considered as construction materials, were studied. Finally, the programme has activities within the Underlying European Fusion Technology Programme, where more fundamental aspects of irradiation effects are investigated.

The programme covers experimental investigation of effect of irradiation on changes in microstructural and mechanical properties using TEM, SEM , PAS and electrical resistivity measurements. Theoretical and simulation studies are also carried out (in collaboration with scientists from Europe, Russia and USA). A workshop on 'Damage Production and Accumulation under Cascade Damage Conditions' was organised at the University of Liverpool, UK.

**Effect of neutron irradiation on mechanical performance of copper alloys**

The first wall and divertor components of ITER will be exposed to an intense flux of 14 MeV neutrons and will experience thermo-mechanical cyclic loading as a result of the cyclic nature of plasma burn operations of the system. Copper alloys are being considered as candidate materials for these components. Therefore, the effect of neutron irradiation on the tensile, fatigue and fracture toughness behaviour of these alloys was investigated.

The fatigue lives of both unirradiated and irradiated (at 350 °C to a dose of ~ 0.3 dpa (displacements per atom)) Cu-$Al_2O_3$ and CuCrZr alloys were determined at 350 °C. The irradiation led to a marked decrease in the lifetime of both alloys, most pronounced in the case of the Cu-$Al_2O_3$ alloy.

Both tensile properties and fracture toughness behaviour of HIP (hot isostatic pressing) joint specimens of Cu-$Al_2O_3$ and CuCrZr alloys with 316 (LN) stainless steel were investigated before and after neutron irradiation. The tensile strength of HIP joint specimens of both copper alloys was considerably lower than for the alloys without HIP treatment. A double HIP thermal cycle reduced the strength even further. Irradiation of the HIP joint specimens with fission neutrons led to a drastic decrease in the ductility of the Cu-$Al_2O_3$ specimens, leaving the ductility of the CuCrZr considerably better than that of the Cu-$Al_2O_3$ specimens.

Notch sensitivity tests carried out on the Cu-$Al_2O_3$ alloy showed that the effective plastic strain to fracture was strongly dependent on the triaxiality of the stress state and decreased with increasing temperature. The large volume fraction of grain boundaries and the presence of alumina particles on these boundaries may be responsible for the observed fracture behaviour.

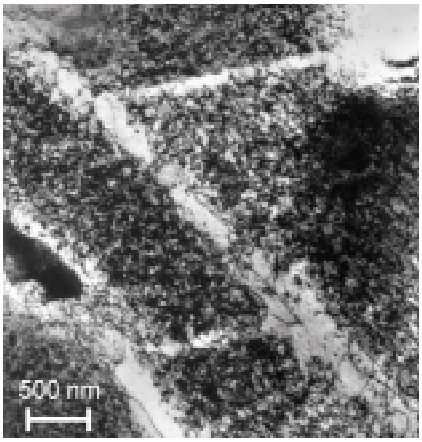
**Tensile and fatigue behaviour of titanium alloys**

The $\alpha$+ß titanium alloy (Ti-6Al-4V) was selected as a candidate material for flexible connectors between the blanket modules and the backplate of ITER. In view of an almost complete lack of knowledge about the effect of irradiation on mechanical properties of this alloy the influence of neutron irradiation on tensile behaviour at 50 °C and 350 °C was evaluated. For comparison, also the α-titanium alloy (Ti-5Al-2.5Sn) was investigated.

Both alloys were irradiated in the as-supplied condition with fission neutrons in the DR3 reactor at Risø National Laboratory to a dose of ~ 0.3 dpa. Unirradiated and irradiated specimens were tensile tested in vacuum at temperatures equal to the irradiation temperatures. The microstructures of the as-supplied and tensile tested (unirradiated) specimens were investigated using optical microscopy and TEM. Fracture surfaces of the tensile tested (unirradiated) specimens were examined using scanning electron microscopy.

The tensile behaviour of the two alloys were quite similar at both temperatures except that the α+ß alloy was slightly stronger and showed somewhat less ductility than the α-alloy. The tensile properties of the α-alloy were much less influenced by the neutron irradiation than that of the α+ß alloy. In the case of the α+ß alloy, irradiation at 50 °C led to a very substantial decrease in the uniform elongation even though the increase in the tensile strength was moderate.

Unirradiated specimens of both α and α+ß alloys were fatigue-tested at 50 °C. For both alloys, the number of cycles to failure increased with decreasing strain amplitude. The fatigue behaviour of the two alloys was found to be quite similar.




Transmission electron microscope picture showing localised plastic flow in the form of 'cleared' channels emanating from large inclusions in a CuCrZr alloy neutron irradiated at 50 °C to a dose level of 0.3 dpa. The irradiated specimen was fatigue-tested at 50 °C at a loading frequency of 0.5 Hz.

### Influence of crystal structure on damage accumulation during neutron irradiation

There is an increasing amount of experimental and theoretical evidence that the evolution of defect microstructure in metals and alloys under neutron irradiation depends on the crystal structure of the material (in addition to temperature and other parameters). To study this dependence in more detail the effects of neutron irradiation on the defect accumulation were investigated in iron, the low activation steel F82H (both body centred cubic), and in copper (face centred cubic) irradiated at temperatures in the range of 50 °C to 350 °C. The specimens were irradiated to doses of 0.2 - 0.3 dpa.

The characterisation of the defect microstructure was carried out using Positron Annihilation Spectroscopy and in some cases electrical conductivity measurements. Positron lifetime measurements may, for example, provide information about cavity densities and sizes, while the electrical conductivity depends on the defect density and configuration and therefore gives an average measure of the overall defect population in a specimen.

The main result of the investigation was that in Cu, void nucleation takes place only at irradiation or annealing temperatures above ~200 °C, whereas in iron and steel voids are formed in the whole temperature range investigated. At the lower irradiation temperatures also smaller, three-dimensional vacancy clusters (microvoids) are formed in iron and steel. In the whole temperature range, the void density in the steel is appreciably lower than that in pure iron. In contrast to the positron results, the electrical conductivity is very different for iron irradiated at 50 °C and at 100 °C. This effect is ascribed to the migration of carbon.

### Modelling of defect accumulation and deformation behaviour under neutron irradiation

In recent years it was demonstrated that in order to treat the problem of defect accumulation under cascade damage conditions appropriately, one has to use the production bias model (PBM). The PBM differs qualitatively from conventional rate theory. It recognises the importance of intra-cascade clustering and the properties of the resulting clusters (thermal stability, one-dimensional (1D) diffusion of clusters of self interstitial atoms (SIA), changes in direction during 1D diffusion, etc.). This required a new type of defect reaction kinetics that allowed accurate calculations of the reaction cross-sections for 1D diffusing SIA clusters with other SIA clusters, vacancy clusters, voids and dislocation segments. This new development made it possible to calculate the temporal evolution of void swelling analytically, including all possible interactions of glissile SIA clusters with all other sinks in the crystal. For the first time, a proper framework for accurate studies of the problem of void swelling even in complicated alloys was provided.

In an effort to validate the cascade induced source hardening (CISH) model, detailed numerical calculations were carried out to determine various aspects of dislocation-loop interactions using three-dimensional dislocation dynamics. These calculations confirm the concept of a 'stand off' distance between a gliding SIA loop and an edge dislocation and the initiation of dislocation decoration with small SIA loops, as envisaged in the CISH model. In order to understand the hardening mechanism, interactions of gliding dislocations (emanating from a source) with coplanar and non-coplanar obstacles (stacking fault tetrahedra and SIA loops) were simulated. The results showed that the upper yield strength of irradiated copper represents the stress level necessary to activate dislocation sources at sites of stress concentration (grain boundaries, particle interfaces, etc.). This is fully consistent with the basic premise of the CISH model.

# Materials Engineering
**– modelling and performance**

*The activities within Materials Engineering are focused on characterisation and modelling of mechanical properties for component design and performance testing. The activities are carried out within two programmes: Properties of Composite Materials which covers metallic, ceramic and polymer matrix composites with fibres or particles, and Mechanical Characterisation and Design of Components which covers experimental and numerical methods that relate material properties to component performance.*

**The highlights from these activities are:**

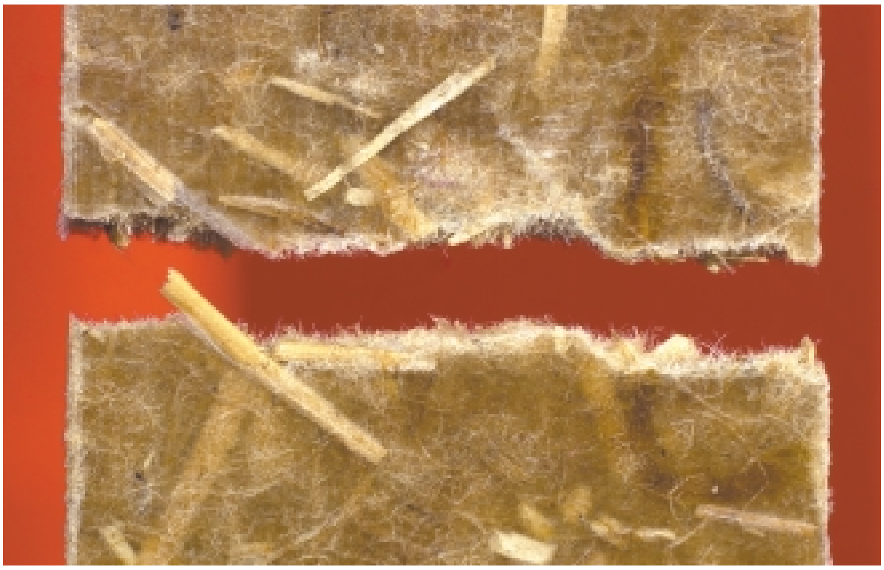
- i reliable calculations of stiffness and strength of plant fibres from experimental composite data,
- ii close correlation between porosity contents as determined by non-destructive measurements (ultrasound, X-rays) and by optical microscopy,
- iii the development and implementation of a new advanced combined end- and shear-loaded test rig for compression testing of fibre composites,
- iv the development of a prototype apparatus for real-time radiographic field inspection of large tubes.

## Properties of Composite Materials

*Characterisation and optimisation of the microstructure (fibre configuration and fibre/matrix interface) of advanced composites is the focus of the programme. The aim is to understand mechanisms of deformation, degradation and failure under mechanical, thermal and chemical loadings. The optimisation relates to the performance of composites in components under service conditions where the components must possess long lifetimes and durability. The materials are composites based on metallic matrices (MMCs), ceramic matrices (CMCs) and polymeric matrices (PMCs). The fibres include carbon, glass, aramid and natural fibres. The composite properties are governed by the microstructures and fibre configurations produced in the fabrication processes. These properties are fundamental to the practical performance of products and components. The activities in this programme are therefore closely related to the activities in other programmes of the Department.*

**Non-destructive characterisation of plant-fibre reinforced polymer composites**

Development and assessment of new non-destructive inspection and characterisation technologies for manufacturing and condition monitoring of fibre reinforced polymer composites is an active research area. The technologies under development are aimed for fast inspection of glass fibre composites, concerning e.g. lay-up defects. An application area in focus is composites based on plant fibres. The work includes the structural assessment and establishment of acceptance criteria for defects. This would lead to industrially applicable inspection methodologies. Emphasis is on radiography using a new high-resolution and high-contrast detector as well as a re-designed lock-in thermography approach, that have been developed in the Department. Software based reconstruction in dual-energy imaging is undertaken to better distinguish between fibres and matrix. This technique is particularly relevant in the case of plant fibre polymer composites, since the fibres and the polymer matrix are relatively similar in elemental composition. An important requirement for the developed technologies is to obtain a quantitative rather than a qualitative non-destructive structural characterisation. For this objective, imaging procedures and



Plant fibre composite panel of flax fibres/polypropylene. A test specimen (20 mm wide) was loaded in tension until failure. The (small) individual fibres can be seen at the fracture zone. The (longer) shives from the flax plant are also seen.

quantitative image processing techniques for radioscopic image acquisition are being developed.

### Properties of plant fibre composites – wheat straw fibres/polypropylene

A long-term aim is to fabricate and characterise composites based on natural fibres, i.e. plant fibres or wood fibres bonded together by a polymeric matrix. The matrix may be a traditional, synthetic polymer or a polymer derived from natural resources. The matrix might be potentially biodegradable and renewable.

The present series of composites are based on the simple approach of using fibres, which are not surface treated, and matrices of conventional thermoplastic polymers such as polypropylene. This will establish a reference for comparison with more advanced fibre treatments and highly developed fabrication processes.

A series of studies were made on materials processed at Risø National Laboratory. Wheat straw was wet oxidised to give fibres of high cellulose content. The fibres were made into mats which were stacked with polypropylene foils before consolidation in an autoclave. The fibres and the composites were characterised by various techniques to get data for chemical composition, cellulose molecular weight, fibre stiffness and strength as well as data for the composite properties stiffness, strength, density and porosity content.

The following key results and properties were recorded. Wet oxidation gave an increase in the cellulose content of wheat straw fibres from 40 % for raw material to 65 - 75 % for treated material. The wet oxidation gave a reduction of the cellulose chain length, with only a minor effect on the stiffness of wheat straw/polypropylene composites. The wheat straw/polypropylene composites had a fibre volume content of about 30 %, and a porosity volume content of 25 - 35 %. The

**Project Funded Research: Materials Engineering**

| Project type / **Project name** | Co-participants |
|---|---|
| Danish Research Councils (STVF, SNF, SJVF) / **Natural Fibres for Polymeric Composites** | • Royal Veterinary and Agricultural University, Denmark<br>• Department of Structural Engineering and Materials, DTU, Denmark |
| The Ministry of Food and Agriculture / **Plant-fibre-based Products** | • Royal Veterinary and Agricultural University, Denmark<br>• Danish Agricultural Advisory Centre, Denmark<br>• Danish Institute of Agricultural Sciences, Denmark<br>• Force Institute, Denmark<br>• Danish Technological Institute, Denmark |
| The Ministry of Environment and Energy / **Wood-fibres, their Structure and Properties with Reference to Composite Materials** | • Royal Veterinary and Agricultural University, Denmark<br>• Institute for Construction and Materials, DTU, Denmark |
| EFP / **Flywheel for Energy Storage-III** | • TERMA Industries A/S, Denmark<br>• NESA A/S, Denmark<br>• DEMEX A/S, Denmark<br>• Institute of Energy Technology, AAU, Denmark |
| Danish Agency for Trade and Industry **Centre for Design of Adhesively Bonded Aluminium Components (CLEA)** | • Hydro Aluminium Tønder, Denmark<br>• LG Trafik A/S, Denmark<br>• Bent Falk Design, Denmark<br>• 3M a/s, Denmark<br>• Teknologisk Institut Århus, Denmark<br>• Arkitektskolen i Århus (AAA), Denmark<br>• Ingeniørhøjskolen i Århus (IHÅ), Denmark<br>• Aalborg Universitet Esbjerg (AUE), Denmark<br>• Alucluster, Denmark |
| EUCLID / **Advanced Techniques for Add-on-Armour** | • Fokker SP, The Netherlands<br>• DEMEX A/S, Denmark<br>• Sistema Compositi, Italy<br>• OTOBREDA, Italy<br>• EN Santa Barbara, Spain<br>• TNO-PML, The Netherlands<br>• Spav, The Netherlands<br>• HB Consultancy, The Netherlands |
| EUCLID / **Long Term Effects on Light Weight Add-on Armour** | • Fokker SP, The Netherlands<br>• DEMEX A/S, Denmark<br>• Sistema Compositi, Italy<br>• DSM, The Netherlands<br>• OTOBREDA, Italy<br>• EN Santa Barbara, Spain<br>• TNO-PML, The Netherlands<br>• Spav, The Netherlands<br>• HB Consultancy, The Netherlands |
| BRITE-EURAM / **Innovative Casting Process of Lighter Steel Components for the Transport Industry** | • Centro Richerche FIAT, Italy<br>• Aerospatiale, France<br>• Ferriere e Fonderie di Dongo, Italy<br>• Gussstahl, Germany<br>• Magma, Germany<br>• RWTH, Germany |
| BRITE-EURAM / **Assessment of Metal Matrix Composites for Innovations** | • Daimler-Chrysler AG, Germany<br>• Centro Ricerche FIAT, Italy<br>• Aerospatiale, France<br>• INTROSPACE GmbH, Germany<br>• EA-Technology Ltd., UK<br>• ISRIM, Italy<br>• Austrian Research Center Seibersdorf GmbH, Austria<br>• CSM Materialteknik AB, Sweden<br>• Technische Universität Wien, Austria<br>• Deutsche Gesellschaft für Materialkunde, Germany<br>• German Aerospace Center, Germany<br>• Universidad Politècnica de Madrid, Spain<br>• Swiss Federal Inst. for Materials Testing and Research, Switzerland<br>• Ecole Polytechnique Fédérale de Lausanne, Switzerland<br>• Institute National des Sciences Appliquées, France<br>• National Physical Laboratory, UK<br>• VTT Manufacturing Technology, Finland<br>• Katholieke Universiteit Leuven, Belgium<br>• University of Cambridge, UK |

*(continues on the next page)*

**Project Funded Research: Materials Engineering (continued)**

| Project type / **Project name** | Co-participants |
|---|---|
| BRITE-EURAM / **Development and Performance Evaluation of a Fast X-Radioscopic and Lock-in Thermographic Non-Destructive Evaluation (NDE) System for Fibre Based Technical Composites (FIBRINS)** | • Photonic Science Ltd., UK<br>• J. B. Plant Fibres Ltd., UK<br>• Carl Bro A/S, Denmark<br>• Sauerwein System Technik GmbH, Germany<br>• VIDROPOL S.A., Portugal<br>• CEDIP S.A., France<br>• Instituto de Soldadura e Qualidade, Portugal |
| BRITE-EURAM / **Thixoforming of Advanced Light Metals for Automotive Components (TALMAC)** | • FIAT SpA, Italy<br>• Volkswagen AG, Germany<br>• Norsk Hydro Aluminium a.s., Norway<br>• Norsk Hydro (Mg Division) a.s., Norway<br>• Pechiney CRV SA, France<br>• EFU AG, Germany<br>• Stampal SpA, Italy<br>• INPG, France<br>• University of Ancona, Italy<br>• SINTEF, Norway |
| BRITE-EURAM / **Hyper-Eutectic Alloys for Automobile Components (HAforAC)** | • Bosch Systemes de Freinage, France<br>• FIAT SpA, Italy<br>• Norsk Hydro Aluminium a.s., Norway<br>• Stampal SpA, Italy<br>• Pechiney CRV SA, France<br>• Fagor Ederlan, S. Coop. Ltd., Spain<br>• CEIT de Guipuzcoa, Spain<br>• Allied Signal Bremsbelag GmbH, Germany<br>• ISRIM, Italy<br>• University of Sheffield, UK |
| BRITE-EURAM / **Development of a Portable Remote Controlled Real-Time Radioscopy System for Quantitative Industrial Inspection of Large Thickness Steel Pipes and Weldings (RAYSQINS)** | • Statoil a.s., Norway<br>• Carl Bro A/S, Denmark<br>• Photonic Science plc, UK<br>• Thomsen Tubes Electroniques SA, France<br>• Sauerwein System-Technik GmbH, Germany<br>• Bundesanstalt für Materialprüfung, Germany<br>• Instituto de Soldadura e Qualidade, Portugal<br>• Institut de Soudure, France |
| BRITE-EURAM / **Graintwist** | • Plansee, Germany/Austria<br>• Mitsui Babcock Energy Ltd., UK<br>• Metall-SpecialRohr GmbH, Germany<br>• University of Cambridge, UK<br>• University of Liverpool, UK |
| EUREKA / **EUROBOGIE** | • EM-Fiberglas a/s, Denmark<br>• Skoda Research, Czech Republic<br>• Bombardier Talbot, Germany<br>• Sciotech, UK<br>• Polymath, UK<br>• English Welsh Scottish Railways, UK<br>• Concargo/Polymer Engineering, UK<br>• SVUM, Czech Republic<br>• IPM, Latvia<br>• Reading University, UK |
| BRITE-EURAM / **Action for Low Weight Automobile Technologies (FLOAT)** | • Rover Group Ltd., UK<br>• GIE Renault, France<br>• FIAT SpA, Italy<br>• Volvo AB, Sweden<br>• Daimler-Chrysler AG, Germany<br>• Austrian Research Center Seibersdorf GmbH, Austria<br>• Fraunhofer Institut, Germany<br>• SEPARIS, France<br>• ISRIM, Italy |
| BRITE-EURAM / **Plant Life Assessment Network (PLAN)** | Thematic Network |

mechanical properties of the composites demonstrated a clear reinforcing effect by the wheat straw fibres, giving an increase of stiffness compared with pure polypropylene. The stiffness and strength values for the wheat straw fibres were comparable with, but slightly lower than those of other natural fibres.

### Measurement and calculation of effective stiffness and strength of plant fibres

Important parameters for design of composite materials with given properties are the stiffness and strength of the fibres. For conventional composites with synthetic fibres such parameters are generally known from tests of single fibres or fibre bundles. For composites based on plant fibres this route has so far not been exploited, partly because of the rather large variability of the fibres, due to origin, handling and characteristica such as variation in diameter, length and branching. It is therefore of interest to arrange for other indirect methods to estimate such fibre properties.

The approach used for plant fibre composites is based on experimental data for composites, and recalculation via composite theory for stiffness and strength. The fibre orientation is included in the model as well as the effect of porosity. Extensive series of composite panels were fabricated for plant fibre composites with jute or flax fibres in a polypropylene matrix. The composite panels were made with a rather wide range of porosities, to allow for safer and more accurate results from the data fitting procedure.

The results of the calculation are the fibre orientation parameter and the fibre strength and stiffness. The resulting values represent average effective values for the functioning of the fibres as reinforcements in the composites.

Test fixture (25 cm wide) for evaluating stiffness and compressive strength of thick laminates made by fibre reinforced plastics.

| Fibre | Density [g/cm$^3$] | Stiffness [GPa] | Strength [MPa] |
|---|---|---|---|
| Jute | 1.5 | 47 ± 3 | 300 ± 20 |
| Flax | 1.5 | 39 ± 3 | 200 ± 20 |
| Glass | 2.6 | 70 | ~ 2000 |

These values are comparable with or a little lower than those reported in the literature. The values for glass fibres are shown for comparison. Although the stiffness and strength are higher on an absolute scale, the ratio between stiffness and density is of the same magnitude. This has positive implications for the use of plant fibres (cellulosic fibres) in materials for light-weight components.

### Compressive strength of thick laminates

Large composite structures made by polymer matrix composite laminates (thickness 5 - 60 mm) are difficult to lay-up, and fibre misalignment is sometimes encountered in thick laminates. The compressive strength is very sensitive to fibre misalignment. Therefore, there is a need for a test method where the compressive strength of thick laminates can be tested and scale effects due to processing can be investigated.

Common methods for compression tests are based on transferring the axial compressive load to the specimen by either end loading or shear loading through tabs. The drawback of these methods for thick composites is that the specimen experiences premature failure due to crushing of the ends or large stress concentrations in the transition zone between tabs and gauge area. A compression test fixture employing combined end and shear loading was developed to avoid these problems.

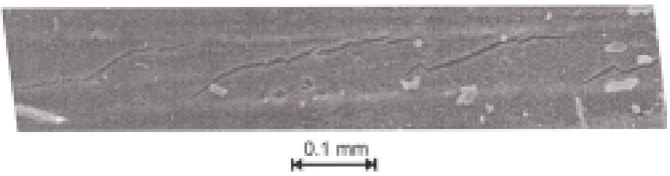
The ratio between shear and end loading is at present constant (1 : 4) and controlled by a hydraulic system. The fixture can accommodate specimens up to a width of 15 mm and a thickness of 25 mm. The gauge length can be varied according to needs. The maximum load is 250 kN. The method is a fast, reliable and operator-independent technique to determine the compressive strength. The technique has been verified by tests on glass, carbon and glass/carbon fibre composites ranging in thickness from 5 to 10 mm. The fixture was developed in a research project on new materials for rotor blades for wind turbines. It is currently being used at the Department as the preferred test method for evaluating compressive strength of polymer matrix composites.

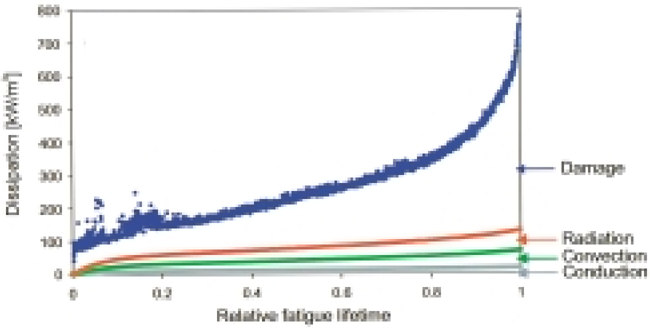
### Quantitative dissipation measurements of fatigue loaded composites

A thermographic experimental set-up was used to analyse the evolution of the temperature field on the surface of polymer matrix composite specimens during cyclic loading. The specimen, which is fastened by the grips of the tensile machine, is enclosed in a well-insulated chamber to allow quantitative analysis of the generated temperature field.

Meanwhile, the mechanical loss is measured by an extensometer. The chamber is also equipped with multiple thermocouples, and options for acoustic emission measurements and control of temperature of the grips and chamber walls. The mechanical losses (extensometry) can then systematically be compared with the heat dissipation by radiation (thermovision), conduction (specimen and grip temperature measurements) and convection (specimen and ambient air temperature). The difference between mechanical loss and heat dissipation can be attributed to microstructural change through damage formation and propagation.

This method has been used to investigate the fatigue degradation and failure of glass/carbon fibre hybrid composites. One result was that heat and damage developed at steady-state with a homogeneous spatial distribution during almost the entire fatigue life. Just imminent to ultimate failure, damage localisation occurred. The localised damage zone propagated rapidly and resulted in final failure. The interest is now directed toward the parameters that control this transition from a distributed to a localised damage state. This can give insight in how the fatigue resistance of the material can be improved from a microstructural point of view. The thermovision equipment plays an important role in this investigation. Together with the instrumented chamber, this set-up provides an excellent tool to




Mechanical dissipation during fatigue of a polymer-matrix composite partitioned into damage formation and various forms of heat dissipation.

quantify the temperatures and hysteresis losses, which is necessary information to conceive predictive models for fatigue degradation and failure.

Accurate temperature measurements are particularly important for polymer matrix composites, since fatigue damage propagation and heat generation have a strong mutual influence, which in turn control the macroscopic properties.

### Modelling of large scale bridging in composites

Crack growth in a plane parallel to the fibre direction can be accompanied by fibre bridging due to misaligned fibres. Fibre bridging is beneficial as it significantly increases the fracture resistance with increasing crack extension (R-curve behaviour). The length of the bridging zone often becomes large in comparison with the specimen dimensions. This is called large scale bridging (LSB). Under LSB the stress intensity factor looses its significance as the loading parameter that connects the overall load to the fracture process zone.

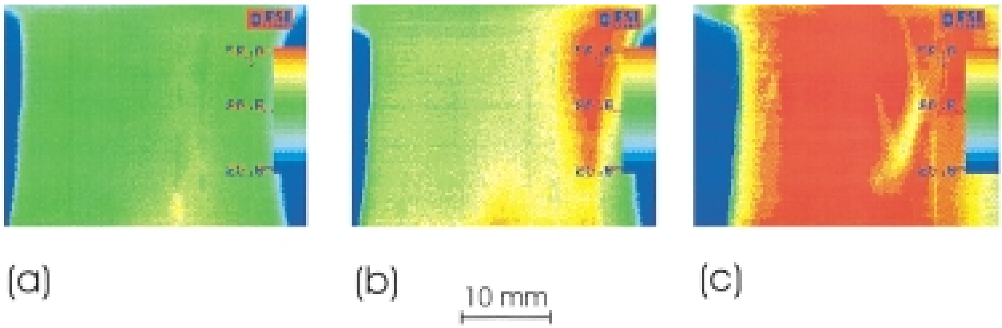
Instead, the path independent J integral can be used to connect the overall (external) load to the process zone. However, evaluating the J integral along the external boundaries of a specimen subjected to LSB usually gives a result that differs from that known from linear elastic fracture mechanics. To elucidate this point, a simple analytical model was developed for a double cantilever beam specimen loaded with wedge forces. The results clearly show that under LSB the nominal fracture resistance (i.e. calculated on the basis of linear elastic fracture mechanics) overestimates the fracture resistance (given by the J integral) significantly.

A numerical study by the finite element method was carried out to model the effect of fibre bridging accurately. The relationship between the crack opening and the bridging stress, the so-called bridging law, had earlier been derived from experiments. In the finite element model the bridging law was implemented by non-linear springs. A very good agreement was found between predicted and measured R-curves, including strong effects of specimen geometry. This result lends confidence to both the measurement approach and the modelling approach. The bridging law can be considered to be a material property.

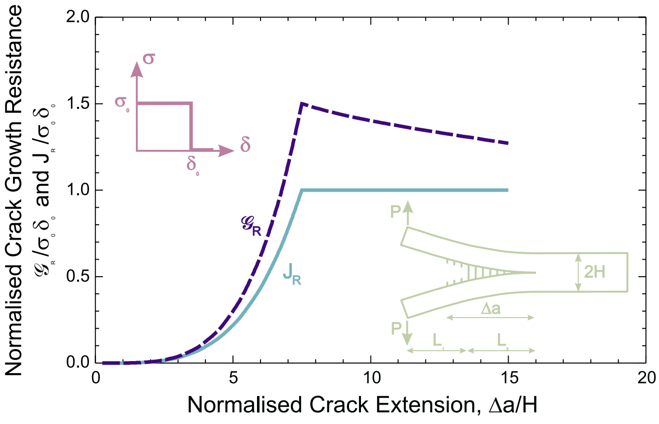
## Mechanical Characterisation and Design of Components

*The programme covers non-destructive and destructive techniques for characterisation of materials and components as well as design and development of specific components. The non-destructive characterisation techniques cover ultrasonic, X-ray, thermography and acoustic emission techniques. Ultrasound is used both for traditional defect detection and for determination of material properties and the influence of environmental effects. A real time X-ray technique for online inspection of heavy steel tubes was developed within the framework of an international programme. Thermography and acoustic emission were used for damage detection in both fundamental materials research and in the development of components.*

Mechanical testing of materials was performed on a commercial basis for several Danish and European companies in parallel with the testing performed within the Department's own project activities. The materials were both traditional metallic engineering materials (tooling materials, aluminium, steel), composite materials




Evolution of temperature field during fatigue of a polymer-matrix composite: (a) first 99 % of fatigue life, (b) localisation prior to failure, and (c) fatigue failure.




Comparison between fracture resistance curves calculated (incorrectly) by linear elastic fracture mechanics, $G_R$, and (correctly) by the J integral, $J_R$. The nominal fracture resistance $G_R$ is significantly higher than the true fracture resistance $J_R$. Unlike $J_R$, $G_R$ does not attain a steady-state value.

(metal matrix composites, polymer matrix composites, natural fibre composites) and elastomers. The amount of commercial testing has increased significantly during 1999, due to the testing facilities and expertise of the Department, a more active marketing effort in this area and an increase in the demand from the industry to have the engineering materials certified.

Projects related to design of components involve rotor blades for wind turbines, composite leaf springs for railway bogies, ballistic add-on armour based on ceramics and polymer matrix composites, adhesively bonded aluminium components and forging die tools.

### Mechanical testing

The Department carries out mechanical testing in connection with programme research contracts and directly for industrial clients. The test results provide the basis for modelling of mechanical properties and of the influences of microstructure. It also serves as a necessary input for industrial design and material qualification.

The increasing use of advanced materials means ever-greater demands on testing techniques. As a result, the mechanical testing laboratory of the Department has developed advanced testing techniques for these materials. During the year special attention has been focussed on compression loading of polymer matrix composites and on fatigue loading. The test activities have expanded and more equipment (two more servohydraulic test machines) has been installed.

The mechanical testing laboratory has the equipment and experience to carry out testing on all types of materials in different environments and at non-ambient temperatures (-196 °C to 1200 °C) under all types of axial and torsional loading. The laboratory is accredited (Reg. no. 210) by DANAK (Danish accreditation body) to carry out mechanical testing, and an increasing number of customers have utilised this service during the year.

### Hypereutectic alloys for automotive components

The importance of advanced light alloys to the transport sector has prompted activities in the development of new alloys and processing techniques for automotive components. The Department is involved in a project focussed on tailoring of specially wear resistant hypereutectic silicon/aluminium alloys for components such as brake drums, brake discs, and motor blocks to be produced via thixoforming, squeeze casting and lost foam techniques in a cost effective way. Microstructural, mechanical and X-radiographic characterisation of a large number of raw and processed alloys have been performed providing information on the composition-processing relationships which allows the selection of suitable alloys.

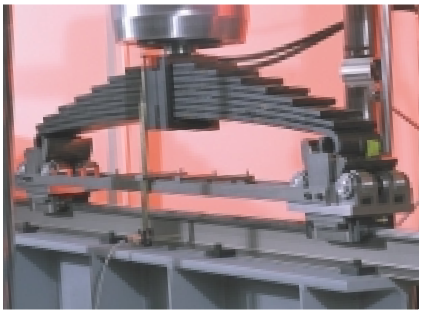
### Metal matrix composites: Thixoforming, microstructure, mechanical properties and quality control

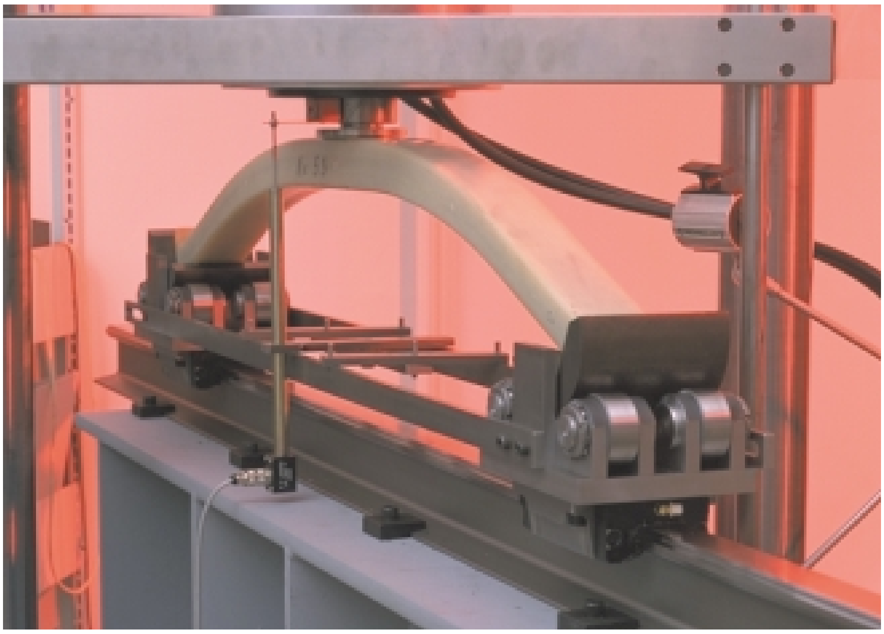
Light materials are of great importance for the transport sector and in particular for the automobile industry. The focus has been on minor components in personal cars manufactured by thixoforming techniques: rear axle supports, break drums, engine brackets and suspension arms. The typical weight of these components is 2 - 4 kg and the goal is a reduction to 1 - 2 kg.

The materials considered were aluminium alloys, aluminium – metal matrix composites and magnesium alloys. The thixoforming process was investigated both experimentally and theoretically to understand the influence of the composition on the rheological behaviour of the semi-solid slurries. The practical experiments were performed on simple generic shapes to gain experience under pilot plant conditions. The work at the Department has focussed on quality control and mechanical characterisation of the thixoformed materials.

The material quality was evaluated by X-radiography and described in terms of microstructure and defects. A close correlation was demonstrated between X-ray measurements and optical microscopy of the porosity content and distribution. The two-line scan profiles across the porosity-region obtained by the two methods showed good agreement. This can be used as validation and calibration for the non-destructive method. Regions of globular grain structure, segregation of eutectics, particle distribution, as well as defects such as voids, porosities and cracks were identified. These observations were of importance in elucidating the effect of microstructure on the rheological behaviour of the alloys in their semi-solid state during thixoforming.

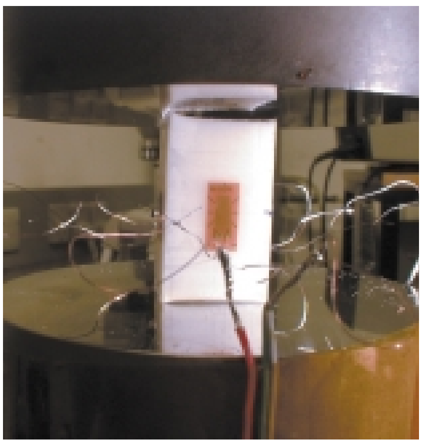
The mechanical properties were measured under tension and under fatigue





The development work on polymer composite leaf springs aims at replacement springs for existing conventional steel springs. In order to establish data for comparison, both new and used conventional steel springs were tested (Length of springs ~1.2 m).

Out-of-plane compression of composite material between parallel platens at low to medium strain rates. The specimen width is 20 mm.



loading. These measurements both served as documentation of the material quality and as the basis for design allowables. In particular new data for the fatigue behaviour of Mg-alloys (AZ91) were established, where a preliminary fatigue limit of about 50 MPa was found.

### Advanced vehicle suspension using fibre-reinforced plastics

Polymer matrix composites are potential materials for suspension elements in road and rail vehicles such as springs and frame elements. They possess good damping and fatigue properties, and they can sustain high strains. A replacement leaf spring is developed for existing railway freight vehicles and for a new bogie frame for the same type of vehicles. The development is focussed on both design and manufacturing, aiming at low costs which will enable this type of springs and bogies to be manufactured competitively. Polymer composite leaf springs for taxi-cabs and large trailers were successfully developed and validated, and industrial production of these springs is now starting up.

The main activities were to characterise the materials manufactured by resin transfer moulding (RTM) and to relate the mechanical properties such as static strength and fatigue performance to the manufacturing parameters. Testing was performed on the leaf spring and spring-like components as well as on smaller coupons cut from the spring.

An important advantage of polymer matrix composites compared with steel is the higher material damping property of the composites, leading to lower dynamic forces between wheel and rail or road at the same speed. This results in less wear on rails or roads. The ride comfort is also higher and maintained throughout the lifetime of the spring, and both internal vehicle noise and environmental noise will be lower.

### Armour materials

Improvement of existing main armour of a personnel carrier was investigated. The add-on armour is placed on the outside of the carrier and consists of ceramic tiles bonded with adhesives to a plate of aluminium or of thermoplastic polymer matrix composite. Optimisation is based on numerical simulations of high-speed impact, requiring an adequate description of the mechanical properties of the ceramic, adhesive, metal and composite materials.

To acquire this, a number of different tests have been performed. The ceramic tile materials were characterised by their flexural strength and fracture toughness. The adhesive materials were tested in tension at different strain rates, and different adhesive/ceramic interfaces were investigated with regard to fracture toughness. The composite materials (high-density polyethylene fibres in an elastomer matrix) were tested at low strain rates in tension, and at different strain rates in shear as well as in compression.

High temperature is likely to change the material properties and the effectiveness of the ballistic armour. Such effects are being evaluated by mechanical tests on armour components after exposure to different environments. After conditioning at various combinations of temperature and moisture, the following properties are evaluated: adhesive/ceramic fracture toughness as well as composite tensile, shear and compression properties. Also the effect of low energy impact (e.g. a tool dropped at low height) will be investigated in terms of change in adhesive and composite properties.

Real time X-ray radioscopic system developed for inspection of large and extensive pipe systems. The diameter of the pipe is about 30 cm. The severity of corrosion of pipes can be measured accurately with the integrated image processing functions.

### Grain twist in commercial steels

The general demand for renewable energy sources has incited development of efficient biomass plants. A high efficiency of power generation in such plants requires the development of heat exchangers capable of gas operating temperatures and pressures of around 1100 °C and 15 - 30 bar, respectively. This, in turn, demands metal operating temperatures up to 1150 °C for the tubing. In collaboration with research partners the Department seeks to develop a tube which offers a sufficiently high creep and oxidation resistance under these conditions.

A coarse grained oxide dispersion strengthened (ODS) steel formed by mechanical alloying is the starting material whose properties are to be optimised. Conventional tubes made from ODS steels have very large grains which are elongated in the tube axis, resulting in excellent axial creep properties.

However, for pressurised tubes, the main goal is to have the grains elongated in the hoop direction so that a high creep resistance is obtained in that direction. The aim is to produce such a microstructure through flow forming techniques followed by controlled annealing.

In order to further the understanding of the grain formation after annealing, local texture measurements using electron back-scattering patterns on the flow formed tubes in the as formed state and after heat treatment were performed. It was shown that the deformed tubes have a texture that varies strongly over the tube thickness. A <100> fibre texture is seen in the surface layer, a virtually random texture is seen in the centre of the tube wall, whereas the inside of the tube wall has a texture dominated by the rotated cube. After heat treatment, the textures sharpen. Torsion deformation tests were performed at 800 °C, and creep tests at 1100 °C. The relations between microstructure, texture recrystallisation behaviour and mechanical properties have been investigated.

### Quantitative X-ray techniques in industrial condition monitoring

An innovative near real-time high energy radioscopic inspection system has been developed. The system incorporates radioscopic inspection of large pipes and other large structures, with particular emphasis on quantification of structural details. The Department has been involved in the detector characterisation and in the development of procedures to determine the pipe wall thickness based on differences in attenuation and on tangential measurement techniques. An expert system to guide the operator towards the efficient use of the equipment was also developed.

Apart from the detector, the system integrates a special isotope camera and a versatile remotely controlled robot with a controlling system involving an expert system. The system also has capability of three-dimensional imaging, an option for stereoscopy and software for image enhancement and for pipe wall thickness calculation.

### Light and elegant constructions of aluminium using adhesion technology

The possibilities of using a combination of adhesion and aluminium profiles in relation to dynamically loaded constructions are being investigated. This is part of an overall solution of adhesion technology related to aluminium constructions from idea and design over production to the problem of disposal.

The Department investigates the mechanical properties of adhesive assemblies, such as strength, fatigue and environmental effects. The Department also introduces non-destructive monitoring (ultrasonics) techniques in production control. These results are used by Danish companies, designers as well as research and educational institutions, who cover expertise in aluminium production, adhesion technology, design, materials and education in the area.

# Materials Technology

**– synthesis, processing and product**

*The work described in this chapter comprises three programmes: Manufacturing Technologies for Composite Materials and Powder Technological Materials, which deal with the development of new materials for a variety of applications for instance the energy and transport sector, and Materials Chemistry which is mainly directed towards solid oxide fuel cells (SOFC) and related electrochemical devices. In the first two programmes, projects are often pursued in collaboration with industry. Consequently, goals are often set not more than three years ahead. These activities are usually initiated by needs or problems encountered in industry, but conceived to be better solved within the research environment of the Department. Much of the research in Materials Chemistry concerns electroceramics and spans all the way from synthesis and characterisation of cell materials, over manufacture of cells, to assembly and testing of small cell stacks. Fundamental research supporting the technological development is carried out in electrochemistry, solid state chemistry and ceramic processing. The contribution of PhD research projects is highly valued in the above areas.*

**Highlights in these programmes are:**

i vacuum consolidation of thermoplastic fibre composites at a 180 °C and one-shot vacuum consolidation of a complete section of a wind turbine blade,

ii development of a light-weight pressure vessel for hydrogen storage,

iii experimental verification of a numerical model for the shape and temperature profiles of spray-formed low alloy steel,

iv determination of the critical cooling rate for a glass-forming alloy,

v development of a solid oxide fuel cell with an anode-supported thin ceramic electrolyte, having total internal resistance of 0.3 Ω $cm^2$ at 850 °C,

vi successful testing of ‘short stacks’, i.e. single repeating stack units, incorporating interconnector plates of ferritic stainless steel,

vii formulation of improved defect chemistry models including a model for calculation of oxygen diffusion coefficients from conductivity relaxation experiments.

## Manufacturing Technologies for Composite Materials

*This programme covers the development and optimisation of new thermoplastic polymer matrix composites (PMCs) and associated process technologies. The aim is to improve occupational health and application properties, and to characterise the materials produced. Special emphasis is on the development and study of three processing technologies: (i) vacuum consolidation, (ii) autoclave consolidation and (iii) press consolidation. For economical and practical reasons, the three technologies are most suitable for different purposes and applications. Vacuum consolidation is suitable for fabrication of larger parts, such as wind turbine blades, autoclave consolidation is suitable for high performance application where weight optimisation (high fibre content) is important, and press consolidation is a fast process suitable for smaller parts such as automobile body parts. Optimisation of the pre-processed raw materials for the different technologies and component types is an important aspect of the work.*

### Vacuum consolidation of thermoplastic composites for wind turbine blades

The melt viscosity of thermoplastic polymers can be as high as several thousand Pa s, which means that it is difficult for the molten plastic to penetrate the fibre bundles and ensure complete wetting of all individual fibres. For the manufacture of a wind turbine blade it is desirable that the consolidation should take place at a low pressure. The processing time is not of primary importance. Therefore, vacuum consolidation of fabrics based on commingled fibres seems to be an attractive and a feasible process technology for wind turbine blades. The vacuum has a multiple function: it consolidates the laminate, it removes the air between the fibres, producing a laminate of high quality and low porosity and, finally, it protects the polymer from degradation during the relatively long time it spends in the molten state.

The process window for a 7 hours process cycle was established for glass/polypropylene commingled fibres. Laminates were consolidated at different temperatures in the range from 170 °C to 225 °C. High materials quality and also high fatigue properties were obtained by process temperatures above 180 °C, while the fatigue properties were significantly inferior with a process temperature of 170 °C.

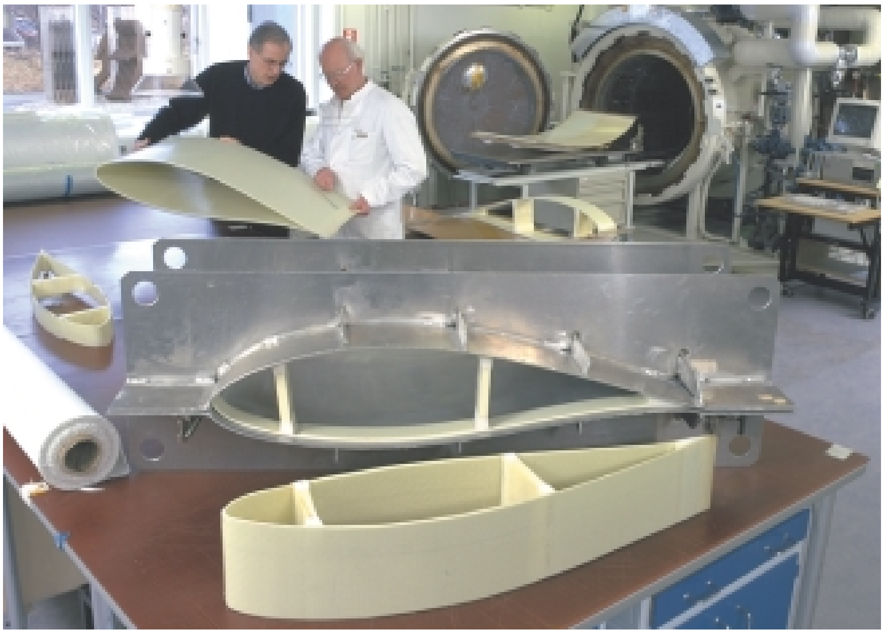
In a metal mould, a smaller but complete section of a blade consisting of an upper skin, a lower skin and two integrated shear webs, was successfully vacuum consolidated in one shot. This manufacturing technology avoids the subsequent assembly of the parts.

### Improvement of quality of plant fibre composites

During the compaction of jute fibre mats composed of short fibres, the fibre volume fraction saturates at a certain compacting pressure. For dry mats, the remaining volume is filled by air. It is not possible to compact the fibre assembly to an arbitrarily high volume fraction of fibres simply by increasing the pressure. For

**Project Funded Research: Materials Technology**

| Project type / **Project name** | Co-participants |
|---|---|
| Danish Agency for Trade and Industry / **Centre for Powder Metallurgical MMC-Materials (COMPOMET)** | • Danish Steel Works Ltd., Denmark<br>• Roulunds A/S, Denmark<br>• Norsænk-Aalykke A/S, Denmark<br>• A/S Hartfelt & Co., Denmark<br>• Scan-Visan A/S, Denmark<br>• Thürmer A/S, Denmark<br>• Danish Technological Institute (DTI), Denmark |
| MUP / **Electro-Ceramic Functional Graded Materials** | • AMP, Denmark<br>• Haldor Topsøe A/S, Denmark<br>• PBI-Dansensor A/S, Denmark<br>• Chemical Institute, KU, Denmark |
| Nordtest / **Fractography of Cyclically Deformed Alumina Ceramics** | • Swedish National Testing and Research Institute, Sweden<br>• University of Tampere, Finland |
| EFP98 / **Low Weight Storage for Hydrogen Cars** | • IRD A/S, Denmark |
| JOULE-THERMIE / **New Generation Wind Turbine Blade** | • Bonus Energy A/S, Denmark<br>• Garrad Hassan & Partners, Ltd., UK<br>• Kemijoki Yo, Finland<br>• VTT, Finland |
| EFP / **DK-SOFC 1996-1999** | • Haldor Topsøe A/S, Denmark<br>• IRD A/S, Denmark<br>• Institute of Chemistry, OU, Denmark<br>• Institute of Chemistry, DTU, Denmark |
| BRITE-EURAM / **Low Cost Fabrication and Improved Performance of SOFC Stack Components (LOCOSOFC)** | • Rolls-Royce, UK<br>• IRD A/S, Denmark<br>• INPG, France<br>• Gaz de France, France<br>• NUVL, UK<br>• Swiss Federal Inst. for Materials Testing and Research, Switzerland |
| Training and Mobility of Researchers Programme / **Alternative Anodes for SOFC** | • British Gas, UK<br>• University of St. Andrews, UK<br>• University of Aveiro, Portugal<br>• University of Patras, Greece<br>• University of Twente, The Netherlands<br>• Research Centre Jülich, Germany |



Sections of a wind turbine blade with and without integrated webs were manufactured in glass fibre reinforced polypropylene. The commingled fibre fabrics were vacuum consolidated at 180 °C for 7 hours to simulate a long process cycle for a larger component.

the jute fibre mats investigated, the saturation fibre volume fraction is about 30 %, corresponding to a weight fraction of about 40 %. A model for the compaction of fibre mats based on this constraint leads to a distinction between structurally governed porosity, which is present irrespective of the processing conditions and a process-governed porosity, which can be eliminated by modifying these conditions.

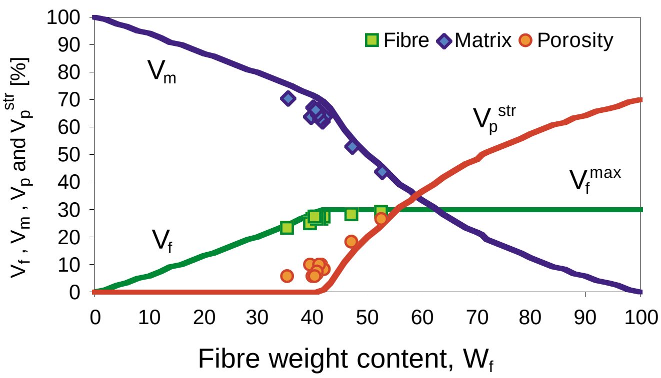
Experiments on jute/polypropylene composites support the model and identify the process-governed porosity at a level of 5 to 10 %, independent of fibre content. When the weight fraction of fibres is below the saturation value, the porosity is process governed. When the saturation value is exceeded, structural porosity is dominant. Improvements in the quality of short fibre composite materials should, therefore, be aimed at process optimisation at low fibre contents and at fibre mat improvement at high fibre content.

### Fibre composite pressure vessel for hydrogen storage

Within a Danish hydrogen-car project, one of the options for the storage of hydrogen onboard cars in in the form as a compressed gas. Commercially available carbon fibre reinforced aluminium pres-

Light and strong fibre reinforced pressure vessels represent an attractive solution for the storage of hydrogen for hydrogen fuelled cars. A polymer lined glass fibre pressure vessel was developed and used for investigation of hydrogen diffusion through the cylinder wall.

Fibre volume fraction $V_f$, matrix volume fraction $V_m$, porosity $V_p$, and structural porosity $V_p^{str}$ as a function of fibre weight content $W_f$ for a jute fibre/polypropylene composite with a maximum fibre content of $V_f^{max}$ = 30 vol. % corresponding to a $W_f^{max}$ of approximately 40 wt %. The full line indicates the model prediction.




sure vessels with a working pressure of 30 MPa are used in the test vehicle. A metal liner inside the pressure vessel ensures hydrogen tightness, but also increases the weight considerably. For example, a 9-litre pressure vessel has a mass of about 5 kg, of which 50 % is due to the metal. To reduce the weight, pressure vessels with polymer and metal-coated polymer liners are under development. If the metal liner inside the 9-litre tank is replaced with a polymer liner, the total weight will be reduced by 40 %. Polymer liners are not, however, 100 % impermeable to hydrogen - the gas slowly diffuses through the liner and the load bearing fibre composite layer. To minimise this, a polymer with good barrier properties against hydrogen is used. Preliminary measurements on polymer lined glass fibre composite pressure vessels of 0.4 litre volume are promising. A hydrogen loss of approximately 0.15 g per month was measured at 10 MPa. An equivalent loss of hydrogen for the 9-litre container was estimated to be 0.8 % over one month for a full tank at 30 MPa. For further reduction of the loss of hydrogen, some polymer liners have been metal-coated. Weight loss measurements are in progress.

## Powder Technology Materials

*This programme is concerned with theoretical and experimental aspects of the production of metal powders and of materials from metallic and ceramic powders. Optimisation of spray forming equipment in parallel with numerical simulation of the spray forming process, were important tasks. The materials covered include metal matrix composites for applications where wear and friction resistance are critical.*

The work on ceramics focussed on optimisation of the fabrication techniques for oxygen sensors and on the development of new techniques for the fabrication of electronic noise filters. A project was initi-


Presentation of fuel element number 1000 in connection with the jubilee.

**Manufacturing of low enriched fuel elements**

In September 1999 the Department celebrated the delivery of fuel element number 1000 to the Danish reactor DR3 and the 25th anniversary of Danish fuel element production. Previously, fuel elements had been bought abroad, but a new fabrication procedure developed in the Department made it economically attractive to produce elements in Denmark.

Over the years, the total economic benefit has been considerable. The design of the elements has not been substantially changed during the years. It is still based on concentric tubes made from sandwich fuel plates with a core containing the uranium. In the late eighties the enrichment of the uranium used for the elements was reduced from high (HEU – high enriched uranium) ~ 90 % to low (LEU – low enriched uranium) ~ 20 % $^{235}U$. This made it necessary to develop a new procedure for production of fuel plates with a ceramic core. Today the cores are cold-pressed bricks made of a powder mixture of $U_3Si_2$ and Al. In 1999, 38 elements were delivered.

ated on development of an electronic system based on ceramic gas sensors for control of industrial processes.

**Hydrogen storage in metal hydrides**

Several mobile hydrogen storage systems for fuelling vehicles are being tested today in various prototypes and demonstration projects all over the world. The hydrogen may be stored in a tank as pressurised gas, liquefied, or absorbed in a metal hydride. The latter method is one of the safest: in case of an accident the hydrogen will be released only slowly from the tank. Furthermore, the storage efficiency can be very high. The Department, in collaboration with industrial and university partners, is involved in the development of a hydrogen storage tank based on metal hydrides for a passenger car with a hydrogen fuelled internal combustion engine. In order to identify storage media that can operate at temperatures provided by the engine cooling system (80 to 90 °C), a number of hydride-forming alloys are being characterised using a high-pressure microbalance. This provides knowledge of activation conditions, reversible storage capacity, working pressures and temperatures, hydriding and dehydriding kinetics, and sensitivity to impurities.

**Metal matrix composites containing graphite**

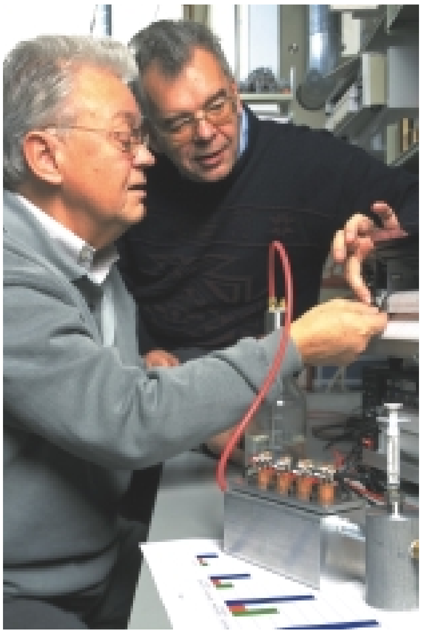
Sintered metal matrix composites (MMCs) containing more than 60 vol. % graphite were developed as a friction material for truck disc brakes. These materials are capable of working at higher temperatures and heat dissipation rates than the conventional polymer-based friction materials. Furthermore, the new materials are more benign to the environment. Efforts have focussed on the behaviour of the individual constituents and of the composites under conditions simulating processing and use. The systems were characterised using X-ray diffraction, thermal analysis, dilatometry, electron microscopy and optical microscopy.

The composites are produced by sintering of uniaxially pressed metal fibres, together with metal, graphite, and ceramic powders. This produces a very anisotropic expansion during binder burn-off and sintering and a very anisotropic microstructure. The graphite basal planes are preferentially oriented perpendicularly to the pressing direction. The expansion of the ‘green’ composite on heating appears to be associated with the burn-off of binder. Irrespective of the heating rate, the major part of the expansion ends at 500 °C. The observed expansion is explained by the release of residual stresses in the structure caused by binder disappearance and softening of the metallic phases. This also explains the large difference in dimensional changes between the directions parallel to the pressing direction (~ 10 %) and perpendicular to it (~ 1 %). Furthermore, the study has elucidated possible reactions and transformations during binder burn-off and sintering under various conditions, providing a basis for processing guidelines.

**Spray-forming of steel based composites**

The aim of producing MMC materials is to combine attractive properties of different materials. However, it is often difficult to achieve the potential benefits from MMCs because of problems like segregation of second phase particles, weakness of interface bonding, porosity etc. The spray forming process has the potential to cope with these problems, and the short processing route, compared with traditional powder metallurgy, makes the process interesting for industry.

Steel-based composites containing $Al_2O_3$ particles were produced in the spray forming equipment of the Department. The application areas for such materials are tools used in agriculture, fishing



The first electronic nose constructed at the Department for odour sensing.

and forestry, where the requirement for wear resistance is high. Adding ceramic particles to steel usually causes embrittlement. To ensure a satisfactory ductility, low alloyed boron steel was chosen as the matrix material.

The matrix material was spray formed by a traditional two-nozzle system. The particles were mixed with a carrier gas outside the spray-forming container and injected into the spray cone by a nozzle placed just underneath the atomisation nozzle. Because of the rapid solidification, segregation of the particles was avoided and a homogeneous distribution of particles was established in the main part of the deposit. The matrix surrounded the particles tightly and there was only limited particle-particle contact. This morphology is understood to be beneficial for the mechanical properties.

Tensile tests showed a decrease in tensile strength and ductility compared with the pure matrix material, but the results were favourable compared with other steel-based composites. The decrease is caused by a crack initiating effect from the particles combined with the smaller tensile strength of the reinforcing particles. Four different materials were compared in a dry sand/rubber wheel abrasion wear test: (i) pure cast matrix material (ii) pure spray formed matrix material (iii) boron steel with 6 vol. % $Al_2O_3$ particles with an average particle size of 43 µm and (iv) boron steel with 7.5 vol. % $Al_2O_3$ particles with an average particle size of 135 µm. The samples were hardened and half of them were subsequently tempered. The best results for the pure materials were obtained in the hardened and tempered state. Conversely, the best results for the composite materials were achieved in the fully hardened state where an increase in wear resistance of up to 300 % was measured. The best result was achieved with the small particles, which indicate that bonding and support of the particles is the limiting parameter at present. Further improvement in the wear resistance is expected by the use of a harder matrix.

### Modelling of spray-forming

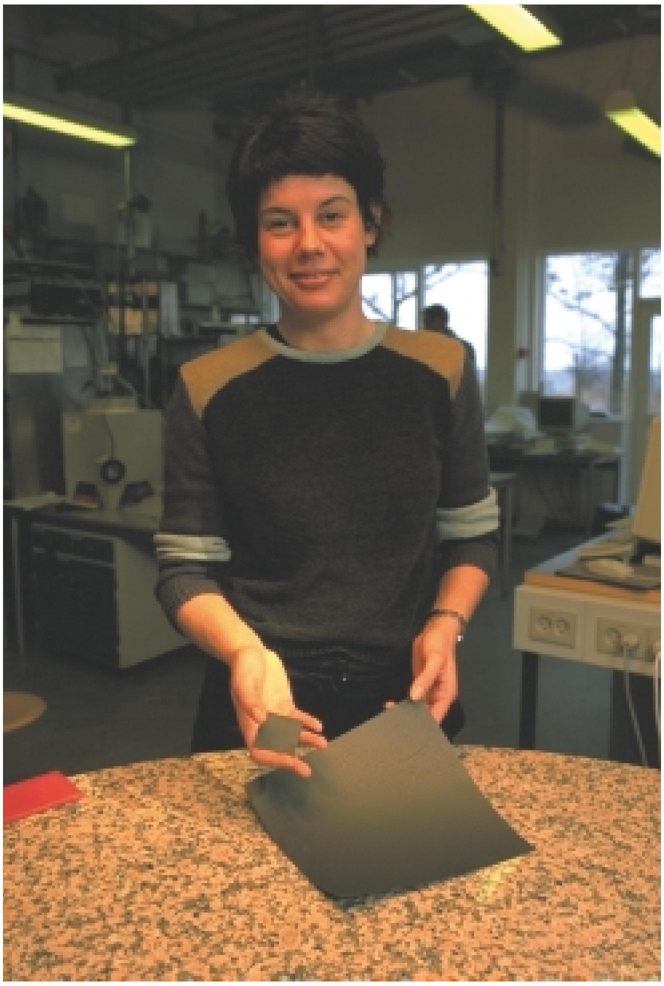
In spray deposition, a molten metal stream is dispersed into droplets using high-energy gas jet. The spray of droplets is directed onto a substrate and consolidated to produce a semi-finished product known as preform in a single processing step. This involves mass transfer, heat transfer, momentum transfer, fluid flow, solidification and undercooling and it is governed by variables such as the droplet size and temperature distribution. All these processes affect the microstructure of the deposited material. An integrated approach for modelling the entire spray forming process was developed. A model developed earlier, which includes the interaction between an array of droplets and the enveloping gas, was coupled to a newly developed deposition model, involving a 2D cylindrical heat flow calculations based on the finite volume principle.

In literature, the coupling between the atomisation and the deposition models has been accomplished using the average temperature of the particles arriving at the surface as the input temperature for the deposited material. However, in real spray forming, the position of powder particles arriving at the surface varies according to particle size: large particles arrive mainly at the centre of the deposited material while smaller particles arrive near the periphery. To overcome the limitation of the previous models, the present model uses an atomisation model with a specific droplet size distribution. These two models are coupled to ensure that the total size distribution is in fact the sum of the 'local' size distributions defined along a radius of the deposited material. The 'local' temperature is then calculated based on the size and temperature of particles arriving in this particular location. Another parameter which determines the yield and the shape of the deposited material, is the sticking efficiency or the fraction of droplets that attach to the preform. This is mainly dependent on the state of the droplets hitting the deposit (liquid, partially liquid or solid) and the substrate configuration. The sticking phenomenon was therefore also incorporated in the deposition model. The overall model is currently compared with experimental results obtained in the spray-forming unit at the Institut für Werkstofftechnik, Bremen.

### Preparation and properties of Mg-Cu-Y-Al bulk amorphous alloys

Metallic glasses have been extensively studied in the last few years, both for their attractive mechanical properties and from a fundamental point of view. These systems are very sensitive to process parameters and to chemical composition, which strongly affects their glass-forming ability. With the right choice of composition and rapid solidification technique, bulk amorphous alloys in the Mg-Al-Y-Cu system can be prepared, with thicknesses of over 2 mm. In a long term perspective, this work is expected to lead to new types of light-metal alloys with high mechanical strengths.

Amorphous alloys were prepared by the relatively simple technique of casting into a copper wedge mould. During solidifica-



10 years of SOFC development is reflected in the two solid oxide fuel cells. The small one (20 $cm^2$, output 6A at 0.7V) was developed in 1990 - 1992 for operation at 1000 °C. The large one (>500 $cm^2$, expected maximum output 1000 A at 0.7V extrapolated from measurement of small cells) is flexible and has a high mechanical strength. It was developed in 1998 - 99 for operation at 750 - 850 °C, and it is the basis for continuation of the national SOFC programme. The continuation includes upscaling through establishment of a pre-pilot-scale fabrication plant.

tion, the temperature was measured as a function of time. The maximum amorphous layer thickness decreased with increasing aluminium concentrations. No significant difference in the thickness of the maximum amorphous layer was found for moulds with and without water cooling. During solidification, a gap formed between the already crystallised materials and the mould, reducing the obtainable cooling rate. This effect that was later confirmed by numerical calculations. The evolution of the microstructures during crystallisation was examined experimentally by X-ray diffraction and differential scanning calorimetry (DSC) for different compositions and annealing temperatures. Using the DSC data, together with direct temperature-time measurements at various positions in the alloy during cooling, has allowed the determination of the critical cooling rate for the formation of an amorphous layer. The value obtained in this way was in good agreement with the critical cooling rate reported in literature. With careful control of the cooling rate, it was possible to obtain selectively amorphous, nanocrystalline or crystalline structures. In those cases where crystallisation occurred, the addition of Al increased the ability of the alloys to maintain nanoscale crystalline particles.

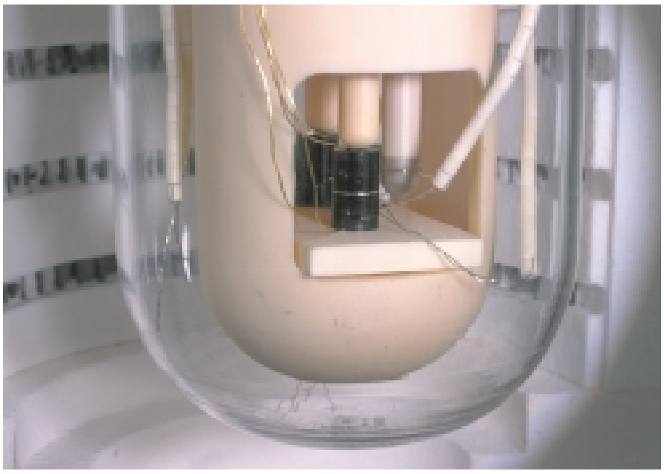
**Ceramic gas sensors**

The electrical resistivity of Mg-doped $SrTiO_3$ depends on the concentration of oxygen in the surrounding gas, a fact exploited in ceramic gas sensors. Work on these sensors has continued with refinement of the screen printing technique for sensor fabrication and with development of a new dip-coating technique. Devices prepared by the latter technique showed promising properties, displaying reduced overshooting when subjected to abrupt changes in the gas composition.

An important property for the application of gas sensors is their selectivity. A study of sensitivity to water vapour showed that the resistance of the sensor materials decreases at very high humidities. Partial proton conduction, introduced under these conditions, was proposed to explain this behaviour. Studies of the influence of $CO_2$ in dry and wet atmospheres were also performed. No influence on the sensor signal was observed in dry $CO_2$, but at relative humidities of above 70 %, a deterioration of the signal was observed, which again could be explained by partial proton conduction. A new project was initiated within the framework of the Danish Sensor Technology Programme. Together with Danish industries and other Danish research institutions, a sensor array based on ceramic gas sensors will be developed for odour detection.

## Materials Chemistry: Development of Solid Oxide Fuel Cells

*Solid oxide fuel cells are all-solid-state devices for generation of electric power by electrochemical combustion of a fuel, normally hydrogen or methane. The technology, which is currently at a pre-commercial level, has the potential of more efficient generation of energy with lower levels of pollution than traditional combustion tech-*



An experimental set-up for the study of electrical characteristics of ceramic interfaces. The set-up allows characterisation at controlled atmosphere and temperature whilst tracking contact pressure and deformations in the contact zone. The picture to the right shows ceramic test pieces and the rods applying contact pressure.

*nologies. The fuel cell programme has the aim to establish a base for a Danish production of SOFC components and consists of three parts: (i) fundamental research in solid state chemistry, including crystallography, defect chemistry and electrochemistry, (ii) fabrication of electroceramic components, using careful process control to obtain components with predetermined properties, and (iii) characterisation, including determination of fundamental materials properties (of powders, ceramics and fabricated electrodes) and cell and stack performance (electrical and mechanical). The expertise established in the fuel cell programme may also find use for the development of other electroceramic components, such as gas sensors and membranes permeable to oxygen or hydrogen. Materials of interest for electrochemical membrane reactors were prepared and characterised. Oxygen sensors were produced and tested, which had good durability to exhaust gases from fossil fuel burning power plants.*

**Fuel cell development**

The development of solid oxide fuel cells with thin yttria stabilised zirconia (YSZ) electrolyte was continued in order to reduce the internal resistance from the value of 0.4 Ω $cm^2$ achieved in 1998 and to expand the cell area. The mechanically strong and highly flexible cells have now reached an internal resistance level of 0.3 Ω $cm^2$ at the operating temperature of 850 °C with test areas around 25 $cm^2$. The size was expanded to more than 400 $cm^2$. Peak effects above 1 W/$cm^2$ have been measured in pure hydrogen with 2 A/$cm^2$. Modelling studies of fabrication show the stack cost in mass fabrication to be well below the EU target for year 2005 of 3500 DKK/kW. A five-year programme for further development of the cell/stack concept was defined. During the first 3 years, a pre-pilot fabrication plant will be established to scale up the laboratory fabrication processes and to complete cell development with respect to durability and reproducibility. A parallel development of materials and structures will be undertaken to enable further reduction of internal resistance and performance improvement at even lower operating temperatures.

**Contact resistances**

Each single cell in an SOFC gives a voltage of 0.5 to 1 V. To obtain a technologically useful voltage, it is necessary to connect several cells in series. In the SOFC design pursued in the Danish SOFC programme, this is done by stacking cells between electrically well conducting interconnector plates. It is found that the performance of the stack does not quite match that expected from the performance of an individual cell. In other words, there is an electrical loss associated with the interfaces between interconnectors and cells. This contact resistance should be minimised in order to maximise stack performance.

The factors that control the contact resistance at the interface between two ceramics are not well understood. Therefore, an activity was launched within the Danish SOFC programme to study this both at fundamental and technological levels. An apparatus was designed for this purpose: a contact dilatometer, in which the electrical characteristics of an interface can be measured at a specified temperature, atmosphere and mechanical load, while the elastic or plastic deformations at the interface are recorded.

The contact between two ceramics was investigated for lanthanum strontium manganite, the cathode material used in state-of-the-art cells. The electrical characteristics of the contact are strongly temperature dependent. Below 700 °C, the contact resistance decreases exponentially with applied polarisation, indicating that the interface represents a potential barrier. The contact resistance was also found to vary inversely with applied pressure over most of the temperature interval 250 to 700 °C. The variation with load was reversible, indicating that only elastic deformation occurs at the interface, in contrast to the behaviour of metals. At temperatures above 700 °C the contact was ohmic and the pressure dependence of the contact resistance was negligible. In this regime, the two pellets simply start sintering together. The contact dilatometer will also be employed to investigate contact layers that are currently used in stacks to minimise contact resistance. The aim is to formulate the specific requirements to the surface morphology of the stack components and to identify optimal profiles of temperature, atmosphere and load to be used during the start-up of stacks.

**Interconnectors**

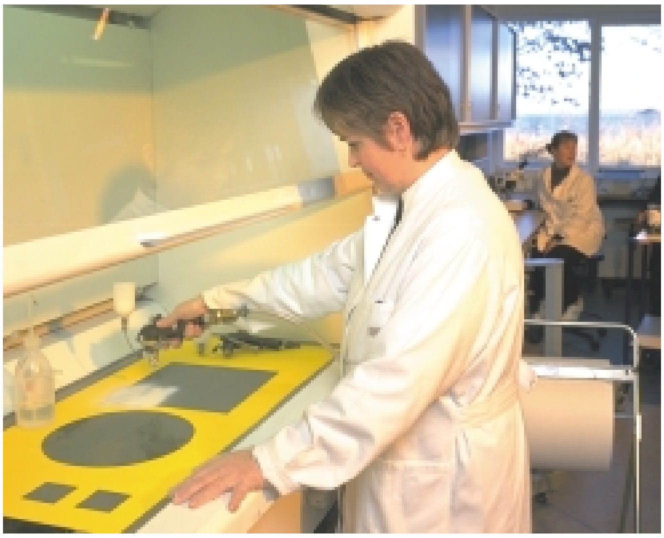
Both ceramics and metals have been investigated as interconnectors for SOFC stacks. In either case, the interconnector material is the most costly part of the stack. The use of cheaper alloys will significantly reduce the stack costs and thereby facilitate the commercialisation of SOFC technology.

When operating a SOFC stack at 850 to 1000 °C, advanced alloys and coatings must be employed to achieve sufficient corrosion resistance. However, recent cell developments have made it possible to lower the operating temperature, thus allowing the use of less oxidation resistant alloys of lower cost. A ferritic steel with small additions of Ce seems to fulfil the requirements for the use in an SOFC stack. Tests have shown that the alloys possess satisfactory corrosion properties. It was shown that by the use of certain contact layers and through controlled start-up conditions, the total contact resistance between cell and interconnector can be kept below 10 % of the total resistance of the stack.

Ceramic interconnectors based on doped lanthanum chromite have also been further developed. The advantage of this system over alloys is its high temperature stability, which allows it to be used at temperatures of 1000 °C or higher. However, a serious problem with the lanthanum chromite based systems was the volume change produced by changes in the partial pressure of oxygen. This causes mechanical stresses in the interconnector plate during operation, which may result in mechanical failure of the stacks. A new composition, subject to a patent application, was developed to meet the requirements of dimensional stability as well as high electronic conductivity under SOFC operating conditions.

**Anodes**

For SOFC operating at reduced temperature (600 to 850 °C), one of the most significant losses occurs during the electrochemical conversion of fuel and oxidant in the respective electrodes. Over the last



Fuel cell development. Air gun spraying of thin YSZ electrolyte layer on a green anode support. The cathode is applied after cofiring, also by air gun spraying.

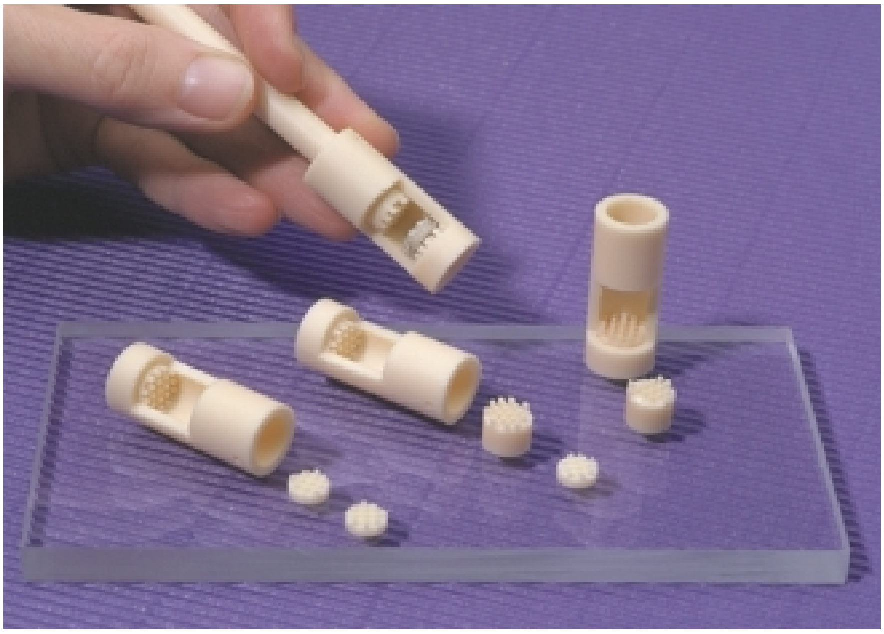
few years development of the traditional Ni/YSZ anode in the Department has contributed to an understanding and a significant reduction of these electrode losses.

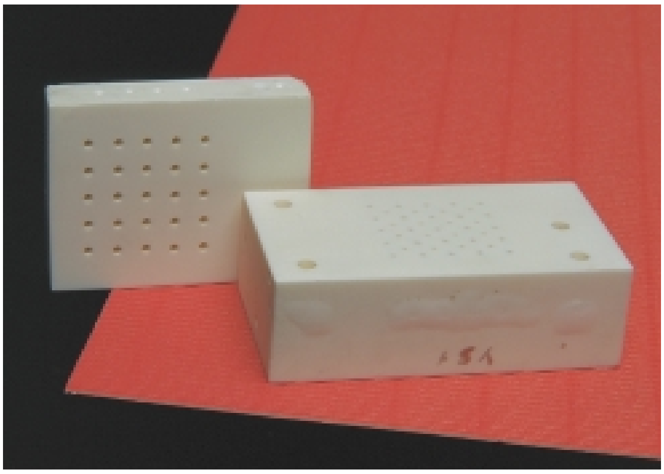
The next generation of anodes for SOFC may well be based on mixed ionic/electronic conducting doped ceria, which, in contrast to Ni, is fairly resistant to accidental oxidation and which is, therefore, applicable in high temperature electrolysers. Simple and cost-effective means for improving these electrodes with respect to lower electrochemical losses and improved mechanical robustness have been identified. An alternative group of materials with similar mechanical properties, doped lanthanum chromites, was investigated. Promising results have been achieved in terms of low electrode losses, but unfortunately, the stability of the electrode performance was poor.

A long-term development effort is the identification of new anode materials. The aim is to develop mixed electronic/ionic conductors with higher electrical conductivity and better mechanical properties as compared with doped ceria. Materials investigated have included solid solutions of pyrochlore structures with high and predominantly electronic conductivity, as well as Ce-modified praseodymium zirconates with lower but equal ionic and electronic conductivity. Various oxides, namely fluorite, rutile, tungsten bronze and pyrochlore materials have been tested as electrodes. The results clearly support the assumption that high ionic conductivity together with electronic conductivity is advantageous for the electrochemical fuel oxidation in the SOFC anode.

### Modelling of defect chemistry and diffusion in non-stoichiometric oxides

The two main models for the description of electronic conductivity in oxides are the small polaron model (electronic carrier localised on a chemical species) and the large polaron model (delocalised carrier). A comparison of these was performed for the cathode material $(La_{1-x}Sr_x)_yMnO_{3\pm\delta}$,





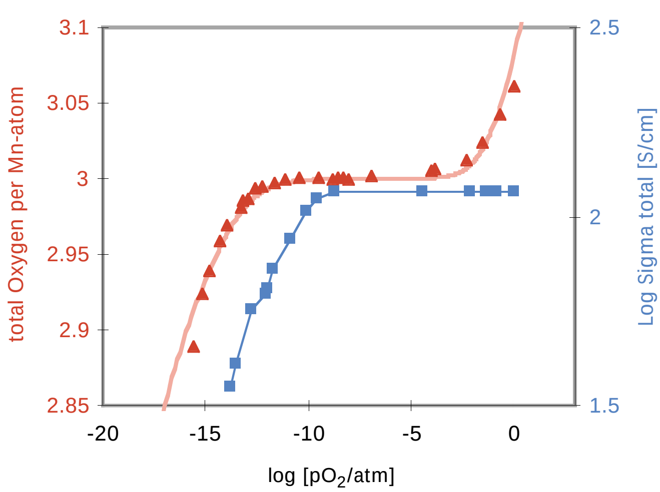
Ceramic components for testing of new fuel cells.

Participants in the Fuel Cell programme of the Department gathering around the recently developed cell type, a flexible and strong cell, with a projected output of 1000 A at 0.7 V at operational temperatures as low as 750 - 850 °C.

based on a sequential solution of the defect equilibrium equations. The stoichiometry dependent on the oxygen partial pressure and the thermodynamic enhancement factor for oxygen diffusion can be described by both models. In order to describe electrical conductivities one must invoke concentration dependent mobilities. The resulting software is now being used at the University of Oslo.

Ongoing work in defect modelling aims to include association between dopants or reduced cations and oxygen vacancies.

Oxygen diffusion in and out of non-stoichiometric oxides can be studied by conductivity relaxation on samples of bar shape: a sample is equilibrated in a given atmosphere, the gas is suddenly changed to a different partial pressure of oxygen and the change in conductivity is recorded monitored. The modelling required to describe this process involves solving finite one-dimensional diffusion coupled with a chemical surface exchange reaction. A spreadsheet routine has been constructed, which allows simultaneous determination of the surface reaction rate and the diffusion coefficient from the conductivity data.




Solution of the defect equilibrium equations for the cathode material $(La_{1-x}Sr_x)_yMnO_{3\pm\delta}$ (LSM) allows the oxygen stoichiometry to be calculated as a function of the oxygen partial pressure ($pO_2$) and compared with experimental data. From the results, the electrical conductivity can be predicted.

▲ Experimental stoichiometry
— Calculated stoichiometry
■ Calculated electronic conductivity

# Commercially Available Technologies

*In 1999, the Department assisted about 35 industrial companies with specific problems (mostly confidential) and collaborated with about 25 Danish and 70 foreign companies in programmes partly funded by the Danish government or the European Union.*

**Electron microscopy and microanalysis**

Two transmission electron microscopes (TEM) and three scanning electron microscopes (SEM) are operated by the Department. Attachments for TEM include energy dispersive X-ray spectrometer (EDS), scanning transmission attachment, serial electron energy loss spectrometer (EELS) and hollow cone illumination. For SEM, EDS and electron back scattering diffraction (EBSD) equipment are available. SEM can be performed at various pressures in different atmospheres (LVSEM, ESEM). Heating and cooling stages, micromanipulator, microinjector and deformation stages are available for *in-situ* observations in ESEM. Electron microscopy is a valuable technique for structural characterisation, such as investigations of:

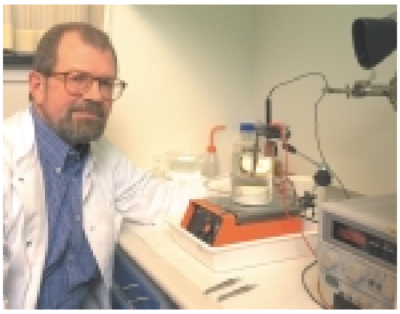
- Grain size and surface texture (orientation imaging microscopy)
- Phase composition and phase distribution (X-ray spectrometry)
- Microdamage mechanisms during mechanical testing (ESEM)

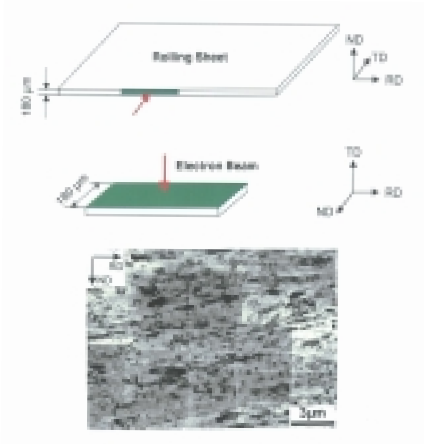
**Powder characterisation**

The Department has experience with cost-effective synthesis of complex oxide-ceramic compounds, usually electroceramics. Control of element dopants allows tailored materials properties. Nano-crystallite sizes are obtained with powder in amounts up to kilogram scale. Subsequent high temperature calcining processes produce desired particle size distributions to suit demands for porosity size distributions and density. X-ray and other techniques provide information on:

- Crystal phases, crystallite size and content of amorphous phase (X-ray diffraction)
- Particle size distribution (laser scattering)
- Powder surface area by gas adsorption (BET)

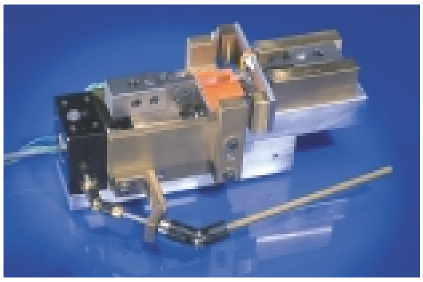
**TEM specimen preparation**
TEM is a powerful tool for studying microstructure of different materials. However, preparation of TEM specimens of thin sheet from edge sections or wires is difficult. A unique technique for preparing TEM specimens from thin sheets or wires (the thickness or the diameter is less than 150 μm) has been developed in the Department.



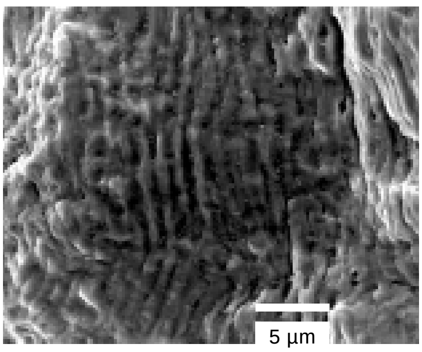



A schematic showing how a TEM foil from the rolling direction (RD)/normal direction (ND) section of a commercial thin rolled aluminium (AA12000) sheet (Alcan Aluminium Ltd.) was prepared. The TEM micrograph shows the microstructure with transverse direction (TD) perpendicular to the picture from the prepared specimen.

PHOTO: NST A/S



A tensile stage for ESEM enables *in-situ* observations of the development of cracks in a high temperature superconductor tape (for superconductor cables, the left cable shown above) as a function of applied strain. The tape (3 mm wide) was made by NST A/S.




Fracture surface of a failed austenitic stainless steel tube. The failure type is identified as a fatigue failure, due to the distinct presence of 'striations'. Part of a commercial investigation carried out for a private company, employing the Department's low vacuum scanning electron microscope.

## Microstructure characterisation

**Method /** *Objective*

**Transmission electron microscopy (TEM): EDS, EELS /** *Microstructure, local crystallographic orientation, local chemical composition*

**Scanning electron microscopy (SEM): EBSD, EDS /** *Microstructure, local crystallographic orientation, orientation maps, local chemical composition*

**Environmental scanning electron microscopy (ESEM): EDS, high temperature, mechanical testing (tension, compression, bending,controlled crack growth) /** *Microstructure, local chemical composition, in-situ investigations of insulators and wet materials, damage and failure mechanisms*

**Positron annihilation spectroscopy (PAS) /** *Point defects, line defects, free volume, interfaces, and gas bubbles*

**Neutron diffraction: ambient and elevated temperature tensile testing /** *In-situ measurements of texture*

**Synchrotron radiation: ambient and high temperature /** *Local crystallographic orientation (3D), local strain state*

**X-ray diffraction; synchrotron radiation /** *Phase characterisation, changes in lattice parameters*

**X-radiography /** *Location of damage, flaws and pores*

**Ultrasonic scanning** / *Detection of flaws*

**Optical microscopy /** *Fibre distribution and orientation, grain size, image analysis*

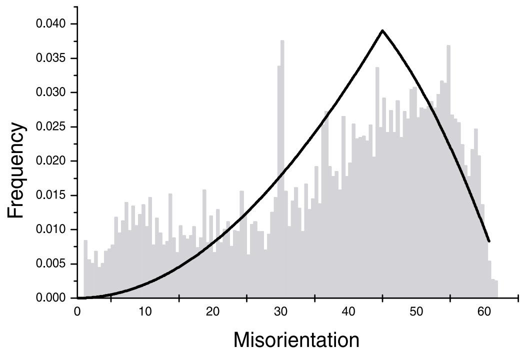
**Matrix digestion /** *Fibre and porosity content in composites*

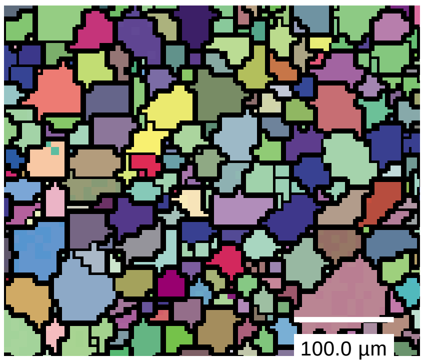
**BET /** *Surface area of powders*

**Light scattering /** *Particle size distribution*

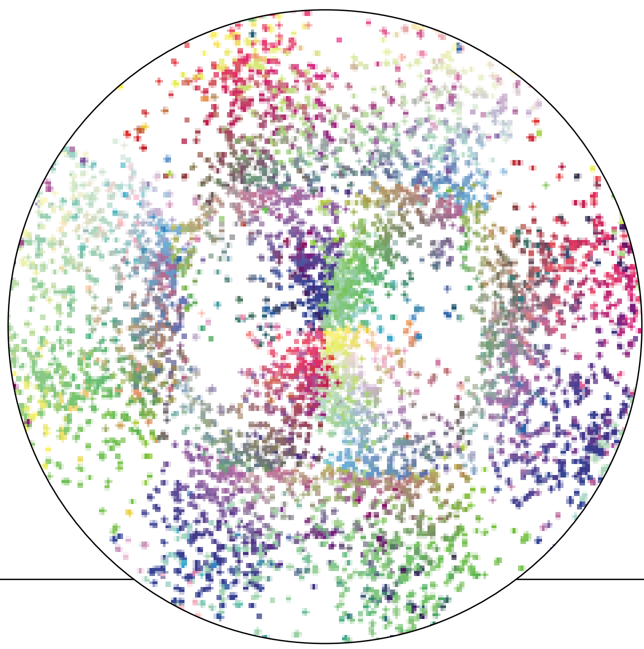
### Crystal orientation microscopy

Through automated analysis of electron back-scattering patterns and computer controlled sample or beam movement, images of local lattice orientations can be obtained rapidly and with high precision in the SEM. The orientation images can be visualised using colours. The images allow one to obtain quantitative data for grain size distributions, grain shape parameters, crystallographic textures and much more.




Crystal orientation image from electrical steel (Pohang Iron & Steel Co. Ltd., Korea) after primary recrystallisation. Thick lines are drawn at high-angle boundaries. The texture is shown as a (100) pole figure using the same colouring. Note the strong peak at 30 ° in the distribution of disorientations. These boundaries are considered to have a particularly high mobility during secondary recrystallisation. The company aims at producing electrical steels with large grains of a very specific orientation and needs to understand the basic mechanisms which produce such grains.



Real time X-ray radioscopic system developed for inspection of large and extensive pipe systems. The concept is commercially exploited by the Carl Bro Industry & Marine Division.

Computerised frame grabber technique for digitising of optical microscopy images for further on-line image processing.

Mechanical testing of PMCs is used for establishing design data for wind turbine rotor blades.

**Non-destructive characterisation techniques**

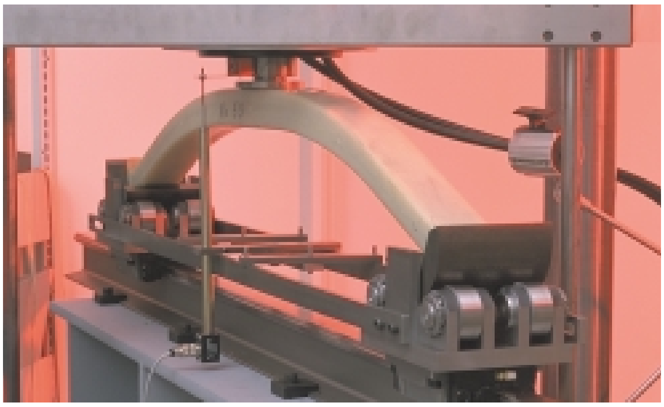
Specimens can be characterised by high resolution X-radiography and ultrasonic immersion scanning. Quantitative image processing procedures for industrial quality control are developed. Thermography is applied for on-line monitoring of damage evolution during mechanical testing, and acoustic emission techniques are applied to correlate damage mechanisms with their noise signature during testing. Typical areas of application include determination of:

- Dimensions of flaws
- Distributions of structure elements
- Dimensional variations of hollow or hidden structures

**Neutron diffraction**

Neutron diffraction provides information about crystal orientation and crystal lattice parameters in most crystalline materials but the techniques are optimised for metals.

- Bulk textures, i.e. the crystallographic orientations in the sample, are measured. Typical sample sizes are in the range 0.5 – 10 $cm^3$. *In-situ* measurements during annealing are performed at temperatures up to 1500 °C. Standard software is used for calculation of parameters related to the mechanical properties from the measured textures.
- Residual stresses and strains can be monitored non-destructively inside large engineering components. Strains are typically monitored to an accuracy of ± $10^{-4}$ in gauge volumes of the order of 10 $mm^3$ at freely selected positions in the sample. Phase specific stresses in multi-phase systems and composite materials can be determined.



Testing of a 1.2 m long glass/polyester leaf spring for vehicle applications. The spring was manufactured by EM Fiberglas A/S.


Techniques for characterisation of materials and components are used in several collaborative projects. Such techniques are non-destructive X-radiography, microstructural characterisation by optical and scanning microscopy, and mechanical testing. The picture shows a demonstrator/prototype of an automotive component (a suspension arm) made in Mg-alloy by thixoforming for Volkswagen AG.

### Macroscopic characterisation

**Method /** *Objective*

**Static tensile, compression, torsion tests /** *Elastic constants, stress-strain behaviour, strength, creep*

**Dynamic tensile, compression, torsion tests damping /** *Fatigue behaviour, damage development, properties*

**Fracture mechanics testing /** *Fracture toughness, fatigue crack propagation*

**Acoustic emission /** *Crack initiation, crack growth, damage development*

**Thermography /** *In-situ detection of damage evolution by heat dissipation*

**Ultrasonic scanning /** *Elastic constants*

**Neutron diffraction: elevated temperature tensile testing /** *In-situ measurements of internal strains, residual stresses*

**Dilatometry /** *Volume changes as function of oxygen partial pressure and temperature, sintering characteristics*

**Thermal analysis /** *Thermal response (expansion, weight, heat evolution)*

**Thermogravimetry; differential thermal analysis; differential scanning calorimetry /** *Characterisation of solid state processes*

**Sensor testing /** *Electrical properties, long term stability*

**Impedance spectroscopy; van der Pauw DC; classical 4 point DC /** *Electrical conductivity, electrochemical activity*

**Direct current vs. voltage (chronoamperometric and potentiodynamic) /** *SOFC performance, critical current density*

### Mechanical characterisation

The Department is accredited by DANAK (Danish accreditation body) to carry out mechanical testing. Testing can be performed in different environments and at non-ambient temperatures (-196 °C to 1200 °C). The load ranges from 20 N to 250 kN. The equipment consists of several servohydraulic test machines, including a combined tension-torsion machine and a resonance test machine for higher frequency testing. Special purpose fixtures have been developed. Testing is carried out in, or in combinations of, the following modes:

- Torsion
- Tension
- Compression
- Bending
- Shearing

### Thermal analysis

Differential thermal analysis (DTA) is used to characterise endothermic or exothermic reactions between –70 °C and 1300 °C. Thermogravimetric analysis (TGA) registrates weight changes during heating up to 1500 °C. Dilatometry is used for measurement of dimensional changes as a function of temperature or oxidising conditions. Amorphous as well as crystalline materials (polymers, alloys, glasses and ceramics) can be characterised. Thermal analysis can be performed in various atmospheres. Typical applications are identification and characterisation of:

- Phase transformations (DTA)
- Chemical reactions (TGA)
- Thermal expansion coefficients (dilatometry)

### Electrical and electrochemical properties

Electrical and electrochemical properties are measured by advanced potentiostats and frequency response analysers (combined AC and DC experiments). The samples can be characterised in a broad range of gas compositions at temperatures up to 1050 °C. Measured properties are:

- Conductivity
- Capacitance
- Current-voltage correlations

Newly established set-up for testing oxygen ion conducting ceramic membranes for production of synthesis gas ($CO + H_2$). The Department has a commercial contract with Haldor Topsøe A/S on development and characterisation of materials for this use.

Measurement of oxygen in non-purified exhaust gas from a power plant fuelled by cheap fossil fuels requires special robust oxygen sensors. The Department constructed such zirconia electrolyte based cells using an electrode of a ceramic conductor instead of a platinum electrode, which is very sensitive to poisoning by sulphur containing gas species. The sensors were tested in Asnæs Power Station, Denmark.

**Ceramic processing**

Several processing techniques are available:

- Biaxial and isostatic pressing
- Tape casting (50 - 1000 μm, sheets for dense or porous flat membranes)
- Ceramic spray painting and dip coating produces thin (5 - 20 μm) layers. Multiple layers can be used for creating material gradients
- Viscous processing produces planar or profiled ceramic sheets

**Processing of polymer matrix composites**

The Department has equipment for production of thermoplastic and thermosetting composite components. Processes include:

- Autoclave and vacuum consolidation and curing (up to 530 °C and 2 MPa)
- Pressing (200 kN, pre-heating in vacuum, fast processing)
- Filament winding

### Processing

**Method /** *Objective*

**Inert gas atomising /**
*Rapidly solidified metal powders*

**Spray forming /**
*Direct formation of compacted structures from liquid particles*

**Inert gas glove box /**
*Handling of reactive materials*

**Uniaxial powder pressing /**
*Green bodies*

**Tape casting; spraying; screen printing; dip coating; cold isostatic pressing /**
*Fabrication of samples, components and devices*

**High shear rolling; profile rolling; planar rolling /**
*Deformation, texture generation*

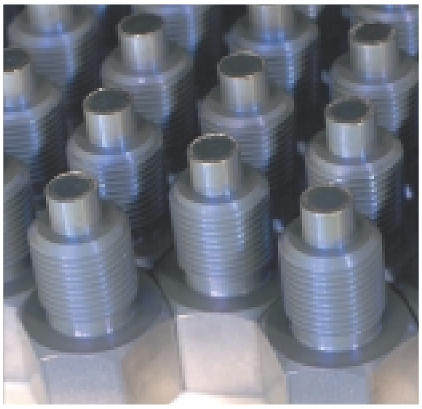
**Heat treatments /**
*Sintering (ceramic and metal parts), recrystallisation*

**Filament winding /**
*Continuous fibre composites (test specimens and components)*

**Autoclave technique /**
*Consolidation or cure of PMCs*

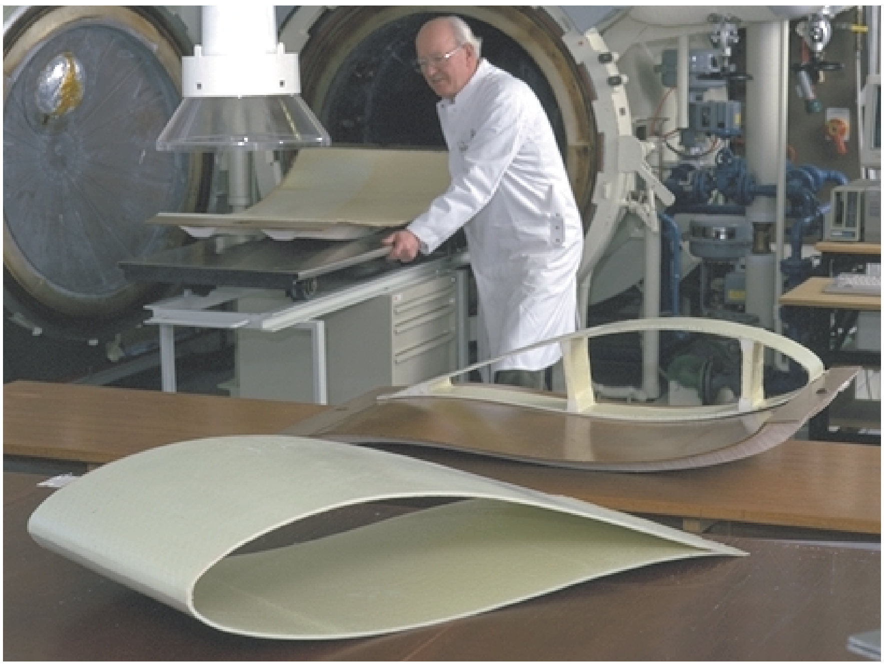
**Press consolidation /**
*Consolidation of PMC composites*

**Grinding by diamond tools /**
*Machining of specimens and components*



Furnace opened after brazing a charge of small nipples for Powder Technologies, Nordborg, Denmark. A silver alloy was used for brazing small ceramic $ZrO_2$ disks to (diameter ~ 5mm) the top of stainless steel nipples.

The technology for manufacturing wind turbine blades of thermoplastic composites in one shot was developed in collaboration with Bonus Energy A/S. The vacuum consolidation technology and the use of metal moulds was demonstrated on a shorter section of a wind turbine blade with integrated webs.



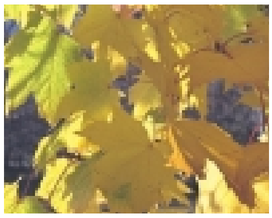
### Joining by vacuum brazing

The Department has several programmable furnaces for heating to 1300 °C in a vacuum better than 5 x $10^{-3}$ Pa. Maximum specimen dimensions are 20 x 20 x 45 $cm^3$. Heating and cooling in a constant flow of inert gas is possible, as well as forced cooling of the furnace atmosphere at any vacuum. The furnaces are generally used for heat-treatment and brazing.

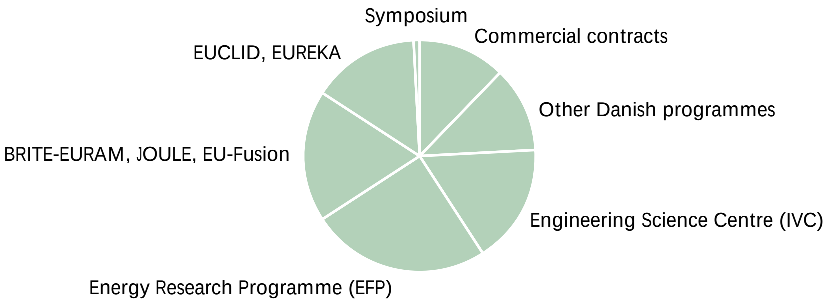
# Finances

The activities of the Department are supported by a combination of government funding, focussed projects funds from national, international and EU programmes and fully commercial industrial contracts.

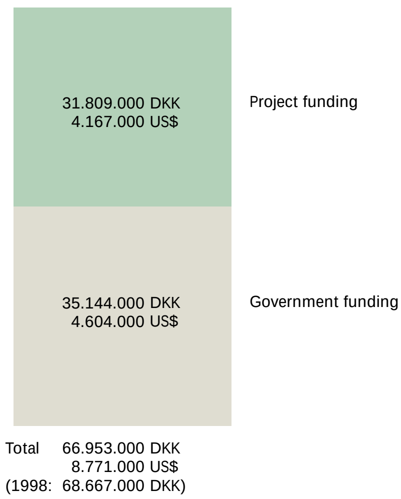
The numbers given are in Danish kroner (DKK). The equivalent amount in US Dollars is also shown (DKK 1000 equal US$ 131, alternatively, US$ 1 equals DKK 7.66).



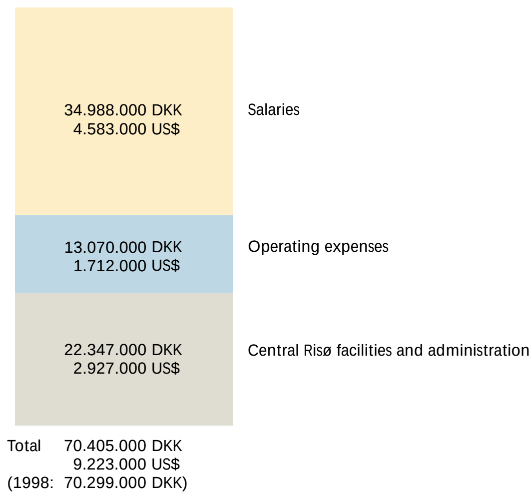
**Project Income**



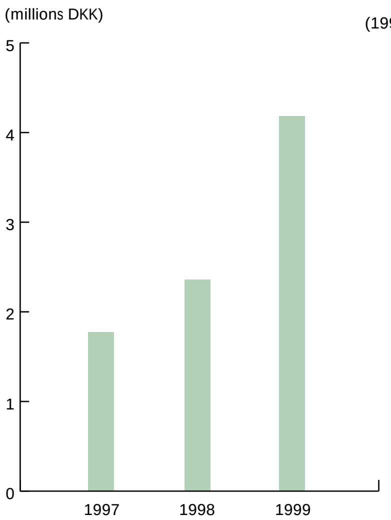

**Income**



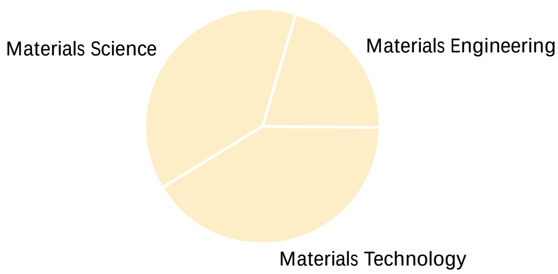

**Expenditure**




**Commercial contracts**




**Research areas (hours)**



# Personnel

## Staff

In 1999 6 members left and 3 new members (*) joined the permanent staff of the Department.

**Head of Department**
Niels Hansen

**Scientific staff**
Adolph, Eivind *until 1 Jun.*
Andersen, Svend Ib
Bagger, Carsten
Bentzen, Janet J.
Bilde-Sørensen, Jørgen B.
Bonanos, Nicholaos
Borring, Jan
Borum, Kaj K.
Brøndsted, Povl
Carstensen, Jesper Vejlø*
Debel, Christian P.
Eldrup, Morten
Gundtoft, Hans Erik
Hendriksen, Peter Vang
Huang, Xiaoxu
Johansen, Bjørn S.
Juul Jensen, Dorte
Jørgensen, Mette Juhl
Kindl, Bruno
Krieger Lassen, Niels C.
Larsen, Peter Halvor
Leffers, Torben
Lilholt, Hans
Linderoth, Søren
Liu, Qing
Liu, Yi Lin
Lorentzen, Torben
Lystrup, Aage S.
Løgstrup Andersen, Tom
Mogensen, Mogens
Nilsson, Tage M.
Pedersen, Allan Schrøder
Pedersen, Ole Bøcker
Poulsen, Finn Willy
Poulsen, Henning F.
Primdahl, Søren
Rheinländer, Jørgen
Singh, Bachu N.
Sørensen, Bent F.
Sørensen, Ole Toft
Toft, Palle
Toftegaard, Helmuth L.
Winther, Grethe

**Postgraduate students**
Andersen, Lotte G.
Glerup, Marianne
Hansen, Jesper Rømer
Hinnum, Benjamin R.*
Højerslev, Christian*
Jensen, Karin Vels*
Lauridsen, Erik M.
Lybye, Dorte
Koch, Søren*
Nielsen, Søren Fæster
Nørbygaard, Thomas
Pedersen, Trine Bjerre*
Petersen, Kenneth
Vegge, Tejs
Zrubcova, Jana*

**Post Docs**
Ahlgren, Erik O. *until 31 May*
Carstensen, Jesper Vejlø *until 1 May*
Cendre, Emmanuelle*
Gamstedt, Kristofer
Grievel, Jean Claude#
Holtappels, Peter W.
Jacobsen, Torben K.# *until 31 Dec.*

#Industrial Post Doc

Laffargue, Denis *until 30 Mar.*
Lawrence, Margulies*
Lyttle, Mark *until 30 Oct.*
Mallon, Stephen*
Mishin, Oleg V. *until 1 Oct.*
Pantleon, Wolfgang
Pryds, Nini H.
Sabin, Tanya*
Schiøtz, Jakob *until 31 Mar.*
Shawen, Tao *until 21 Dec.*

**Consultant**
Waagepetersen, Gaston

**Technical staff**
Adrian, Frank
Borchsenius, Jens F. S. *until 1 Sep.*
Christensen, Lasse *until 6 Sep.*
Frederiksen, Henning
Gravesen, Niels
Nørregaard
Hersbøll, Bent
Hinnum, Benjamin *until 31 Mar.*
Huld, Peder*
Jensen, Knud
Jensen, Palle V.
Jespersen, John
Kjøller, John
Klitholm, Cliver
Larsen, Bent
Larsen, Jan
Larsen, Kjeld J. C.
Lindbo, Jørgen
Mikkelsen, Claus
Nielsen, Birgitte
Nielsen, Palle H.
Nilsson, Helmer
Nygaard, Birgit Herup
Olesen, Preben B.

*(continues on the next page)*

## Visiting Scientists

*(staff continued)*

Olsen, Benny F.
Olsen, Carina Nykjær *until 28 Feb.*
Olsen, Henning
Olsen, Ole
Olsson, Jens O.
Paulsen, Henrik
Pedersen, Niels Jørgen
Sandsted, Kjeld
Strauss, Torben R.
Sørensen, Erling
Vogeley, Erik
Aagesen, Sven

**Office staff**
Dreves Nielsen,
Elsa *until 28 Feb.*
Kiler, Diana*
Hoffmann Nielsen, Lis
Lauritsen, Grethe Wengel
Mortensen, Jytte
Sørensen, Eva M.
Thomsen, Ann
Voss, Anita

**Apprentices**
Hammershøj Olsen, Casper
Klein, Roland until 8 Jan.
Nilsson, Jesper *until 31 Jul.*

An important ingredient in modern research is international collaboration. In 1999 members of the Departments staff worked at other institutions. Similarly many guests visited or worked in the Department for a shorter or longer period or presented some of their work in a colloquium.

**Staff members visiting other institutions**

*Only visits of three or more days are listed.*

**Jesper Rømer Hansen** University of Missouri, Rolla, MI, USA. 9 January - 10 May.

**Niels Hansen** Sandia National Laboratory, Livermore, CA, USA. 27 February - 14 March.

**Xiaoxu Huang** Kyoto University, Kyoto, Japan. 17 - 21 July.

**Mogens Mogensen** University of Waikato, Hamilton, New Zealand. 8 - 29 January.

**Bachu N. Singh** University of California Los Angeles, CA, USA. 10 - 21 May. Pacific Northwest National Laboratory, WA, USA. 18 - 22 October.

**Scientists visiting the Department**

*Only visits of three or more days are listed.*

**Dr. Claire Y. Barlow** University of Cambridge, UK. 30 August - 14 September.

**Mr. John Bradley** University of St. Andrews, UK. 9 August - 10 September.

**Dr. Vincent Branger** CEA Saclay, Gif-sur-yvette, France. 18 - 25 April.

**Dr. Mark Daymond** ISIS, UK. 31 January - 7 February.

**Dr. Dan J. Edwards** Pacific Northwest National Laboratory, Richland, WA, USA. 11 - 25 July.

**Dr. Mike Fitzpatrick** The Open University, UK. 31 January - 8 February.

**Dr. Stanislav I. Golubov** Institute of Physics and Power Engineering, Obninsk, Russia. 6 August - 30 September.

**Mr. Lars Hartmann** University of Kiel, Germany. 20 - 22 January.

**Mr. Byung-Deng Hong** POSCO Technical Research Laboratories, Pohang, Korea. 31 March - 20 November.

**Dr. Darcy A. Hughes** Sandia National Laboratory, Livermore, CA, USA. 31 July - 28 August.

Department meeting at the local beach.

**Ms. Karen James** University of Newcastle, UK. 18 - 22 November.

**Dr. Andreas Kaiser** University of St. Andrews, UK. 23 July- 13 August.

**Dr. Alexandra Kelaidopoulou** BG-Technology, UK. 13 - 24 September.

**Mr. Kevin S. Knight** Rutherford Appleton Laboratory, UK. 10 - 14 May.

**Dr. Alex Korsunsky** University of Oxford, UK. 18 - 22 November.

**Prof. Amar N. Kumar** Indian Institute of Technology, India. 17 - 28 May.

**Ms. Bérangére Leclercq** Ecole Nationale Supérieure de Céramique Industrielle (ENSCI), Limoges, France. 21 June - 20 September.

**Ms. Helena Magnusson** Swedish Institute for Metals Research, Stockholm, Sweden. 25 August - 24 September.

**Mr. Alexander Madgwick** University of Manchester, UK. 16 - 24 August.

**Dr. Mori** University of Manchester, UK. 16 - 24 August.

**Dr. Marie-Helene Monthon** CEA Saclay, Gif-sur-yvette, France. 18 - 25 April.

**Prof. Frank R.N. Nabarro** University of the Witwatersrand, Johannesburg, South Africa. 6 - 20 September.

**Mr. Cyril Nedaud** Ecole Nationale Supérieure de Céramique Industrielle (ENSCI), Limoges, France. 21 September - 31 December.

**Dr. Masato Ohnuma** National Research Institute for Metals, Tsukuba, Japan. 1 January - 26 February, 12 May - 30 June.

**Dr. Judy Pang** University of Manchester, UK. 6 - 11 April.

**Dr. H.G. Priesmeyer** University of Kiel, Germany. 20 - 22 January.

**Mr. Ulrich Rutinau** University of Kiel, Germany. 20 - 22 January.

**Prof. George Saada** Laboratoire d'Etude des Microstructures, CNRS-ONERA, Châtillon, France. 9 August - 6 September.

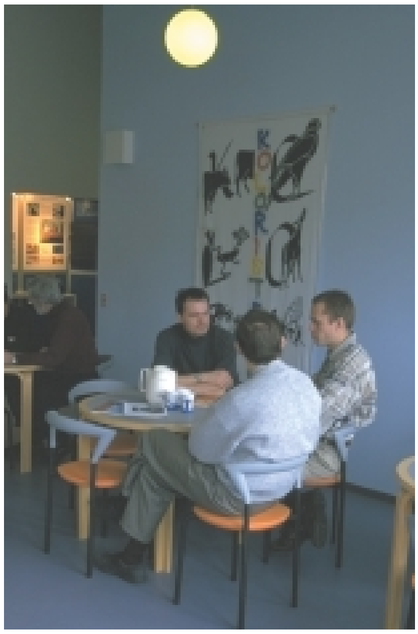
**Mr. Jérôme Senaneuch** Ecole des Mines, St. Etienne, France. 20 May - 20 August.

**Mr. Peter Staron** University of Kiel, Germany. 20 - 22 January.

**Prof. Jim F. Stubbins** University of Illinois, Urbana, IL, USA. 11 - 24 May.

**Dr. Robert M. Suter** Carnegie Mellon University, Pittsburgh, USA. 28 June - 23 July.

**Dr. Helmut Trinkaus** Research Centre Jülich, Germany. 7 - 11 August.



**Mr Jonathan Tuck** University of Newcastle, UK. 18 - 22 November.

**Dr. Roy Vandermeer** Alexandria, VA, USA. 26 August - 23 September.

**Prof. John A. Wert** University of Virginia, Charlottesville, VA, USA. 1 January - 31 December.

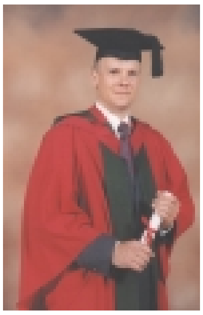
**Dr. Steven F. Zinkle** Oak Ridge National Laboratory, TN, USA. 24 August - 3 September.

**Ms. Jana Zrubcova** Slovak Academy of Science, Bratislava, Slovakia. 20 October - 31 December.

# Community Activities

## Educational Activities

*The Department is strongly involved in the education of students at different levels. The involvement ranges from postgraduate and undergraduate research projects, in collaboration with universities, to summer jobs for university students and one-day courses for high school students. Several of the projects are carried out in collaboration with industrial companies, both through 'Industrial PhD projects' in which students work an apreciable part of their time in the company, and through the summer jobs to university students who work on joint projects between the Department and industrial companies, spending time at both places. In addition, many staff members of the Department act as external university teachers and examiners. Most of the postgraduate students are funded by the Danish Research Academy, the Engineering Science Centre or the Department, while a few are funded from other sources.*



Peter Halvor Larsen received his Ph.D. from University of Sheffield, UK.

## Postgraduate (PhD) Projects

### PhD projects finished during 1999

**Michael S. Brown**
'Anodes for solid oxide fuel cells'.
University of Waikato, Hamilton, New Zealand.
Supervisors: Nigel Sammes (UW), *Mogens Mogensen*

**Peter Halvor Larsen**
'Sealing materials for solid oxide fuel cells'.
Materials Research Department, University of Sheffield, UK.
Supervisor: Peter F. James (US)

**Søren Primdahl**
'Nickel/yttria-stabilised zirconia cermet anodes for solid oxide fuel cells'.
University of Twente, Holland.
Supervisors: Henk Verweij, Henny J.M. Bouwmeester (UT), *Mogens Mogensen*

**Rolf Jarle Åberg**
'Studies of kinetics and reaction mechanisms on Ni-YSZ anodes for solid oxide fuel cells (SOFC)'.
Norwegian University of Technology and Science, Trondheim, Norway.
Supervisors: Reidar Tunold (NTNU), *Mogens Mogensen*

### On-going Industrial PhD Projects

**Eva Mogensen**
'Low temperature sintering of piezo-electrics'.
Lead zirconate-titanate perovskites find use in capacitors, piezoelectrics and actuators. The state of the art components are fabricated with costly platinum electrodes. The aim of the present project is to modify precursor powders with or without additions of sintering aids, such that co-sintering with relatively low-melting silver alloys can be achieved.
The Technical University of Denmark, Lyngby.
Supervisors: Erling Ringgård (Ferroperm A/S), Jacob W. Høj (DTU), *Finn Willy Poulsen*

**Lene Pedersen**
'Relations between composition, fabrication and properties for MMC friction materials for trucks'.
The aim of the project is to develop guidelines regarding optimal sintering conditions for MMC materials for friction applications based on MMC components and desired functional properties.
The Technical University of Denmark, Lyngby.
Supervisors: Erik Simonsen (A/S Roulunds Fabriker), Stuart Clyens (DTI), Knud Aage Thorsen (DTU), *Allan Schrøder Pedersen*

**Kenneth Petersen**
'Development of the spray-forming process for production of steel-based composites'.
The aim of the project is to identify the correlation between the applied, experimental parameters and the properties of the resulting material. This is done by studying the effect of parameter variations on the obtained micro-structures. In particular the alloy solidification is studied with regard to the presence of ceramic particles. The project focuses on wear properties, strength and toughness.
The Technical University of Denmark, Lyngby.
Supervisors: Jan Laurberg List (Danish Steel Works), Stuart Clyens (DTI), Knud Aage Thorsen (DTU), *Allan Schrøder Pedersen*

**Séverine Ramousse**
'Development of high temperature stable friction materials (HT-fricmats)'.
The overall main objective of the project is to develop high temperature stable friction materials, which have a stable friction characteristic under severe temperatures without a comprehensive run-in.
The Technical University of Denmark, Lyngby.
Supervisors: Jesper Valentin (OBTEC A/S), Jacob W. Høj (DTU), *Ole Toft Sørensen*

### Ongoing PhD Projects

**Lotte Gottschalck Andersen**
'Structural properties of superconducting BiSCCO/Ag tapes during cooling'.
Tapes containing the high temperature superconductor Bi-2223 will be characterised by a combination of SEM, TEM, synchrotron XRD and magneto-optic measurements. The goal is

to identify detrimental effects on the superconducting critical current during cooling of the tapes and to optimise the cooling procedure.
The Technical University of Denmark, Lyngby.
Supervisors: Jørgen Bindslev Hansen (DTU), *Henning Friis Poulsen*

**Marianne Glerup**
'Raman spectroscopy and X-ray diffraction on oxides at high temperature'.
The PhD study deals with vibrational spectroscopy (Infrared and Raman) of doped oxides. Aliovalently doped oxides have structurally disordered oxide vacancies and the possibility of accomodating protonic defects. Correlations are established between defect chemistry, structural findings from diffraction and the vibrational spectra.
University of Copenhagen, Denmark.
Supervisors: Rolf W. Berg (DTU), Ole Faurskov Nielsen (KU), *Finn Willy Poulsen*

**Jesper Rømer Hansen**
'Structural and electrical properties of electron conducting perovskites'.
Materials such as $La_{1-x}Sr_xCr_{1-y}Mb_yO_{3-\delta}$ with Mb=V, Sn, Ti, Zr may be used as interconnect for solid oxide fuel cells. The materials exhibit expansion on reduction. In this project the nature of this expansion is investigated with the aim of finding means to minimize the effect.
The Technical University of Denmark, Lyngby.
Supervisors: Torben Jacobsen (DTU), Mogens Mogensen, *Finn W. Poulsen, Peter Vang Hendriksen*

**Benjamin Rask Hinnum**
'Mobility of cations in oxide conductors'.
The mobilities of cations in SOFC materials such as yttria stabilised zirconia (YSZ) and strontia doped lanthanum manganite (LSM) are low but not zero. This may result in redistribution of material over prolonged periods in electrical and thermal gradients and may eventually cause failures of the SOFC. This redistribution in YSZ and LSM is studied in electrical and thermal gradients over periods of many weeks at accelerated conditions. Also the determination of the mobilities through measurements of diffusion coefficients is attempted.
The Technical University of Denmark, Lyngby.
Supervisors: Torben Jacobsen (DTU), *Mogens Mogensen, Carsten Bagger*

**Christian Højerslev**
'The influence of microstructure on the fatigue properties of high strength materials for cold forging tools'.
Ultra high strength martensitic steels are used in prestressed industrial forging tools. Lifetimes of the tools are limited due to very high strain cycles. The microstructural influence on fatigue behaviour is studied in order to optimise the material selection.
The Technical University of Denmark, Lyngby.
Supervisors: Marcel Somers (DTU), *Povl Brøndsted, Jesper Vejlø Carstensen*

**Karin Vels Jensen**
'Interfaces in composite electrodes for SOFC and high temperature electrolysers'.
The relation between the structure of the electrolyte-electrode interface and the electrical losses by passage of current through the interface is investigated.
The Technical University of Denmark, Lyngby.
Supervisors: Ib Chorkendorff (DTU), *Mogens Mogensen, Carsten Bagger, Jørgen Bilde-Sørensen*

**Søren Koch**
'Contacting of ceramic materials'.
The contact resistance in a SOFC stack may contribute significantly to its internal resistance and thus to the electrical loss during the operation of the SOFC. Whereas metal electrical contacts have been studied extensively, very little is known about contacting ceramics. Therefore, the purpose of this study is to identify the mechanisms causing the contact resistance between two ceramics and between a ceramic and a metal. Special equipment, a contact dilatometer, has been developed for the purpose of combined measurements of contact resistance and dimensional changes with the specimens in atmospheres and at temperatures relevant to SOFC operations.
The Technical University of Denmark, Lyngby.
Supervisors: Torben Jacobsen (DTU), *Carsten Bagger, Peter Vang Hendriksen, Mogens Mogensen*

**Erik Mejdal Lauridsen**
'Nucleation and grain growth studies by use of a 3D X-ray microscope'.
The aim is to use a novel 3D X-ray microscope at the ESRF synchrotron for studies of the kinetics of individual grains during recrystallisation of prototype metals. Focus is on both instrumentation and materials science applications.
University of Copenhagen, Denmark.
Supervisors: Jens Als-Nielsen (KU), *Henning Friis Poulsen, Dorte Juul Jensen*

**Dorthe Lybye**
'Structural and electrical properties of perovskites'.
Oxides with perovskite structures such as $LaAlO_3$, $LaGaO_3$ and $LaScO_3$ doped with SrO and MgO are studied. The main purpose of the study is to find relationships between the detailed crystal structures and the conductivities of the materials.
The Technical University of Denmark, Lyngby.
Supervisors: Kurt Nielsen (DTU), *Mogens Mogensen, Finn Willy Poulsen*

**Søren Fæster Nielsen**
'Organized structures in deformed aluminium'.
The dislocation structure in compressed aluminium is studied by synchrotron radiation and electron microscopy. The main goal is to make *in-situ* studies of the evolution of the dislocation structure and the crystallographic orientations when the samples are compressed.
University of Copenhagen, Denmark.
Supervisor: Erik Johnson (KU), *Torben Leffers*

**Thomas Nørbygaard**
'Structure and characteristics of general and special grain boundaries'.
The aim of the project is to achieve an understanding of the connection between grain boundary structure and boundary properties. Cu-Ni(2%) samples with inactive surface markers (a fine-meshed grid) are tested under diffusional creep conditions and studied by SEM and TEM. Bubble formation behavior in copper ►

implanted with helium at elevated temperatures is investigated with TEM.
University of Copenhagen, Denmark.
Supervisors: Erik Johnson (KU), *Jørgen B. Bilde-Sørensen*

**Trine Bjerre Pedersen**
'Modelling of residual stresses in spray-formed structures'.
The aim of the project is to develop a thermomechanical model for the prediction of residual stresses and deformations in spray formed products. Based on model results for temperature and geometry of the spray formed material a finite volume model for the stresses and the deformations will be developed as well. Analyses of the stresses and deformations will be used as the basis for the predictions of the formation of cracks and distortions of the preform. This will improve the understanding of the resulting microstructure and thereby the mechanical properties of the material.
The Technical University of Denmark, Lyngby.
Supervisors: Jesper Hattel (DTU), *Nini H. Pryds*

**Jens Pålsson**
'Integration of a Solid Oxide Fuel Cell (SOFC) into a gas turbine process. Formulation and implementation of an SOFC - gas turbine system model'.
A combination of an SOFC and a gas turbine (GT) is a promising route for power production with high efficiency. A mathematical model of the energy- and mass-flows of a combined SOFC/GT system is formulated. The different characteristics of the SOFC and the gas turbine lead to different requirements for the system layout and operation. The model is used to compare the performance of systems with different layouts and eventually to optimize the system layout and operation.
Lund Institute of Technology, Sweden.
Supervisors: Tord Torisson, Lars Sjunnesson (LTH), *Peter Vang Hendriksen*

**Azra Selimovic**
'Integration of an SOFC into a gas turbine process. Formulation and implementation of an SOFC stack model'.
Detailed system design of a combination of an SOFC and a gas turbine requires modelling of the whole system, including SOFC, gas turbine, heat exchangers, blowers etc. For this to be meaningful a trustworthy description of the SOFC stack in the system is needed. The aim of this project is to develop an SOFC stack model that includes enough details of the coupled heat-, mass-, and charge-transfer processes in the stack to provide a good representation of its characteristics. Yet, the model must be sufficiently simple to allow it to be integrated in the system model.
Lund Institute of Technology, Sweden.
Supervisors: Tord Torisson, Lars Sjunnesson (LTH), *Peter Vang Hendriksen*

**Tejs Vegge**
'Defect dynamics at the atomic scale'.
The mechanical properties of materials are to a very large extent determined by the structural and dynamic properties of lattice defects such as point defects (vacancies, impurities), line defects (dislocations), two-dimensional defects (boundaries) and/or the interaction between these defects. The project focuses on the theoretical understanding of the dynamics of the defects on the basis of an atomic-scale description with special reference to the dislocation behaviour in copper.
The Technical University of Denmark, Lyngby.
Supervisors: Karsten Wedel Jacobsen (DTU), *Torben Leffers, Ole Bøcker Pedersen*

### Undergraduate Projects

**Roger Bachmann**
'Oxygen permeation measurements on mixed conducting membranes'.
Swiss Federal Institute of Technology (ETH), Zürich, Switzerland.
Supervisor: *Peter Vang Hendriksen*

**Jens Høgh**
'Electrical and structural properties of doped lanthanum chromite'.
The Technical University of Denmark, Lyngby.
Supervisors: Jacob W. Høj (DTU), Jesper Rømer Hansen, *Peter Vang Hendriksen, Peter Halvor Larsen*

**Pernille Høy**
'Crystal chemistry in doped $LaCrO_3$ materials under different oxygen partial pressures'.
University of Aarhus, Denmark.
Supervisors: Bernard Grobéty, Sidsel Grundtvig (AU), *Peter Halvor Larsen*

**Karsten Jessen**
'Ceramic electron conductors as electrodes for piezo-electric devices'.
The Technical University of Denmark, Lyngby.
Supervisors: Jacob W. Høj (DTU), *Mogens Mogensen*

**Olivia Redon**
'Fatigue dissipation and failure in glass fibre/carbon fibre hybrid composites'.
Luleå University of Technology, Sweden.
Supervisors: Lars Berglund (LTU), *Kristofer Gamstedt*

**Richard Schmidt**
'$CO_2$ sensor with an open reference electrode based on yttria stabilized zirconia'.
The Technical University of Denmark, Lyngby.
Supervisors: Jacob W. Høj (DTU), *Ole Toft Sørensen*

**Gwon Yau Wong**
'Techniques for characterising oxygen exchange kinetics on a mixed conductor in a $pO_2$ gradient'.
Imperial College of Science, London, UK.
Supervisor: *Peter Vang Hendriksen*

## Summer Students

The Department offers summer jobs to university students. The aims of the employment are (i) to motivate the students for more advanced courses in materials science, (ii) to strengthen the Department's contacts to university students and (iii) to strengthen collaboration with Danish industry. During the projects, the students spend time both at an industrial company and in the Department. Two of the students had their project accepted by the university as one of their courses and one student continued with an undergraduate project in the Department.

**Flemming Hjorth**
'Structure and formability of austenitic stainless steel'.
The Technical University of Denmark, Lyngby.
Supervisors: Andy Horsewell (DTU), Helmut Broe-Richter, Morten Flytkjær (Danfoss), *Grethe Winther*

**Jesper Vester Nielsen**
'Structure and formability of steels: Influence of structure elements on mechanical anisotropy'.
The Technical University of Denmark, Lyngby.
Supervisors: Andy Horsewell (DTU), Lene A. Jensen (Grundfos), *Grethe Winther*

**Richard Schmidt**
'Testing of Mg-doped strontium titanate sensors'.
Supervisors: Henning Jensen (PBI-Dansensor A/S), *Ole Toft Sørensen*

**Bo Sell**
'Synthesis of perovskites and measurement of oxygen diffusion'.
Supervisors: Niels Christiansen (Haldor Topsøe A/S), *Peter Vang Hendriksen, Mogens Mogensen*

**Kim Vardinghus-Nielsen**
'Crack growth studies in porous ceramics'.
Supervisors: Niels Christiansen (Haldor Topsøe A/S), *Bent F. Sørensen*

## High School Students

In an effort to increase the interest among high school students for science and technology the Department has continued the one-

day courses in materials science for students from high schools around the country. The courses deal with polymer chemistry and polymer based fiber composites. This provides the students with some knowledge about the production and the properties of materials along with the experience that science can be fascinating. This year 66 students participated in the courses. They came from the following schools: Roskilde Tekniske Gymnasium (HTX), Skive Gymnasium (2 classes), Vordingborg Gymnasium (2 classes) and Himmelev Gymnasium.

## External Lecture Courses

**Svend Ib Andersen**
**Jørgen B. Bilde-Sørensen**
**Morten Eldrup**
**Allan Schrøder Pedersen**
**Bent F. Sørensen**
**Ole Toft Sørensen**
**John A. Wert**
'Introduction to new materials'. University of Aarhus and Aalborg University, Denmark.
20 April - 5 May. (distance learning course)

**Christian P. Debel**
'Fracture mechanics – Theory and case stories'. Materials Science Course, The Engineering College of Copenhagen, Ballerup, Denmark. 17 May.

**Jørgen B. Bilde-Sørensen**
'Lattice defects, microstructure and the mechanical properties of materials'. PhD course at The Technical University of Denmark, Lyngby. Spring term.

'Electron microscopy and microanalysis'. Course no. 10455 at The Technical University of Denmark, Lyngby. Fall term.

'Low vacuum and environmental scanning electron microscopy - basic principles' and

'Energy-dispersive X-ray spectrometry in the low vacuum and environmental scanning electron microscopes'. LV-ESEM'99: a course on low vacuum and environmental scanning electron microscopy arranged by Chalmers University of Technology, Gothenburg, Sweden, in collaboration with Hitachi, JEOL, Leo, Oxford Instruments and Philips. 19 - 21 October.

**Wolfgang Pantleon**
'Dynamical systems' and 'General aspects of computer simulation'. Winter School: 'Simulation Techniques in Materials Science', Freiberg University of Mining and Technology, Germany. 22 – 26 February.

**Ole Bøcker Pedersen**
'Lattice defects, microstructure and the mechanical properties of materials'. PhD course at the Technical University of Denmark, Lyngby. Spring term.

'Fatigue and associated microstructural aspects'. NATO Advanced Study Institute: 'Multiscale Phenomena in Plasticity: From Experiments to Phenomenology, Modelling & Materials Engineering'. Ouranopoulis, Greece. 8 - 19 September.

**Ole Toft Sørensen**
'Introduction to modern thermal methods' and 'Sample controlled TA'. Summerschool: 'Advanced Techniques in Thermal Analysis and Calorimetry of Solid State Reactions'. Risø, 26 – 28 May.

## External Examiners

**Svend Ib Andersen**
**Povl Brøndsted**
**Christian P. Debel**
**Morten Eldrup**
**Hans Lilholt**
**Søren Linderoth**
**Aage Lystrup**
**Finn Willy Poulsen**
**Ole Toft Sørensen**
Members of the officially appointed corps of Danish university examiners (MSc and BSc).

**Svend Ib Andersen**
PhD examiner. University of Aalborg, Denmark.

**Povl Brøndsted**
PhD examiner. The Technical University of Denmark, Lyngby.

**Niels Hansen**
PhD examiner. Ecole des Mines, St. Etienne, France.

**Dorte Juul Jensen**
PhD examiner. Freiberg University of Mining and Technology, Germany.

**Hans Lilholt**
PhD examiner. University of Aalborg, Denmark.

**Søren Linderoth**
PhD examiner. The Technical University of Denmark, Lyngby.

**Mogens Mogensen**
PhD examiner. University of Twente, Holland.

**Finn Willy Poulsen**
PhD examiner. University of Oslo, Norway.

**Ole Toft Sørensen**
PhD examiner. The Technical University of Denmark, Lyngby.
PhD examiner. University of Aalborg, Denmark.

## Conferences and Colloquia

### 20th Risø International Symposium: 'Deformation-Induced Microstructures: Analysis and Relation to Properties'

The Symposium was held at Risø from 6 - 10 September with 74 participants from industry, research institutes and universities in 16 countries.

The theme of the Symposium was the formation of microstructures during monotonic and cyclic deformation at low and high temperatures as well as during creep or in fatigue and fracture, where microstructures form locally at crack fronts. The Symposium gave a high priority to papers addressing the ultimate aim of microstructural investigations: to establish links from microstructures, including relevant atomic-scale investigations, via micromechanics to macromechanics. The 560 page proceedings containing 12 invited papers and 46 contributed papers were available at the Symposium and can be purchased from the Department.

The Symposium was organized by the Department in collaboration with the Engineering Science Centre (IVC) at Risø for Structural Characterization and Modelling of Materials.

Organisers: *Jørgen B. Bilde-Sørensen, Jesper Vejlø Carstensen, Niels Hansen, Dorte Juul Jensen, Grethe W. Lauritsen, Torben Leffers, Wolfgang Pantleon, Ole Bøcker Pedersen, John A. Wert and Grethe Winther*

### Nordic Workshop on Polymer Matrix Composite Materials and Mechanics

A small workshop with participants from Denmark, Sweden and Latvia was organised at Risø 26-27 August 1999. The meeting included presentations and discussions on topics such as natural-fibre composites, fracture mechanics of composite materials, fatigue characterisation and life prediction.

Organisers: *Kristofer Gamstedt, Bent F. Sørensen*

### International Summer School: Advanced Techniques in Thermal Analysis and Calorimetry of Solid State Reactions

The purpose of this summer school, which was organised at Risø 26 - 28 May, was to discuss and to present the latest developments in thermal analysis and calorimetric techniques for studies of solid state reactions. The programme comprised introductory lectures as well as demonstrations of techniques by leading equipment manufacturers. The summer school attracted about 60 participants, including 20 PhD students, from the Nordic countries.

Organiser: *Ole Toft Sørensen*

The symposium excursion to the Great Belt bridge exhibition.

### Colloquia

**Prof. Golam Newaz**
Wayne State University, Detroit, USA.
'Role of external constraints on energy absorption in unidirectional polymeric composite tubes'.
22 March.

**Mr. Kevin S. Knight**
ISIS Facility, Rutherford Appleton Laboratory, UK.
'Structural phase transitions, real and imaginary, in doped and undoped barium cerate'.
12 May.

**Dr. John A. Sutliff**
GE Corporate R & D, USA.
'Quantification of engineering materials using the automated-EBSP technique'.
25 May.

**Prof. Z.Q. Sun**
University of Science and Technology Beijing, China.
'Neutron diffraction study on site occupation of substitutional elements at sublattices in $Fe_3Al$ intermetallics'.
29 July.

**Prof. W. Mao**
University of Science and Technology Beijing, China.
'Analysis of misorientation distribution in polycrystalline materials by using ODF data'.
29 July.

**Prof. George Saada**
CNRS-ONERA, Chatillon-France.
'Stress field at interfaces'.
17 August.

**Prof. George Saada**
CNRS-ONERA, Chatillon-France.
'Plastic deformation of face centred cubic metals from elementary processes to modelling'.
31 August.

**Ms. Anja Bieberle**
Nichtmetallische Werkstoffe, ETH Zürich, Switzerland.
'On the way of understanding SOFC anodes'.
6 September.

**Prof. Frank R.N. Nabarro**
University of the Witwatersrand, Johannesburg, South Africa.
'Grain size, stress and creep mode'.
16 September.

## Participation in Committees

Many staff members have accepted special responsabilities by joining Danish or international committees, such as Research Programme Committees, Advisory Committees, Editorial Committees, Conference Committees or Boards of Professional Societies.

### Research Programme Committees

**Tom Løgstrup Andersen**
The Steering Committee of the Non-food program 'Plant Fibre Products – Defibration and Composites'.

**Carsten Bagger**
The Steering Committee of the Danish Solid Oxide Fuel Cell Programme.

**Nikolaos Bonanos**
Management Committee of EU COST Action 525 'Advanced Electronic Ceramics: Grain Boundary Engineering'.

**Niels Hansen**
The Steering Committee of the Danish Solid Oxide Fuel Cell Programme.
EFDA Technology Sub-Committee. Brussels, Belgium.

**Peter Vang Hendriksen**
Management Committee of EU COST Action 525 'Advanced Electronic Ceramics: Grain Boundary Engineering'.

**Dorte Juul Jensen**
The Programme Committee of FREJA (Female Researchers in Joint Action). Copenhagen.

**Torben Leffers**
The Ministry of Research Programme Committee for Materials Research. Copenhagen.
The Review Committee for Materials Engineering and Environmental Matters under the European Synchrotron Radiation Facility. Grenoble, France.
The Coordination Committee for the Danish Materials Technology Development Programme. Copenhagen.
The Board of Ingeniørvidenskabelig Fond og G. A. Hagemanns Mindefond. Copenhagen.

**Hans Lilholt**
The Project Management Committee of the EU BRITE Project: 'Thixoforming of Advanced Light Metals for Automotive Components (TALMAC)'. (Chairman).
The Steering Committee of the Non-food program 'Plant Fibre Products – Defibration and Composites'. (Chairman).

**Søren Linderoth**
The Steering Committee of the Danish Solid Oxide Fuel Cell Programme.
The Project Management Committee of the EU BRITE-EURAM project 'Low-cost Fabrication and Improved Performance of Solid Oxide Fuel Cell Stack Components'.

**Torben Lorentzen**
The Project Management Committee of the EU BRITE-EURAM project 'Residual Stress Standard using Neutron diffraction (RESTAND)'.
The Steering Committee of the EU-funded thematic network 'Training Industry in Neutron Strain Scanning (TRAINSS)'.
The Project Management Committee of the EU project 'Welding of Airframes using Friction Stir Welding (WAFS)'.
The Project Management Committee of the EU project 'Joining Dissimilar Materials and Composites using Friction Stir Welding (JOIN-DMC)'.

**Aage Lystrup**
The Steering Committee of the Danish Solar Cell Hybrid Car project.

**Mogens Mogensen**
The Steering Committee of the Danish Solid Oxide Fuel Cell Programme.
The Project Management Committee of the EU BRITE-EURAM project 'Low-cost Fabrication and Improved Performance of Solid Oxide Fuel Cell Stack Components'. (Chairman).
The Project Management Committee of the EU Training and Mobility of Researchers Network Project 'Alternative Oxide Anodes for Direct Oxidation of Methane in SOFCs'.

**Finn Willy Poulsen**
The Electrochemical Energy Conversion Programme under the Nordic Energy Research Programme. Ås, Norway. (Chairman).

**Henning Friis Poulsen**
The Steering Committee of the EFP-98 Programme on Danish Superconductors. Copenhagen.

**Jens Olsson**
The Board of Governors of Risø National Laboratory. (Staff representative).

**Jørgen T. Rheinländer**
The Project Management Committee of the EU BRITE-EURAM project 'Development of a portable remote controlled real-time radioscopy system for quantitative industrial inspection of large thickness steel pipes and welds (RAYSQUINS)'. (Chairman).

## Advisory Committees

**Eivind Adolph**
Technical Assessor, DANAK. Copenhagen.

**Niels Hansen**
Advisory Group for Advanced Energy Technologies. The Danish Ministry of Environment and Energy, Copenhagen.
Advisory Group for Hydrogen Research. The Danish Ministry of Environment and Energy, Copenhagen.
Technical Assessor. DANAK, Copenhagen.
Reference Group for the BRITE-EURAM Programme. The Danish Ministry of Trade and Industry, Copenhagen.
The Advisory Committee of the Engineering Science Centre (at Risø) for Structural Characterization and Modelling of Materials.
The COST Technical Committee on Materials. Brussels, Belgium.
Technical Scientific Advisory Board, GKSS Forschungszentrum. Geesthacht, Germany.

**Dorte Juul Jensen**
The Advisory Committee of the Engineering Science Centre (at Risø) for Structural Characterization and Modelling of Materials.

**Torben Leffers**
Advisory Group for the EU Programme on Competitive and Sustainable Growth. The Danish Ministry of Research, Copenhagen.

**Hans Lilholt**
Committee for associate professor appointment. University of Aalborg, Denmark.

**Torben Lorentzen**
VAMAS committee, Technical Working Area TWA20, Measurement of Residual Stresses.
ISP-7 Scheduling Panel, ISIS-facility. Rutherford Appleton Laboratory, UK.

**Finn Willy Poulsen**
Expert and Danish contact IEA Annex SOFC collaboration.

**Henning Friis Poulsen**
Review board of the European Synchrotron Research Facility. Grenoble, France.

**Jørgen T. Rheinländer**
Technical Assessor. DANAK, Copenhagen.

**Bachu N. Singh**
Expert Group on Structural Materials, EU Fusion Technology Programme. Brussels, Belgium.
Task Force Materials, EU Fusion Technology Programme. Brussels, Belgium.

**Ole Toft Sørensen**
ICTAC Standardization Committee.

## Editorial Committees

**Jørgen B. Bilde-Sørensen**
Editorial Board of 'Microscopy Research and Techniques'.

**Povl Brøndsted**
Editorial Board of 'Advanced Composites Letters'.

**Morten Eldrup**
Advisory Board of 'Materials Science Forum'.

**Niels Hansen**
Editorial Board of 'Revue de Metallurgie'.
Editorial Board of 'Monographs in Materials Science'.

**Dorte Juul Jensen**
Advisory board of 'Zeitschrift für Metallkunde'.
Associate editor of 'Acta Materialia' and 'Scripta Materialia'.

**Peter Halvor Larsen**
Editorial board of 'Journal of the Danish Ceramic Society'.

**Torben Leffers**
Editorial board of 'Textures and Micro-structures'.

**Hans Lilholt**
Editorial Board of 'Advanced Composite Materials'.
Editorial Board of 'Composite Science and Technology'.
Editorial Board of 'Polymers and Polymer Composites'.
Editorial Board of 'Applied Composite Materials'.

**Søren Linderoth**
Advisory Board of 'Diffusion and Defect Data'.

**Torben Lorentzen**
Editorial board of 'Journal of Neutron Research'.

**Mogens Mogensen**
Contributor to 'The Great Danish Encyclopedia'. Copenhagen.

**Allan Schrøder Pedersen**
Editorial Panel of 'Powder Metallurgy'.

**Ole Bøcker Pedersen**
Contributor to 'The Great Danish Encyclopedia'. Copenhagen.

**Jørgen T. Rheinländer**
Editorial Board of 'Svejsning', the Journal of the Danish Welding Society.

**Bent F. Sørensen**
Editorial Board of 'Key Engineering Materials'.

**Ole Toft Sørensen**
Editorial Board of 'Journal of Thermal Analysis'.
Editorial Board of 'Journal of the European Ceramic Society'.

The Project Management Committee of the EU BRITE-EURAM project 'Development and performance evaluation of a fast X-radioscopic and lock-in thermographic non-destructive evaluation (NDE) system for fibre based technical composites (FIBRINS)'. (Chairman).

The Project Management Committee of the EU BRITE-EURAM project 'Hyper-eutectic alloys for automotive components (HAforAC)'.

The Steering Committee of the EU-funded thematic network 'Plant life assessment network (PLAN)' (Cluster Co-ordinator).

## Conference Committees

**Nikolaos Bonanos**
International Advisory Board for International Conferences on Solid State Protonic Conductors.

**Morten Eldrup**
International Advisory Committee for International Conferences on Positron Annihilation.

**Niels Hansen**
International Advisory Committee for EUROMAT 2000. Topical-European Conference. France.

International Advisory Committee for 'Dislocations 2000'. June 2000. Washington, USA.

International Advisory Committee for International Conference on Advanced Materials Processing (ICAMP 2000). New Zealand.

International Advisory Committee for 'Advanced Materials and Technologies ' (AMT'2001). Poland.

**Dorte Juul Jensen**
International Committee of the International Conferences on the Strength of Materials (ICSMA).

International Committee of the International Conferences on Textures of Materials (ICOTOM).

**Peter Halvor Larsen**
Advisory Committee for the Sixth Conference & Exhibition of the European Ceramic Society 1999.

Organizing Committee of the 12th International Conference on Thermal Analysis and Calorimetry. August 2000. Copenhagen, Denmark.

**Hans Lilholt**
International Advisory Committee for the 5th International Conference 'Deformation and Fracture of Composites', March 1999. London, UK.

International Advisory Committee for the 12th International Conference on Composite Materials (ICCM-12), July 1999. Paris, France.

International Advisory Committee for the 11th International Conference on Mechanics of Composite Materials (MCM-2000), June 2000. Riga, Lithuania.

**Torben Lorentzen**
International Scientific Committee of the International Conference on Stress Evaluation. December 2000. Reims, France.

**Mogens Mogensen**
International Advisory Committee of the 12th International Conference on Solid State Ionics. June 1999. Thessaloniki, Greece.

**Wolfgang Pantleon**
Scientific Committee of the International Workshop 'Local Lattice Rotations and Disclinations in Microstructures of Distorted Crystals'. April 2000. Rauschenbach, Germany.

**Ole Bøcker Pedersen**
Advisory Board for the International Symposium on Materials Ageing and Life Management. October 2000 (ISOMALM 2000). Kalpakkam, India.
Scientific Committee of the 2nd International Conference on Fatigue of Composites. June 2000. Atlanta, USA.
International Scientific Committee of the International Conference on Fatigue Damage of Structural Materials III. September 2000. Hyannis, USA.

**Finn Willy Poulsen**
Scientific Committee of the 4th European SOFC FORUM. July 2000. Lucerne, Switzerland.

**Bachu N. Singh**
Technical Programme Committee of 9th International Conference on Fusion Reactor Materials. October 1999. Colorado Springs. CO, USA.
Advisory Board for the International Symposium on Materials Ageing and Life Management (ISOMALM 2000). October 2000. Kalpakkam, India.
Organizing Committee of the International Workshop on Dislocation-Defect Interactions in Irradiated Materials. April 2000. Toledo, Spain.

**Ole Toft Sørensen**
Organizing Committee of the 12th International Conference on Thermal Analysis and Calorimetry. August 2000. Copenhagen. (Chairman).

## Professional Societies

**Svend Ib Andersen**
The European Structural Integrity Society. Delft, The Netherlands.

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

**Jørgen B. Bilde-Sørensen**
Member of the board of the Scandinavian Society for Electron Microscopy.

**Povl Brøndsted**
The Executive Committee of the Danish Metallurgical Society. (Chairman).

**Peter Halvor Larsen**
The Executive Committee of the Danish Ceramic Society. (Chairman).
The Executive Committee of the Danish Society for Thermal Analysis and Calorimetry.
The Executive Committee of the Nordic Society for Thermal Analysis and Calorimetry.
Council of the European Ceramic Society.

**Hans Lilholt**
International Committee of Composite Materials. Philadelphia, USA.
Council and Executive Committee of the European Society for Composite Materials. London, UK.

**Mogens Mogensen**
The Executive Committee of the Danish Electrochemical Society.
The Executive Committee of the High Temperature Materials Division of the Electrochemical Society. Pennington, NJ, USA.

**Wolfgang Pantleon**
Working Group 'Computer Simulation - Modelling in Materials Science' of the German Society for Materials Science. (Vice-Chairman).

**Søren Primdahl**
The Executive Committee of the Danish Electrochemical Society.

**Bent F. Sørensen**
The Executive Committee of the Danish Ceramic Society.

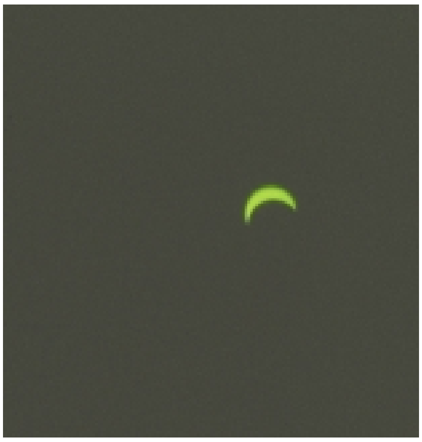
**Ole Toft Sørensen**
Nordic Society for Thermal Analysis and Calorimetry.

**Anita Voss**
Secretary, the Danish Ceramic Society.

## Exhibition

The Department participated in the DSEi '99 Exhibition in Chertsey, Surrey, UK, 14 - 17 September 1999. DSEi is an abbreviation for Defence Systems and Equipment international and the exhibition had around 500 stands. A stand was shared with European companies and institutions participating with the Department in different research programs (EUCLID) on light weight armour for ballistic protection. The Department showed methods for materials testing of composite materials, ceramics and adhesives.

Partial solar eclipse 11 August.



# Published Work

*For a public research centre the most important way of communicating its scientific results is to publish them in the scientific literature. The number of publications in 1999 was 146, similar to that of 1998. In addition to the publications listed below, members of the Department delivered about 85 presentations at conferences and meetings where no proceedings were subsequently published and wrote 11 internal reports.*

*Some papers are co-authored with scientist from other institutes, as a result of national and international collaboration. Names of authors from the Department are written in italics. Note that journal articles are sorted by the origin of publications (abroad or Denmark), not by language.*

## International Publications

*The list of international publications covers articles in international journals, books and reports, as well as patent applications and foreign books.*


1. *Andersen, L.G.; Poulsen, H.F.; Frello, T.*; Andersen, N.H.; Zimmermann, M. von, Cooling behavior of BSCCO/Ag tapes. IEEE Trans. Appl. Superconduct. (1999) v. 9 p. 2758-2761

2. Andersen, N.H.; Zimmermann, M. von; Frello, T.; Kall, M.; Mønster, D.; Lindgård, P.-A.; Madsen, J.; Niemöller, T.; *Poulsen, H.F.*; Schmidt, O.; Schneider, J.R.; Wolf, T.; Dosanjh, P.; Liang, R.; Hardy, W.N., Superstructure formation and the structural phase diagram of $YBa_2Cu_3O_{6+x}$. Physica C (1999) v. 317-318 p. 259-269

3. *Appel, C.C.; Bonanos, N.*, Structural and electrical characterisation of silica-containing yttria-stabilised zirconia. J. Eur. Ceram. Soc. (1999) v. 19 p. 847-851

4. *Appel, C.C.*; Botton, G.A.; *Horsewell, A.*; Stobbs, W.M., Chemical and structural changes in manganese-doped yttria-stabilized zirconia studied by electron energy loss spectroscopy combined with electron diffraction. J. Am. Ceram. Soc. (1999) v. 82 p. 429-435

5. *Bonanos, N.; Poulsen, F.W.*, Considerations of defect equilibria in high temperature proton-conducting cerates. J. Mater. Chem. (1999) v. 9 p. 431-434

6. *Brøndsted, P.; Johansen, B.S.*, Measurement of damage progress in fibre reinforced polymer materials. Plast. Rubber Compos. (1999) v. 28 p. 458-462

7. Bødker, F.; Mørup, S.; Charles, S.W.; *Linderoth, S.*, Surface oxidation of cobalt nanoparticles studied by Mössbauer spectroscopy. J. Magn. Magn. Mater. (1999) v. 197 p. 18-19

8. *Clausen, B.; Leffers, T.; Lorentzen, T.; Pedersen O.B.*; Houtte, P. Van, The resolved shear stress on the non-active slip systems in Taylor/Bishop-Hill models for FCC polycrystals. Scr. Mater. (2000) v. 42 p. 91-96

9. *Clausen, B.; Lorentzen, T.*; Bourke, M.A.M.; Daymond, M.R., Lattice strain evolution during uniaxial tensile loading of stainless steel. Mater. Sci. Eng. A (1999) v. 259 p. 17-24

10. Edwards, D.J.; *Singh, B.N.; Toft, P.; Eldrup, M.*, Post-irradiation annealing response of pure copper irradiated at 100 deg C. In: Fusion materials. Semiannual progress report for the period ending June 30, 1999. DOE/ER-0313/26 (1999) p. 121-125

11. Flükiger, R.; Huang, Y.; Marti, F.; Dhalle, M.; Giannini, E.; Passerini, R.; Bellingeri, E.; Grasso, G.; *Grivel, J.-C.*, Observation of the Bi,Pb(2223) reaction mechanism and alternative ways of producing tapes with new filament configurations. IEEE Trans. Appl. Superconduct. (1999) v. 9 p. 2430-2435

12. Frello, T.; *Poulsen, H.F.; Andersen, L.G.*; Andersen, N.H.; Bentzon, M.D.; Schmidberger, J., An in situ study of the annealing behaviour of BiSCCO Ag tapes. Supercond. Sci. Technol. (1999) v. 12 p. 293-300

13. *Gamstedt, E.K.*; Berglund, L.A.; Peijs, T., Fatigue mechanisms in unidirrectional glass-fibre-reinforced polypropylene. Compos. Sci. Technol. (1999) v. 59 p. 759-768

14. *Gamstedt, E.K.*; Sjögren, B.A., Micromechanisms in tension-compression fatigue of composite laminates containing transverse plies. Compos. Sci. Technol. (1999) v. 59 (no.2) p. 167-178

15. *Gamstedt, E.K.*; Talreja, R., Fatigue damage mechanisms in unidirectional carbon-fibre-reinforced plastics. J. Mater. Sci. (1999) v. 34 p. 2535-2546

16. *Grivel, J.-C.*; Eltsev, Y.; Andersson, M.; Rapp, O.; Erb, A.; Walker, E.; Flükiger, R., First-order melting transition observed from resistivity measurements in ultra-pure $YBa_2Cu_3O_{7-\delta}$ single crystals with high twin boundary density. Physica C (1999) v. 322 p. 203-208

17. *Hansen, N.*; Hutchinson, B.; Houtte, P. van; *Juul Jensen, D.*, Deformation microstructures and textures in steels - Discussion. Phil. Trans. R. Soc. London A (1999) v. 357 p. 1485

18. *Hansen, N.; Juul Jensen, D.*, Development of microstructure in FCC metals during cold work. Phil. Trans. R. Soc. London A (1999) v. 357 p. 1447-1469

19. *Hansen, U.*, Damage development in woven fabric composites during tension-tension fatigue. J. Compos. Mater. (1999) v. 33 p. 614-639

20. Hattel, J.H.; *Pryds, N.H.*; Thorborg, J.; Ottosen, P., A quasi-stationary numerical model of atomized metal droplets. 1: Model formulation. Model. Simul. Mater. Sci. Eng. (1999) v. 7 p. 413-430


21. Heinisch, H.L,; *Singh, B.N.*; Golubov, S.I., Kinetic Monte Carlo studies of the effects of one-dimensional glide on the reaction kinetics of interstitial clusters. In: Fusion materials. Semiannual progress report for the period ending December 31, 1998. DOE/ER-0313/25 (1999) p. 271-274

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25. *Holtappels, P.*; Haart, L.G.J. de; Stimming, U., Reaction of hydrogen water mixtures on nickel-zirconia cermet electrodes 1. DC polarization characteristics. J. Electrochem. Soc. (1999) v. 146 p. 1620-1625

26. *Holtappels, P.*; Haart, L.G.J. De; Stimming, U.; Vinke, I.C.; *Mogensen, M.*, Reaction of $CO/CO_2$ gas mixtures on Ni-YSZ cermet electrodes. J. Appl. Electrochem. (1999) v. 29 p. 561-568

27. *Holtappels, P.*; Vinke, I.C.; Haart, L.G.J. de; Stimming, U., Reaction of hydrogen/ water mixtures on nickel-zirconia cermet electrodes. 2. AC polarization characteristics. J. Electrochem. Soc. (1999) v. 146 p. 2976-2982

28. Howe, A.; Ricks, R.A.; Brown, L.M.; Beynon, J.H.; *Hansen, N.*, The deformation models needed by the aluninium industry - Discussion. Phil. Trans. R. Soc. London A (1999) v. 357 p. 1528-1529

29. *Huang, Y.D.*; Yang, W.Y.; Sun, Z.Q, Improvement of room temperature tensile properties for $Fe_3Al$-based alloys by thermo-mechanical and annealing processes. Mater. Sci. Eng. A (1999) v. 263 p. 75-84

30. Jiang, J.Z.; *Poulsen, F.W.*; Mørup, S., Structure and thermal stability of nanostructured iron-doped zirconia prepared by high-energy ball milling. J. Mater. Res. (1999) v. 14 p. 1343-1352

31. *Juhl Jørgensen, M.; Primdahl, S.; Mogensen, M.*, Characterisation of composite SOFC cathodes using electrochemical impedance spectroscopy. Electrochim. Acta (1999) v. 44 p. 4195-4201

32. *Juul Jensen, D.*; Shercliff, H.R.; Beynon, J.H., Modelling of microstructure evolution in hot deformation - Discussion. Phil. Trans. R. Soc. London A (1999) v. 357 p. 1642-1643

33. Kapranos, P.; *Rheinländer, J.T.*, Quantitative NDE by X-ray radiography for optimisation of the thixocasting process. Insight (1999) v. 41 p. 25-30

34. Kek, D.; *Bonanos, N.*, Electrochemical H-D isotope effect at metal-perovskite proton conductor interfaces. Solid State Ionics (1999) v. 125 p. 345-353

35. Klimanek, P.; Cyrener, K.; Germain, C.; Jenkner, K.; Martin, U.; Oettel, O.; Ostwaldt, A.; Ostwaldt, D.; *Pantleon, W.*, Flow behaviour and microstructure of the heat-resistant steels X20CrMoV12.1 and X5NiCrTiAl32.20 (Alloy 800). In: Microstructure and mechanical properties of metallic high-temperature materials. Mughrabi, H.; Gottstein, G.; Mecking, H.; Riedel, H.; Tobolski, J. (eds.), (Wiley-VCH, Weinheim, 1999) (Sonderforschungsberichte der Deutschen Forschung) p. 272-290

36. *Krieger Lassen, N.C.*, Source point calibration from an arbitrary electron backscattering pattern. J. Microsc. (1999) v. 195 p. 204-211

37. *Larsen, P.H.*; Berg, R.W.; *Poulsen, F.W.*, The influence of $SiO_2$ addition to 2MgO-$Al_2O_3$-3.3$P_2O_5$ glass. J. Non-cryst. Solids (1999) v. 244 p. 16-24

38. Lebensohn, R.A.; *Leffers, T.*, The rules for the lattice rotation accompanying slip as derived from a self-consistent model. Textures Microstruct. (1999) v. 31 p. 217-230

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42. *Lybye, D.; Bonanos, N.*, Proton and oxide ion conductivity of doped $LaScO_3$. Solid State Ionics (1999) v. 125 p. 339-344

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44. *Marina, O.A.; Bagger, C.; Primdahl, S.; Mogensen, M.*, A solid oxide fuel cell with a gadolinia-doped ceria anode: Preparation and performance. Solid State Ionics (1999) v. 123 p. 199-208

45. *Marina, O.A.; Mogensen, M.*, High-temperature conversion of methane on a composite gadolinia-doped ceria-gold electrode. Appl. Catal. A (1999) v. 189 p. 117-126

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47. *Mishin, O.V.*, Statistical characteristics of grain boundary ensembles in variously textured copper. J. Mater. Sci. (1998) v. 33 p. 5137-5143

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49. *Mogensen, M.*; Pearce, J.H.; Walker, C.T., Behaviour of fission gas in the rim region of high burn-up $UO_2$ fuel pellets with particular reference to results from an XRF investigation. J. Nucl. Mater. (1999) v. 264 p. 99-112

50. Ohnuma, M.; Hono, K.; Onodera, H.; Pedersen, J.S.; *Linderoth, S.*, Cu clustering stage before the crystallization in Fe-Si-B-Nb-Cu amorphous alloys. Nanostruct. Mater. (1999) v. 12 p. 693-696

51. Ohnuma, M.; *Pryds, N.H.; Linderoth, S.; Eldrup, M.; Schrøder Pedersen, A.*; Pedersen, J.S., Bulk amorphous $(Mg_{0.98}Al_{0.02})_{60}Cu_{30}Y_{10}$ alloy. Scr. Mater. (1999) v. 41 p. 889-893

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53. Phillips, R.J.; *Bonanos, N.; Poulsen, F.W.; Ahlgren, E.O.*, Structural and electrical characterisation of $SrCe_{1-x}Y_xO_{xi}$. Solid State Ionics (1999) v. 125 p. 389-395

54. *Poulsen, F.W.*, Method for calculating ionic and electronic defect concentrations in proton containing perovskites. J. Solid State Chem. (1999) v. 143 p. 115-121

55. *Poulsen, H.F.; Andersen, L.G.*; Frello, T.; Prantontep, S.; Andersen, N.H.; Garbe, S.; Madsen, J.; Abrahamsen, A.B.; Bentzon, M.D.; Zimmermann, M. von, In situ study of equilibrium phenomena and kinetics in a BiSCCO Ag tape. Physica C (1999) v. 315 p. 254-262

56. *Primdahl, S.; Mogensen, M.*, Gas diffusion impedance in characterization of solid oxide fuel cell anodes. J. Electrochem. Soc. (1999) v. 146 p. 2827-2833

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60. Richert, M.; *Liu, Q.; Hansen, N.*, Microstructural evolution over a large strain range in aluminium deformed by cyclic-extrusion-compression. Mater. Sci. Eng. A (1999) v. 260 p. 275-283

61. *Sarraute, S.; Toft Sørensen, O.; Sørensen, B.F.*; Hansen, E.R., Microstructure dependent thermophysical properties of Ni-Zn ferrite-$BaTiO_3$ functionally graded ceramics. J. Mater. Sci. (1999) v. 34 p. 99-104

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63. *Singh, B.N.*, Atomic displacements and defect accumulation during irradiation with energetic particles: An autobiographical review. Radiat. Eff. Defects Solids (1999) v. 148 p. 383-446

64. *Singh, B.N.*, Damage production, accumulation and materials performance in radiation environment. J. Comput.-Aided Mater. Des. (1999) v. 6 p. 195-214

65. *Singh, B.N.; Horsewell, A.; Toft, P.*, Effects of neutron irradiation on microstructure and mechanical properties of pure iron. J. Nucl. Mater. (1999) v. 271/272 p. 97-101

66. *Singh, B.N.*; Stubbins, J.F.; *Toft, P.*, The influence of neutron irradiation on the fatigue performance of OFHC copper and a dispersion strengthened copper alloy. J. Nucl. Mater. (1999) v. 275 p. 125-137

67. Skov-Hansen, P.; Bay, N.; Grønbæk, J.; *Brøndsted, P.*, Fatigue in cold-forging dies: Tool life analysis. J. Mater. Proc. Technol. (1999) v. 95 p. 40-48

68. *Tao, S.W.*; Wu, Q.Y.; Zhan, Z.L.; Meng, G.Y., Preparation of $LiMO_2$(M=Co,Ni) cathode materials for intermediate temperature fuel cells by sol-gel processes. Solid State Ionics (1999) v. 124 p. 53-59

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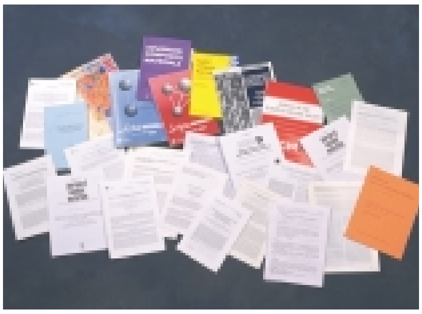

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71. *Toft Sørensen, O.*, RCTA techniques used in studies of solid state reactions in inorganic compounds. J. Therm. Anal. Cal. (1999) v. 56 p. 17-26

72. *Toftegaard, H.*, Simulated stiffness determination from simple compression tests on a thick laminate. Composites A (1999) v. 30 p. 849-858

73. Wang, W.G.; Han, Z.; Skov-Hansen, P.; Goul, J.; Bentzon, M.D.; Vase, P.; *Liu, Y.L.*, High critical current Ag and Ag alloy sheathed multifilament Bi-2223 tapes. IEEE Trans. Appl. Superconduct. (1999) v. 9 p. 2613-2616

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76. Zhou, X.-H.; *Toft Sørensen, O.*; Cao, Q.-X.; Xu, Y.-L., Study on electrical conduction mechanism of Mg-doped $SrTiO_3$ ceramics. J. Inorg. Mater. (1999) v. 164 (no.6) p. 927-932

## Danish Publications

*Danish publications are defined as articles in Danish patents, journals, books and reports, as well as reports and books published in Denmark.*

1. *Andersen, S.I.* (ed.), Svinghjul for energilagring. Svinghjul 3. Slutrapport, fase 1. (Forskningscenter Risø. Afdelingen for Materialeforskning, Roskilde, 1999) vp.

2. *Bagger, C.; Primdahl, S.; Juhl Jørgensen, M.*, Fastoxid brændselscelle. DK Patentansøgning PA 1999 00623

3. *Bilde-Sørensen, J.B.; Carstensen, J.V.; Hansen, N.; Juul Jensen, D.; Leffers, T.; Pantleon, W.; Pedersen, O.B.; Winther, G.* (eds.), Deformation-induced microstructures: Analysis and relation to properties. Proceedings. 20. Risø international symposium on materials science, Risø (DK), 6-10 Sep 1999. (Risø National Laboratory, Roskilde, 1999) 560 p.

4. *Brøndsted, P.*; Grønning Sørensen, K. (eds.), Metallurgiske processer til design af strukturer og egenskaber. Dansk Metallurgisk Selskabs vintermøde, Horsens (DK), 6-8 Jan 1999. (DMS, Lyngby, 1999) 378 p.

5. *Larsen, P.H.*, Sealing materials for solid oxide fuel cells. Risø-R-1105(EN) (1999) 187 p. (Ph.D. thesis)

6. *Lybye, D.; Mogensen, M.*, Strukturelle og elektriske egenskaber af oxidionledende perovskitter. Tidsskr. Dansk Keramisk Selskab (1998) v. 1 (no.2) p. 28-34

7. Lynov, J.P.; *Singh, B.N.* (eds.), Association Euratom - Risø National Laboratory annual progress report for 1998. Risø-R-1136(EN) (1999) 46 p.

8. *Winther, G.; Hansen, N.* (eds.), Materials Research Department annual report 1998. Risø-R-1098(EN) (1999) 64 p.

9. *Mogensen, M.*, Improving durability of SOFC stacks (''IDUSOFC''). Publishable final report. 1/1 1996 to 31/12 1998. (Risø National Laboratory. Materials Research Department, Roskilde, 1999) 10 p.

10. *Primdahl, S.*, Nickel/yttria-stabilised zirconia cermet anodes for solid oxide fuel cells. Risø-R-1137(EN) (1999) 199 p. (Ph.D. thesis)

11. *Primdahl, S.; Juhl Jørgensen, M.*, Introduktion til keramiske brændselsceller. Tidsskr. Dansk Keramisk Selskab (1999) v. 2 (no.1) p. 13-20

12. *Toft Sørensen, O.*, Keramiske gassensorer. Dansk Kemi (1999) v. 80 (no.10) p. 24-27

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## Publications in Proceedings

*This list contains papers published in conference proceedings, published by international or Danish publishers.*

1. *Bagger, C.; Linderoth, S.; Mogensen, M.; Hendriksen, P.V.; Kindl, B.; Primdahl, S.; Larsen, P.H.; Bonanos, N.; Poulsen, F.W.; Juhl Jørgensen, M.*, Status of Danish solid oxide fuel cell R and D. In: Proceedings. 6. International symposium on solid oxide fuel cells (SOFC 6), Honolulu, HI (US), 17-22 Oct 1999. Singhal, S.C.; Dokiya, M. (eds.), (Electrochemical Society, Pennington, NJ, 1999) (Proceedings volume PV 99-19) p. 28-35

2. Barlow, C.Y.; *Liu, Y.L.*, High-strain deformation of aluminium containing small alumina particles. In: Deformation-induced microstructures: Analysis and relation to properties. Proceedings. 20. Risø international symposium on materials science, Risø (DK), 6-10 Sep 1999. Bilde-Sørensen, J.B.; Carstensen, J.V.; Hansen, N.; Juul Jensen, D.; Leffers, T.; Pantleon, W.; Pedersen, O.B.; Winther, G. (eds.), (Risø National Laboratory, Roskilde, 1999) p. 261-267

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4. *Brøndsted, P.; Andersen, S.I.; Lilholt, H.*, Fatigue and damping properties of glass/polyester materials for vehicle leaf springs. In: Proceedings. 7. Euro-Japanese symposium on composite materials and transportation, Ecole des Mines de Paris (FR), 1-2 Jul 1999. (French Association for Composite Materials (AMAC), Ecoles de Mines de Paris, 1999) 10 p.

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7. Crostack, H.-A.; Fischer, G.; Soppa, E.; Schmauder, S.; *Liu, Y.L.*, Localization of strain in metal-matrix composites. In: ICCE 6. 6. International conference on composites engineering, Orlando, FL (US), 27 Jun - 3 Jul 1999. Hui, D. (ed.), (University of New Orleans, New Orleans, 1999) p. 223-224

8. *Gamstedt, E.K.*, Fatigue in composite laminates - a qualitative link from micromechanics to fatigue life performance. In: DURACOSYS '99. Extended abstracts, Brussels (BE), 11-14 Jul 1999. Cordon, A. (ed.), (Free University of Brussels, Brussels, 1999) p. 1-5

9. *Gamstedt, E.K.; Brøndsted, P.*, Damage dissipation and localization during fatigue in unidirectional glass/carbon fibre hybrid composites. In: Proceedings of the 7. International fatigue congress. Vol. 3. Fatigue '99, Beijing (CN), 8-12 Jun 1999. Wu, X.R.; Wang, Z.G. (eds.), (EMAS, Cradley Heath, 1999) p. 1731-1736

10. Ghoniem, N.M.; *Singh, B.N.*, Dislocation dynamics study of the onset of plastic instabilities in irradiated materials. In: Deformation-induced microstructures: Analysis and relation to properties. Proceedings. 20. Risø internatio-

nal symposium on materials science, Risø (DK), 6-10 Sep 1999. Bilde-Sørensen, J.B.; Carstensen, J.V.; Hansen, N.; Juul Jensen, D.; Leffers, T.; Pantleon, W.; Pedersen, O.B.; Winther, G. (eds.), (Risø National Laboratory, Roskilde, 1999) p. 41-59

11. *Gundtoft, H.E.; Borum, K.K.*, Characterization of composites by ultrasonic-scanning. Examples and experience from participation in European and national projects. In: ICCE 6. 6. International conference on composites engineering, Orlando, FL (US), 27 Jun - 3 Jul 1999. Hui, D. (ed.), (University of New Orleans, New Orleans, 1999) p. 273-274

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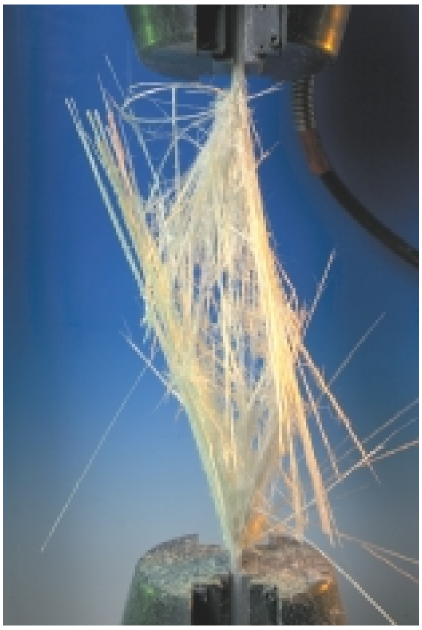

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Trees fell for the hurricane 3 December

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1. *Leffers, T.; Pryds, N.H.*, Gør metaller bedre. Risønyt (1999) (no.4) p. 10-11

# Abbreviations

## Organisations, etc.

| | |
|---|---|
| **AAU** | Aalborg University |
| **ASTM** | American Society for Testing and Materials |
| **AU** | University of Aarhus |
| **BRITE** | An EU programme, Basic Research in Industrial Technology for Europe |
| **DANAK** | Danish accreditation body |
| **DTI** | Danish Technological Institute |
| **DTU** | The Technical University of Denmark |
| **EFP** | The Energy Research Programme of The Danish Ministry of Environment and Energy |
| **ESRF** | European Synchrotron Radiation Facility |
| **EUCLID** | European Cooperation for the Long term In Defense |
| **EUREKA** | A Europe-wide Network for Industrial R&D |
| **ITER** | International Thermo-nuclear Experimental Reactor |
| **IVC** | Engineering Science Centre. A programme under The Danish Technical Research Council |
| **JOULE** | Joint Opportunities for Unconventional or Long-term Energy Supply. An EU programme on non-nuclear energy and rational exploitation of energy |
| **KU** | University of Copenhagen |
| **LTH** | Lund Institute of Technology |
| **MUP** | The Danish Materials Technology Programme |
| **NTNU** | Norwegian University of Technology and Science |
| **OU** | University of Odense |
| **PSO** | Public Service Obligation |
| **SJVF** | The Danish Agricultural & Veterinary Research Council |
| **SNF** | The Danish Natural Science Research Council |
| **STVF** | The Danish Technical Research Council |
| **VAMAS** | Versaille Project on Advanced Materials and Standards |

## Technical abbreviations

| | |
|---|---|
| **BET** | A method for measuring surface area of powders named after Brunauer, Emmett and Teller |
| **CISH** | Cascade induced source hardening |
| **CM** | Centre of mass |
| **CMC** | Ceramic matrix composite |
| **DKK** | Danish kroner |
| **dpa** | Displacements per atom |
| **DSC** | Differential scanning calorimetry |
| **DR3** | Danish Reactor 3. A nuclear research reactor at Risø |
| **DTA** | Differential thermal analysis |
| **EBS** | Electron back-scattering |
| **EBSD** | Electron back-scattering diffraction |
| **EDS** | Energy dispersive (X-ray) spectrometry |
| **EELS** | Electron energy loss spectrometry |
| **ESEM** | Environmental scanning electron microscope |
| **GNB** | Geometrically necessary boundary |
| **HEU** | High enriched uranium |
| **HIP** | Hot isostatic pressing |
| **IDB** | Incidental dislocation boundary |
| **ISB** | Intense slip bands |
| **ISL** | Intense slip lines |
| **LEU** | Low enriched uranium |
| **LSB** | Large scale bridging |
| **LSM** | Lanthanum strontium manganite. A ceramic material used in SOFCs |
| **LVSEM** | Low vacuum scanning electron microscope |
| **MMC** | Metal matrix composite |
| **NEB** | Nudged elastic band |
| **ODS** | Oxide dispersion strengthening |
| **PAS** | Positron annihilation spectroscopy |
| **PBM** | Production bias model |
| **PMC** | Polymer matrix composite |
| **PSB** | Persistent slip band |
| **RTM** | Resin transfer moulding |
| **SEM** | Scanning electron microscope |
| **SIA** | Self interstitial atom |
| **SOFC** | Solid oxide fuel cell |
| **TEM** | Transmission electron microscopy |
| **TGA** | Thermogravimetric analysis |
| **YSZ** | Yttria stabilized zirconia |
| **3D** | Three-dimensional |
| **3DXRD** | Three-dimensional X-ray diffraction |

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ISBN 87-550-2644-3
ISBN 87-550-2645-1 (Internet)
ISSN 0106-2840
ISSN 1397-8071

Also available on Internet. URL-address:
http://www.risoe.dk/afm/index.htm