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Winther, Grethe; Hansen, Niels

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Materials Research Department

Annual Report 2000

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ABSTRACT

Selected activities of the Materials Research Department at Risø National Laboratory during 2000 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 industrial collaboration, educational activities and academic activities, such as collaboration with other research institutions, committee work and a list of publications. Furthermore, the main figures outlining the funding and expenditures of the Department are given. Lists of staff members and visiting scientists are included.

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Editors:	Grethe Winther Niels Hansen
Editorial Board:	Nikolaos Bonanos Morten Eldrup Kristofer Gamsted Torben Leffers Wolfgang Pantleon Eva Sørensen
Journalistic consultant:	Jørgen Hornemann
Photos:	Boye Koch Helmer Nilsson Preben B. Olesen
Layout:	Finn Hagen Madsen
Repro and printing:	Holbæk Center-Tryk
Cover illustrations:	Microstructures of textiles

Materials Research Department
Risø National Laboratory
P.O. Box 49
Frederiksborgvej 399
4000 Roskilde
DENMARK

Phone	+45 4677 5700
Fax	+45 4677 5758
E-mail	materials@risoe.dk
URL address	http://www.risoe.dk/afm/index.htm

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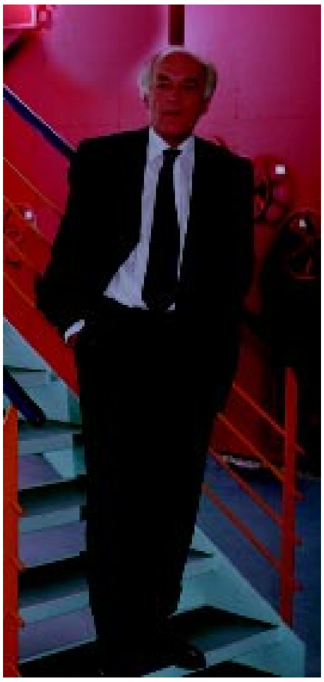
Introduction



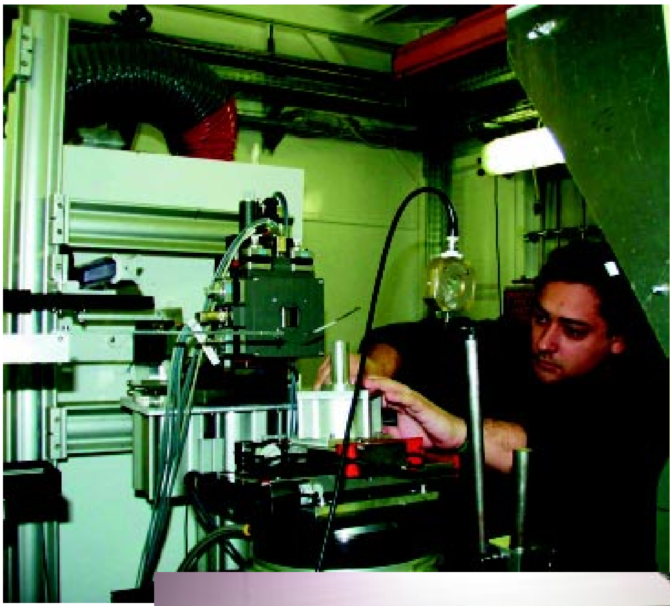
Dr. Niels Hansen, founder and Head of the Department, retired on 31st December 2000 having reached the obligatory age of retirement. The pictures are from the Department's tribute to him in December.

The year 2000 has in many ways been regarded as a special year. For the Department it was extraordinary successful scientifically, technically as well as financially. In our basic research a focal point was our first generation three-dimensional X-ray diffraction (3DXRD) microscope operating at the European Synchrotron Radiation Facility in Grenoble, France. A number of critical in-situ experiments have been carried out and new insight has been gained studying processes taking place during deformation and annealing of metals. The results point to several ways in which established physical models can be expanded and improved.

Other achievements in basic research relate to atomistic modelling of ultrafast plastic deformation in collaboration with the Department of Physics at the Technical University of Denmark and the characterisation of amorphous alloys produced by rapid solidification. Within solid oxide fuel cells (SOFC), the Department has succeeded in establishing a full 3-year programme for the development of cells and small fuel cell stacks as well as for underlying long-term research. The programme is sponsored by the Ministry of Energy and Environment (about 20%), whereas the rest of the DKK 100 mill. programme is financed by the power utilities (Elkraft System, Eltra), industry (Haldor Topsøe A/S) and Risø. As part of the programme a 300 m^2 prepilot plant is being built in the Department for operation in the beginning of 2001. The technical work will be carried out in a close collaboration between the Department and Haldor Topsøe A/S, the latter being responsible for bringing the SOFC technology to the market.

Yet another important achievement has been the procurement of funding for a new analytical 300 kV transmission electron microscope in collaboration with researchers from University of Copenhagen, University of Aarhus and Technical University of Denmark. The funding is made up of grants from the Danish Research Councils with a substantial contribution from Risø. The new microscope will be placed in the Department and operated by the Department as a national centre facility. Finally, the 21st Risø Symposium on Materials Science was held with more than 100 participants from all over the world. The theme this year was "Recrystallisation – Fundamental Aspects and Relations to Deformation Microstructure".

The new projects and improved financing for energy projects sponsored by the Public Service Obligation (PSO) programme have raised the turnover to about DKK 40 mill. (a 25% increase over 1999) and a surplus of about 5% of the turnover was anticipated. However, the Risø Board unexpectedly decided in September not to restart the DR3 reactor after a shut down of about half a year. The result of this decision was cancellation of several contracts which eliminated the surplus. This is a onetime revision and good economy is foreseen for the coming years with a project



New and upcoming facilities

Top: The first generation three-dimensional X-ray diffraction microscope became operational in 1999 and has so far resulted in 19 scientific publications.
Middle: A prepilot plant for ceramic processing will be ready for use in April 2001.
Bottom: A new analytical transmission electron microscope will be installed in October 2001.

turnover of about DKK 40 mill. and a surplus of about DKK 2 mill. which is needed for new equipment and temporary staff.

Follow-up on the 1997 International Evaluation

The Department, as the rest of Risø, was internationally evaluated in the beginning of 1997 and will be evaluated again in the beginning of 2001. The 1997 evaluation acknowledged the international standing of the Department and also its role in "establishing connections from the Danish environment to major European companies". The evaluation committee also found it to be "an important future task for the Department to increase the commercial results from its research activities". This advice together with others may be summed up as a general recommendation to strengthen the links between the Department and industry. This has been dealt with in several ways. (i) The Department has hired two external senior consultants to advice on various approaches in order to increase the contacts between the Department and the industrial and energy sectors, respectively. (ii) Staff have visited various key industries and in 1999 the Department started to publish a newsletter in Danish. The newsletter published three times a year has been very effective in creating contacts both to "old" and "new" customers. (iii) A number of PhD students under the auspices of the Engineering Science Centre was given projects of industrial relevance in collaboration with an industrial company. (iv) A number of summer jobs (about 5 a year) has been created for undergraduate students in a collaboration with industry on problems of current industrial interest.

It is of course difficult to relate initiatives and results directly, but the facts show that the number of industrial partners has increased significantly during the last four years, in Denmark from 27 to 39 for externally funded projects and the commercial income has raised from about DKK 1.8 mill. to DKK 9.3 mill.

Advisory Committee

The Advisory Committee to the Department had its first meeting in 2000. The committee has 8 external members: 4 from industry and 4 from academia. Half of the members are from Denmark and half from the United Kingdom and Germany. An introductory meeting was held in March followed by a second meeting in November to discuss many of the programmes for the coming year. A recent decision by the Risø Management to split the Department of Materials Physics and Chemistry into a Polymer Department and a Physics Group and integrate the latter (about 20 persons) with the Department from 1st January 2001 was also discussed.

The Past and the Future

Research and development of the Department has since its founding been directed towards the energy sector: Initially concentrated on components (especially fuel elements and pressure vessels) for nuclear reactors and in the last 25 years directed towards (i) energy production (fuel cells, wind turbine blades and fusion materials), (ii) energy transport (high T_c superconductors) and (iii) energy storage (fly wheels and metal hydrides). The energy areas of research are still of great importance for the Danish and international society as long term solution of many of the energy related problems (e.g. clean energy production and energy storage) are still missing. Another area is materials and components for the transport sector to reduce pollution and to improve economy and safety. Projects focus especially on light and strong materials, such as polymer-based composites and light alloys based on aluminium and magnesium. Most of the energy and

At the end of its second meeting, the Advisory Committee to the Department drafted a number of comments where those of more general nature are given below:

> *"During the meeting the committee received an excellent going through of the running research programmes through seven presentations.*
>
> *After the presentations the committee had a closed meeting without participation of the members from Risø and concluded the following:*
>
> *It is the opinion of the committee that the research of the Materials Research Department is still at a high level and strongly placed internationally. To a large extent it excels other Danish materials science groups.*
>
> *The committee recommends steps to further co-operation with Danish and international industry.*
>
> *It is valued that the Department through PhD students contributes to education in an area that we find is undersupplied with education possibilities in Denmark."*

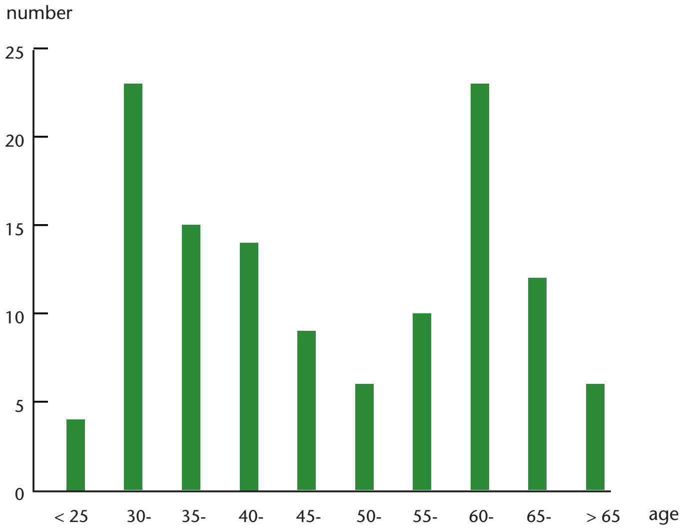
transport projects have a strong industrial participation of both Danish and European companies. Here the Department has a double role; to establish close links to the Danish partners and to bridge European and Danish research and development. This role of the Department is expanding.

The Department has also since its founding combined its energy mission with educational and research activities in collaboration with Danish and foreign universities. Funding for educational activities, especially through the Engineering Science Centre, has allowed a number of graduate students (at present 17) to carry out their PhD studies in the Department. However, the Engineering Science Centre ends in 2002 and there is no apparent solution as to how a large PhD programme can be continued after that year. Besides PhD training the Department is involved in many other educational activities ranging from undergraduate training to post doctoral research projects. With respect to international contacts the Post Doc. programme is especially important as it attracts many graduates from many different countries. Finally, to motivate young people, high school students and teachers are trained in the Department.

The positive trend in collaboration with utilities and industry together with the educational activities have fulfilled almost all the challenges mentioned in the introduction of last year's annual report. The last challenge was to maintain basic research in the Department at an international level. A shortlisting by the Danish National Research Foundation of a large research proposal entitled "Metal Structures in 4 dimensions" is a very positive development in this respect.

In conclusion the Department is well prepared for the coming years. There is at present a good balance between basic research, energy and industry research and educational activities. Also there is a very strong international collaboration which is considered vital for the future of the Department. The balance we see today between the different activities may change in the future. However, the Department in its lifetime has adapted to many changes and at present it is strongly based with a competent, experienced and motivated staff having a broad age distribution, a large number of Danish and international contacts, a healthy economy and an acknowledged scientific and technical status.

The Department's staff and their age distribution



Materials Science

- Theory and characterisation

This section covers the research activities within three programmes: (i) Modelling of Materials and Structures of Materials, (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:

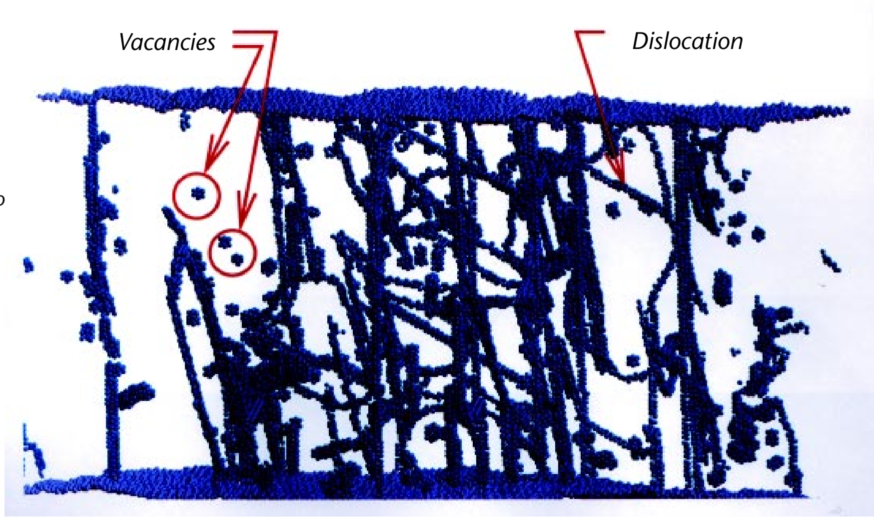
i. Determination of cross-slip rates by molecular-dynamics simulation
ii. Atomistic modelling of ultra-fast plastic deformation
iii. Formulation of a multiscale dislocation theory for cyclic plasticity and fatigue of wavy slip and planar slip materials in terms of edge dipole destabilisation, point defect diffusion and cross slip of jogged and unjogged screw dislocations
iv. Experimental identification and statistical modelling of correlations between disorientations in neighbouring dislocation boundaries
v. In-situ 3D X-ray diffraction on individual grains in the bulk of aluminium polycrystals during deformation and recrystallisation
vi. Determination of lifetime and number of cycles to failure as a function of holdtime in creep-fatigue interaction experiments on a dispersion hardened copper alloy before and after irradiation
vii. Investigation of the dynamics of dislocation interaction in copper with interstitial clusters decorating the dislocations and stacking fault tetrahedra in the matrix, using 3D dislocation dynamics methodology

Modelling of Materials and Structures of Materials

This programme focuses on the formulation of micromechanical models for the mechanical behaviour of materials with due consideration of the actual microstructures. In the latest years there has been an increasing input from modelling at the atomic scale, in collaboration with Department of Physics at DTU. Atomic-scale modelling and its relation to phenomena observed at coarser scales is one of the topics selected for this annual report. The second topic is a multiscale theory of fatigue. Of other active research areas, one may list the following: plastic deformation of polycrystals, especially for face centred cubic materials with low stacking fault energy, analytical modelling of effects of inclusion orientation distribution on the internal stresses in composites, diffusion creep with emphasis on the behaviour of different types of grain boundaries, and electron microscopical investigation of delocalisation of dislocation cores.

Measurement of residual stresses/internal stresses by neutron diffraction is one of the research themes within the programme. The special advantage of neutron diffraction, as compared with traditional stress measurements by X-ray diffraction, is that neutrons penetrate into the bulk of the materials whereas traditional X-rays only monitor the stresses in a thin surface layer. Hard X-rays, as produced in synchrotrons, also penetrate into the bulk, and synchrotron radiation has the added advantage of a very high intensity so that one can collimate the beam very narrowly and hence provide a spatial resolution that cannot be achieved with neutrons. For instance, one may measure the stresses in individual grains. The intensity advantage of synchrotron radiation has already been exploited in preliminary experiments. The closedown of DR3 at Risø will obviously lead to further focus on stress measurements with synchrotron radiation.

The Engineering Science Centre for Structural Characterisation and Modelling of Materials, financed jointly by the Danish Technical Research Council and Risø, is managed within the programme. The centre consists of a core financed by the research council and a shell financed by Risø. In the past the core activities have been concentrated in this programme, while the shell activities have been distributed in various programmes. The centre is now well into its second and last five-year period, which focuses on activities which may continue after the end of 2002 when the funding from the research council will end. This has enforced a redistribution of the available resources so that a larger part of the funding goes to promising activities in other programmes such as the three-dimensional X-ray diffraction microscope, superconductors, fatigue of tool materials for cold forging, composite materials and modelling of the spray-forming process.



Copper foil computer-deformed at ultra-high rate. Atoms at the surface are shown in lilac. Atoms next to a vacancy or a dislocation core are shown in turquoise. All other atoms are invisible.

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 • NKT Cables A/S, Denmark • NKT Research Centre A/S, Denmark • Dept. of Appl. Eng. Design and Production, DTU, Denmark • Dept. of Electric Power Eng., DTU, Denmark • Research Assoc. of Danish Electric Utilities
Danish Natural Science Research Council Danish Centre for X-ray Synchrotron Radiation (DanSync)	• Novo Nordisk A/S, Denmark • CISMI, Symbion, Denmark • Condensed Matter Physics and Chemistry Department, Risø • Danish Space Research Institute, Denmark • Department of Physics, DTU, Denmark • H. C. Ørsted Laboratory, KU, Denmark • Haldor Topsøe A/S, Denmark • Department of Chemistry, AU, Denmark • Institute of Chemistry, KU, Denmark • Royal Danish School of Pharmacy, Denmark • Department of Chemistry, DTU, Denmark
GROWTH Welding of Airframes by Friction Stir (WAFS)	• British Aerospace (Operations) Ltd, UK • GIE Aerospatiale Matra CCR, France • DaimlerChrysler Aerospace Airbus GmbH, Germany • Alenia Aerospazio-Un' Azienda Finmeccanica SpA (IMPT), Italy • Construcciones Aeronauticas SA, Spain • SA Belge de Constructions Aeronautiques (SABCA), Belgium • Alenia Aerospazio-Un' Azienda Finmeccanica SpA (PU), Italy • Pechiney S.A., France • Institut de Soudure, France • DaimlerChrysler AG • Deutsches Zentrum für Luft- und Raumfahrt eV, Germany • GKSS - Forschungszentrum Geesthacht GmbH, Germany • DaimlerChrysler Aerospace AG, Germany
Competitive and Sustainable Growth Joining Dissimilar Materials and Composites by Friction Stir Welding	• CRF, Italy • EADS, Germany • ISMIE, Portugal • ISQ, Portugal • Rutherford Appleton Laboratory, UK • University of Manchester, UK • University of Cambridge, UK
Large Installation Programme (LIP) Neutron Diffraction	• Condensed Matter Physics and Chemistry Department, Risø • DR3, Risø, Denmark
BRITE-EURAM Graintwist	• Plansee, Germany/Austria • Mitsui Babcock Energy Ltd., UK • Metall-SpecialRohr GmbH, Germany • University of Cambridge, UK • University of Liverpool, UK

Continues on next page

Atomistic studies of dislocation dynamics

Cross slip is a thermally activated process of great importance for monotonic and cyclic plasticity. The activation energy for cross slip of non-jogged and jogged screw dislocations has been determined by atomic-scale modelling. In order to estimate actual cross slip rates one must also know the pre-exponential in the Arrhenius expression. Pre-exponentials for simple thermally activated transport processes involving few atoms like vacancy diffusion are fairly well known and of the order of 10^{13} s^{-1}. Cross slip, on the other hand, involves a fairly large number of atoms along the dislocation line, and the pre-exponentials for such processes have not been investigated before by modelling at the atomic scale. The annihilation (in copper) of non-jogged screw-dislocation dipoles by cross slip was investigated with molecular dynamics in collaboration with Department of Physics at DTU. In practice such an investigation is only possible for very narrow dipoles - with low activation energy for cross slip. The investigated dipole height was six {111} interplanar distances. The times for annihilation were determined from 19 computer experiments for different temperatures in the range 225-375 K. From an Arrhenius plot one could calculate an activation energy (0.29 eV) very close to the activation energy determined earlier by the 'nudged elastic band' method (0.32 eV). The pre-exponential turned out to be $2{\cdot}10^{23}$ $m^{-1}s^{-1}$ or $5{\cdot}10^{13}$ s^{-1} per Burgers vector along the dislocation.

Expressed in the latter way the pre-exponential for cross slip comes close to conventional pre-exponentials for simple processes.

As part of the association with the Academic Frontier Research Centre for Ultra-High-Speed Plastic Deformation in Hiroshima the ultra-fast deformation of initially dislocation-free copper was investigated by atomic-scale modelling, again in collaboration with Department of Physics at DTU. It turns out that dislocations are generated spontaneously and move subsequently to create plastic deformation. This apparently contradicts the idea forwarded from the above centre, that ultra-fast plastic deformation takes place by a mechanism without dislocation motion. During computer deformation a large number of vacancies are generated in agreement with experimental observations at the centre.

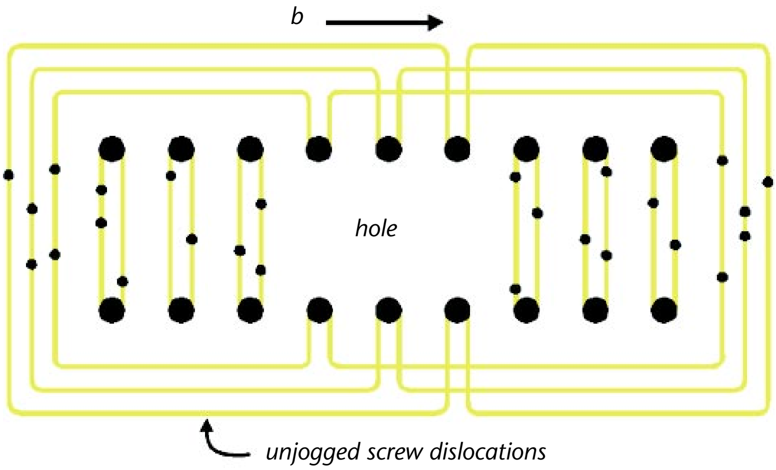
In two cases, results of atomistic studies have found applications in the investigation of materials behaviour at coarser scales. In 1968 the activation energy for the rolling-texture transition in face centred cubic materials was estimated on the basis of the dependence of the texture in Cu-5%Zn on strain rate and temperature. The experimental activation energy is 0.72 eV. Since the texture transition also depends on stacking fault energy, cross slip is an obvious candidate for the thermally activated process behind the texture transition. However, in 1968 the activation energy for cross slip was not really known and, therefore, the experimentally determined activation energy had limited significance. Now it is known from atomistic modelling that the activation energy for cross slip of jogged screw dislocations in 'computer copper' (which has a stacking fault energy similar to that of real Cu-5%Zn) is 0.87 eV, close to the experimental value of 0.72 eV. Thus, there is now a solid basis for ascribing the texture transition to cross slip. The other case, referring to the identification of the thermally activated process governing the macroscopic strength and microscopic strain localisation in metal fatigue, will be reported in the following.

BRITE-EURAM Residual Stress Standard using Neutron Diffraction (RESTAND)	• Rolls-Royce - Gas Turbines, UK • Volkswagen AG, Germany • British Aerospace - Airbus, UK • Sintech Keramik, Germany • Schunk Kohlenstofftechnik, Germany • AEA Technology, UK • Rutherford Appleton Laboratory, UK • Hahn-Meitner Institut, Germany • Institute Laue Langevin, France • NFL (Studsvik), Sweden • Joint Research Centre Petten, The Netherlands • University of Salford, UK • Imperial College, UK • University of Cambridge, UK
BRITE-EURAM Thematic Network Training Industry in Neutron Strain Scanning (TRAINSS)	• British Aerospace, UK • CRF, Italy • FLAMATEL, Italy • ISQ, Portugal • DaimlerChrysler AG, Germany • NEL, UK • Nuova, Italy • PSA, France • SNCF, France • Technatom, Spain • Rutherford Appleton Laboratory, UK • University of Manchester, UK • The Open University, UK • Uppsala University, Sweden • University of Ancona, Italy • Katholieke Universitiet Leuven, Belgium • Kiel University, Germany • ENSAM, France • Laboratoire Léon Brillouin, France
EU-Fusion Technology Programme (EU-FTP) Next Step Technology. International Thermonuclear Experimental Reactor R & D (ITER) 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: - Pacific Northwest National Laboratory, USA - University of Illinois, USA - VTT Manufacturing Technology, Finland
EU-FTP Underlying Technology Effects of Irradiation on Physical and Mechanical Properties of Metals and Alloys	• EU-sponsored, in collaboration with: - Oak Ridge National Laboratory, USA - London University, UK - Research Centre Jülich, Germany - Inst. of Physics and Power Engr., Russia - National Research Institute for Metals, Japan - Pacific Northwest National Laboratory, USA
EU-FTP Long Term Materials Programme Effects of Irradiation on Deformation Behaviour of Iron and Low Activation Steels	• EU-sponsored, in collaboration with: - CRRP, Switzerland - Research Centre Jülich, Germany - AEA Technology, UK - UCLA, USA

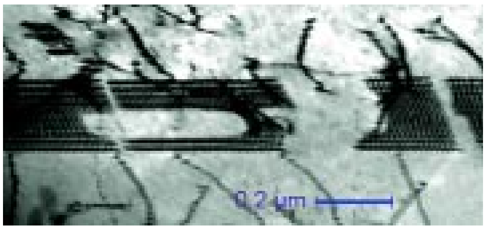
Multiscale theory and characterisation of fatigue in wavy and planar slip materials

Observation of fatigue damage shows that ductile materials spend by far the longest part of their fatigue lives in a process involving several length scales with initiation, growth and coalescence of microcracks into macroscopic cracks. The transformation of slip bands into microcracks must be characterised and modelled on the microscale before the rate of damage can be determined.

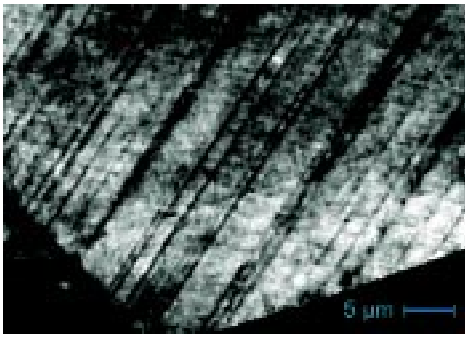
The channelling contrast method (CCM) is a novel multiscale characterisation method, which complements traditional transmission electron microscopy (TEM). In particular, the CCM allows large areas of the dislocation microstructures just beneath polished surfaces to be investigated, for example with a view to selecting sites for high-resolution observation by atomic force microscopy. In collaboration with the Dresden University of Technology the CCM was for the first time applied to a planar slip material, polycrystalline α-brass. The CCM was combined with the electron back-scattering diffraction technique to correlate the crystallographic orientation of individual crystal grains with characteristic dislocation microstructures recognised from TEM observations. Only grains near <100> and <111> orientations contained planar structures on more than one slip system. Even in polycrystals deformed at plastic strain amplitudes as high as $5 \cdot 10^{-3}$,



a) Sketch of the process of destabilisation of a planar array of narrow edge dipole loops (EDLs). The habit plane of the array is inclined by a small angle to the primary slip plane, and superjogs and elementary jogs are indicated by large and small black dots. The emission of jogged edge and unjogged screw dislocations leaves 'holes' in the array.



(b) TEM micrograph of a planar EDL array on the primary slip plane in cyclically hardened polycrystalline α-brass. The holes are interpreted to form by the destabilisation process sketched in (a).



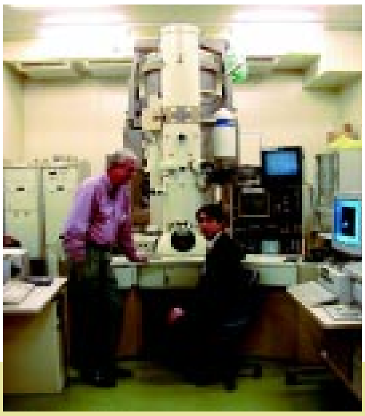
(c) SEM channelling contrast micrograph of planar arrays of primary EDLs viewed close to the specimen surface at a large angle to their habit plane in a grain of polycrystalline α-brass.

most of the grains contain structures dominated by primary dislocations.

A multiscale theory of fatigue in planar and wavy slip materials was formulated on the basis of the idea that slip line initiation requires destabilisation of primary finite edge dislocation dipoles (edge dipole loops, EDLs) forming by cross-slip processes. In planar slip materials, planar arrays of EDLs form during a primary cyclic hardening stage resembling stage I of tensile deformation. The theory shows that destabilisation leaves holes in the arrays, as confirmed by TEM. The holes are associated with cyclic softening and formation of slip lines with limited persistence. In wavy slip materials the primary hardening stage gives way to rapid hardening controlled by the internal stresses carried by regularly spaced walls dense with EDLs. The destabilisation of these walls releases mobile edge dislocations, which subsequently reorganise the destabilised structure during a strain carried by the edge dislocations on the slip line.

This instability model has the important feature that the gliding edge segments of destabilised EDLs leave trailing screw dislocations with no jogs. The additional 'annihilation strain' (produced on slip lines when the unjogged screws glide to nanoscale annihilation by cross-slip) can therefore be determined with perfectly justified use of the atomistic computations of activation energies for annihilation of unjogged screw dislocation dipoles. Thus, without introducing any free parameters, the multiscale theory accounts accurately for the whole range of local plastic shear strains displayed by slip lines in copper.

The partial internal stress-, instability- and cross-slip models are all reconciled and combined in the multiscale theory. While internal stresses and instability account for the rate of production of slip lines and their strain amplitudes, they do not account for the temperature dependence of the saturation stress. The saturation stress is seen simply as the stress required to destabilise a jogged screw dislocation dipole at the point of annihilation. This idea immediately suggests why the saturation stress is strongly dependent on temperature, but it involves the annihilation height, which is a dynamical parameter requiring atomistic modelling. Atomistic modelling shows that the screw dipole height may be reduced by jog migration to the point of spontaneous annihilation. This gradual process is non-conservative. It requires a steady supply of jogs and hence diffusion of point defects, which may be produced during slip instability. Alternatively, thermal fluctuations may overcome the energy barrier against initiation of cross-slip at the jog, which leads to a purely conservative annihilation process. The actual rates of the two possible recovery processes must be calculated atomistically, so as to identify the thermally activated process governing the saturation stress, i. e. the fatigue endurance limit.



Touring in Japan for demonstration of the new microscope

New analytical transmission electron microscope

In addition to the microscopes already present in the Department a 300 kV JEOL 3000F transmission electron microscope (TEM) will be purchased equipped with field emission gun (FEG), parallel electron energy loss spectrometer and imaging filter, energy-dispersive X-ray spectrometer, scanning transmission unit and other attachments. This will be the first FEG-TEM in the public sector in Denmark, and it will greatly improve the potential for Danish researchers for the characterisation of materials – in particular in the field of chemical analysis at the nanometre level. The installation is expected to begin in October 2001.

The purchase is based on grants from the research council programme 'Expensive apparatus' and from the Danish Technical Research Council, supplemented with a substantial contribution from Risø. The applications were written together with researchers from University of Copenhagen, University of Aarhus and the Technical University of Denmark. The new microscope will be placed at Risø as a centre facility with access for microscopists from universities, research laboratories or industrial laboratories.

Local Structure and Properties

This program was established in 1997 and the overall aims have constantly been the understanding of the development of local structure and local texture during mechanical and thermal treatments as well as the understanding of relations between structure and properties. The work has developed from being focused on quantitative characterisation of typical polycrystalline metals to studies of (i) model materials for example single crystals of selected crystallographic orientations, and (ii) industrial materials often hot-deformed. At present, a key effort is modelling. With the knowledge achieved during the previous years, there is now a basis for developing a new type of models. Such a model for plastic deformation incorporates the observed micrometre scale grain subdivision, an important phenomenon previously not considered. A goal for the future is the development of a model, which simultaneously predicts the evolution in structure and local texture during deformation.

It is characteristic for the programme that significant breakthroughs in understanding have followed the development of new experimental techniques. During the period from 1988 to 1996 a method to automate the indexing of electron back-scattering patterns was developed. Also methods for automatic indexing of Kikuchi patterns in TEM were developed. These two developments formed the necessary basis for the scientific progress summarised above. The newest experimental technique developed within the group is a so-called 3D X-ray Diffraction (3DXRD) microscope operating at the European Synchrotron Radiation Facility in Grenoble, France. This allows non-destructive measurements of crystallographic orientations and internal strains in small local volumes within the bulk of typical metal samples. The instrument became operative in the summer of 1999. Since focus has been on in-situ experiments during tensile deformation and during annealing. Specific features of the microstructure, for example grain boundaries, can be located with a precision of approximately 25 µm. A future aim is to improve this spatial resolution. An important task is also the development of software for fast, automatic analysis of the often very large data sets (up to 10 Gbyte/h) collected using this technique.

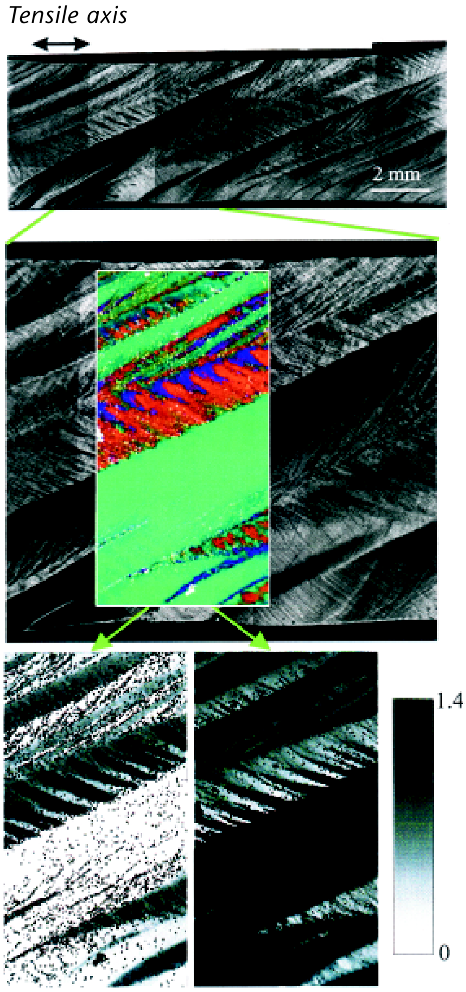
An overall trend for the future of the programme is to exploit the new insight offered by the 3DXRD microscope and perform experiments furthering the fundamental understanding of the basic processes occurring during plastic deformation and annealing. Two themes new to the programme will be initiated, namely investigations of multiphase alloys and nano-scale structures. These themes have been selected based on their timeliness, their connection to the present work and their need for the techniques available today or under development.

Correlation between disorientations in deformation structures

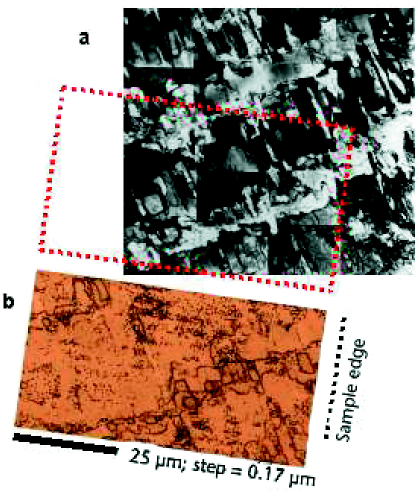
Mobile dislocations are the carriers of plastic deformation. They are emitted from dislocation sources. Dislocations of opposite sign created in the same event move in opposite direction and split up. After travelling along their free path, they become immobilised and gather into dislocation rotation boundaries. A surplus of dislocations of one sign leads to an orientation difference across the boundary. Owing to the pairwise emission of mobile dislocations of opposite sign and their limited free path, the disorientations across neighbouring boundaries are not independent. The occurrence of a spatial correlation between the disorientations is modelled by analogy to queuing theory. Electron back-scattering diffraction (EBSD) and X-ray diffraction show experimental evidence for such a correlation between disorientation angles across neighbouring boundaries in a deformation structure. The orientation difference across several boundaries increases with their number. As predicted by the model, the disorientation angle across several boundaries saturates after a few boundaries, and no evidence for long-range accumulation of disorientations is obtained.

EBSD versus TEM for characterisation of deformation structures

Deformation structures consist of dislocation boundaries with spacings (≥ 0.1µm) and disorientations (≥ 0.1°). Quantitative measurements of boundary spacings and disorientation angles have traditionally been done using TEM owing to its excellent spatial (less than a few nanometres) and angular (< 0.1°) resolutions. Automatic EBSD in a scanning electron microscope (SEM) offers an alternative tool. However, spatial and angular resolutions are 50 nm and 1°, even in a modern field emission gun (FEG) SEM. To investigate the capability and limitation of EBSD, the deformation structure of a tensile deformed aluminium single crystal was examined. TEM revealed a structure of bands containing well-aligned dislocation boundaries. In some bands more than 70% of the boundaries have disorientation angles less than 1°, in others only about 30%. EBSD orientation images of exactly the same area revealed the band structure. An alignment of boundaries with larger disorientation angles was also observed, but a correspondence with those seen in TEM was not obtained. Moreover, all dislocation boundaries with disorientation angles less than 1° are completely missing in the EBSD images. For a complete characterisation of dislocation structures by EBSD, both spatial and angular resolutions have to be improved, but the angular resolution seems to be the more serious limitation.



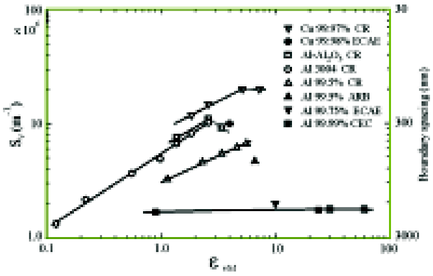
Aluminium single crystal deformed 30% in tension. The upper part is a reflected light image of the strained crystal. The middle part shows a portion of the reflected light image with a crystal rotation axis map overlaid on the locations where the crystal orientations were measured by EBSD. The lower portion shows two shear amplitude maps calculated by a crystal mechanics analysis from the crystal orientation map (dark = larger shear amplitude).



Aluminium single crystal deformed 30% in tension. Deformation structure revealed by (a) TEM and (b) EBSD. In the TEM image, a band structure of alternating dark and bright contrast is seen. Within the bands well aligned dislocation boundaries are formed. The frame marks the area examined by EBSD in a FEG-SEM. In the EBSD image, dark lines show disorientations larger than 1° between neighbouring measured points. A band structure corresponding to that seen in TEM can be identified. However, most of the details within the bands are not revealed.

Macroscopic deformation patterns in deformed single crystals

Crystal orientation patterns in single crystals deformed in tension and rolling subdivide into crystal orientation domains of millimetre scale down to dislocation cells on a micrometre scale. The objective is to identify the degree to which the various levels of subdivision are controlled by externally imposed constraints associated with the deformation process and which represent a spontaneous material response. As an example, an aluminium single crystal with highly polished surfaces is deformed to a strain of 30% in tension. The surface roughness developed during straining is coupled to an underlying pattern of crystal rotations. From a crystal mechanics analysis, the pattern of shear on the available slip systems can be deduced. It shows that the macroscopic scale deformation band pattern is solely a consequence of the external constraints imposed by the specimen grips or by the contact with the rolls in the case of rolled specimens. This analysis provides a mechanism for understanding texture and property inhomogeneities in technologically important materials.



Total surface area of boundaries per unit volume S_v as a function of the plastic strain after different deformation processes. S_v is inversely proportional to the boundary spacing. Additions of alloying elements (compare (Al 3004 CR) and Al 99.5 CR) or particles (compare (Al-Al2O3 CR) and Al 99.5 CR) refine the structures at a given strain.

High strain deformation microstructures

Grain subdivision during deformation results in a distribution of low and high angle deformation induced boundaries surrounding nanometre to micrometre scale volumes. With increasing deformation, the boundaries refine the microstructure and the fraction of high angle boundaries increases. These effects of deformation are of both scientific and technical interest, as they may strongly improve the mechanical properties of metals and alloys.

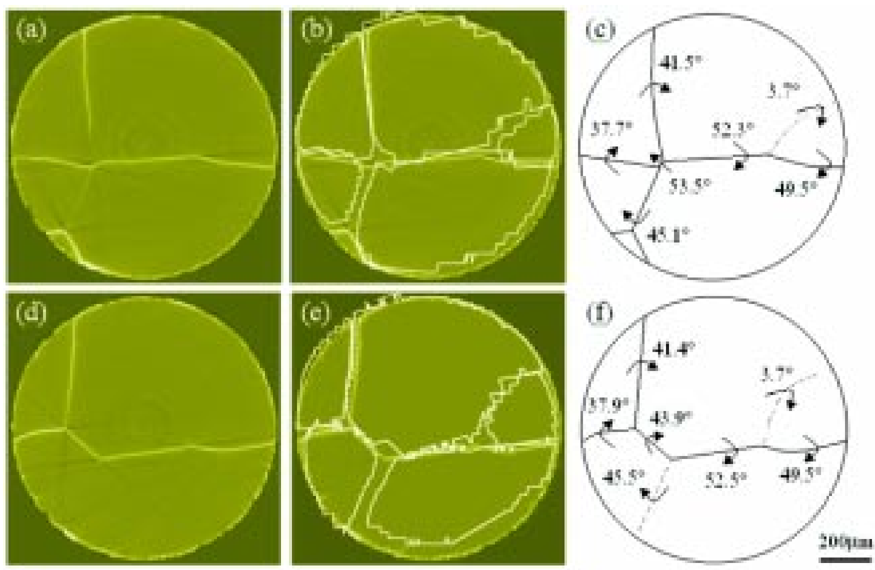
Different deformation modes and material types modify the boundary morphology, spacings and disorientation angles. A continuous structural refinement is achieved with increasing strain, but very large strains are necessary to obtain the very fine structure. For example a boundary spacing of about 200 nm requires a strain of about 10. This corresponds to a reduction in thickness in rolling from about 50 cm to a thickness of about 20 µm. These results point to ways of optimising structures and properties by further exploring the relationship between processing, chemical composition, structure and mechanical properties.

Lattice rotation of individual grains by 3DXRD

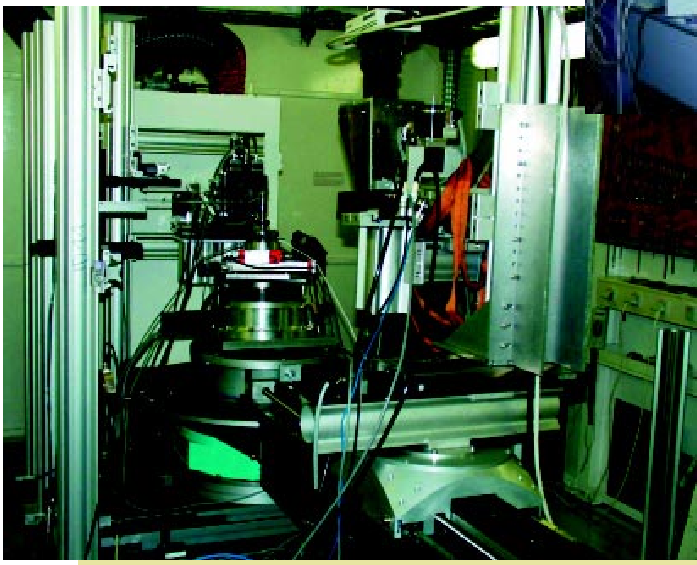
For the first time lattice rotations of individual grains deeply embedded in a polycrystal have been measured during deformation using 3DXRD. The investigated sample is aluminium with a grain size of 300 µm strained up to 11% in tension. Comparison with predictions of two classical models (Taylor and Sachs) for four grains reveals that neither model is completely correct, but that the experimental rotation directions fall in between the predictions of the two models. The experimental rotation rates are slower or comparable to the predicted. The experimental spread of orientations developed during deformation due to grain break-up is of the order of 2°, in the range of disorientations across deformation induced dislocation boundaries found by TEM for the same material. Such data, combined with the possibility of characterising neighbouring grains, provide the first possibility to test model predictions on individual grains rather than on average bulk textures. They are thus the key to resolving major fundamental issues like the role of grain interaction and the number of active slip systems.

X-ray tomography

When an aluminium sample is exposed to liquid gallium, the latter will penetrate into the bulk of the sample along the grain boundaries. The large difference in the linear attenuation coefficient between Al and Ga makes it possible to visualise the Ga-decorated grain boundaries by using synchrotron X-ray tomography. The spatial resolution is less than 1µm, which makes the technique ideal for studying the wetting process. An issue presently being discussed is the penetration of different types of grain boundaries by liquid Ga. The orientation of the individual grains can be determined by the X-ray tracking technique and the calculated disorientations make it possible to determine if a correlation exists between the amount of liquid Ga in the grain boundaries and the disorientations between neighbouring



Sections through a cylindrical polycrystalline aluminium sample. The two sections (100 µm apart) are reconstructed tomographically in (a) and (d) where the grey lines correspond to the position of liquid gallium. The white superimposed lines in (b) and (e) correspond to the grain boundaries determined with the tracking technique. (c) and (f) are schematic illustrations of the grain boundaries. The boundaries sketched as dotted lines are determined only by the tracking technique. The calculated disorientations between neighbouring grains are denoted at the boundaries.



Looking inside the sample!

At Risø a 3-Dimensional X-ray Diffraction (3DXRD) microscope was developed for fast and non-destructive characterisation of the individual grains inside bulk materials such as powders or polycrystals. The underlying method is diffraction with monochromatic and focused hard X-rays (50-100 keV), enabling 3D studies of millimetre to centimetre thick specimens. With a typical resolution of 5x5x50 μm^3, hundreds of grains can be studied simultaneously with respect to:

- position and volume
- crystallographic orientation
- elastic strain
- 3D grain shapes (for coarse-grained materials only)

The 3DXRD microscope is established at the European Synchrotron Facility (ESRF) in Grenoble. A 25 kN stress-rig and two furnaces are available, enabling the grain kinetics to be studied while deforming or annealing the samples. The initial work has mainly focused on nucleation, grain growth and deformation of pure metals, where the grains are large. A number of external users are actively engaged in the use of the instrument, with topics ranging from recrystallisation of commercial aluminium, phase transitions in TRIP (transformation induced plasticity) steel, processing of ceramics, residual strain in thick coatings, kinetics of superconducting coated conductors, ion-implantation and crack propagation.

grains. The combination of the two X-ray techniques provides new information on the penetration process of liquid Ga in solid Al.

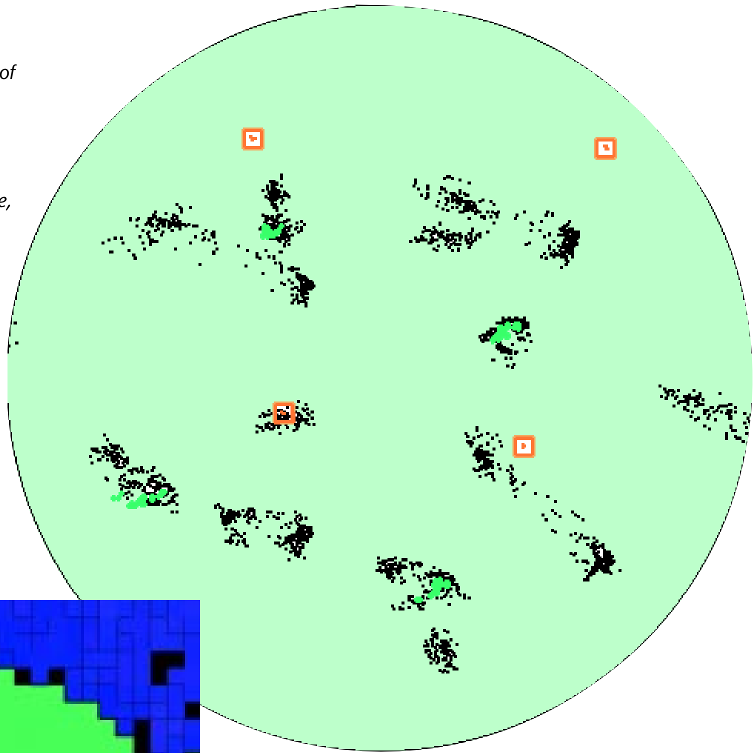
Software algorithm for grain detection

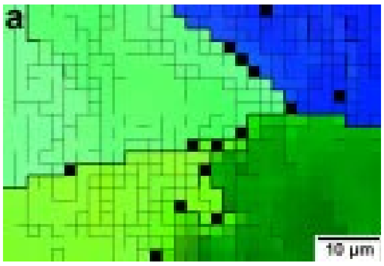
A typical data set from the 3DXRD consists of Bragg reflections from hundreds of grains. A fast algorithm has been developed for retrieving individual grains. The major task is to seek out the right subsets of reflections (each consisting of 10 to 30 reflections) which constitute individual grains. The combinations to test are enormous and a different approach is used depending on the symmetry of the space group. Typically, 86,400 different crystallographic orientations of a grain are calculated and compared with the data. The required execution time is short and almost independent of the number of Bragg reflections in the data set. In fact, the algorithm is nearly fast enough to process data at the rate of data recording. The algorithm has been used on various data sets, e.g. alumina. The intensities of the Bragg reflections for each of the recovered grains are consistent, indicating that the set of reflections indeed originates from the same volume.

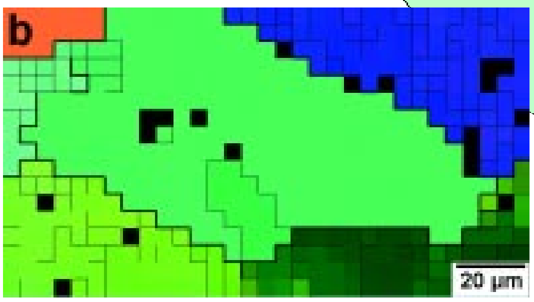
Nucleation at triple junctions

The potency of triple junctions as nucleation sites for recrystallisation is investigated and also the orientation relationships between nuclei and the original deformed grains. EBSD has been used to map the orientations present around triple junctions after cold rolling and after subsequent annealing to produce some small nuclei. It is the first study to examine triple junction nucleation in such detail and to observe exactly the same areas before and after annealing using EBSD. It has verified that triple junctions are preferred sites for nucleation, compared with grain boundaries and grain interiors. Also, results have shown not only nuclei in orientations found before annealing, but a significant number rotated between 10° and 50° about a <111> pole relative to orientations measured in the deformed structure. This contradicts conventional nucleation theory, although it supports other recent findings on single crys-

Below: Orientation, (a) before and (b) after annealing, showing a recrystallisation nucleus at the triple junction. Lines indicate changes of orientation from point to point: the thickest line >10°, the thinnest >2°. Black points were measured with low confidence. Left: 111 pole figure showing orientations detected in the deformed structure (black points) with the orientations of two nuclei shown in green and orange, corresponding to the nuclei that appear in those colours on the orientation maps. The orange nucleus is rotated approximately 20° about a <111> pole.







tals and bicrystals. These effort will continue to determine how and at what stage in the deformation and annealing process these orientations arise.

Grain twist in superalloy tubes

The combination of excellent high temperature mechanical properties and superior oxidation resistance makes iron-based oxide-dispersion strengthened (ODS) alloys a suitable material for components subjected to high stresses and corrosive environments at temperatures well above 1000 °C. The ferritic ODS alloy PM 2000 is therefore being considered as structural material for heat exchanger tubings where a high efficiency demands gas operating temperatures and pressures around 1100 °C and 15-30 bar, respectively. The high pressure requires good creep properties in the hoop direction, which are not obtained with standard extruded tubes because the recrystallised grains become strongly elongated along the tube axis. A torsional deformation process known as flow forming is currently being investigated for its potential to twist the grains and make them elongate along the hoop direction. The Department contributes to an EU-funded project by characterising microstructures and textures of the tubes using EBSD. In particular, the recrystallisation behaviour of the material was studied. It was shown that the desired formation of very coarse grains is obtained when the as-formed tube has a homogeneous microstructure and texture across the tube wall.

Characterisation of superconductors

The Department participates in a Danish programme for utilisation of superconductors with high critical temperatures (high T_c) in the electric power sector. Superconducting cables for power transport are based on tapes containing the compound $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ (Bi-2223). The tapes are produced at Nordic Superconductor Technologies A/S (NST) by using the powder-in-tube technique, where a randomly orientated powder of $(Bi,Pb)_2Sr_2CaCu_2O_x$ (Bi-2212) and secondary phases are filled into a silver tube. The powder is converted into Bi-2223 by a series of drawing, rolling and annealing steps. The aim of the Department in close collaboration with NST and the Department of Condensed Matter Physics and Chemistry at Risø is to further the understanding of the material and increase the critical current density by optimising the processing of the tape. By TEM (partially at the University of Antwerp RUCA), insight in the current path on a submicron scale is gained including grain thickness, colony thickness and grain boundary angles. Investigations of the grain boundaries on an atomic scale and of the intergrowth of Bi-22(n-1)n phases with n≠3 are performed by high-resolution TEM. The microstructure of tapes with different process parameters has been compared.

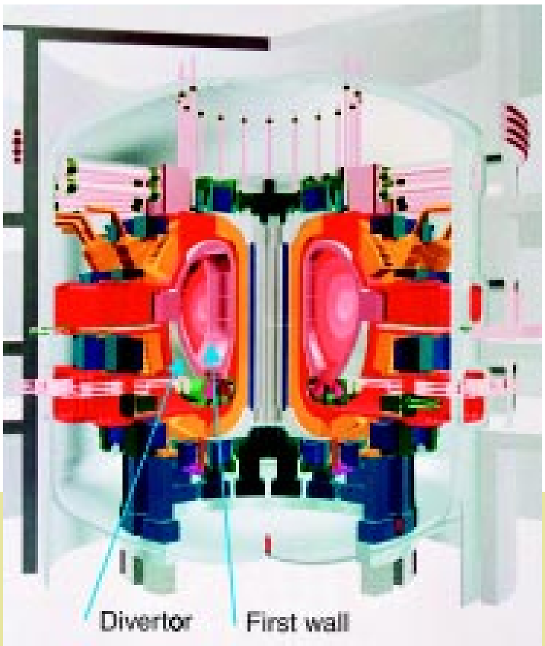
Irradiation Damage, Defects and Fusion Materials

The activities, within the association between Euratom and 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 Thermo-nuclear Experimental Reactor (ITER) programme. The activities in the Department in this area were concentrated on the effect of irradiation on copper alloys, which possess the high thermal conductivity needed in components such as the first wall and divertors. Similar investigations were carried out on titanium alloys.

The 'Long Term Technology Programme' covers activities dealing with problems related to design and construction of the demonstration fusion reactor (DEMO). At the Department, irradiation effects on iron and low activation steels (candidate materials for the first wall) 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 investigations of the effect of irradiation on changes in microstructure using TEM, SEM, positron annihilation spectroscopy (PAS) and electrical resistivity measurements and mechanical properties using tensile, creep-fatigue interaction and fracture toughness tests. Theoretical and simulation studies are also carried out in collaboration with scientists from Europe, Russia and the USA.



Construction materials for fusion reactors

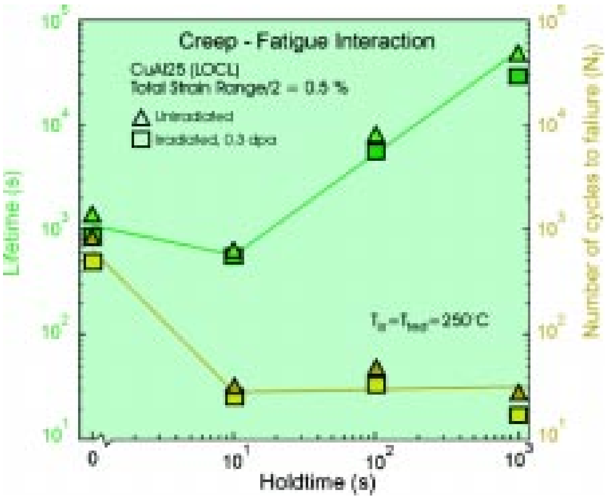
The availability of suitable materials for the first wall and divertor components is an essential requirement for the design, construction and safe and economic operation of a fusion reactor. This requires a thorough assessment of the effect of manufacturing processes and of neutron irradiations on physical and mechanical properties of materials to be employed in these components. Materials exposed to an intense flux of fusion neutrons are likely to suffer from the loss of ductility and volumetric expansion (i.e. swelling). Consequently, the useful lifetime of in-vessel components such as the first wall and divertor may be significantly reduced. For the development of new and radiation resistant materials, it is therefore essential to pursue both theoretical and experimental investigations to establish a proper understanding of the effect of neutron irradiation on microstructural evolution and its impact on physical and mechanical properties.

Effect of neutron irradiation on creep-fatigue interaction and fracture toughness of copper alloys

The first wall and divertor components of ITER will be exposed to an intense flux of fusion neutrons and will experience thermo-mechanical cyclic loading as a result of the cyclic nature of plasma burn operations of the system. Consequently, the structural materials in the reactor vessel will have to endure not only cyclic loading but also stress relaxation and microstructural recovery (creep) during the 'plasma-on' and 'plasma-off' periods. Since copper alloys are being considered as candidate materials for the first wall and divertor components in ITER, we have investigated the creep-fatigue interaction behaviour of a dispersion hardened copper alloy ($Cu\text{-}Al_2O_3$) in the unirradiated and irradiated condition. Fatigue specimens were neutron irradiated at 250 °C to 0.3 dpa (displacement per atom) in the DR3 reactor at Risø. All tests were carried out at 250 °C. In these experiments, the creep-fatigue interaction condition was simulated by applying a certain holdtime on both tension and compression sides of the cyclic loading.

A holdtime of 10 seconds led to a significant decrease in the number of cycles to failure, particularly at the low level of the applied strain value. At a given strain level, no further decrease was observed at longer holdtimes of 100 and 1000 seconds. The neutron irradiation to a dose level of 0.3 dpa does not seem to affect the creep-fatigue interaction behaviour in any significant way.

According to the current design of ITER, copper alloys will be joined to the stainless steel primary wall module using Hot



Creep-fatigue response of a dispersion hardened copper alloy ($Cu\text{-}Al_2O_3$) before and after neutron irradiation as a function of hold time during fatigue test with a loading frequency of 0.5 Hz. A hold time of even 10 seconds causes a significant reduction in the fatigue lifetime as well as the number of cycles to failure.

Isostatic Pressing (HIP) technique. In order to assess the structural integrity of the first wall module, it is essential to determine the fracture toughness behaviour of such joints before and after neutron irradiation. For this purpose, HIP joint specimens of $Cu\text{-}Al_2O_3$ and CuCrZr alloys with 316 stainless steel were irradiated at 50, 200 and 350 °C to a dose level of 0.3 dpa. The initiation fracture toughness of the irradiated and unirradiated specimens was determined at these temperatures using three-point bend tests. A marked decrease in the fracture toughness of the HIP joint specimens of both copper alloys was observed after neutron irradiation. The strength mismatch between the base alloys (i.e. copper alloys and stainless steel) seems to dominate the fracture behaviour of the joint specimens. The increase in strength mismatch causes stronger localisation of plasticity in the softer alloy and also at the joint interface.

Fatigue and fracture toughness behaviour of titanium alloys before and after neutron irradiation

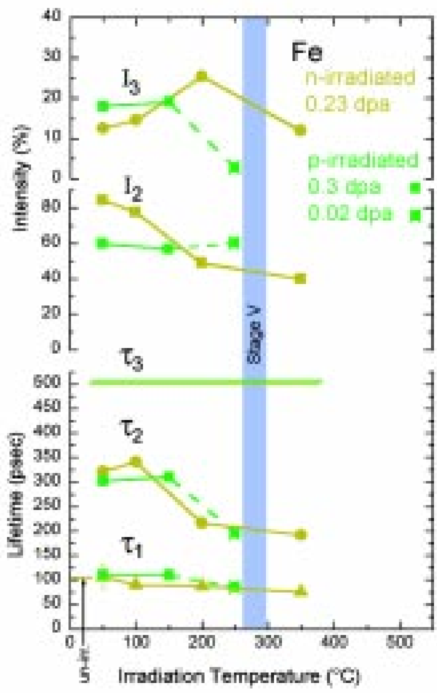
Titanium alloys are being considered as candidate materials for flexible mechanical connectors between the blanket modules and the backplate of ITER. Since very little is known about the effect of neutron irradiation on mechanical properties of these alloys, it was decided to carry out screening investigations to determine the effect of neutron irradiation on tensile, fatigue and fracture toughness behaviour of Ti-5Al-2.5Sn (α) and Ti-6Al-4V (α+β) alloys at 50 and 350 °C.

Both fatigue and fracture toughness specimens were irradiated in the DR3 reactor at Risø to a dose level of 0.3 dpa at 50 and 350 °C. Pre- and post-irradiation mechanical tests were carried out at 50 and 350 °C. In general, the fatigue lifetime of α and α+β alloys is rather similar in both unirradiated and irradiated conditions. However, the fatigue lifetime of the α-alloy is found to be longer than that of the α+β alloy at lower strain range values at 50 °C and at higher strain range values at 350 °C. As regards the effect of neutron irradiation, the fatigue lifetime of the α-alloy at 50 °C and that of the α+β alloy at 350 °C does not seem to be affected by neutron irradiation to 0.3 dpa. However, the lifetime of the α-alloy at high strain range values is somewhat reduced due to irradiation at 350 °C. In the case of the α+β alloys, on the other hand, neutron irradiation seems to cause a noticeable increase in the lifetime at low strain range values.

The fracture toughness of these alloys both in the unirradiated and irradiated conditions was determined using three-point bend tests at 50 and 350 °C. In the unirradiated conditions, both alloys had rather similar fracture toughness values at 50 °C. However, at 350 °C, the α+β alloy exhibit higher initiation fracture toughness than the α-alloy. At 50 °C, irradiation causes a significant reduction in the fracture toughness of both alloys. In both alloys, once the cracks are nucleated, they grow in a stable fashion. However, at 350 °C the reduction in fracture toughness due to irradiation is not as large as at 50 °C and both alloys are more ductile at 350 °C.

Void formation in neutron and proton irradiated pure iron

The problem of void formation in bcc metals and alloys has been the subject of investigations in a number of laboratories. With the aim of obtaining a better under-



Iron irradiated with reactor neutrons (olive-green) and 600 MeV protons (dark green). The figure shows the variation of positron lifetimes and their intensities with irradiation temperature. The lifetimes τ_2 and τ_3 are characteristic for different types of defects. At low temperatures (50 to 150°C) both voids (τ_3 = 500 ps as shown by straight lines) and micro-voids (τ_2 ~300 - 350 ps) are formed, while at higher temperatures no micro-voids are seen. The rather low value of τ_2 (~200 ps) at and above 200 °C indicates the presence of vacancy clusters. The intensities τ_2 and τ_3 of the lifetime components give information about the defect densities.

standing of these problems, such as the influence of irradiation temperature, annealing treatment and the presence of gas, a series of experiments on pure iron (and on steel) have been initiated.

The microstructure of pure iron after neutron and 600 MeV proton irradiation was studied by positron annihilation spectroscopy. PAS has the advantage that it is sensitive to vacancy-type defects such as single vacancies, two-dimensional vacancy clusters (i.e. loops) and three-dimensional vacancy agglomerates (i.e. voids and gas bubbles). With PAS it is possible to detect sub-microscopic voids (microvoids) and get information about their sizes, since the lifetime of positrons trapped in such microvoids increases with the cavity size in the range from mono-vacancies up to agglomerates of about 50 vacancies. In addition, the technique provides information about the density of such cavities.

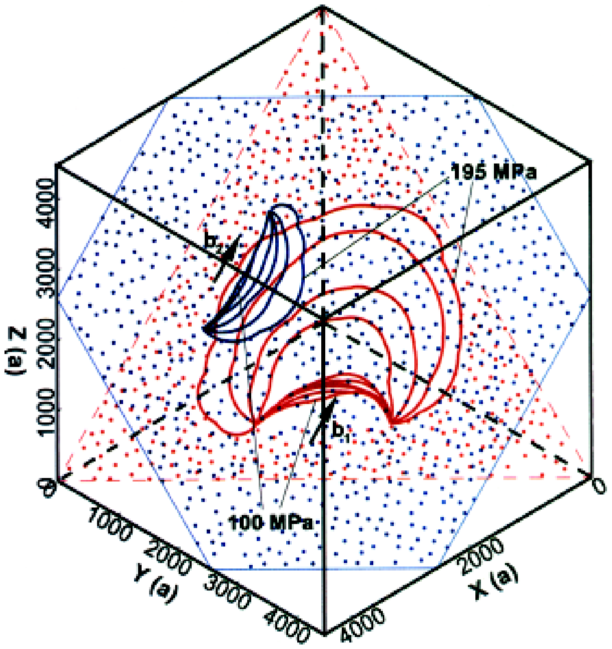
The results show that in general both neutron and 600 MeV proton irradiations of iron (to doses of 0.23 and 0.3 dpa, respectively) lead to the formation of both microvoids and voids at an irradiation temperature as low as 50 °C and up to about 200 °C. At temperatures above about 200 °C, no microvoids are formed and the void density decreases with increasing temperature.

The microvoids typically have average sizes of about 10 to 15 vacancies, while the voids on average contain more than about 50 vacancies. The density of the voids is 3 to 8 times lower than that of the microvoids, both being in excess of 10^{23} m^{-3} for irradiation temperatures below 200 °C. If the total microvoid and void population is considered, the average cavity size is larger in the proton than in the neutron irradiated iron. Although the details of the microstructures differ for the two types of irradiation, the created void populations are qualitatively the same.

Modelling of defect accumulation and plastic deformation behaviour under neutron irradiation

Within the framework of the production bias model (PBM), the damage accumulation is expected to be sensitive to features of the primary damage production, namely the intracascade clustering of vacancies and self-interstitial atoms (SIAs). Since the intracascade clustering is dependent on the level of recoil energy, according to the PBM the defect accumulation in the form of clusters and voids must then be sensitively dependent on the recoil energy. That this is indeed the case has been demonstrated recently by experimental investigations. Using the parameters of these experiments, the evolution of the defect microstructure has been calculated. The calculated results on cluster and void densities and sizes, void size distributions at different doses and the dose dependence of void swelling are in good agreement with the experimental observations. The analysis of these results shows that the origin of the effect of recoil energy lies in the intracascade clustering of SIAs and the properties of SIA clusters, as expected from the model.

For a comprehensive modelling of damage accumulation within the framework of the PBM, it was found necessary to consider the consequences of the possibility that small SIA clusters may change their direction while performing 1D diffusion. This would disturb the global 1D reaction kinetics and increasing frequency of such direction changes would induce a continuous transition from 1D to 3D reaction kinetics. Recently, a general interpolation formula was derived to describe this transition. The resulting function represents a simple one-parametric master curve (interpolating between the pure 1D and 3D cases) for the dependence of the sink strength on the main spatial scales involved in the process. This functional relationship has been determined using kinetic Monte Carlo simulations. The results of these simulations confirm the master curve dependence predicted by the analytical treatment.



Results of 3-D dislocation dynamics simulations showing spread of plastic slip emanating from two interacting Frank-Read sources in copper irradiated with fission neutrons at 100 °C to a dose level of 0.1 dpa. The dots represent stacking fault tetrahedra with a density of $4.5x10^{23}$ m^{-3} and 2.5 nm in size. Simulated crystal size is 4500 a (1.62 µm) with the lattice constant a (0.3615 nm). The Frank-Read sources are separated by 20 a (7.2 nm).

The main thesis of the cascade induced source hardening model that the upper yield stress and subsequent plastic flow instability are caused by a sudden unpinning of dislocations from the atmosphere of small defect clusters has been further investigated using the method of 3D dislocation dynamics. The dynamics of dislocation interaction with SIA clusters in the dislocation decoration and with stacking fault tetrahedra in the matrix were investigated. The critical stress to free trapped dislocations from pinning atmospheres can be a factor of about two smaller than values obtained in the original calculations of the cascade induced source hardening model, where dislocation interactions were assumed to be rigid. The calculated unlocking stress values are in general in agreement with experimental observations and suggest that unlocking of heavily decorated dislocations will be most prevalent in areas of high stress.

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: (i) Properties of Composite Materials which covers metal, ceramic and polymer matrix composites, and (ii) Mechanical Characterisation and Design of Components which covers experimental and numerical methods that relate material properties to component performance.

Highlights are as follows:

i. Fabrication of plant fibre composites with high content of unidirectional fibres and strengths reaching about 300 MPa
ii. Installation of a laser extensometer for mechanical testing with very high sensitivity under contact-free conditions
iii. Characterisation and evaluation of the influence of microstructures on the performance of tool steels
iv. Establishment of a number of commercial contracts with Danish wind energy companies

Picture supplied by LM-glasfiber A/S.

Properties of Composite Materials

The activities on composite materials focus on the relations between microstructure and properties, and the physical and mechanical mechanisms for deformation, strength and fracture. The modelling of properties of composite materials and performance of components has taken an increasing importance in the developments towards design procedures involving materials properties. The special option of tailoring properties for composite materials through detailed design of the microstructure is used to optimise properties for given applications.

The processing technologies constitute an important activity serving both the materials development activities inside the Department and the fabrication methods of interest to the industrial community in Denmark and Europe. Several techniques are in progress to develop intermediate products, e.g. fibre mats, and to fabricate composites. These techniques include filament winding, press and autoclave consolidation.

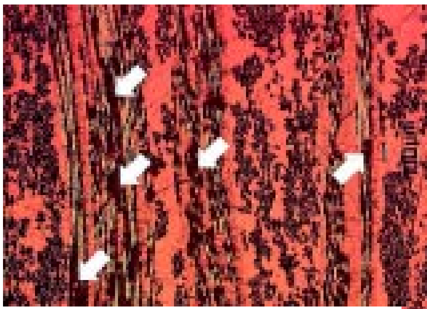
Extensive characterisations of composite materials and of the constituents (fibres and matrix) are carried out. The microstructure, defects, damage and properties are recorded and quantified. The characterisation techniques include microscopy, nondestructive evaluation and mechanical testing. A special effort is made to correlate the measurements from different techniques to obtain a better and more extensive description, both for validation and calibration of the experimental methods, and for quantitative microstructural characterisation for modelling of the properties.

The design and performance of components is studied both by experiments and by finite element analysis. Because of their high specific strengths, composites offer an excellent possibility for light components and products for an energy conscious society. In the energy sector activities are in progress to use composite materials under very high-cycle fatigue loading in rotor blades for large wind turbines for generation of electricity. The high specific strength is also exploited in flywheel constructions for very high rotational velocities and thus for large and efficient energy storage. Hydrogen is a valuable and useful carrier of energy, which should be stored in pressure vessels, and composites offer an efficient and light material for such storage vessels. In the transport sector work is in progress towards use of composites for springs in vehicles. The suspension and damping characteristics are promising and the weight of springs can be reduced significantly.

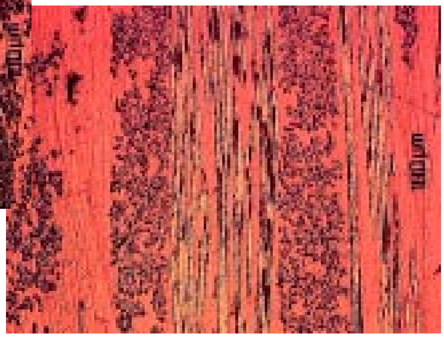
Development of composites based on natural cellulose fibres with a polymeric matrix, which can be synthetic or based on renewable resources, is an activity motivated by environmental considerations. The aim is to achieve strength and stiffness comparable to traditional composites.

Effects of processing conditions on microstructure and fatigue performance of glass fibre/ polypropylene composites

(a): Cross-section of Twintex® glass fibre/polypropylene material processed at 170°C. The arrows indicate small black areas where the matrix is broken off due to a poor wetting/bonding between fibre and matrix.



(b): Cross-section of Twintex® glass fibre/ polypropylene material processed at 180°C with acceptable microstructure.



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 • Department of Structural Engineering and Materials, DTU, Denmark
The Ministry of Food and Agriculture Plant-fibre based Products	• Royal Veterinary and Agricultural University, Denmark • Danish Agricultural Advisory Centre, Denmark • Danish Institute of Agricultural Sciences, Denmark • Force Institute, Denmark • Danish Technological Institute, Denmark
The Ministry of Environment and Energy	• Royal Veterinary and Agricultural University, Denmark • Department of Structural Engineering and Materials, DTU, Denmark
PSO-2079 Fatigue Strength and Life Time of Wind Turbine Components	• Elsamprojekt A/S, Denmark • Wincon A/S, Denmark • NEG-MICON, Denmark
EFP Flywheel for Energy Storage-III	• TERMA Industries A/S, Denmark • NESA A/S, Denmark • DEMEX A/S, Denmark • 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 • ALU-B7, Denmark • JILKO, Denmark • LG Trafik A/S, Denmark • Bent Falk Design, Denmark • 3M a/s, Denmark • Teknologisk Institut Århus, Denmark • Arkitektskolen i Århus (AAA), Denmark • Ingeniørhøjskolen i Århus (IHÅ), Denmark • Aalborg Universitet Esbjerg (AUE), Denmark • Alucluster, Denmark
EUCLID Advanced Techniques for Add-on-Armour	• Fokker SP, The Netherlands • DEMEX A/S, Denmark • Sistema Compositi, Italy • OTOBREDA, Italy • EN Santa Barbara, Spain • TNO-PML, The Netherlands • Spav, The Netherlands • HB Consultancy, The Netherlands
EUCLID Long Term Effects on Light Weight Add-on Armour	• Fokker SP, The Netherlands • DEMEX A/S, Denmark • Sistema Compositi, Italy • DSM, The Netherlands • OTOBREDA, Italy • EN Santa Barbara, Spain • TNO-PML, The Netherlands • Spav, The Netherlands • HB Consultancy, The Netherlands
BRITE-EURAM Innovative Casting Process of Lighter Steel Components for the Transport Industry	• Centro Richerche FIAT, Italy • Aerospatiale, France • Ferriere e Fonderie di Dongo, Italy • Gussstahl, Germany • Magma, Germany • RWTH, Germany

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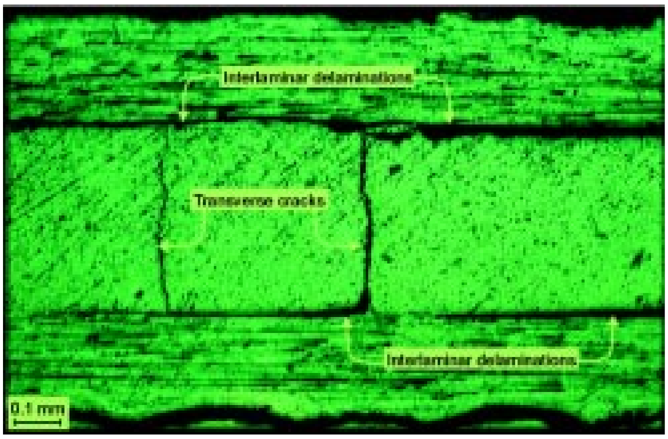
Effects of processing conditions on microstructure and fatigue performance of glass fibre/ polypropylene composites

Microscopy and tension-tension fatigue tests have been carried out for determination of the effects of process conditions for the commercially available glass fibre reinforced thermoplastic composite material Twintex®. The glass fibre/polypropylene fabric is woven with commingled roving composed of glass and polypropylene fibres.

A low process temperature is generally desirable. Not only can energy and cost to heat the material be saved, but the variety of usable tooling and bagging materials is also expanded. Laminates were produced by vacuum consolidation at selected process temperatures of 170, 180 and 190°C. Microscopy of polished cross-sections of the 170°C laminate shows areas where the matrix separated from the glass fibres indicating a poor wetting of the glass fibre. A visually acceptable material quality was observed for laminates processed at 180 and 190°C. Tensile fatigue tests were performed at 5 Hz at room temperature with a stress ratio of R = 0.1. The results show that the fatigue properties of the material do not change if the process temperature is in the range from 180 to 190°C, but drop for the material processed at 170°C, as expected from the microscopic evaluation. A process temperature of 180°C can therefore be recommended for vacuum consolidation.



Micrograph of an edge replica of a cross-ply $[0_2,90_2]_S$ laminate made of carbon-fibre reinforced epoxy subjected to fatigue. The interaction of transverse cracking and delamination was shown to be responsible for the sequence effect in variable amplitude loading of this material.

Project type/Project name	Co-participants
BRITE-EURAM Assessment of Metal Matrix Composites for Innovations	• Daimler-Chrysler AG, Germany • Centro Ricerche FIAT, Italy • Aerospatiale, France • INTROSPACE GmbH, Germany • EA-Technology Ltd., UK • ISRIM, Italy • Austrian Research Center Seibersdorf GmbH, Austria • CSM Materialteknik AB, Sweden • Technische Universität Wien, Austria • Deutsche Gesellschaft für Materialkunde, Germany • German Aerospace Center, Germany • Universidad Politècnica de Madrid, Spain • Swiss Federal Inst. for Materials Testing and Research, Switzerland • Ecole Polytechnique Fédérale de Lausanne, Switzerland • Institute National des Sciences Appliquées, France • National Physical Laboratory, UK • VTT Manufacturing Technology, Finland • Katholieke Universiteit Leuven, Belgium • University of Cambridge, UK
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 • J. B. Plant Fibres Ltd., UK • Carl Bro A/S, Denmark • InnospeXion ApS, Denmark • Sauerwein System Technik GmbH, Germany • VIDROPOL S.A., Portugal • CEDIP S.A., France • Instituto de Soldadura e Qualidade, Portugal
BRITE-EURAM Hyper-Eutectic Alloys for Automobile Components (HAforAC)	• Bosch Systemes de Freinage, France • FIAT SpA, Italy • Norsk Hydro Aluminium a.s., Norway • Stampal SpA, Italy • Pechiney CRV SA, France • Fagor Ederlan, S. Coop. Ltd., Spain • CEIT de Guipuzcoa, Spain • Allied Signal Bremsbelag GmbH, Germany • ISRIM, Italy • University of Sheffield, UK
EUREKA EUROBOGIE	• EM-Fiberglas A/S, Denmark • Skoda Research, Czech Republic • Bombardier Talbot, Germany • Sciotech, UK • Polymath, UK • English Welsh Scottish Railways, UK • Concargo/Polymer Engineering, UK • SVUM, Czech Republic • IPM, Latvia • Reading University, UK
BRITE-EURAM Action for Low Weight Automobile Technologies (FLOAT)	• Rover Group Ltd., UK • GIE Renault, France • FIAT SpA, Italy • Volvo AB, Sweden • Daimler-Chrysler AG, Germany • Austrian Research Center Seibersdorf GmbH, Austria • Fraunhofer Institut, Germany • SEPARIS, France • ISRIM, Italy
BRITE-EURAM Plant Life Assessment Network (PLAN)	Thematic Network
JOULE-THERMIE New Generation Wind Turbine Blade	• Bonus Energy A/S, Denmark • Garrad Hassan & Partners, Ltd., UK • Kemijoki Yo, Finland • VTT, Finland

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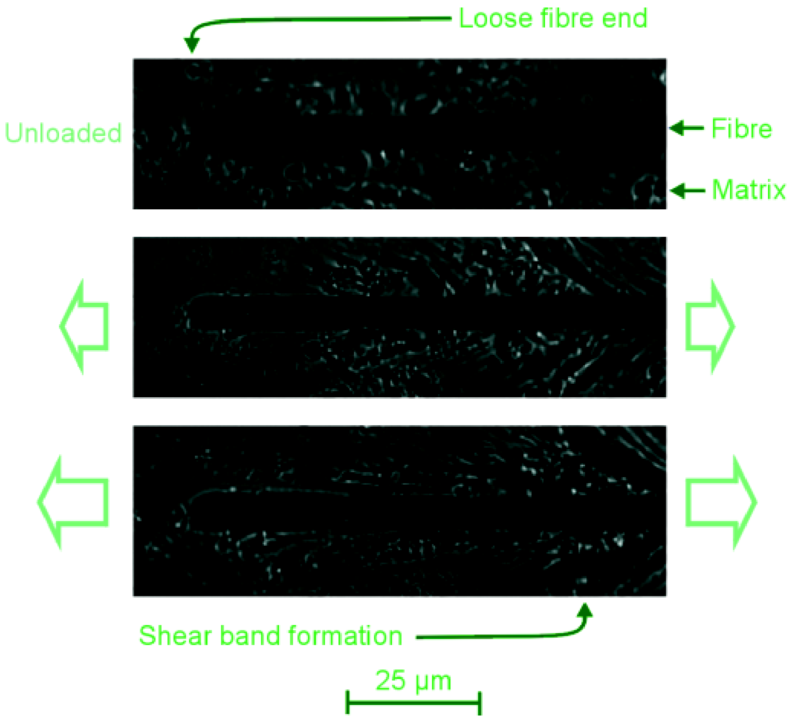
Sequence effects in fatigue of composite materials

In variable amplitude loading, marked sequence effects occur in polymer-matrix composites, which are not as common in most metallic structural materials. Macroscopic criteria such as the Palmgren-Miner rule fail to describe the fatigue behaviour of composites, since these materials sometimes show premature failure if a block of high amplitude loading is followed by a block of low amplitude loading, whereas in other cases the opposite effect takes place. Empirical macroscopic laws are based on extensive and costly testing. A better way forward would be to identify and model the underlying damage accumulation up to failure. The physical reasons for the sequence effect has been investigated experimentally in a cross-ply laminate of carbon-fibre reinforced epoxy. It was found that the reason for the sequence effect was the mutual influence of two different damage mechanisms. Transverse cracking was found to be an initiatory mechanism, which interacted with progressive interlaminar delamination initiated from the transverse cracks. The transverse cracking dominated at high loads, whereas delamination dominated at low loads. Macroscopically, high-low block loading resulted in shorter lifetimes than a low-high sequence, which can be explained by a more rapid growth of damage where delaminations propagate from transverse cracks. Results from modelling concur with the observed physical explanation. Ongoing efforts focus on generalisation to other material lay-ups, with the aim to reduce the degree of empiricism in the macroscopic models used in design of composite structures against fatigue.



Deformation at the end of a single carbon fibre embedded in an unsaturated polyester matrix. As increasing load is applied along the fibre direction, inelastic matrix deformation takes place in the vicinity of the carbon fibre. A quantitative measure of the maximum interfacial stress can be obtained by micro-Raman spectroscopy in conjunction with microscopic observations

Chemical tailoring of the interface in carbon fibre/unsaturated polyester composites

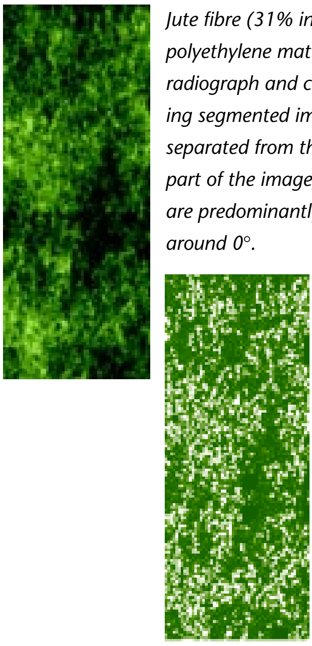
New experimental techniques have made it easier to chemically tailor the interface between fibre and matrix in composites. This can be achieved by synthesis of an unsaturated polyester which is likely to bond covalently to the functional groups on the carbon fibre surface. In a joint study with Aalborg University and an industrial manufacturer of polyester resins, the interfacial shear strength has been obtained by means of micro-Raman spectroscopy in tensile specimens with single carbon fibres embedded in various polyester matrices of various chemical compositions. This technique allows direct measurements of the strain along individual carbon fibres from the strain-induced shifts of the Raman spectrum. From the strain profiles, the interfacial strength can be determined. It has been shown that larger relative amounts of maleic anhydride at the expense of orthophthalic anhydride and 1,2-propylene glycol as precursors, resulted in a stronger interface, probably due to increased chemical bonding between the polyester unsaturations and the carboxylic end groups of the fibre. This methodology unlocks potentials for rational polymer synthesis to tailor desired interfacial properties in polymer-matrix composites.

Characterisation of natural fibres and related composite materials

The natural fibres for potential reinforcements are jute, flax and hemp. The fibres have been characterised by chemical composition, especially cellulose content, and by molecular weight. Both high cellulose content and high molecular weight are believed to be beneficial for fibres of high stiffness and strength. For a wide range of natural fibres, the molecular weight has been found to decrease with increasing cellulose content. All strength and stiffness values for practical fibres are significantly lower than the theoretical stiffness and strength values for cellulose fibres, which indicates that there is potential for improvements in the properties of fibres.

Composites based on jute, flax and hemp have been fabricated by press consolidation. Both fibre mats with medium length fibres in random orientation, and long bundles of unidirectional fibres have been investigated. The processing technologies for natural fibre composites are under constant development. Work has been initiated to modify the fibre/matrix interface and interphase in composites based on polypropylene matrix. The fabrication of aligned fibre composites has been improved, and the packing of aligned fibre bundles has been studied in an attempt to increase the volume fraction of fibres in the composites. This is a much needed development towards high strength composites with low porosity contents to optimise mechanical performance.

Project type/Project name	Co-participants
EUCLID Survivability , Durability and Performance of Naval Composite Structures	• Danyard Aalborg, Denmark • Force Institute, Denmark • Det Norske Veritas AS, Norway • FiReCo AS, Norway • Umeo Mandal AS, Norway • DERA, UK • Vosper Thornycroft Ltd, UK • Marconi Marine Ltd, UK • Southampton University, UK • Newcastle University, UK • DCN, France • DCE, France • Ifremer, France • Medysys, France • TNO, The Netherlands • Royal Shelde Group, The Netherlands • Cetena, SpA, Italy • Fincantieri Cantieri Navali Italiani, SpA, Italy • CSI, Italy • SIR Industriale SpA, Italy • Otobreda, Italy • IDS, Italy
BRITE-EURAM Improvement of Service Life and Reliabiltiy of Cold Forging Tools with Respect to Fatigue Damage due to Cyclic Plasticity (COLT)	• Krupp Presta AG, Liechtenstein • STRECON, Danfoss, Denmark • Universität Erlangen, Germany • Böhler Edelstahl, Austria • Rockfield Software Ltd, UK • C3M, Slovenia



Jute fibre (31% in weight)/ polyethylene mat: digitised radiograph and corresponding segmented image (fibres separated from the remaining part of the image). The fibres are predominantly aligned around 0°.

Analysis of X-ray pictures of composite materials

An active research area is the assessment of critical defects and development of non-destructive inspection and characterisation technologies of glass- and plant-fibre reinforced polymer composites. Emphasis is placed on a new high-resolution high-contrast radiographic system. The work includes flaw detection and quantification to establish a relationship between flaws and mechanical properties. Relevant data can be provided to evaluate the manufacturing processes involved in the production. Imaging procedures, e.g. dual-energy imaging to better distinguish between matrix and fibres, and quantitative image processing techniques are being developed. Extraction of quantitative data from digitised radiographs has been accomplished with three main objectives:

- To determine the amount and orientation of fibres and fibre bundles
- To determine the void content
- To quantify the structural homogeneity of the samples.

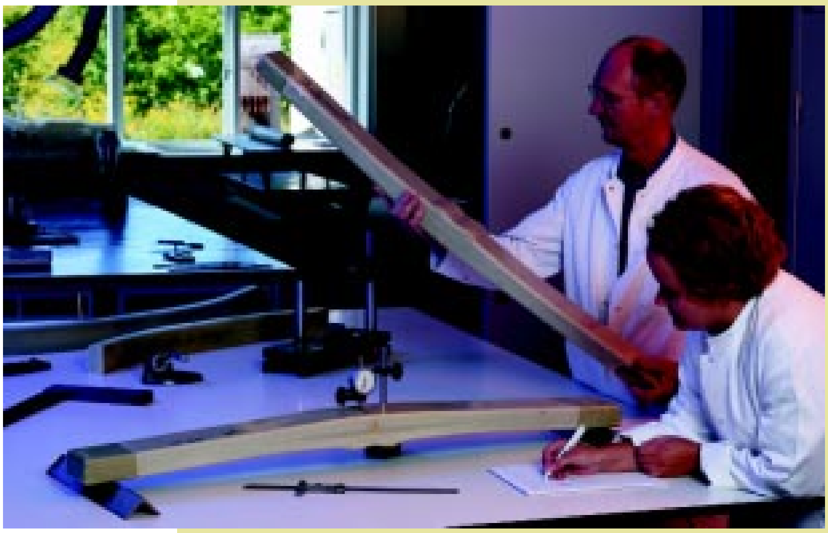
The segmentation process, allowing the separation of fibres, or voids, from the remainder of the image is a crucial step because of its influence on the determination of parameters characteristic to the materials. Procedures based on background correction, mathematical morphology and thresholding have been established.

Industrial applications

Train suspension based on fibre composites

The advantages of using fibre composite springs instead of steel leaf springs are weight savings, constant spring characteristics throughout the lifetime and higher damping properties. A steel leaf spring for a smaller freight wagon weighs 120 kg, while an equivalent glass-fibre composite spring weighs only 40 kg. Corrosion between the leaves in a steel spring causes degradation in the damping performance, and the stiffness changes through the lifetime. This is not the case for a composite spring, where the properties remain constant throughout the lifetime. The higher damping properties of a composite suspension leads to lower loads on both rails and wagons, which in turn lead to lower maintenance costs and noise reduction.

Glass-fibre composite springs are being developed within a European project. The Danish company EM Fiberglass A/S has developed the manufacturing technology for production of the bottom spring element. The Department performs proof testing of springs and characterisation of the materials properties, such as damping, fatigue of undamaged material and fatigue after impact. Initial test results show that the spring properties match extremely well with the predicted design performance. Dynamic full-scale tests at Skoda Heavy Industries in the Czech Republic on a wagon fitted with composite suspension have proven to be very positive, with a very significant reduction in load and noise.



Glass fibre composite spring elements for two-part suspension elements for train freight wagons are examined before testing at 100 kN.

Two add-on armour panels are mounted on the side of an armoured personel carrier (M-113) as part of a test run by the Danish Defence Research Establishment to investigate field conditioning of add-on armour. Similar panels are conditoned by the Department to compare the performance of field conditioned and laboratory conditoned armour.

Armour materials

Ballistic protection is an important issue for Danish peace-keeping forces. Together with a number of European companies such as Demex A/S, the Department is participating in research programs to optimise design and materials selection for lightweight armour. The armour in question is used to improve the existing armour of personnel carriers. The add-on armour is placed on the outside of the carrier and consists of ceramic tiles bonded with an adhesive to a plate of aluminium or a 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, the adhesive, the metal and the composite. To achieve this, a number of different material tests have been performed. Different adhesive/ceramic interfaces were tested with regard to fracture toughness. The composite material (high-density polyethylene fibres in an elastomer matrix) was tested at low strain rates in bending, shear and compression. Focus is on various operational conditions such as temperature, humidity, low energy impact and vibrations, because the conditions may change the material properties and the effectiveness of the armour.

Mechanical Characterisation and Design of Components

The objective of this programme is to characterise and model mechanical properties in new and industrial materials and to gather knowledge for use in design. The mechanical properties and the design data are to be applied in development of specific components and in tailoring new materials. The mechanical testing activities cover all types of standard testing (static, fatigue, creep and impact). In addition of carrying out specialised testing on any type of material the laboratory is an accredited test laboratory by Danish Accreditation, DANAK, which is the national accreditation body.

Micromechanics and finite element modelling are used to demonstrate and understand the influence of basic material structures on the observed damage behaviour and macroscopic properties. For instance, models for fibre bridging in composite materials have been modelled and experimentally verified in specially developed test set-ups. Image analyses of X-ray pictures have been improved to automated quantification and to gain contrast information. Thermography has been used in several mechanical fatigue tests and it enables monitoring of damage evolution during tests. A non-contacting extensometer based on laser and video measurements has been installed. Accurate mechanical tests on thin foils as well as ultra-high-strength materials can be offered.

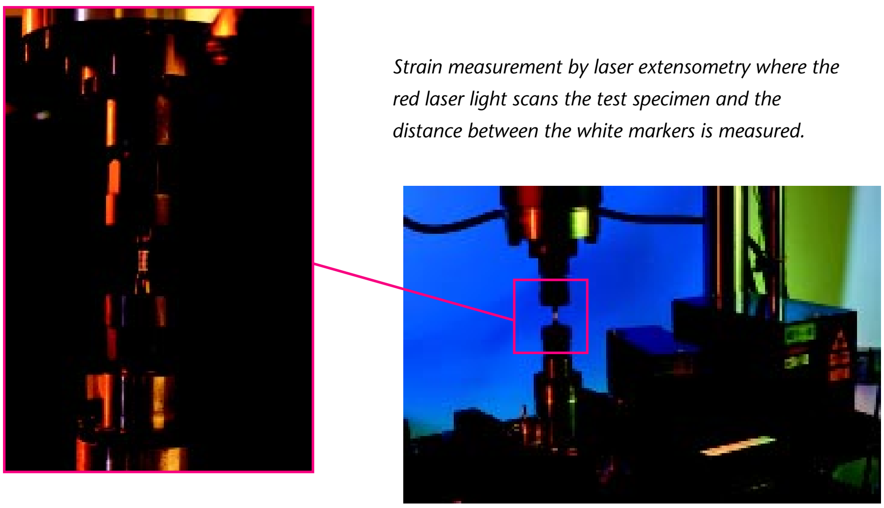
Joint research programmes are a major activity. A large project on cyclic plasticity and microstructural optimisation of high strength tool materials was started in collaboration with steel producers, tool manufacturers and end-users. A project was started, which focuses on the microstructural influence on fatigue crack initiation. Joint European projects are currently running in all fields of the programme activities.

Industrial research and services constitute a large part of the activities. Accredited testing is carried out routinely for a large number of industrial companies. Most of the projects are related to design of components. This includes rotor blades for wind turbines, ballistic add-on armour, composite leaf springs, adhesively bonded aluminium constructions, forging, oil tools, automotive parts, flexible oil pipe lines etc.

Integration of the activities in non-destructive characterisation and experimental mechanical testing has been initiated and wider use of materials mechanics in design of industrial components is foreseen. This will also involve an even broader horizontal collaboration between the different research programmes in the Department.

Laser extensometry

Non-contact deformation measuring techniques have developed rapidly over the last years. Faster computers have improved the applications from simple static testing to full strain-controlled fatigue testing. The mechanical testing laboratory at the De-



Strain measurement by laser extensometry where the red laser light scans the test specimen and the distance between the white markers is measured.

partment has purchased an automated laser extensometer. It consists of a 400 Hz laser scanner and a receiver, which enables strain monitoring effectively at 25 – 50 Hz. Reliable software gives the extensometer reading in digital and/or analogue signals.

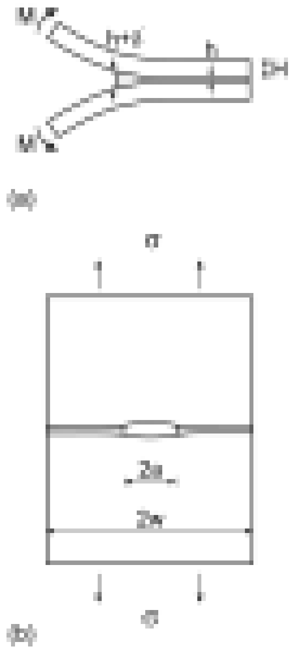
A software interface to control a servo-hydraulic test machine in strain control has been developed and implemented. It enables low-cycle fatigue tests to be performed in strain control at lower frequencies (< 0.5 Hz) based on the non-contacting measurements. The laser extensometer also allows direct measurement of properties of ultra high-strength materials, tool steels, composites with very thin fibres and foils where contact extensometry is not possible.

Large scale bridging in composites

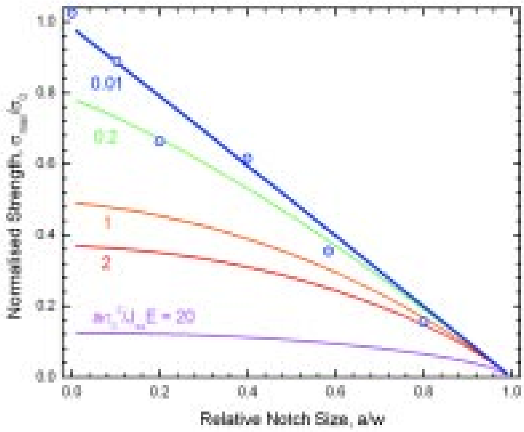
Fibre-bridged cracking in composites results in rising crack growth resistance with increasing crack extension (R-curve behaviour). Several cracking problems involve large-scale bridging and linear elastic fracture mechanics is no longer valid because the fracture process zone is large compared with specimen dimensions. Experiments have shown that R-curves depend on specimen geometry.

Under large-scale bridging, the failure process should be described by a bridging law, i.e. the relationship between the local bridging traction resulting from the bridging ligaments and the local crack opening displacement. The bridging law can be derived by evaluation of the J integral along the external boundaries of the specimen.

Experiments on unidirectional carbon/epoxy composites showed specimen-to-specimen variations in the measured bridging laws and R-curves. A numerical study by the finite element method was carried out to model the effect of fibre bridging accurately. Lower and upper bound curve fits to the measured bridging law were implemented into the numerical model. The corresponding R-curves were then obtained by numerical calculations. Excellent agreement was found between the bounds of the experimental and theoretical R-curves, lending confidence to the use of bridging laws as material properties and to the consistency of the experimental and theoretical approach as a whole.



Test specimens used for characterising adhesive joints: (a) a double cantilever beam sandwich specimen used for determining the bridging law, and (b) bonded panels containing a central notch used to measure notch sensitivity.



Measured tensile strength of butt-joints containing a central notch shown as a function of notch size. Strength, predicted by the notch sensitivity parameter α is shown as lines. The relevant value of α was determined to be about 0.01 from independent measurements of the bridging law.

Bridging law and notch sensitivity of adhesive joints

We have previously characterised the fracture resistance of composites with large-scale fibre bridging by a cohesive zone or a bridging law. An experimental study was recently performed to investigate to determine whether it is useful to characterise the strength of bonded joints by a bridging law, according to which the bridging stress depends only on the crack opening. Double cantilever beam specimens were loaded by pure bending moments. The bridging law was found to be highly nonlinear. The maximum bridging stress, determined independent by a direct tensile experiment of a butt-joint, differed less than 3 % from the maximum bridging stress determined by a J integral approach.

The bridging law was related to the notch sensitivity of bonded panels by a notch sensitivity index, α. The strength of bonded panels having a central notch was predicted solely from the bridging law parameters and α. This strength was also determined experimentally. A very good agreement was found between the predicted and measured strength values, suggesting that bridging laws are indeed useful for characterising adhesively bonded joints.

Adhesively bonded aluminium components

The Centre for Design of Adhesively Bonded Aluminium Components, CLEA, is a joint venture between Danish companies, universities and research institutes. The objective is to demonstrate the use of adhesive bonding in aluminium components and production. The activities involve designers, materials researchers, and construction and production engineers in order to focus on the use of adhesive bonding from the initial design. This enables quantitative integration of the bonding in the resulting product. A number of factors, e.g. surface treatment, joint design and protection, are important for the optimal bonding. The project aims to reduce the reluctance in industry to use adhesive bonds in load carrying parts by demonstrating that suitable design and processing can help adhesive bonds to be advantageous solutions, both technically and economonically.

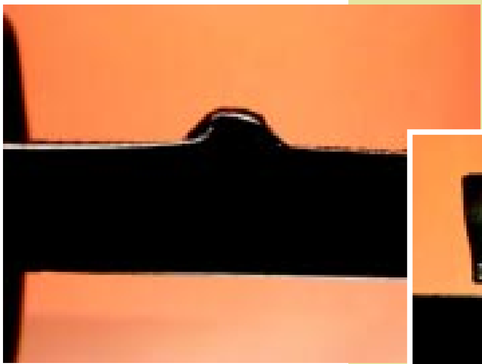
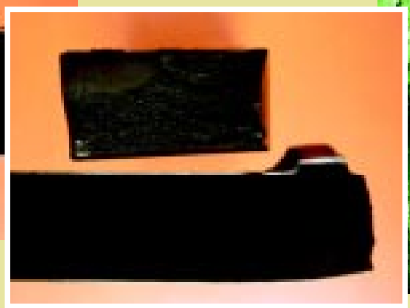
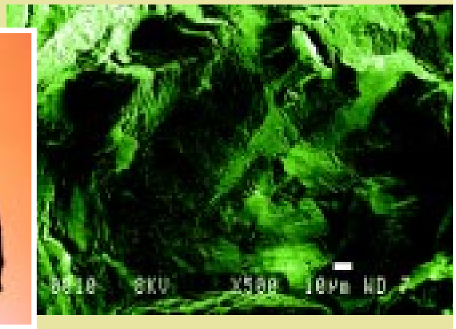
The work in the Department is concentrated on destructive testing and non-destructive characterisation of the joints. The main non-destructive technique used is ultrasonic scanning using pulse-echo technique. By measuring the ultrasonic reflection from the adhesion, the quality of the adhesion can be judged.

Performance and microstructure of tool steels

Fatigue mechanisms in tool steels

Tool steels are highly alloyed steels with high strength and hardness, and their typical microstructure consists of a dispersion of hard carbides in a matrix of tempered martensite. Depending on the heat treatment, different amounts of primary and secondary carbides as well as retained austenite are present, and each of these phases may have strong influence on the mechanical properties of the steel. When subjected to cyclic loading, tool steels eventually fail because fatigue microcracks induce brittle fracture. An improvement of the fatigue performance is therefore narrowed down to a suppression of crack initiation.

Low cycle fatigue tests were carried out on different commercial high-speed steels. A decrease of the mean stress, i.e. compressive prestress, clearly increases the fatigue life of a given tool steel, which is explained by a suppression of crack initiation due to compressive loads. Microstructural characterisation of the different tool steels showed that in the tempered condition the volume fractions of retained austenite were lower than 2-3 %. Retained austenite is not expected to have a dominating effect on the fatigue crack formation. Instead, hard phases like carbides and dislocation dense martensite laths are suggested as possible crack forming features during cyclic deformation.

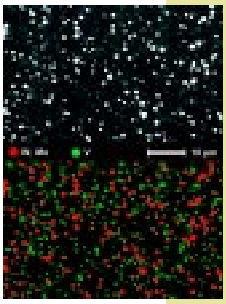




Specimen before and after fracture and microstructure of fracture surface

Analyses of carbides in tool steels

Most commercial tool steels contain carbon and alloying elements of magnitudes sufficient to harden the steel matrix and form alloy carbides during heat treatment. Typical alloying elements are tungsten, molybdenum, chromium and vanadium, which are all strong carbide-forming elements. Carbides in the hardened and tempered condition contribute to the desired properties of the steel depending on their composition, morphology and distribution. Primary carbides, which are present in the as delivered condition and which do not dissolve during heat treatment, are distinguished from secondary carbides which form during tempering. The primary carbides provide resistance to abrasive wear, whereas the secondary carbides improve the hardness.

Microstructural characterisation of carbides in different commercial high-speed steels was carried out using a combination of techniques, including X-ray and electron diffraction, light optical microscopy, scanning electron microscopy and transmission electron microscopy. In all steels, an isotropic distribution of spherical shaped carbides was found and two different types were identified: MC-type V_8C_7 and M_6C-type (Fe, Mo, W)$_6$C. No evidence was found for the presence of chromium-based carbides in the tempered condition, notwithstanding a chromium content of more than 4.0 % by weight. The presence of secondary carbides of a few nanometres is expected.



SEM micrograph (top) showing the carbide distribution in a commercial tool steel grade S390 ISOMATRIX from Böhler Edelstahl. Two types of carbides, (Fe, Mo, W)$_6$C and V_8C_7, have been identified in the EDS-map (bottom), which are coloured red and green, respectively.

Materials Technology
–synthesis, processing and products

The work described in this chapter comprises two programmes: (i) Powder Technological Materials, which deals with the development of new materials for a variety of applications, for instance the energy and transport sector, and (ii) Materials Chemistry, which is mainly directed towards solid oxide fuel cells (SOFC) and related electrochemical devices. In the first programme, activities are usually initiated by needs or problems encountered in industry, but conceived to be better solved within the research environment of the Department. In the second programme, which mainly concerns electroceramics, the activities cover the whole range from synthesis and characterisation of cell materials, to manufacture of cells and to assembly and testing of small cell stacks. Fundamental research supporting technological development is carried out in electrochemistry, solid state chemistry and ceramic processing.

Highlights are as follows:

i. Preparation of metal matrix composites with high wear resistance and satisfactory forgeability
ii. Observation of superplasticity in magnesium-based bulk amorphous alloys at temperatures between those of glass transition and crystallisation
iii. Study of contacts in SOFC stacks, leading to a lower resistance between current collectors and electrodes
iv. Derivation of a tentative model for oxygen reduction at SOFC cathodes
v. Study of the local environment of dopant ions in oxide based ceramics by means of vibrational spectroscopy

Powder Technological Materials

During the 90's the majority of activities in this programme concerned the classical powder technological areas of powder metallurgy and ceramics. Several new projects were started within a programme New Materials for Danish Industry under two Danish Materials Research and Development Programmes designated MUP I and II. Besides participating in several framework programmes, members of the Powder Technological Materials group have managed the Centre for Powder Metallurgy and the Centre for Advanced Technical Ceramics. Much valuable work has been initiated in these centres and in the framework programmes and a clear line can be drawn from this work to the present projects.

Two examples will be mentioned here. The first is the work on rapidly solidified alloys, which is based on laboratory scale equipment for production of gas atomised metal powders. The equipment was established in a centre project and has since been reconstructed to allow spray forming of alloys and metal matrix composites. Collaboration with Danish industry has lead to the development and characterisation of new, spray formed, wear resistant Fe-based matrix-ceramic composite materials. The formability of the materials has been demonstrated by forging experiments. The materials are expected to find use in industry, in agriculture and in forestry.

Another example is the work on gas sensors based on electroceramics. New oxide ion conducting materials were developed, with improved mechanical properties for oxygen sensors, as were improved techniques for the fabrication of oxygen sensors based on semiconductors of Mg-doped $SrTiO_3$. These materials and techniques are now being applied by Danish industry for production of oxygen sensors.

Preparation and characterisation of amorphous and nanocrystalline lightweight alloys has been started recently in the programme, as a spin-off of the work with spray forming techniques. This activity has rapidly lead to important technical results. These alloys become amorphous at modest cooling rates and show a glass transition temperature as much as 100 K below the crystallisation temperature. In this, they differ from traditional glassy metals, which must be cooled very rapidly to inhibit crystallisation. The materials show extreme plastic properties near the glass transition temperature, which suggest a great potential for industrial application.

The accumulation of energy by the storage of hydrogen in metals or carbon structures has been within the Department's expertise for many years. An increased need for energy storage is being experienced by industry and, more specifically, by the electrical power generating utilities. The level of activity work is rising rapidly at Risø and the Powder Technological Materials programme is expected in the very near future to become a major player within this field.

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 • Roulunds A/S, Denmark • Norsænk-Aalykke A/S, Denmark • A/S Hartfelt & Co., Denmark • Scan-Visan A/S, Denmark • Thürmer A/S, Denmark • Danish Technological Institute (DTI), Denmark
Danish Agency for Trade and Industry Centre for Powder Sensor Technology	• dk Teknik, Energi&Miljø, Denmark • Force, Denmark • Royal Veterinary and Agricultural University, Denmark • PBI-Dansensor A/S, Denmark • Rockwool A/S, Denmark
EFP98 Low Weight Storage for Hydrogen Cars	• IRD A/S, Denmark • Dept. of Chemistry, DTU, Denmark
EFP DK-SOFC 1996-1999	• Haldor Topsøe A/S, Denmark • IRD A/S, Denmark • Institute of Chemistry, OU, Denmark • Institute of Chemistry, DTU, Denmark
EFP2000 DK-SOFC 2000-2002	• Haldor Topsøe A/S, Denmark • IRD A/S, Denmark • Elsamprojekt A/S, Denmark • Energi E2 A/S, Denmark • Eltra, Denmark • Elkraft System a.m.b.h., Denmark
BRITE-EURAM Low Cost Fabrication and Improved Performance of SOFC Stack Components (LOCOSOFC)	• Rolls-Royce, UK • IRD A/S, Denmark • INPG, France • Gaz de France, France • NUVL, UK • Swiss Federal Inst. for Materials Testing and Research, Switzerland
Training and Mobility of Researchers Programme Alternative Anodes for SOFC	• British Gas, UK • University of St. Andrews, UK • University of Aveiro, Portugal • University of Patras, Greece • University of Twente, The Netherlands • Research Centre Jülich, Germany
FP5 Development of Energy Efficient and Innovative Ceramic Membrane Reforming Technology for Synthesis gas production (CERAM-GAS)	• L'Air Liquide S.A., France • Snamprogetti S.p.A., Italy • Politecnico de Torino, Italy
Swedish National Energy Administration Integration of SOFC into a Gas Turbine Process	• Lund Institute of Technology, Sweden • Power Utility Sydkraft, Sweden
FP5 Scale Up of a Multi-Functional Solid Oxide Fuel Cell to Multi-Tens of Kilowatt Levels (MF-SOFC)	• Rolls-Royce plc, UK • Imperial College, UK • Advanced Ceramics ltd., UK • Gaz de France, France

Continues on next page

Spray forming of metal matrix composites

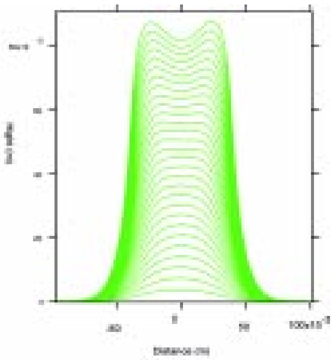
This work has covered the effect of added ceramic particles, and of their mean size, on the wear resistance and tensile strength of spray-formed steel-based materials. The work was carried out in the Department's spray-forming unit. Composites with a low alloyed steel matrix containing alumina particles were prepared by introducing ceramic paricles into the spraying plume. Cast hot-rolled metallic material without particles, spray-formed material without particles and the spray-formed composites with an average ceramic particle size of 46 and 134 µm were compared in terms of their wear resistance and tensile strength. It was found that the addition of alumina particles to the steel improved its wear resistance by a factor 2 to 3, but reduced the tensile strength and the strain to failure. However, the metal matrix composite materials still had satisfactory ductility. The composite was found to have good properties when subjected to hot-forging. This is very important for the application potential of these materials, where shaping problems are traditionally percieved as an obstacle to their use.

An integrated numerical model of the spray forming process

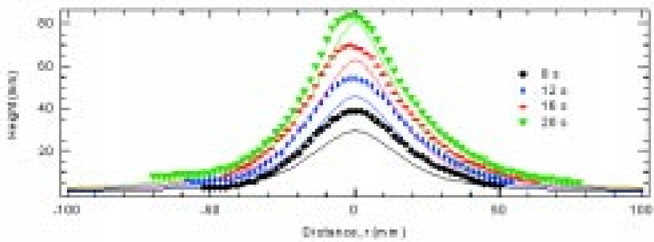
An integrated approach for modelling the entire spray forming process has been developed. The basis for the analysis is a recently developed model, which includes the interaction between an array of droplets and the enveloping gas. The deposition model has been formulated using a 2D cylindrical treatment of heat flow. This model was coupled with an atomisation model via a log-normal droplet size distribution. The coupling between atomisation and deposition was accomplished by ensuring that the total size distribution of the spray is the sum of local size distributions along the radius. Excellent agreement was observed between the calculation and the experimental results.

Spray forming with a stationary substrate produces a bell-shaped preform. To model the shape of a spray-formed billet, one has to take into account the combined effect of spray forming variables, namely the spray distribution, angular velocity of rotation, velocity of withdrawal, spray angle and eccentric distance. At present, these are treated by a 2D numerical model which predicts the evolution of billet geometry. This model was tested by comparing its predictions with experimental results from literature. It is planned to extend the model to 3D and enlarge it by including thermal parameters for the spray forming process.

Spray formed specimen



Modelled evolution of the shape at different times (rod like form)



Comparison between the calculated and the measured shape of the deposited material at different deposition times. The solid lines represent the prediction of the model while the experimental data are represented by symbols. (bell-shaped form).

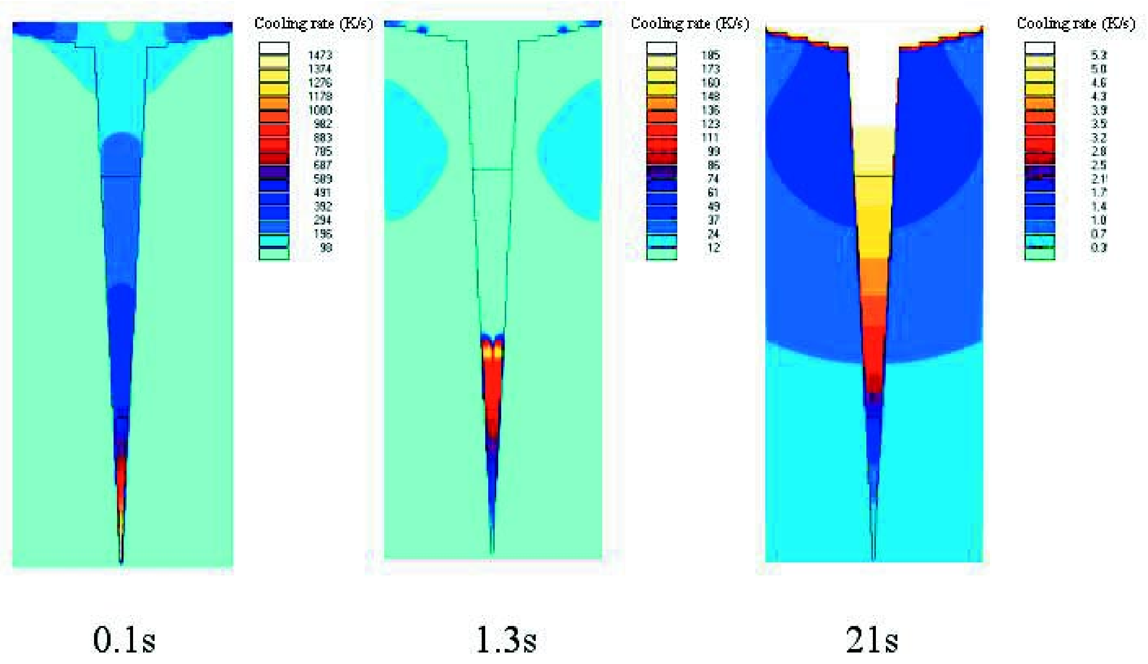
Bulk amorphous alloys

The conditions that lead to the formation of bulk amorphous states were studied by preparation and investigation of Mg-Cu-Al-Y alloys in their amorphous, super-cooled-liquid and crystalline states.

A crucial parameter in the production of bulk amorphous alloys is the rate at which the molten alloy is cooled. To obtain a wide range of cooling rates in a single operation, molten alloys were cast in a wedge shaped copper mould. The alloy was amorphous in the lower, narrow part of the mould, where the cooling rate is, high and crystalline in the upper part where this was lower. By measuring the temperature as a function of time at different positions in the mould and modelling the process, a quantitative description of the cooling behaviour was obtained. Using differential scanning calorimetry, the glass transition and crystallisation temperatures were determined for different heating rates and alloy compositions. Thus, a cooling rate temperature-transformation diagram was constructed, by which it is possible to make an estimate of the critical cooling rate for the formation of the amorphous phase. For a Mg60Cu30Y10 alloy, this rate is ~ 100 K/s. The addition of 5 at% Al increases the critical rate by an order of magnitude, resulting in a reduction of the obtainable thickness of amorphous specimens from ~3 mm for the Al free alloy to a fraction of a mm for the Al containing one.

The distribution of the crystalline and amorphous phases was investigated on a cross-section of an as-cast wedge shaped specimen using microfocus X-ray diffraction. As predicted by the computer simulations, amorphous regions were

Project type/Project name	Co-participants
NATO Science for Peace Programme Lithium Ion Rechargeable Batteries	• National Institute of Chemistry, Univ. of Ljubljana, Slovenia • Graz University, Austria • Danionics A/S, Denmark
INTAS Hydrogen in Oxide Systems	• University of Oslo, Norway • Imperial College, UK • Aristotle University of Thessaloniki, Greece • IPTI, St. Petersburg, Russia • Ural State University, Ekatarinburg, Russia • Russian Academy of Science, Ekatarinburg, Russia • Inst. of Atomic Energy, Almaty, Kazakhstan
Researchers Training Network High Temperature Proton Conductors	• University of St. Andrews, UK • University of Aveiro, Portugal • University of Patras, Greece • University of Oslo, Norway • University of Edinburgh, UK • Imperial College, UK • EPFL, Lausanne, Switzerland • Research Centre Jülich, Germany • CSIC, Madrid, Spain
European Science Foundation Optimisation of Solid State Electrochemical Processes for Hydro Carbon Oxidation (OSSEP)	• University of St. Andrews, UK • University of Aveiro, Portugal • University of Karlsruhe, Germany • University of Oslo, Norway • University of Delft, The Netherlands • Imperial College, UK • EPFL, Lausanne, Switzerland • University "La Sapienza", Rome, Italy • CSIC, Madrid, Spain



Computer simulation of the cooling rates in a Mg-Cu-Y specimen and in the surrounding wedge-shaped mould at different times during cooling of the specimen.

found close to the mould-sample interface, while crystalline regions were in the middle of the specimen. These investigations were made in collaboration with National Research Institute for Metals, Japan, and the Technical University of Denmark.

Superplastic behaviour of bulk amorphous alloys

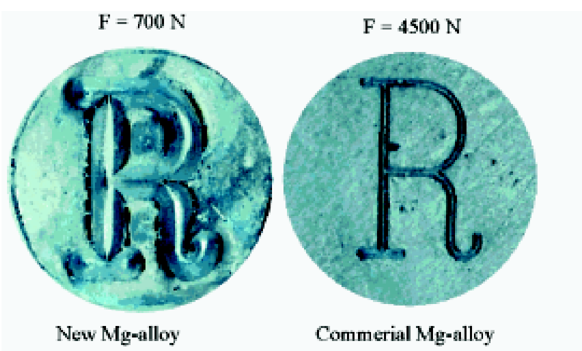
The very low flow stress observed in amorphous alloys, and their superplastic behaviour near the glass transition temperature, makes it possible to process them like oxide glasses. In fact, their elongation in the supercooled liquid state can be several hundred percent. However, these systems, unlike oxide glasses, are not thermally stable, and this may lead to crystallisation of the amorphous phase near the glass transition temperature.

Recently the Department initiated studies of Mg-based bulk amorphous alloys. A preliminary study of the superplasticity of these types of materials demonstrated that, with a relatively low load, it was possible to produce extensive deformation in the supercooled liquid region. The time dependence of the transition from the amorphous to the crystalline state has been investigated using X-ray diffraction at temperatures between the glass transition and crystallisation. This work forms the basis for selection of an optimum temperature in the supercooled liquid region for a potential industrial forming process of the Mg-alloys.

Electronic noses

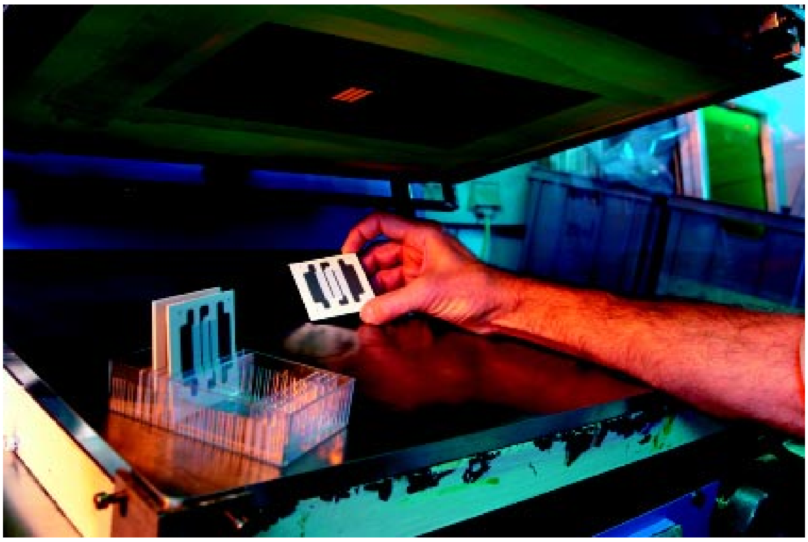
A new project on development of sensor array systems for odour detection, the so-called electronic noses, was started within the Danish Sensor Technology Programme. Compared with single sensor systems, electronic noses have the advantage that a characteristic signal pattern, a fingerprint, can be determined even for complex gas mixtures, as for instance the exhaust gases from industrial chimneys. These systems have important applications in process control and environmental protection.

In collaboration with Danish industry, a laboratory nose system based on the previously developed Mg-doped $SrTiO_3$ oxygen sensor and on commercial metal oxide sensors was constructed and tested. Principal component analysis of sensor signals obtained with pure gases, gas mixtures and industrial exhaust gases showed that the signal patterns could be resolved for each case. The experience obtained with this system will be useful in developing commercial electronic nose systems.



Deformation of Mg-based alloys: To the left an easily deformed bulk amorphous Mg-alloy (supercooled liquid region) and to the right a commercial Mg-alloy. Both samples were deformed at a temperature of ~436 K, with a load that is shown above each sample.

Materials Chemistry: development of solid oxide fuel cells



Fuel cells were introduced in the Department's energy related research programmes in 1989 because of their efficient, silent and pollution free properties for electric power generation. Discussions with potential sponsors resulted in the selection of the Solid Oxide Fuel Cell (SOFC), which has a relatively high operating temperature, 700 to 1000αC. Electrical efficiencies of 50 to 60% can be obtained from stand-alone high-temperature fuel cell plants, moreover, the temperature of exhaust gases makes the waste heat a high-value product. Since this type of cell is tolerant to the fuel gas components, it is possible to reform natural gas at the anode. This eliminates the need for auxiliary plants to produce and purify hydrogen; integrated reformers could operate on petrol or diesel. In a potential hydrogen-fuelled society, SOFCs could operate directly on hydrogen.

The development of fuel cells requires simultaneous efforts in several disciplines. Firstly, there is the need for a fundamental understanding of defect chemistry, electrochemistry, catalysis, ceramics and the high temperature corrosion of metals. Secondly, there is the design of cell components, the development of cost effective technologies for their fabrication, and the modelling of their electrical/mechanical performance. Thirdly, there are the economics of fuel cell operation. A large number of experimental techniques are used for the characterisation of the physical, chemical and electrical properties of these materials and devices.

Collaboration was established with Danish industries and universities, namely Haldor Topsøe A/S and ERL (now IRD A/S), the University of Southern Denmark and the Technical University of Denmark. This work has been performed within a series of projects referred to as DK-SOFC and sponsored by the Danish Energy Agency, Elsam and Elkraft. In parallel, a number of EU projects, bilateral development projects and educational activities were initiated to expand the available knowledge base.

The construction and test of a $^1/_2$ kW cell stack at 1000°C over 2000 h in 1995-96 was a most important milestone, demonstrating a high level of expertise in the relevant areas. The development in 1998-99 of a new type of anode supported cell, with excellent mechanical strength and low internal resistance at temperatures below 850°C has provided the basis for a five year continuation of the DK-SOFC projects. The production of cell stacks at a commercially acceptable cost will depend on a further reduction of the operating temperature to a level where metallic cell interconnection components can be used.

Electrodes

A considerable effort is made to improve the performance of the electrodes as these are responsible for a substantial part of the cell resistance. Three examples of recent work are given.

The traditional anode is a cermet consisting of yttria stabilised zirconia (YSZ) and nickel. Electrons and oxide ions are transported through Ni and YSZ, respectively, to the reaction sites located at the YSZ-Ni contact points. Substitution of YSZ e.g. by gadolinia doped ceria, may expand the area of reaction to the whole surface of the ceramic component, because ceria is both oxide ion conducting and electron conducting. By careful control of the microstructure and interface with YSZ, performances similar to those of good Ni/YSZ cermets have been obtained.

The intimate physical contact between electrolyte and electrode is essential for SOFC performance and durability. The contact may be improved by the presence of anchoring particles on the electrolyte surface. A material providing excellent anchoring properties has been identified and is the subject of a patent application.

A large number of composite cathodes based on lanthanum strontium manganate (LSM) and YSZ have been investigated using electrochemical impedance spectroscopy. A model derived for the oxygen reduction process includes five rate limiting processes. Two processes are tentatively ascribed to transport of oxide ions through the YSZ phase of the composite, two are related to elementary processes in oxygen reduction and the last to diffusion in a stagnant gas layer above the electrode structure.

Cell testing

The anode supported SOFC mentioned above has been operated in hydrogen with high water contents and at high currents ((1 A/cm^2) to simulate the conditions found in a true SOFC cell stack under operation. Under such conditions cells are found to be sensitive to the content of foreign phases and impurities. Nevertheless, cells were produced with an internal resistance of less than 0.30 Ωcm^2 at 850°C. This value is promising for the commercialisation of SOFCs.

In operation and testing of such cells, it is essential to obtain adequate gas sealing and good current collection over the cell surface. Compression forces on the seals and the current collectors must be applied in ways that are compatible with mass production of stacks. A number of test facilities have been designed and are being developed. A novel contacting structure has been developed to meet the above demands. A patent application for this structure is pending.

In an European project, MF-SOFC, the Department has started constructing test facilities for development of the anode supported cells and for testing multi-cell modules, as developed by Rolls-Royce for industrial power production applications.

High temperature corrosion

Ferritic steel alloys are being investigated as interconnect materials in SOFC stacks. Their thermal expansion coefficients (TEC) are close to those of the main SOFC components. Studies at the Department have revealed that both optimum TEC matching and oxidation resistance is obtained for Fe-Cr steels with ~20 at% Cr. Experimental contacts have been set up between foils of Fe-22Cr and electrode current collectors (the Ni-containing anode current collector and the LSM cathode current collector). The contacting procedure has been optimised such that contact resistances of less than 50 m(cm2 have been achieved, corresponding to about 10% of the total resistance. The findings have been confirmed in a short SOFC stack test.

Durability tests are now being addressed. The morphology of the oxide scale is found to depend strongly on the corroding atmosphere and in particular on the water vapour content. During long periods of operation, the structure and composition of the scale will change. The effects of this and minor, but important, alloy impurities are being investigated.

Mechanical properties of ceramic materials for fuel cells

Most of the materials used in solid oxide fuel cells are brittle ceramics. Therefore, an important part of achieving durable fuel cells is to measure relevant thermo-mechanical properties and use them in mechanical design against fracture. For fuel cell stacks, which are usually co-sintered at a high temperature, the most severe problem is cracking due to stresses arising from differences in the thermal expansion coefficients of the various layers.

Thus, one must determine the elastic properties of each material, the residual stress in each layer and the fracture resistance of each material and interface. Recently, the Young's modulus and Poisson's ratio of the porous NiO/YSZ composite layer were determined by uniaxial tensile tests of tapecast specimens.

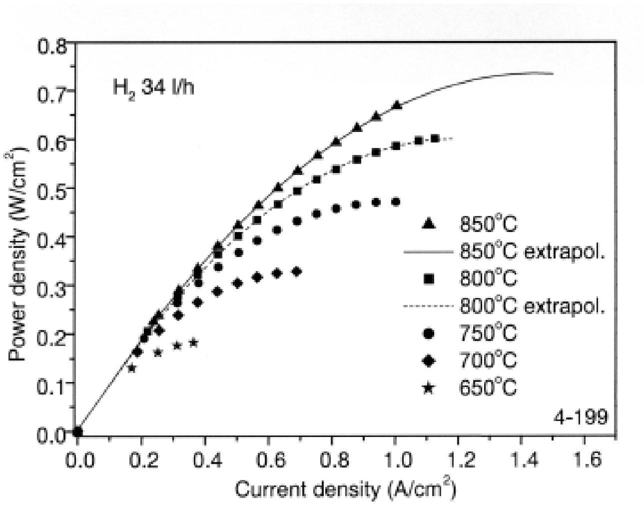
Focus is now on the measurement of residual stresses by means of bilayer experiments, and of fracture resistance of interfaces by means of double cantilever beam experiments, where the specimens are subjected to pure bending moments. Stable crack growth experiments at high temperature are planned for the future. Post-mortem investigations indicate that attention should be directed toward the causes of interfacial failure along the electrolyte/cathode interface.

New electrically conducting ceramics

Work on both ionic and electronic conduction in oxide ceramics with perovskite and fluorite structures has been carried out. The two main aims are to identify the parameters that control the various types of conductivity; and to find new materials which better fulfil the numerous requirements for the practical applications in an SOFC.

With respect to oxide ion conductivity in perovskites and fluorites we have found that a very important factor is the lattice stress, in other words, the oxide ion conductivity can be maximised if the lattice is stress free. Doping with oxides of lower valent metals is necessary for the creation of charge carriers (oxide ion vacancies). The dopant cation must fit snugly into the host lattice, i.e. have a matching ionic radius. This in turn implies that the symmetry of these structures usually remains close to cubic.

For electronic conductors, high symmetry seems also to be of great importance. This rule, combined with rules about relations between materials properties and thermal expansion, has resulted in the identification of new perovskites with an electronic conductivity about 500 S/cm. TEC matching with the YSZ electrolyte is better in the case of these compounds than in the case of strontium doped lanthanum cobaltite, the preferred material for current collection from the SOFC cathode.



The power density as a function of current density of a one-cell stack of a Ni/YSZ anode supported thin electrolyte cell with a graded LSM/YSZ cathode and metallic Fe-22Cr as the interconnector. The smooth lines are second order polynomial fits.

The new pilot plant under construction.

Establishment of pilot plant facility

An economically viable industrial production of advanced ceramic devices requires a fundamental understanding of the process parameters and their impact on the final product. It is practically impossible to obtain this knowledge in small-scale experimental laboratory productions, which suffer from problems of reproducibility. To overcome this obstacle, a pre-pilot ceramic processing plant is being established in the Department in close collaboration with the Danish company Haldor Topsøe A/S. The plant will be ready for operation in April 2001. Facilities will be available for the following processing steps:

- Powder processing
- Slurry fabrication for volumes of up to 25 litres
- Shaping and deposition techniques for thin layers
- Tape-casting, continuous on a 20 metre long tapecaster
- Screen printing of areas of up to 60x60cm^2
- Spraying, air or ultrasonically driven
- Sintering in furnaces with 1.2 m^3 chambers

The pre-pilot plant is initially aimed for use in the Danish Solid Oxide Fuel Cell project for scaling-up the production of anode-supported flat plate cells. The main themes for the three-year project period have been determined as:

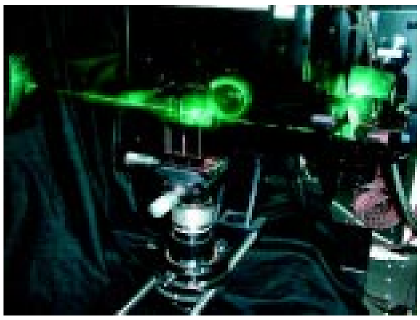
- Optimisation of quality/price relations
- Maximisation of durability and lifetime
- Minimisation of rejection
- Establishment of non-destructive characterisation methods

The tapecaster to be installed

Probing of defects and local disorder in ceramics by vibrational spectroscopy

Electric, magnetic and many other physical properties of ceramics can be tailored by acceptor doping or donor doping, which means substitution of host ions by lower valent or higher valent ions, respectively. This strategy is applied in optimisation of ceramics for use in SOFCs and in sensors. Diffraction gives information only about the average structure. Even though many ions in the doped material have a lower coordination number than in the undoped material, the diffraction patterns of doped oxides usually resemble the pattern of the host system.

To study the local arrangement of ions and vacancies one may use vibrational spectroscopy. While infrared absorption or reflection spectroscopy cannot at present be used at the operating temperature of SOFCs, Laser Raman spectroscopy (inelastic scattering of photons) can be performed up to 1000°C. Three important structure types are being investigated by this technique: fluorites $A_{1-x}Mf_xO_{2-y}$, perovskites $A_{1-x}Mf_xBO_{3-y}$ and pyrochlores $A_2Mf_xB_{2-x}O_{7-y}$. Extra vibrational modes, observed only in the doped systems, have been assigned to formation of new local structures.

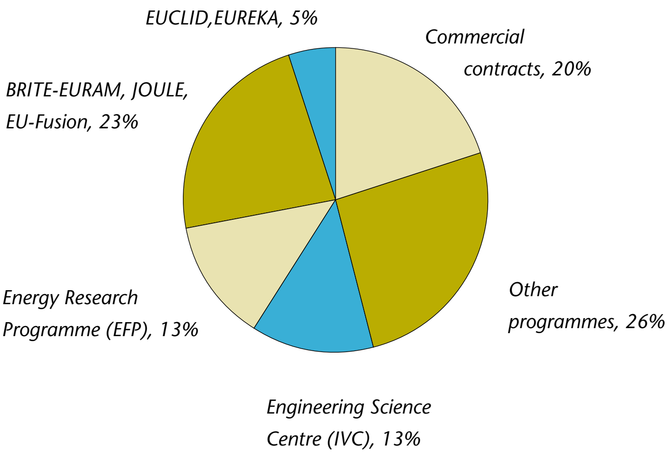


Excitation of Raman spectrum from a jewel sized zirconia single crystal with 514.5 nm light from an Ar-ion laser. Although the crystal is cubic one observes many more vibrational modes, than predicted from factor group analysis.

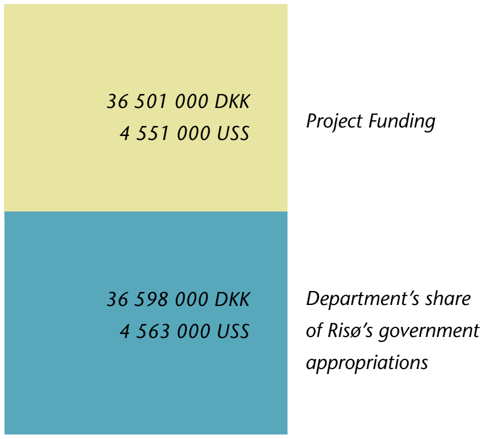
Finances

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 contracts.
The numbers given are in Danish kroner (DKK). The equivalent amount in US Dollars is also shown.

Project income

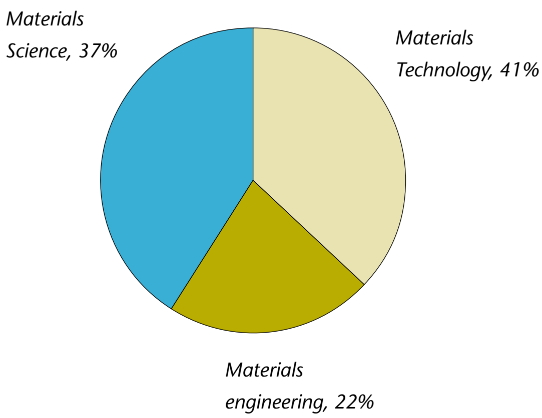


Income

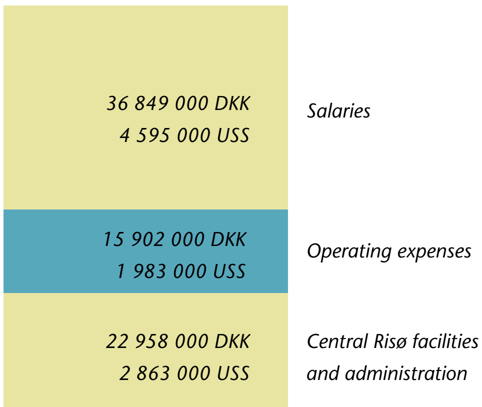


Total 73 099 000 DKK
9114 000 US$
(1999: 66 953 000 DKK)

Research areas (hours)



Expenditures



Total 75 709 000 DKK
9 441 000 US$
(1999: 70 405 000 DKK)

Staff

A person leaving (#) or joining (*) the Department during 2000 is counted as $^1/_2$ man year.

Head of Department

Niels Hansen

Academic staff (43.5 man years: 34.5 scientific officers and 9 experimental officers)

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
Engbæk, Preben*#
Gundtoft, Hans Erik
Hendriksen, Peter Vang
Huang, Xiaoxu
Jacobsen Krogsdal, Torben*
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.
Pantleon, Wolfgang*
Pedersen, Allan Schrøder
Pedersen, Ole Bøcker
Poulsen, Finn Willy
Poulsen, Henning F
Poulsen, Jørgen*.
Primdahl, Søren
Pryds, Nini*
Rheinländer, Jørgen#
Singh, Bachu N.
Sørensen, Bent F.
Sørensen, Ole Toft
Toft, Palle
Toftegaard, Helmuth L.
Winther, Grethe
Wert, John A.*

+) deceased January 2001

Professor John A. Wert joined the Department in March 2000 as a Research Professor especially within the fields of deformation processes, structure and properties of alloys and theoretical modelling. Before coming to Risø, John A. Wert has been Professor at University of Virginia, Charlottesville, USA for 15 years and 6 years at Rockwell International Science Centre in California.

Postgraduate students (14.5 man years)

Andersen, Lotte G.
Christiansen, Jesper*
Dinesen, Anders R
Glerup, Marianne
Hansen, Jesper Rømer#
Hinnum, Benjamin R.
Højerslev, Christian
Jensen, Karin Vels
Lauridsen, Erik M.
Lybye, Dorte#
Koch, Søren
Mikkelsen, Lars
Nielsen, Søren Fæster#
Nørbygaard, Thomas
Pedersen, Trine Bjerre
Petersen, Kenneth
Vegge, Tejs

Post Docs (9.5 man years)

Cendre, Emmanuelle
Friehling, Peter*
Gamstedt, Kristofer
Grievel, Jean Claude#
Hatchwell, Charles E.*
Holtappels, Peter W. #
Jacobsen, Torben K#
Jessen, Claus Qvist*
Margulies, Lawrence
Mallon, Stephen
Nielsen, Søren Fæster*
Pantleon, Wolfgang#
Pryds, Nini H#
Sabin, Tanya*
Schmidt, Søren*

Consultant

Waagepetersen, Gaston

Technical staff (31 man years)

Abdellahi, Ebtisam*
Adrian, Frank
Frederiksen, Henning
Gravesen, Niels Nørregaard
Hansen, Krystyna Anna*
Hersbøll, Bent#
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.
Olsen, Benny F.
Olsen, Henning
Olsen, Ole
Olsson, Jens O.
Paulsen, Henrik
Pedersen, Niels Jørgen
Sandsted, Kjeld
Stenkilde, Pia Sørup*
Strauss, Torben R.
Sørensen, Erling
Vogeley, Erik
Udbjørg Lund, Charlotte*
Aagesen, Sven

Office staff (6 man years)

Kiler, Diana#
Hoffmann Nielsen, Lis#
Lauritsen, Grethe Wengel
Nielsen Pihl, Jytte
Sørensen, Eva M.
Thomsen, Ann
Voss, Anita

Apprentices (1 man year)

Jacobsen, Rasmus*
Madsen H, Christian*

Prizes

In 2000, a member of the Department received two prizes, bringing the number of prizes and other special recognitions received during the past three years up to 14.

Niels Hansen was awarded the Poul Bergsøe Medal for his life-long contribution to materials science in Denmark.

Niels Hansen was awarded the Robert F. Mehl Medal for his outstanding work.

Niels Hansen (right) receives the Poul Bergsøe Medal from Povl Brøndsted (left), chairman of Danish Metallurgical Society.

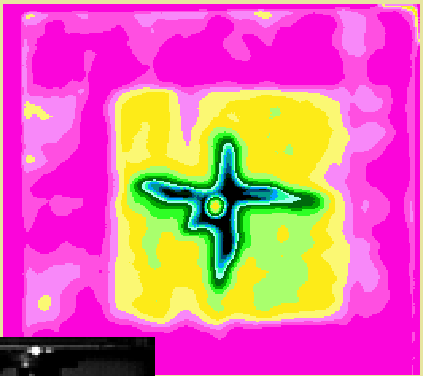
Industrial Activities

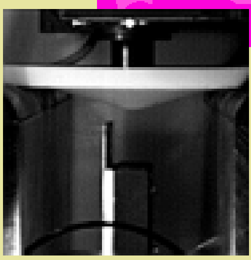
Collaboration with Industry

In 2000 the Department collaborated with about 55 Danish and 65 foreign industrial companies – either in externally funded projects or on a commercial basis.

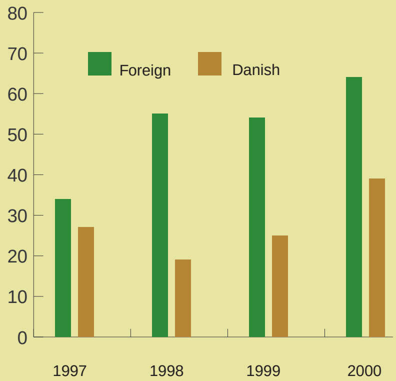
Externally funded industrial projects

Industrial companies participate in projects with external funding from e.g. the Danish government or EU.





Industrial partners in externally funded projects



Add-on armour is developed in a EUCLID project involving Danish DEMEX A/S and a number of foreign companies. The extent of damage after an impact test in the laboratory is visualised by ultrasonic image.

Industrial summer jobs for students

Summer jobs for undergraduate students have now been offered in co-operation with industry for three years, involving a total of 15 students. An increasing number of summer jobs are financed by industrial companies and the projects often serve as feasibility studies for potential larger projects. For example, one of the projects this year has been extended so that the student is still employed part time in the Department. This year Ellen and Valdemar Leth Christiansen's Family Foundation also contributed to the projects.

The students this year and their projects were:

Casper Thorning
'Recrystallisation and annealing of Titanium and Titanium alloys'.Supervisors: John Christensen (Danfoss), *Povl Brøndsted*

Lars Rindorf
'Development and testing of gas sensors for odour sensing'. Supervisors: Henning Jensen (PBI-Dansensor), *Ole Toft Sørensen*

Jan Høgsberg
'Crack growth in layered porous materials'. Supervisors: Niels Christiansen (Haldor Topsøe A/S), *Peter Vang Hendriksen*

Jens Høgh
'Characterisation of ceramic oxygen membranes'. Supervisors: Niels Christiansen (Haldor Topsøe A/S), *Peter Vang Hendriksen*

Jesper Holm
'FEM modelling of damping in fibre composites'. Supervisors: Stig Black (EM Fiber Glas A/S), *Bjørn Sejr Johansen*

Ann Marie Earwaker
'Assessment of techniques for fixing single yarn or roving in building up of fibre preforms for manufacturing of fibre composites'. Supervisor: *Aage Lystrup*

The increasing industrial demand for techniques developed in the Department is reflected in the fact that two new companies have been started by scientists from the Department in 2000. Jørgen Tyge Rheinländer's InnospeXion ApS (right) performs non-destructive evaluation, primarily using X-rays, and Torben Lorentzen's DanStir ApS (left) offers expertise with friction stir welding technology.

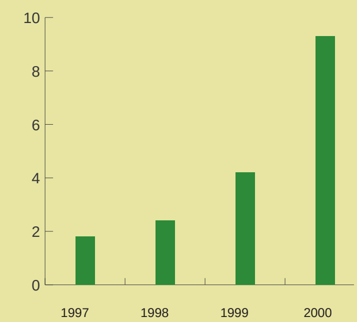
Wind turbine blade manufacturers are major partners in commercial projects (picture supplied by LM-Glasfiber A/S).

Commercial projects

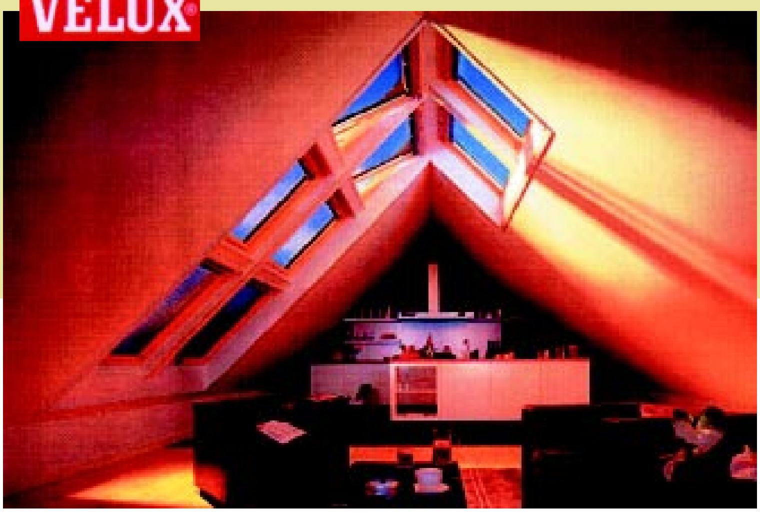
Collaboration on commercial basis is also increasing very rapidly – mostly with Danish companies and in confidentiality. In 2000 the major subjects of these projects have been

- Microscopy (both characterisation and courses)
- Mechanical testing (e.g. LM-Glasfiber A/S, LM-Industri A/S, Bonus Energy A/S, Vestas A/S, Grundfos A/S, Danfoss A/S, MAN B&W, Scan-Plast A/S)
- Process technology for composite polymeric materials (e.g. Trevira Neckelman A/S, Comfit Restore, Velux A/S)
- Process technology for ceramic materials (e.g. Haldor Topsøe A/S)

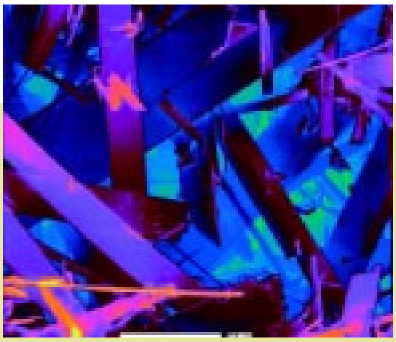
Commercial contracts



Materials and technology for manufacturing of accessories for windows based on fibre reinforced thermoplastic have been investigated on commercial basis for VELUX A/S.



Commercially Available Technologies



X-ray spectrometry of sod from an oven reveals that the oven has been polluted by molybdenum. The ESEM picture, which is taken without any sample preparation (like deposition of a carbon layer), shows crystals with the characteristic shape of MoO_3.

Electron microscopy and microanalysis

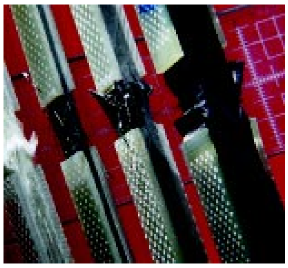
Two transmission electron microscopes (TEM) and three scanning electron microscopes (SEM) are operated by the Department. Attachments for TEM include energy dispersive X-ray spectrometer (EDS), scanning transmission attachment, serial electron energy loss spectrometer (EELS) and hollow cone illumination. 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)

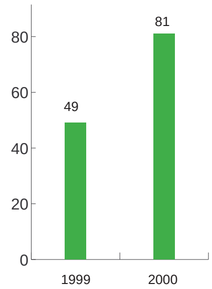
Powder characterisation

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)



Specimens of polymer matrix fibre composites after testing in compression



Number of accredited materials tests

Mechanical characterisation

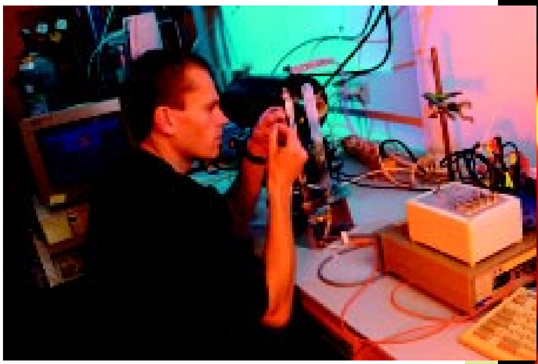
The Department is accredited by DANAK (Danish accreditation body) to carry out mechanical testing. Testing can be performed in different environments and at non-ambient temperatures (-196 °C to 1200 °C). The load ranges from 20 N to 250 kN. The equipment consists of several servohydraulic test machines, 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

Ceramic processing

Several processing techniques are available:

- Biaxial and isostatic pressing
- Tape casting (thin, 50 - 1000µm, 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



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

Set-up for measurement of electrical conductivity of a bar shaped sample. Tracking temporal changes in conductivity after a sudden change in atmosphere it is possible to deduce the oxide ion conductivity and the rate of the surface exchange process. The method is used in a project with Haldor Topsøe A/S aiming at making membrane reactors for production of synthesis gas.

3-Dimensional X-ray Diffraction (3DXRD) Microscope

A 3-Dimensional X-ray Diffraction (3DXRD) Microscope has been developed for fast and non-destructive characterisation of individual grains inside bulk materials (powders or polycrystals). The underlying method is diffraction with monochromatic and focused hard X- rays, enabling 3D studies of millimetre – centimetre thick specimens with a typical resolution of 5x5x50 µm^3. A 25 kN stress-rig and 2 furnaces are available, enabling the grain kinetics to be studies while deforming or annealing the samples.

Hundreds of grains can be studied simultaneously with respect to

- Position and volume
- Crystallographic orientation
- Elastic strain
- 3D grain shapes (for coarse-grained materials only)

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)

X-ray inspection of motor parts produced by FIAT reveals small cavities with a size up to 2x1x0.5 cm^3 (dark spots in the framed parts of the picture).

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

Filament winding technique has been used to manufacture fibre composite pressure vessels for hydrogen storage. The cylinders are wound around a polymer liner instead of the usual metal liner in order to minimise the weight. For a 9-litre cylinder, the weight is reduced from 5 to 3 kg at the expense of a measured hydrogen loss of around 1% per month. The hydrogen loss is completely eliminated if the polymer liner is coated with a thin metal film.

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

Spray forming of alloys and composites

Equipment for spray forming of alloys and metal matrix composites is available in the Department. The accessible melting temperature range is from room temperature up to 1700 °C and the melting capacity is currently two litres. Both light metals and iron based metals can be handled.

Joining by vacuum brazing

The Department has several programmable furnaces for heating to 1300 °C in a vacuum better than 5×10^{-3} Pa. Maximum specimen dimensions are $20 \times 20 \times 45$ 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.

Two wear parts drop forged from spray formed material by the Danish company Norsaenk-Aalykke. To the right: Forged pure metal. To the left: Forged metal matrix composite part. A tendency to crack formation is observed at the edges of the metal matrix composite part.

Educational Activities

The Department is strongly involved in the education of students at different levels. The involvement ranges from postgraduate and undergraduate courses and research projects, in collaboration with universities and industry, to one-day courses for high school classes. In addition, many staff members of the Department act as external university examiners.

PhD projects finished during 2000

Jesper Rømer Hansen
'Structural and electrical properties of electron conducting perovskites'.
Technical University of Denmark.
Supervisors: Torben Jacobsen (DTU), *Mogens Mogensen, Finn W. Poulsen, Peter Vang Hendriksen*

Mette Juhl Jørgensen
'Lanthanum manganate based cathodes for solid oxide fuel cells'.
Keele University, UK.
Supervisors: Kevin Kendall (Keele University), *Mogens Mogensen*

Dorthe Lybye
'Structural and electrical properties of perovskites'.
Technical University of Denmark.
Supervisors: Kurt Nielsen (DTU), *Mogens Mogensen, Finn Willy Poulsen*

Søren Fæster Nielsen
'Organised structures in deformed aluminium'.
University of Copenhagen, Denmark.
Supervisor: Erik Johnson (KU), *Torben Leffers*

Work,
PhD defence
and celebration!

Ongoing industrial PhD projects

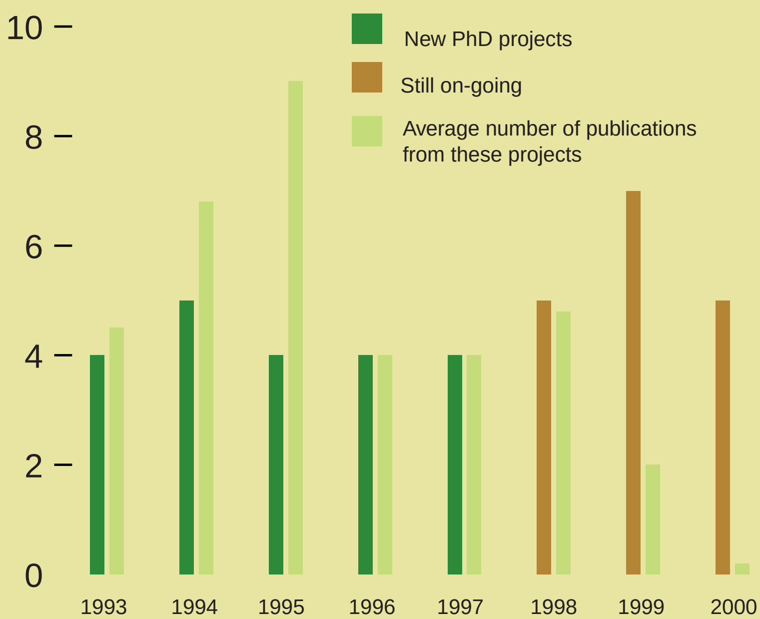
Eva Ravn Nielsen
'Low temperature sintering of piezo-electrics'.
Technical University of Denmark.
Supervisors: Erling Ringgård (Ferroperm A/S), Jacob W. Høj (DTU), *Finn Willy Poulsen*

Lene Pedersen
'Relations between composition, fabrication and properties for MMC friction materials for trucks'.
Technical University of Denmark.
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'.
Technical University of Denmark.
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)'.
Technical University of Denmark.
Supervisors: Jesper Valentin (OBTEC A/S), Jacob W. Høj (DTU), *Ole Toft Sørensen*

Over the past five years the Department has educated 21 PhDs and supervised 34 undergraduate projects in collaboration with both Danish (Technical University of Denmark, University of Copenhagen and University of Aarhus) and foreign universities.



Ongoing PhD projects

Lotte Gottschalck Andersen
'Structural properties of superconducting BiSCCO/Ag tapes during annealing'.
Technical University of Denmark.
Supervisors: Claus Schelde Jacobsen (DTU), *Henning Friis Poulsen*

Jesper Christiansen
'Dislocation interactions with surfaces and grain boundaries'.
Technical University of Denmark.
Supervisors: Jakob Schiøtz, Karsten Wedel Jacobsen (DTU), *Torben Leffers*

Anders Reves Dinesen
'Magnetic properties of amorphous and nanocrystalline alloys'
Technical University of Denmark.
Supervisors: Steen Mørup (DTU), *Søren Linderoth, Nini H. Pryds*

Marianne Glerup
'Raman spectroscopy and X-ray diffraction on oxides at high temperature'.
University of Copenhagen, Denmark.
Supervisors: Rolf W. Berg (DTU), Ole Faurskov Nielsen (KU), *Finn Willy Poulsen*

Benjamin Rask Hinnum
'Mobility of cations in oxide conductors'.
Technical University of Denmark.
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'.
Technical University of Denmark.
Supervisors: Marcel Somers (DTU), *Povl Brøndsted, Jesper Vejlø Carstensen*

Karin Vels Jensen
'Interfaces in composite electrodes for SOFC and high temperature electrolysers'.
Technical University of Denmark.
Supervisors: Ib Chorkendorff (DTU), *Mogens Mogensen, Carsten Bagger, Jørgen B. Bilde-Sørensen*

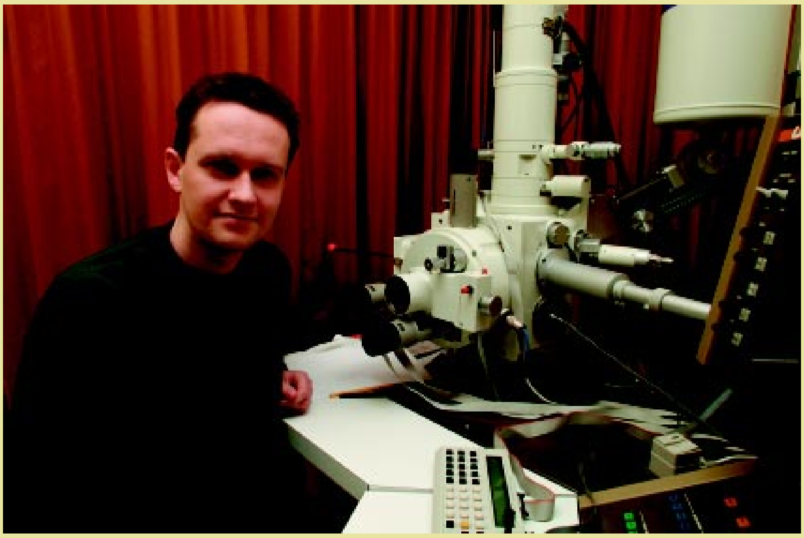
Søren Koch
'Contacting of ceramic materials'.
Technical University of Denmark.
Supervisors: Torben Jacobsen (DTU), *Carsten Bagger, Peter Vang Hendriksen, Mogens Mogensen*

Erik Mejdal Lauridsen
'Nucleation and grain growth studies by use of a 3D X-ray microscope'.
University of Copenhagen, Denmark.
Supervisors: Jens Als-Nielsen (KU), *Henning Friis Poulsen, Dorte Juul Jensen*

Bo Madsen
'Evaluation of properties of aligned plant fibre composites – an experimental modelling study'.
Technical University of Denmark.
Supervisors: Lars Damkilde, Preben Hoffmeyer (DTU), Anne Belinda Thomsen (PBK, Risø), *Hans Lilholt*

Lars Mikkelsen
'Oxidation of iron-chromium alloys'.
University of Southern Denmark, Odense.
Supervisors: Eivind Skou (USD), *Søren Linderoth, Mogens Mogensen, Peter Halvor Larsen*

Thomas Nørbygaard
'Structure and characteristics of general and special grain boundaries'.
University of Copenhagen, Denmark.
Supervisors: Erik Johnson (KU), *Jørgen B. Bilde-Sørensen*



Trine Bjerre Pedersen
'Modelling of residual stresses in sprayformed structures'.
Technical University of Denmark.
Supervisors: Jesper Hattel (DTU), *Nini H. Pryds*

Jens Pålsson
'Integration of a SOFC into a gas turbine process. Formulation and implementation of an SOFC - gas turbine system model'.
Lund Institute of Technology, Sweden.
Supervisors: Tord Torisson, Lars Sjunnesson (LTH), *Peter Vang Hendriksen*

Henrik Schmidt
'Modelling of mechanical and metallurgical properties of Friction Stir Welded Joints'
Technical University of Denmark.
Supervisors: Jesper Hattel (DTU), *John A. Wert*

Azra Selimovic
'Integration of a SOFC into a gas turbine process. Formulation and implementation of an SOFC stack model'.
Lund Institute of Technology, Sweden.
Supervisors: Tord Torisson, Lars Sjunnesson (LTH), *Peter Vang Hendriksen*

Tejs Vegge
'Defect dynamics at the atomic scale'.
Technical University of Denmark.
Supervisors: Karsten Wedel Jacobsen (DTU), *Torben Leffers, Ole Bøcker Pedersen*

Undergraduate and graduate projects

John Algers
'Free volume characterisation of palacos bone cement'.
Lund Institute of Technology, Sweden.
Supervisors: Frans H. J. Maurer (LTH), *Morten Eldrup*

Gilberto Formentini
'Fatigue behaviour of thixoformed aluminium alloys'.
University of Ancone, Italy.
Supervisor: *Yi-Lin Liu*

Emilie Ferrie
'RAYSQUINS detector - Inspection of pipe'.
Lyon National Institute of Applied Sciences, France.
Supervisor: *Emmanuelle Cendre*

Susanne Holm
'X-ray spectrometry in the environmental scanning electron microscope'.
Roskilde Technical School, Denmark.
Supervisor: *Jørgen B. Bilde-Sørensen*

Jens Høgh
'Characterisation of doped Lanthanum Chromite'.
Technical University of Denmark.
Supervisors: Jacob W. Høj (DTU), *Peter Vang Hendriksen*

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

Tom Würlitzer
'Ceramic electron conductors as electrodes for piezo-electric devices'.
Technical University of Denmark.
Supervisors: Erling Ringgaard (Ferroperm A/S), Jacob W. Høj (DTU), *Peter Halvor Larsen*

External lecture courses

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

'Electron back scattering diffraction in the scanning electron microscope'.
'Energy-dispersive X-ray spectroscopy in the low vacuum and environmental scanning electron microscopes'. Course at SEM 2000: Scanning electron microscopy - imaging and microanalysis. Chalmers Technical University, Gothenburg, Sweden. 17 - 19 October.

'Scanning electron microscopy'. Course given at ADC Denmark, Farum. 18 September.

Kristofer Gamstedt
'Polymers and polymer composites'. Undergraduate course in Applied Materials Science at Malmö University, Sweden. 24 November - 8 December.

Aage Lystrup
'Polymer based fibre composites'. Course given at Risø for students from Frederiksberg Technical School, Denmark. 10 November.

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

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.

Jørgen B. Bilde-Sørensen
PhD examiner. University of Copenhagen, Denmark.

Nikolaos Bonanos
PhD examiner. Oslo University, Norway.

Povl Brøndsted
PhD examiner. Technical University of Denmark.
PhD examiner. University of Aalborg, Denmark.

Dorte Juul Jensen
PhD examiner. Norwegian University of Technology and Science, Norway.

Hans Lilholt
PhD examiner. University of Aalborg, Denmark.

Søren Linderoth
PhD examiner. Technical University of Denmark.

Ole Bøcker Pedersen
PhD examiner. Katholieke Universiteit Leuven, Belgium.

Bent F. Sørensen
PhD examiner. Luleå University of Technology, Sweden.

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

Over the past five years 304 high school students from 27 schools (in 2000: 51 students from 4 schools) have participated in one-day courses in the Department on polymer chemistry and polymer based fibre composites. In 2000, the Department received funding from Ellen and Valdemar Leth Christiansen's Family Foundation which allowed the purchase of a vacuum oven to dry the synthesised polymer.

Academic Activities

Collaboration with universities and research institutes

Collaboration with the Department of Physics at the *Technical University of Denmark* was formalised in 1998 and members of the staff from both institutions have offices at the other institution. In total 4 senior scientists from Risø and DTU participate in this collaborative research. The Department also has formally established projects with other parts of the Technical University of Denmark, covering 4 of the university's 5 sectors. Other Danish universities and research institutes in joint projects are *University of Copenhagen*, *Royal Veterinary and Agricultural University*, *Royal Danish School of Pharmacy*, *University of Aalborg*, *University of Aarhus*, *University of Southern Denmark, Danish Agricultural Advisory Centre, Danish Technological Institute* and the *FORCE Institute*.

In 2000, Danish universities participated in 25 % of the Department's externally funded research projects. Externally funded projects also involved about 70 foreign universities and research institutions from 18 countries. As a result of these and less formalised projects, about 20 % of the Department's publications for the last five years have had Danish co-authors from outside Risø and 50 % have had foreign co-authors.

Visiting Scientists

Only visits of three or more days are listed.

Staff members visiting other institutions

Svend Ib Andersen
Laboratoire Matériaux Endommagement Fiabilité et Ingénierie des Procédés, ENSAM, Talence, France. 1 March - 31 August.

Jesper Vejlø Carstensen
Technical University of Dresden, Germany. 29 March - 7 April.

Niels Hansen
Sandia National Laboratory, Livermore, CA, USA. 18 March - 1 April.

Xiaoxu Huang
University of Science and Technology, Beijing, China. 22 May - 17 June.

Søren Linderoth
National Research Institute for Metals, Tsukuba. Japan. 28 August – 8 September.

Bachu N. Singh
University of Liverpool, UK. 18 - 23 March.
Indira Ghandi Centre for Atomic Research, Kalpakkam, India. 9 - 14 October.
Oak Ridge National Laboratory, TN, USA. 10 - 17 December.

Bent F. Sørensen
Luleå University of Technology, Sweden. 20 - 24 March.

Grethe Winther
University of Vienna, Austria. 3 – 5 July.

Scientists visiting the Department

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

Mr. Marcello Cabibbo
University of Ancona, Italy. 1 March - 10 May.

Mr. Robert Dominko
National Institute of Chemistry, Ljubljana, Slovenia. 3 July - 3 September.

Dr. Dan J. Edwards
Pacific Northwest National Laboratory, Richland, WA, USA. 28 June - 19 July.

Mr. Patrik Fernberg
Luleå University of Technology, Sweden. 28 October - 3 November.

Dr. Andy Godfrey
Sandia National Laboratory, Livermore, CA, USA. 8 May - 30 June.

Dr. Howard Heinisch
Pacific Northwest National Laboratory, Richland, WA, USA. 10 - 17 September.

Dr. Darcy A. Hughes
Sandia National Laboratory, Livermore, CA, USA. 1 - 29 July.

Prof. John T. S. Irvine
University of St. Andrews, UK. 7 - 10 November.

Prof. Shiori Ishino
University of Tokyo, Japan. 10 - 15 September.

Dr. Roberts Joffe
Luleå University of Technology, Sweden. 3 - 7 May.

Conferences

21st Risø International Symposium

The title of the International Symposium of this year was "Recrystallisation – Fundamental Aspects and Relations to Deformation Microstructure". It was held at Risø National Laboratory 4 -8 September with 104 participants from universities, research institutes and industry in 21 countries.

The developments of advanced automatic characterisation techniques have within the field of recrystallisation led to lots of new experimental data but less on the fundamental mechanisms governing recrystallisation. To differentiate this Risø Symposium from the series of international conferences on recrystallisation, which have been held four times at various locations, it was decided to focus on the fundamentals of recrystallisation. At the Symposium this challenge was accepted in most of the papers, and key themes were i) grain boundary migration, ii) relations between deformation microstructure and nucleation, iii) advanced recrystallisation modelling, and iv) recrystallisation in metals optimised for mechanical properties. Also special attention was given to microstructure in highly strained materials and on methods that link scientific understanding to industrial practice.

The Symposium was organised by the Department in collaboration with the Engineering Science Centre at Risø for Structural Characterisation and Modelling of Materials.

Organisers: N. Hansen, X. Huang, D. Juul Jensen, E.M. Lauridsen, G.W. Lauritzen, N.C. Krieger Lassen, T. Leffers, W. Pantleon, T.J. Sabin, J.A. Wert

Dr. Sarah Lillywhite
University of Birmingham, UK. 13 March - 9 April.

Prof. Oleg B. Naimark
Institute of Continuous Media Machanics of the Russian Academy of Sciences, Perm, Russia. 16 - 20 December

Mr. Cyril Nedaud
Ecole Nationale Supérieure de Céramique Industrielle, Limoges, France.
1 January - 30 June.

Dr. Tatsuya Okada
Tokushima University, Japan. 3 July - 15 September.

Prof. James F. Stubbins
University of Illinois, Urbana, IL, USA. 20 February - 4 March.

Dr. Roy Vandermeer
Alexandria, VA, USA. 21 August - 9 September.

Prof. John A. Wert
University of Virginia, Charlottesville, VA, USA. 16 January - 28 February.

Dr. Erlin Zhang
Harbin Institute of Technology, China. 1 October - 31 December.

Ms. Jana Zrubcova
Slovak Academy of Science, Bratislava, Slovakia. 1 January - 18 April and 23 October - 22 November.

The Department practised African dancing at the annual Christmas party

Participation in committees

Research Programme Committees

Tom Løgstrup Andersen
Steering Committee of the Non-food program ‘Plant Fibre Products – Defibration and Composites’.

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

Janet J. Bentzen
Project Management Committee of the EU BRITE-EURAM project ‘Hypereutectic Al/Si Alloys for Automotive Components (HAforAC)’.

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

Povl Brøndsted
Steering Committee of the Danish Center project CLEA.

Niels Hansen
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’.

Project Management Committee of the EU project ‘Development of energy Efficient and Innovative Ceramic Membrane Reforming Technology for Synthesis Gas Production (CERAM –GAS)’.

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

Torben Leffers
Ministry of Research Programme Committee for Materials Research. Copenhagen, Denmark.

Board of Ingeniørvidenskabelig Fond og G. A. Hagemanns Mindefond. Copenhagen, Denmark.

Hans Lilholt
Project Management Committee of the EU BRITE project ‘Thixoforming of Advanced Light Metals for Automotive Components (TALMAC)’. (Chairman).

Steering Committee of the Non-food program ‘Plant Fibre Products – Defibration and Composites’. (Chairman).

Steering Committee of the Danish Materials Science Programme project ‘High performance hemp fibres and improved fibre network for composites’.

Research and Industrial Support Group for the research programme ‘Structurally graded polymeric materials and filled polymers’. Aalborg University, Denmark.

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

Project Management Committee of the EU project ‘Scale Up of a Multi-Functional Solid Oxide Fuel Cell to Multi-Tens of Kilowatt Levels (MF-SOFC)’.

Torben Lorentzen
Project Management Committee of the EU BRITE-EURAM project ‘Residual Stress Standard using Neutron diffraction (RESTAND)’.
Steering Committee of the EU-funded thematic network ‘Training Industry in Neutron Strain Scanning (TRAINSS)’.

Project Management Committee of the EU project 'Welding of Airframes using Friction Stir Welding (WAFS)'.

Project Management Committee of the EU project 'Joining Dissimilar Materials and Composites using Friction Stir Welding (JOIN-DMC)'.

Mogens Mogensen
Steering Committee of the Danish Solid Oxide Fuel Cell Programme.

Project Management Committee of the EU BRITE-EURAM project 'Low-cost Fabrication and Improved Performance of Solid Oxide Fuel Cell Stack Components'. (Chairman).

Project Management Committee of the EU Training and Mobility of Researchers

Network project 'Alternative Oxide Anodes for Direct Oxidation of Methane in SOFCs'.

Project Management Committee of the EU Training and Mobility of Researchers Network project 'Investigation of High Temperature Solid Proton Conductors of Relevance to Fuel Processing and Energy Conversion Applications'.

Steering Committee of the European Science Foundation program 'Optimisation of Solid State Electrochemical Processes for Hydrocarbon Oxidation (OSSEP)'.

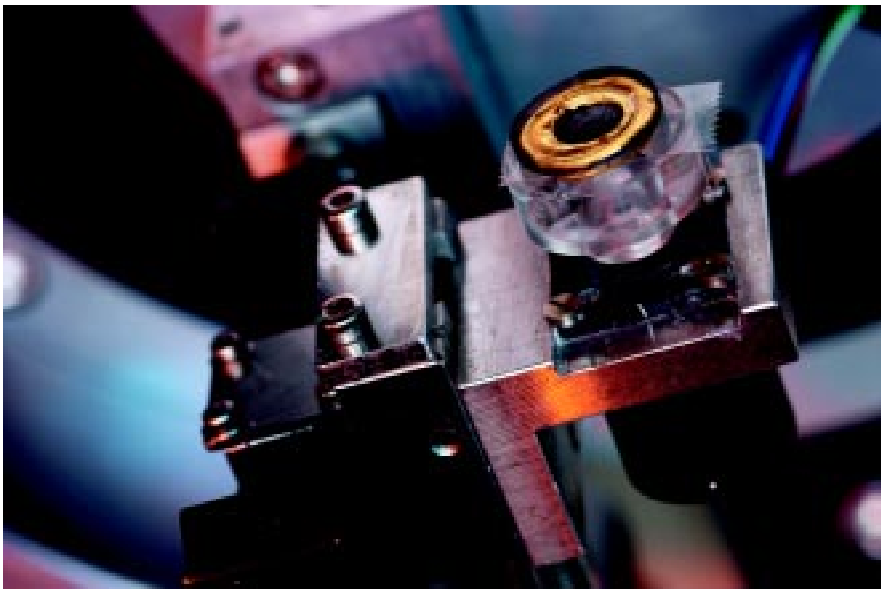
NATO Science for Peace Programme 'Lithium Ion Rechargeable Batteries'. (Project director).

Tage M. Nilsson
Management Committee of the EU BRITE-EURAM project 'Innovative Casting Process of Lighter Steel Components for the Transport Industry (ICARO)'.

Management Committee for the EU BRITE-EURAM Thematic Network 'Action for Low Weight Automotive Technologies (FLOAT)'.

Management Committee for EU BRITE-EURAM project 'Development and Performance Evaluation of a Fast X-Radioscopic and Lock-in Thermographic Non-Destructive Evaluation System for Fibre Based Technical Composites (FIBRINS)' (Chairman).

Management Committee for the EU GROWTH project 'Joining Dissimilar Materials and Composites by Friction Stir Welding (Join-DMC)'.



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

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

Jørgen T. Rheinländer
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).

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 System for Fibre Based Technical Composites (FIBRINS)'. (Chairman).

Project Management Committee of the EU BRITE-EURAM project 'Hyper-Eutectic Alloys for Automotive Components (HAforAC)'.

Steering Committee for the EU-funded Thematic Network 'Plant Life Assessment Network (PLAN)'. (Cluster Co-ordinator).

Advisory Committees

Niels Hansen
Advisory Group for Hydrogen Research. The Danish Ministry of Environment and Energy.

Technical Assessor. DANAK, Copenhagen, Denmark.

Advisory Committee of the Engineering Science Centre (at Risø) for Structural Characterization and Modelling of Materials.

COST Technical Committee on Materials. Brussels, Belgium.

Technical Scientific Advisory Board, GKSS Forschungszentrum. Geesthacht, Germany.

Torben K. Jacobsen
Expert Group on Certification of Rotor Blades for Wind Turbines. The Danish Ministry of Environment and Energy.

Dorte Juul Jensen
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.

Review Committee for Materials Engineering and Environmental Matters under the European Synchrotron Radiation Facility. Grenoble, France.

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.

Scientific Advisory Committee of the European Spallation Source (ESS).

Scientific Advisory Committee of ESS in Scandinavia.

Aage Lystrup
Advisory Committee for Research and Education. The Danish Plastics Federation, Section for Composites.

Mogens Mogensen
Advisory Board for the NATO Science for Peace Programme 'Lithium Ion Rechargeable Batteries'.

Finn Willy Poulsen
Expert and Danish contact. IEA Annex SOFC Collaboration.

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

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 Standardisation Committee.

Editorial Committees

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

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 Microstructures'.

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'.

The Department learned how to put out fires

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

Mogens Mogensen
Contributor to 'The Great Danish Encyclopedia'.

Allan Schrøder Pedersen
Editorial panel of 'Powder Metallurgy'.

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

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 and Calorimetry'.
Editorial board of 'Journal of the European Ceramic Society'.

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. Gaithersburg, MD, USA.

International advisory committee for International Conference on Advanced Materials Processing (ICAMP 2000). November 2000. New Zealand.

International advisory committee for 'Advanced Materials and Technologies' (AMT'2001). Poland.

Scientific committee of the European Research Conference on Plasticity of Materials. September 2000. Acquafredda, Italy.

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
Organising committee of the 12'th International Conference on Thermal Analysis and Calorimetry. August 2000. Copenhagen, Denmark.

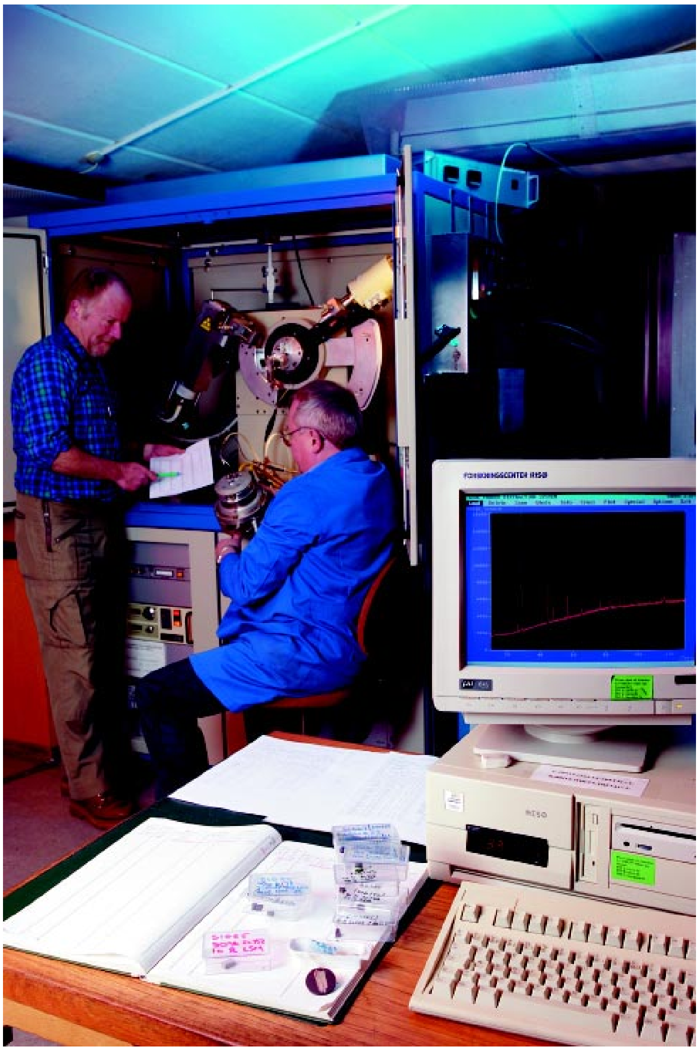
International advisory committee of the 7'th Conference and Exhibition of the European Ceramic Society. September 2001. Brugge, Belgium.

Hans Lilholt
International advisory committee for the 11'th International Conference on Mechanics of Composite Materials (MCM-2000). June 2000. Riga, Latvia.

International advisory committee for the 6'th International Conference on Deformation and Fracture of Composites. April 2001. Manchester, UK.

International advisory committee for the 13'th International Conference on Composite Materials (ICCM-13). June 2001. Bejing, China.

Søren Linderoth
Organising committee of the Nanoworkshop on Nanomagnetism. May 2000. Lyngby, Denmark.



Torben Lorentzen
International scientific committee of the International Conference on Stress Evaluation. December 2000. Reims, France.

Mogens Mogensen
International advisory board of the International Conference on Solid State Ionics - Materials and Processes for Energy and Environment. July 2001. Cairns, Australia.

Co-organiser of the 4th International Symposium on Ionic and Mixed Conducting Ceramics; part of Joint ECS - ISE Meeting. September 2001. San Francisco, CA, USA.

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 (ISOMALM 2000). October 2000. Kalpakkam, India.

Scientific committee of the 2'nd International Conference on Fatigue of Composites. June 2000. Williamsburg, VA, USA.

International scientific committee of the International Conference on Fatigue Damage of Structural Materials III. September 2000. Hyannis, MA, USA.

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

Bachu N. Singh
Organising committee of the International Workshop on Dislocation-Defect Interactions in Irradiated Materials. April 2000. Toledo, Spain.

Organising committee of the International Workshop on Production and Accumulation of Defects under Cascade Damage Conditions. September 2000. Barcelona, Spain.

Advisory board for the International Symposium on Materials Ageing and Life Management (ISOMALM 2000). October 2000. Kalpakkam, India.

Programme committee and publication committee of the 10'th International Conference on Fusion Reactor Materials. October 2001. Baden Baden, Germany.

Ole Toft Sørensen
Organising committee of the 12th International Conference on Thermal Analysis and Calorimetry. August 2000. Copenhagen, Denmark. (Chairman).

Professional Societies

Janet J. Bentzen
Executive committee of the Danish Society for Materials Research and Testing.

Jørgen B. Bilde-Sørensen
Board of the Scandinavian Society for Electron Microscopy.

Executive committee of the Danish Society for Materials Research and Testing.

Povl Brøndsted
Executive committee of the Danish Metallurgical Society. (Chairman).

Niels Hansen
Danish Academy of Technical Sciences.

Dorte Juul Jensen
Danish Academy of Technical Sciences.

Peter Halvor Larsen
Executive committee of the Danish Ceramic Society. (Chairman).

Executive committee of the Danish Society for Thermal Analysis and Calorimetry.

Executive committee of the Nordic Society for Thermal Analysis and Calorimetry.

Council of the European Ceramic Society.

Council of the International Confederation for Thermal Analysis and Calorimetry.

Hans Lilholt
International Committee of Composite Materials. Philadelphia, PA, USA.

Council and Executive Committee of the European Society for Composite Materials. London, UK.

Mogens Mogensen
Executive Committee of the Danish Electrochemical Society. (Vice-President).

Executive Committee of the High Temperature Materials Division of the Electrochemical Society. Pennington, NJ, USA.

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

Ole Toft Sørensen
Nordic Society for Thermal Analysis and Calorimetry.

Published Work

The number of publications in 2000 was 151, similar to that of 1999. In addition, members of the Department delivered about 80 presentations at conferences and meetings where no proceedings were subsequently published and wrote 28 internal reports.
Some papers are co-authored with scientists from other institutions, as a result of national and international collaboration. Names of authors from the Department are written in italics. Note that articles are sorted by the origin of publication (abroad or Denmark), not by language.

International Publications

1. Almazouzi, A.; Rubia, T.D. de la; *Singh, B.N.;* Victoria, M., Basic aspects of differences in irradiation effects between fcc, bcc and hcp metals and alloys. Editors' summary. J. Nucl. Mater. (2000) v. 276 p. 295-296

2. Almazouzi, A.; Victoria, M.; Rubia, T.D. de la; *Singh, B.N. (eds.)*, Proceedings. International workshop on basic aspects of differences in irradiation effects between FCC, BCC and HCP metals and alloys, Asturias (ES), 15-20 Oct 1998. (Elsevier, Amsterdam, 2000) (Journal of Nuclear Materials, v. 276) 306 p.

3. Anderegg, T.S.; *Wert, J.A.*, Effect of strain state on a yield anisotropy model for materials strengthened by plate-shaped precipitates. Mater. Sci. Forum (2000) v. 331-333 p. 1267-1272

4. Armstrong, W.D.; *Lilholt, H.*, The time dependent, super-viscoelastic behavior of NiTi shape memory alloy fiber reinforced polymer matrix composites. Mater. Sci. Eng. B (2000) v. 68 p. 149-155

5. Breuer, D.; Klimanek, P.; *Pantleon, W.*, X-ray determination of dislocation density and arrangement in plastically deformed copper. J. Appl. Cryst. (2000) v. 33 p. 1284-1294

6. Brown, M.; *Primdahl, S.; Mogensen, M.*, Structure/performance relations for Ni/yttria-stabilized zirconia anodes for solid oxide fuel cells. J. Electrochem. Soc. (2000) v. 147 p. 475-485

7. Edwards, D.J.; *Singh, B.N.*, Overaging of Outokumpu CuCrZr. In: Fusion materials. Semiannual progress report for the period ending June 30, 2000. DOE/ER-0313/28 (2000) p. 176-182

8. Edwards, D.J.; *Singh, B.N.; Toft, P.*, Radiation hardening in HCP titanium alloys. In: Fusion materials. Semiannual progress report for the period ending June 30, 2000. DOE/ER-0313/28 (2000) p. 214-220

9. *Eldrup, M.; Schrøder Pedersen, A.;* Ohnuma, M.; *Pryds, N.H.; Linderoth, S.*, Bulk amorphous alloys: Preparation and properties of $(Mg_{0.98}Al_{0.02})_x(Cu_{0.75}Y_{0.25})_{1-x}$. Mater. Sci. Forum (2000) v. 343-346 p. 123-128

10. *Eldrup, M.; Singh, B.N.*, Study of defect annealing behaviour in neutron irradiated Cu and Fe using positron annihilation and electrical conductivity. J. Nucl. Mater. (2000) v. 276 p. 269-277

11. Fiori, F.; Girardin, E.; Giuliani, A.; *Lorentzen, T.;* Pyzalla, A.; Rustichelli, F.; Stanic, V., Neutron diffraction measurements for the determination of residual stresses in MMC tensile and fatigue specimens. Physica B (2000) v. 276-278 p. 923-924

12. *Gamstedt, E.K.*, Effects of debonding and fiber strength distribution on fatigue-damage propagation in carbon fiber-reinforced epoxy. J. Appl. Polym. Sci. (2000) v. 76 p. 457-474

13. *Gamstedt, E.K.*, Fatigue in composite laminates - a qualitative link from micromechanics to fatigue life performance. In: Recent developments in durability analysis of composite systems. Cardon, A.H.; Fukuda, H.; Reifsnider, K.L.; Verchery, G. (eds.), (A.A. Balkema Publishers, Rotterdam, 2000) p. 87-100

14. Ghoniem, N.M.; *Singh, B.N.;* Sun, L.Z.; Rubia, T.D. de la, Interaction and accumulation of glissile defect clusters near dislocations. J. Nucl. Mater. (2000) v. 276 p. 166-177

15. Ghoniem, N.M.; *Singh, B.N.;* Sun, L.Z.; Rubia, T. D. de la, Dislocation decoration with nano-scale defect clusters in irradiated metals. In: Fusion materials. Semiannual progress report for the period ending December 31, 1999. DOE/ER-0313/27 (2000) p. 222-223

16. Ghoniem, N.M.; Sun, L.Z.; *Singh, B.N.;* Tong, S.-H., Investigations of radiation hardening and plastic instability in FCC metals. In: Fusion materials. Semiannual progress report for the period ending December 31, 1999. DOE/ER-0313/27 (2000) p. 226-228

17. *Glerup, M.;* Nielsen, O.F.; Christensen, D.H., Laser frequency dependence of broad backgrounds in Raman spectra of Pd/Mg(Al)O. J. Mol. Struct. (2000) v. 509 p. 165-170

18. Godfrey, A.; *Krieger Lassen, N.C.*, Unsupervised approval criteria for automated EBSP investigation of deformed metals. J. Microsc. (2000) v. 197 p. 249-259

19. Golubov, S.I.; *Singh, B.N.;* Trinkaus, H., Defect accumulation in fcc and bcc metals and alloys under cascade damage conditions. Towards a generalisation of the production bias model. J. Nucl. Mater. (2000) v. 276 p. 78-89

20. Heinisch, H.L.; *Singh, B.N.;* Golubov, S.I., Kinetic Monte Carlo studies of the effects of Burgers vector changes on the reaction kinetics of one-dimensionally gliding interstitial clusters. J. Nucl. Mater. (2000) v. 276 p. 59-64

21. Heinisch, H.L.; *Singh, B.N.;* Golubov, S.I., The effects of one-dimensional glide on the reaction kinetics of interstitial clusters. J. Nucl. Mater. (2000) v. 283-287 p. 737-740

22. Heinisch, H.L.; *Singh, B.N.;* Golubov, S.I., 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, 1999. DOE/ER-0313/27 (2000) p. 211-213

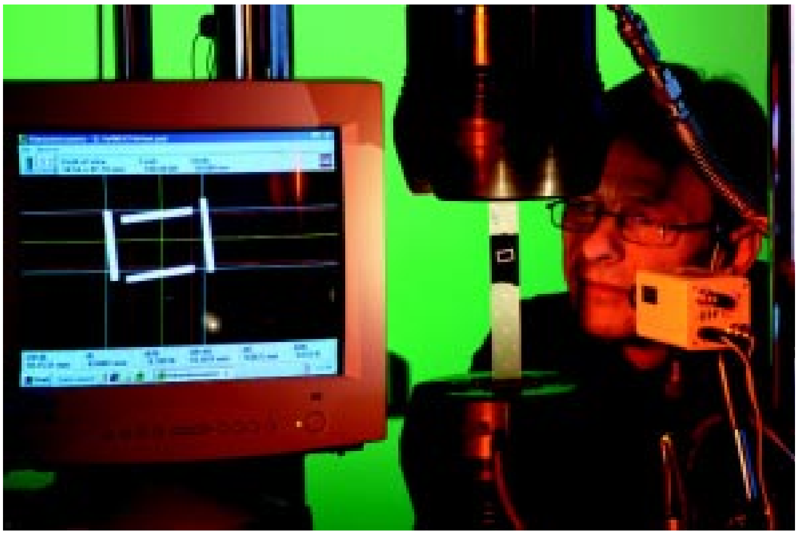
23. *Hendriksen, P.V.; Larsen, P.H.; Mogensen, M.; Poulsen, F.W.;* Wiik, K., Prospects and problems of dense oxygen permeable membranes. Catal. Today (2000) v. 56 p. 283-295

24. Hirade, T.; Maurer, F.H.J.; *Eldrup, M.*, Positronium formation at low temperatures: The role of trapped electrons. Radiat. Phys. Chem. (2000) v. 58 p. 465-471

25. *Holtappels, P.; Poulsen, F.W.; Mogensen, M.*, Electrical conductivities and chemical stabilities of mixed conducting pyrochlores for SOFC applications. Solid State Ionics (2000) v. 135 p. 675-679

26. *Huang, X.; Pryds, N.H.*, Crystallography and morphology of cementite precipitates formed during rapid solidification of a ferritic stainless steel. Acta Mater. (2000) v. 48 p. 4073-4082

27. *Huang, X.; Pryds, N.H.*, The effect of cooling rate on the microstructures formed during solidification of ferritic steel. Metall. Mater. Trans. A (2000) v. 31 p. 3155-3166



28. Hughes, D.A.; *Hansen, N.*, Microstructure and strength of nickel at large strains. Acta Mater. (2000) v. 48 p. 2985-3004

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30. *Jensen, K.V.; Primdahl, S.; Mogensen, M.*, Studies of theNi/YSZ interface in solid oxide fuel cell anodes. In: Mass and charge transport in inorganic materials. Fundamentals to devices. Part B. Vincenzini, P.; Buscaglia, V. (eds.), (Techna, Faenza, 2000) (Advances in Science and Technology, 29) p. 1443-1449

31. *Juhl Jørgensen, M.; Holtappels, P.; Appel, C.C.*, Durability test of SOFC cathodes. J. Appl. Electrochem. (2000) v. 30 p. 411-418

32. Kek, D.; *Bonanos, N.; Mogensen, M.;* Pejovnik, S., Effect of electrode material on the oxidation of H_2 at the metal-$Sr_{0.995}Ce_{0.95}Y_{0.05}O_{2.970}$ interface. Solid State Ionics (2000) v. 131 p. 249-259

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34. Kumar, A.N.; *Sørensen, B.F.*, Fracture resistance and stable crack-growth behavior of 8-mol%-yttria-stabilized zirconia. J. Am. Ceram. Soc. (2000) v. 83 p. 1199-1206

35. Käll, M.; Zimmermann, M. von; Andersen, N.H.; Madsen, J.; Frello, T.; *Poulsen, H.F.;* Schneider, J.R.; Wolf, T., Anisotropic dynamical scaling in a weakly 3D system: The case of oxygen ordering in $YBa_2Cu_3O_{6.5}$. Europhys. Lett. (2000) v. 51 p. 447-453

36. *Lauridsen, E.M.; Juul Jensen, D.; Poulsen, H.F.;* Lienert, U., Kinetics of individual grains during recrystallization. Scr. Mater. (2000) v. 43 p. 561-566

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39. Lindhagen, J.E.; *Gamstedt, E.K.;* Berglund, L.A., Application of bridging-law concepts to short-fibre composites. Part 3: Bridging law derivation from experimental crack profiles. Compos. Sci. Technol. (2000) v. 60 p. 2883-2894

40. *Liu, Q.; Wert, J.; Hansen, N.*, Location-dependent lattice rotation and shear strain in rolled aluminium single crystals of cube and Goss orientations. Acta Mater. (2000) v. 48 p. 4267-4279

41. *Liu, Y.L.;* Wang, W.G.; *Kindl, B.*, (Bi,Pb) $2Sr_2Ca_2Cu_3O_x$ phase formation in a silver-sheathed multifilament tape. *Physica C* (2000) v. 341-348 p. 2579-2580

42. *Lybye, D.; Poulsen, F.W.; Mogensen, M.*, Conductivity of A- and B-site doped $LaAlO_3$, $LaGaO_3$, $LaScO_3$ and $LaInO_3$ perovskites. Solid State Ionics (2000) v. 128 p. 91-103

43. *Mishin, O.V.;* Bay, B.; *Juul Jensen, D.*, Through-thickness texture gradients in cold-rolled aluminum. Metall. Mater. Trans. A (2000) v. 31 p. 1653-1662

44. *Mishin, O.V.; Lauridsen, E.M.; Krieger Lassen, N.C.;* Brückner, G.; Tschentscher, T.; Bay, B.; *Juul Jensen, D.; Poulsen, H.F.*, Application of high-energy synchrotron radiation for texture studies. J. Appl. Cryst. (2000) v. 33 p. 364-371

45. *Mogensen, M.; Lybye, D.; Bonanos, N.; Hendriksen, P.V.*, Oxide ion transport in solid oxide fuel cells. In: Mass and charge transport in inorganic materials. Fundamentals to devices. Part B. Vincenzini, P.; Buscaglia, V. (eds.), (Techna, Faenza, 2000) (Advances in Science and Technology, 29) p. 1261-1272

46. *Mogensen, M.; Primdahl, S.; Juhl Jørgensen, M.; Bagger, C.*, Composite electrodes in solid oxide fuel cells and similar solid state devices. J. Electroceram. (2000) v. 5 p. 141-152

47. *Mogensen, M.;* Sammes, N.M.; Tompsett, G.A., Physical, chemical and electrochemical properties of pure and doped ceria. Solid State Ionics (2000) v. 129 p. 63-94

48. Nagai, Y.; Kakimoto, M.; Hyodo, T.; Fujiwara, K.; Ikari, H.; *Eldrup, M.;* Stewart, A.T., Temperature dependence of the momentum distribution of positronium in MgF_2, SiO_2, and H_2O. Phys. Rev. B (2000) v. 62 p. 5531-5535

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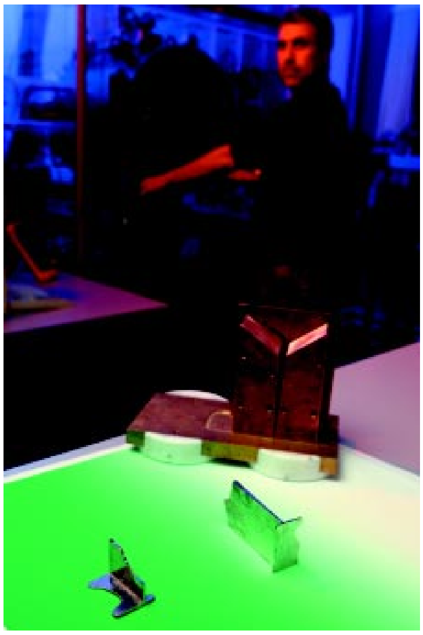
50. Nielsen, S.A.; *Toftegaard, H.*, Ultrasonic measurement of elastic constants in fiber-reinforced polymer composites under influence of absorbed moisture. Ultrasonics (2000) v. 38 p. 242-246

51. *Nielsen, S.F.;* Wolf, A.; *Poulsen, H.F.;* Ohler, M.; Lienert, U.; Owen, R.A., A conical slit for three-dimensional XRD mapping. J. Synchrotron Radiat. (2000) v. 7 p. 103-109



52. Osetsky, Y.N.; Bacon, D.J.; Serra, A.; *Singh, B.N.;* Golubov, S.I., Stability and mobility of defect clusters and dislocation loops in metals. J. Nucl. Mater. (2000) v. 276 p. 65-77

53. Osetsky, Y.N.; Bacon, D.J.; Gao, F.; Serra, A.; *Singh, B.N.*, Study of loop-loop and loop-edge dislocation interactions in bcc iron. J. Nucl. Mater. (2000) v. 283-287 p. 784-788

54. Osetsky, Y.N.; Serra, A.; *Singh, B.N.;* Golubov, S.I., Structure and properties of clusters of self-interstitial atoms in fcc copper and bcc iron. Phil. Mag. A (2000) v. 80 p. 2131-2157

55. *Pantleon, W.;* Stoyan, D., Correlations between disorientations in neighbouring dislocation boundaries (Erratum Acta Mater. 2000, v. 48, p. 4179). Acta Mater. (2000) v. 48 p. 3005-3014

56. *Pedersen, O.B.; Carstensen, J.V.*, Internal stresses and dislocation dynamics in cyclic plasticity and fatigue of metals. Mater. Sci. Eng. A (2000) v. 285 p. 253-264

57. *Poulsen, F.W.*, Defect chemistry modelling of oxygen-stoichiometry, vacancy concentrations, and conductivity of $(La_{1-x}Sr_x)_yMnO_{3((}$. Solid State Ionics (2 000) v. 129 p. 145-162

58. *Poulsen, F.W.; Glerup, M.; Holtappels, P.*, Structure, Raman spectra and defect chemistry modelling of conductive pyrochlore oxides. Solid State Ionics (2000) v. 135 p. 595-602

59. *Primdahl, S.; Mogensen, M.*, Durability and thermal cycling of Ni/YSZ cermet anodes for solid oxide fuel cells. J. Appl. Electrochem. (2000) v. 30 p. 247-257

60. *Primdahl, S.; Sørensen, B.F.; Mogensen, M.*, Effect of nickel oxide/yttria-stabilized zirconia anode precursor sintering temperature on the properties of solid oxide fuel cells. J. Am. Ceram. Soc. (2000) v. 83 p. 489-494

61. *Pryds, N.H.; Eldrup, M.;* Ohnuma, M.; *Schrøder Pedersen, A.;* Hattel, J.; *Linderoth, S.*, Preparation and properties of Mg-Cu-Y-Al bulk amorphous alloys. Mater. Trans. JIM (2000) v. 41 p. 1435-1442

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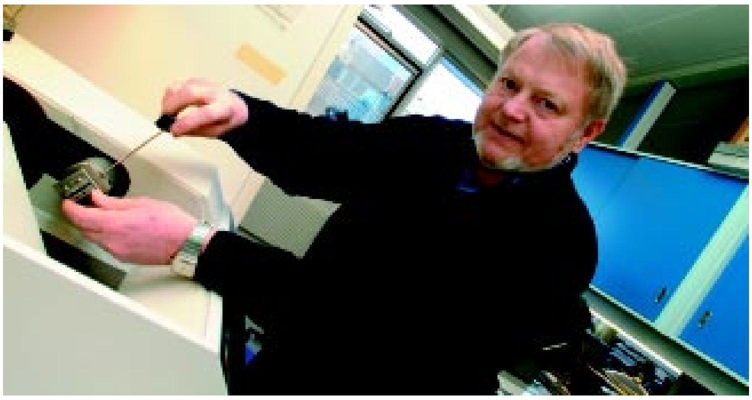

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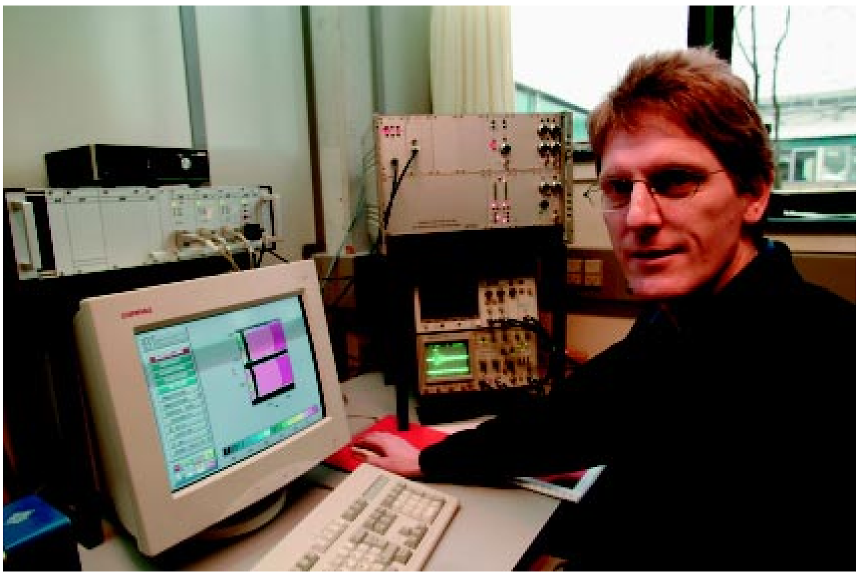

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Abbreviations

BET	method named after Brunauer, Emmett and Teller
CCM	channelling contrast method
CLEA	Centre for Design of Adhesively Bonded Aluminium Components
DANAK	Danish Accreditation
dpa	displacements per atom
DR3	Danish Reactor 3 (at Risø)
DTA	differential thermal analysis
DTU	Technical University of Denmark, Lyngby
DTI	Danish Technological Institute
EBSD	electron back-scattering diffraction
EDL	edge dipole loop
EDS	energy dispersive (X-ray) spectrometry
EELS	electron energy loss spectrometry
ESEM	environmental scanning electron microscopy
ESRF	European Synchrotron Radiation Facility, Grenoble
EXAFS	Extended X-ray absorption fine structure
HIP	hot isostatic pressing
ITER	International thermo-nuclear experimental reactor
KU	University of Copenhagen
LSM	lanthanum strontium manganate
LTH	Lund Institute of Technology
LTU	Luleå University of Technology
LVEM	low vacuum scanning electron microscopy
PAS	positron annihilation spectroscopy
PBK	Plant Biology and Biogeochemistry Department at Risø
PBM	production bias model
SDU	University of Southern Denmark
SEM	scanning electron microscopy
SIA	self-interstitial atoms
SOFC	solid oxide fuel cells
TEC	thermal expansion coefficient
TEM	transmission electron microscopy
TGA	thermogravimetric analysis
USD	University of Southern Denmark
YSZ	yttria stabilised zirconia
1D	one dimensional
3D	three dimensional
3DXRD	three dimensional X-ray diffraction