Technical University of Denmark

Materials Research Department annual report 1998

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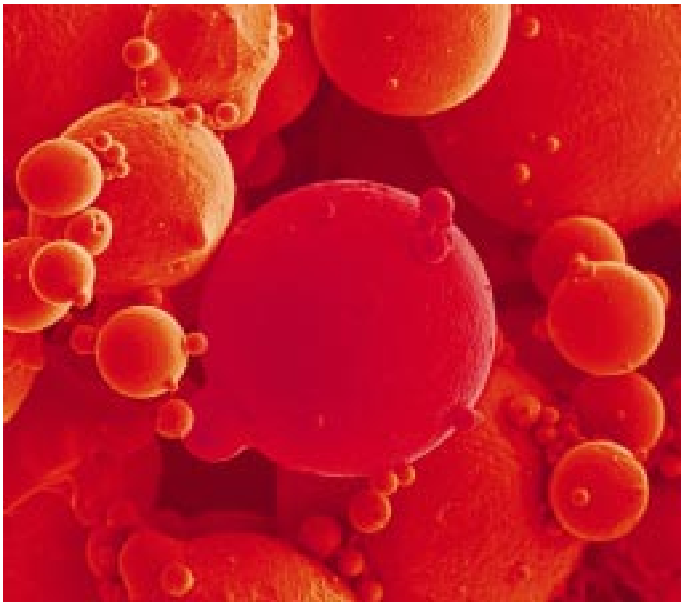
Publication date:
1999

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

Materials Research Department Annual Report 1998

RISØ

Published by
Materials Research Department
Risø National Laboratory
April 1999
Risø-R-1098(EN)



Materials Research Department

Annual Report 1998

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ABSTRACT

Selected activities of the Materials Research Department at Risø National Laboratory during 1998 are described. The scientific work is presented in five chapters: Materials Science, Materials Engineering, Materials Technology, Materials Chemistry and Fusion Materials. A survey is given of the Department's collaboration with national and international industries and research institutions. Furthermore, the main figures outlining the funding and expenditure of the Department are given. Lists of staff members, visiting scientists and educational activities are included.

Risø-R-1098(EN)

Published by the Materials Research Department, Risø National Laboratory April 1999

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Cover photo: Microstructure of spray-deposited metal and reaction products on ceramics.

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ISBN 87-550-2506-4; ISBN 87-550-2507-2 (internet)
ISSN 0106-2840
ISSN 1397-8071

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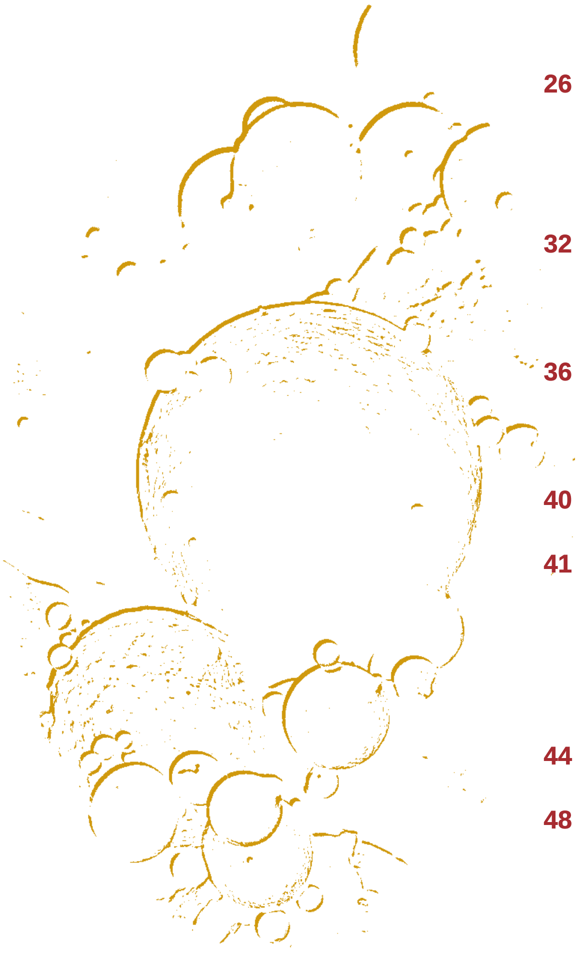


INTRODUCTION
– Materials Research Department 1998

The work in the Materials Research Department has as its primary objective to understand materials behaviour in order to improve the performance of products.

1998 has been a successful year thanks to many new developments, where several goals have been reached in a fruitful collaboration with many groups outside the Department, at Risø, in Denmark, and internationally.

Achievements

- **New techniques.** A 3D X-ray microscope has been developed and is being commissioned at ESRF, in France. This instrument is unique in the world, as it can give information from very small volumes in the bulk of a material. We foresee that, in collaboration with ESRF and other groups, we can reach new understanding in many different fields. Initially, the Department will concentrate on plastic deformation and recrystallisation of metals and alloys and solid state reactions in solid oxide fuel cells and high temperature superconductors in collaboration with Danish industry.

- **New technology.** Fabrication of bulk amorphous samples in large dimensions has been achieved in collaboration with the National Research Institute for Metals, Tsukuba, Japan. The chill cast alloys have volumes in the cubic centimetre range, allowing their suitability for new applications to be investigated.

  Manufacturing of functionally graded ceramics, i.e. ceramics with a continuous change in composition, has led to several applications, e.g. in oxygen sensors and noise filters. These have been developed in collaboration with Danish industry within the Danish Materials Technology Programme (MUP 2).

  Introduction of radically new technologies in the solid-oxide fuel cells programme has led to very significant improvement in performance and to a large reduction in manufacturing costs. The development of a cell having an internal resistance of 0.4 $\Omega\cdot cm^2$ at 850 °C brings us back into the world league.

- **Advanced composites.** Development of a Risø patented flywheel for energy storage (under the sponsorship of the Danish Ministry of Environment and Energy). A wheel made of a glass-reinforced polymer reached in December a speed of 18,000 rotations per minute. Next step will be a doubling of this speed which will increase the storage capacity of the wheel by four times.

- **Strong materials.** The dynamic properties of tool steel have been analysed for STRECON, a company in the Danfoss group. These steels are

Henning Friis Poulsen (right) receives the 'Hede-Nielsen-Prize' from Director Leif Hede Nielsen(left).

Prizes and other special recognitions

In 1998 a number of private foundations have awarded prizes or other donations to members of the Department in recognition of their work:

'Civilingeniør Frederik Leth Christiansens Almennyttige Fond' has supported *Aage Lystrup* in acquiring a 'Computer controlled XYZ-milling machine for cutting test specimens in fibre composites' (January).

The 'Tagea Brandts Rejselegat' (Tagea Brandt's Grant) to *Dorte Juul Jensen* as 'An outstanding female scientist' (17 March).

The 'Hede-Nielsen-Prize' to *Henning Friis Poulsen* for his work on 'Synchrotron instrumentation for hard X-rays and application of this technique to optimisation of high-temperature superconducting tapes' (28 May).

The 'Patentprisen' (Patent Prize) to *Carsten Bagger, Bruno Kindl and Mogens Mogensen* for their patent 'Solid Oxide Fuel Cell with cathode of LSM and YSZ' (20 August).

'Director, dr. techn. A.N. Neergaard's and Wife's Foundation' has supported *Torben Lorentzen*'s work on a textbook: 'Introduction to characterisation of residual stress by neutron diffraction' (13 September).

used for cold forging and also for manufacturing diamonds under very high pressure. The research has led to a tenfold increase in the lifetime of commercial tools.

- **Recyclable materials.** The development of new thermoplastic composites, e.g. for wind turbine blades and automobile body parts, reduces the usage of materials posing an occupational health hazard and enables these products to be recycled.
  With the specific aim of improving recycling of products in the automotive and packaging industries, composites have been produced where glass fibres have been replaced by natural plant fibres.

- **Patents.** Three new patents have been filed in 1998. Two in the fuel cells area (one of which is together with the company Haldor Topsøe) and one in the area of wind turbine blades (together with the Wind Energy and Atmospheric Physics Department, Risø).

- **Symposia.** The 19th Risø International Symposium on Materials Science with the title *Modelling of Structures and Mechanics of Materials from Microscale to Products* and a Workshop on *Damage Production and Accumulation under Cascade Damage Conditions* have been arranged at Risø.

Collaboration with the Department of Physics at the Technical University of Denmark (DTU) was formalised in 1998. Staff from the Department now has an office at DTU where these paintings are to be placed. DTU staff also has an office in the Department.

Challenges

Today the Department faces a number of challenges. Some of these are expansions of what we already do, while others require a new approach. In the former category we and our partners in the Danish solid oxide fuel cells programme have to bring the research from the laboratory into the development phase, leading to application within a period of 5 to 10 years. This task requires international collaboration, which is foreseen both bilaterally and multilaterally within EU's 5th Framework Programme. This programme is in itself another challenge because it requires highly focussed research and strong industrial involvement.

A new challenge is the political demand in Denmark that Risø's research is not only relevant, but also that it demonstrates its relevance by attracting customers who are prepared to pay. We are confident that the long experience the Department has gained in working with industry has prepared us well to meet this challenge. In 1998 we identified areas where we believe there will be a market and are at present analysing the market potential in the following areas: Design, manufacturing and testing of composite materials/components, high temperature materials (including ceramics), and finally advanced characterisation and modelling of materials.

Another challenge also has its origin in a political demand, namely that the education in natural and technical sciences is strengthened, especially at the technical universities. The goal, namely to produce more and better qualified graduates, requires new initiatives, such as involving staff from government research institutes in teaching at the universities. We are already prepared for this through our different educational activities, which range from teaching high school students and high school teachers to external teaching of courses at the universities. In addition, a much stronger involvement is foreseen through a recent agreement with the Department of Physics at the Technical University of Denmark for joint research in fields of common interest, including atomic scale modelling, nanostructural and amorphous materials and electron microscopy. This collaboration, which also involves teaching, is expected to increase from the present level of four senior scientists. This arrangement is a pioneering activity which may be followed up with similar arrangements with other departments at the Technical University of Denmark and with other Danish universities.

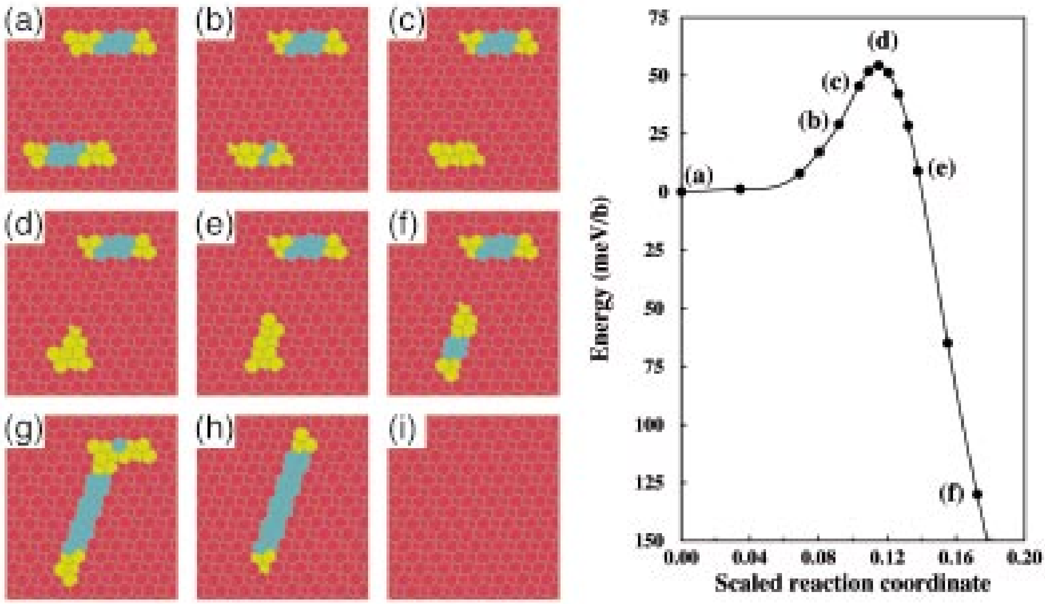
The many initiatives and the new activities require a qualified and motivated staff and good scientific equipment. We have that at present, but we are 'selling the family silver' collected over a long period. Long range research is becoming difficult to finance, the time allowed for young scientists to get a proper scientific training is becoming insufficient and key equipment needs replacement. However, the good results in 1998 nourish our optimism and it is our hope that our activities, especially our collaboration with the Danish universities in the coming years, will be so useful to society that the 'family silver' can be replenished.

MATERIALS SCIENCE
- theory and characterisation

This section covers the research activities within two programmes: *Materials Models and Materials Structures* and *Local Structure and Properties*. The focus is on the basic understanding of materials properties (predominantly the 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 two programmes finds applications in other programmes aiming more directly at technological applications.
Among the scientific highlights one may point to
(i) three-dimensional atomic-scale modelling of the annihilation of screw dislocations of opposite sign,
(ii) experimental confirmation of theoretical expectations concerning grain boundary activity during diffusional creep,
(iii) studies of anisotropic dislocation dynamics in cyclic plasticity of copper at temperatures above 200 K,
(iv) successful modelling of low-energy dislocation boundary stability, subdivision of single crystals and yield stress anisotropy,
(v) development and assembly of a 3D X-ray microscope at the European Synchrotron Radiation Facility (ESRF) in France.

Project Funded Research: Materials Science

Project type	Project name	Co-participants
Danish Technical Research Council, 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)	• 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
Danish Materials Technology Programme (MUP)	Materials Processing, Properties and Modelling Centre (MPPM)	• Dept. of Appl. Eng. Design and Production, DTU, Denmark • Department of Solid Mechanics, DTU, Denmark • Department of Production, AAU, Denmark
Large Installation Programme (LIP)	Neutron Diffraction	• Condensed Matter Physics and Chemistry Department Risø • DR3, Risø
BRITE-EURAM	Improvement of Quality and Productivity for Rolled and Extruded Aluminium Products through Microstructure and Texture Modelling (REAP)	• Hydro Aluminium a.s., Norway • Pechiney Recherche, France • Gränges AB, Sweden • Norwegian Univ. of Science and Technology, NTNU, Norway • Ecole des Mines de Saint-Etienne, France • Swedish Institute for Metal Research, Sweden • Norwegian Institute of Technology, SINTEF, Norway
BRITE-EURAM	Residual Stress Standard using Neutron Diffraction (RESTAND)	• Rolls Royce - Gas Turbines, UK • Sintech Keramik, Germany • Schunk Kohlenstofftechnik, Germany • Volkswagen GmbH, Germany • British Aerospace - Airbus, UK • AEA Technology, Harwell, UK • Rutherford Appleton Laboratory, UK • Hahn-Meitner Institute, Germany • Institute Laue Langevin, France • NFL (Studsvik), Sweden • Joint Research Centre Petten, The Netherlands • University of Cambridge, UK • Imperial College London, UK • University of Salford, UK



Left: Selected configurations from atomistic simulation of screw dipole annihilation. Atoms in the perfect atomic lattice are coloured red, while yellow and blue atoms form Schockly partial dislocations and stacking faults, respectively.
Right: The energy as a function of a scaled reaction coordinate with 0 corresponding to the initial state (a) and 1 to the final perfect crystal (i). The transition state is seen in (d).

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

In 1998 the 19th Risø International Symposium on Materials Science with the title 'Modelling of Structure and Mechanics of Materials from Microscale to Product' was organised. The symposium successfully fulfilled its aim, namely to demonstrate the relations between models at different length scales and the trend to combine different length scales in the models.

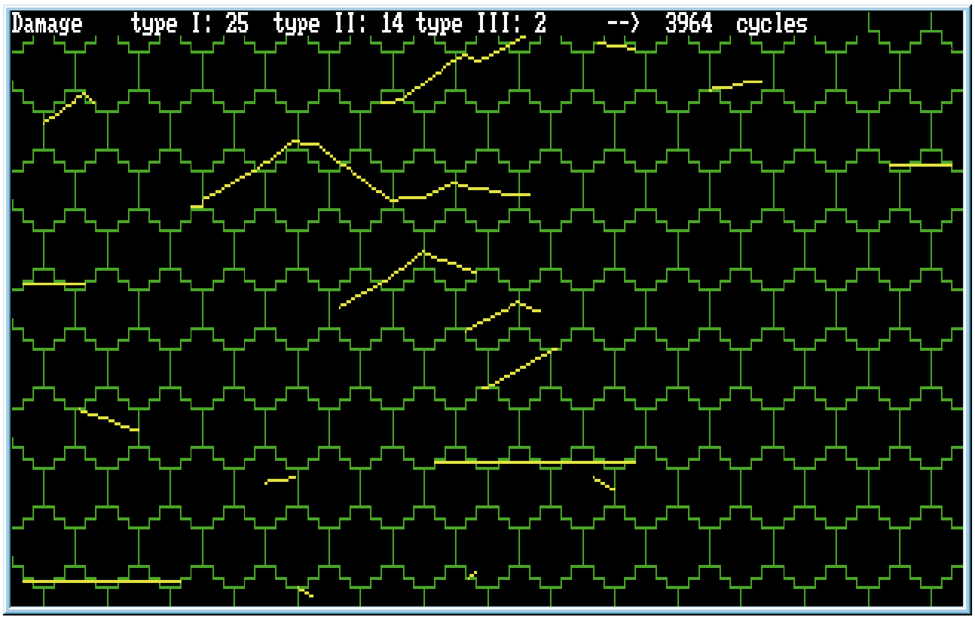
The Engineering Science Centre for Structural Characterisation and Modelling of Materials, financed jointly by the Danish Technical Research Council and Risø, is managed within the programme. The centre consists of a core and a shell. So far, the core has mainly been positioned within this programme whereas the shell has covered a wider range of department activities. The centre is now in its second and last 5-year period, during which the activities should be 'anchored' so that they can continue after the last centre period. In this connection some of the core activities are being transferred to other programmes, but the centre of gravity of the core is still within this programme.

In 1998 a formal agreement was reached on collaboration with the Departments of Physics and Process Technology at DTU within atomic scale modelling, electron microscopy and nanostructured materials. Furthermore, the programme has been associated with the newly established 'Academic Frontier Research Center for Ultra-High-Speed Deformation' in Hiroshima together with the programme *Irradiation damage, defects and fusion materials*.

Atomic scale modelling of the annihilation of screw dislocations

The dislocation density of deformation-induced microstructures is ultimately limited by annihilation of dislocations. The annihilation may occur either by climb at high homologous temperatures, where the rate of self-diffusion is high, or by cross slip at lower temperatures. The annihilation of screw dislocation dipoles by cross slip is especially important for low temperature cyclic deformation, where dislocations are generated continuously over cumulative plastic shears far exceeding those of monotonic plastic deformation.

The minimum stable height of screw dipoles (annihilation height) is a key parameter in modelling of plasticity. Experimentally, the minimum height of screw dipoles observed by electron microscopy in cyclically saturated copper provides an upper limit of about 50 nm to the annihilation length at room temperature. It is generally believed that cross slip is a thermally activated process and that the activation energy for annihilation decreases with decreasing dipole height, although the experimental values for the minimum stable a



screw dipole heights are rather ambiguous.

The structure and annihilation of screw dislocation dipoles were studied by atomistic computer simulations in collaboration with the Department of Physics at DTU. The approach was to start with the smallest possible dipole height and then increase the dipole height as much as necessary and possible within the computational limitations. A standard energy minimisation procedure showed that screw dislocations of opposite sign separated by up to 4 close packed planes annihilate spontaneously, while higher screw dipoles appear to be stable. In the regime of stable dipoles, the annihilation mechanism was studied using the 'nudged elastic band' (NEB) method of finding the minimum energy transition paths through configuration space.

The activation energy of annihilation was determined as a function of dipole heights up to 5.2 nm and found to be linear in inverse dipole height for heights larger than 1.2 nm. The activation energy for screw dipole annihilation may be seen as the barrier against cross slip of a screw dislocation in the presence of an opposite screw dislocation. The results suggest that the activation energy for cross slip is modified proportionally to the stress from that dislocation.

Dislocation dynamics, microstructural evolution and the link to fatigue

The dislocation microstructures produced by cyclic plasticity in metals appear to result from cross slip of mobile screw dislocations and trapping of edge dislocations into dipoles. The dislocations are organised in arrays that form metastable hard regions in the relatively soft metal. The length scale of the microstructures displays a strong temperature dependence, which seems to be related in a simple way to the annihilation of cross slipping screw dislocations. The metastability of the microstructures leads to continued formation of slip bands, which are sites of surface microcracking in fatigue.

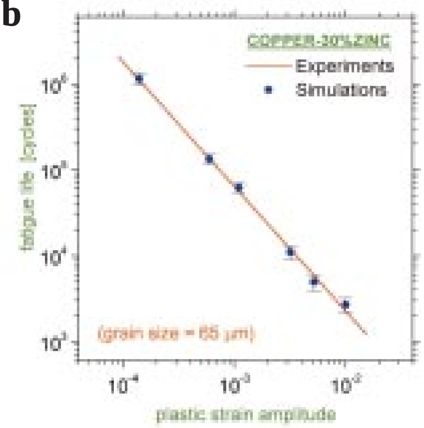
This general picture was critically examined in the light of quantitative information on the microstructures and the sizes and shapes of macroscopic hysteresis loops for single crystals and polycrystals of Cu and polycrystals of Cu-Zn. For temperatures below 200 K, the size of hysteresis loops could be linked quantitatively to the microstructure of slip bands in terms of two contributions: a static contribution from the regions of crystal hardened by edge dipole arrays and a dynamic contribution from the remaining regions hardened by mobile screw dislocations. The dynamic contribution reflects the maximum dislocation density set by the screw dipole annihilation height and simply corresponds to passage of screw dislocations on glide planes separated by this height.

At higher temperatures, the dynamic contribution exceeds this value. The excess dynamic term could be represented in terms of a reduced effective spacing of the hard edge dipole walls. In dislocation terms, the excess dynamics possibly reflect dislocation intersection processes. Subsequent analysis of the shapes of hysteresis loops for the single crystals revealed that the excess dynamic contribution is anisotropic with respect to forward and reverse stressing.

The additional complicating effects of polycrystallinity and chemical composition on hysteresis loop shapes were examined for polycrystalline Cu and Cu-Zn. The results fit into a multiscale account of fatigue, in which modelling at the atomic scale delivers input to microstructurally related models and damage models at coarser length scales and correspondingly higher levels of complexity.

Characterisation and modelling of damage evolution and fatigue life

Slip bands form continuously during fatigue of industrial materials as an aspect of microstructural evolution. The slip bands lead to both transgranular and intergranular microcracking, but the b



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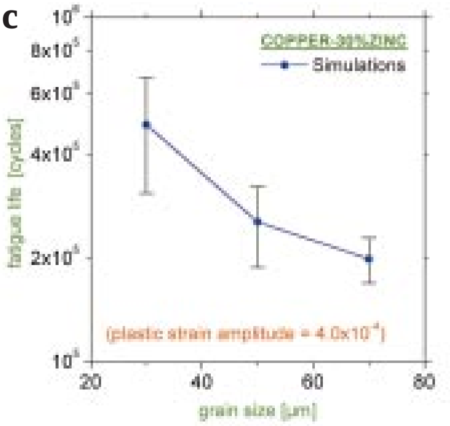


a) Simulated surface damage on Cu-30%Zn fatigued at constant plastic strain amplitudes.
b) Simulated fatigue life curve for Cu-30%Zn.
c) The simulation predicts a grain size effect on the fatigue life.
All standard deviations correspond to 30 simulations.

details in the transition from slip bands to microcracks are still not well understood. On the other hand, it appears that the fracture mechanics approach to fatigue, based on a single crack growth relation, is insufficient for a realistic account of fatigue life.

Damage characterisation shows that the material spends by far the longest part of its fatigue life in a complex damage process of initiation, growth and coalescence of microcracks culminating in the formation of a fatal fatigue crack. A numerical approach based on statistical physics was therefore adopted to simulate damage evolution and fatigue life. The approach uses random crack initiation based on a Monte Carlo approach, and it invokes a number of physical damage parameters to account for the growth and interaction of microcracks. The work was done in collaboration with Ecole des Mines de St-Etienne, France, where the approach originated.

In practice, the damage simulation features a simplified grain structure. The grains consist of 36 unit cells forming a quasi-hexagon. During random crack initiation, each unit cell is continuously monitored for initiation, propagation and coalescence of microcracks. The growing microcracks are classified as type I, II and III according to their surface length and growth behaviour, and experimentally determined crack growth relations are used to correlate the simulated crack growth with an equivalent increment in the number of cycles. The simulation continues until the longest crack exceeds the 'fatal' crack length of ~12 grain sizes.

Damage characterisation showed that intergranular crack growth is the dominant crack growth mode in Cu-30%Zn, which was studied experimentally and simulated as a model system for single-phase planar slip materials, such as the austenitic stainless steels. Qualitatively, this result matches the relatively high intergranular stresses associated with the dominant single slip behaviour, which is observed even at plastic strain amplitudes as high as $5 \cdot 10^{-3}$. The simulated damage evolution and fatigue endurance for Cu-30%Zn are in excellent agreement with experiment.

Cyclic softening and microstructures in high nitrogen duplex steels

Austenitic-ferritic duplex stainless steels are important industrial materials consisting of wavy slip ferritic phase and planar slip austenitic phase. They have specific chemical compositions, crystallographic textures, phase morphology and dislocation microstructures, induced by thermomechanical processing and subsequent plastic deformation. Recent progress in the development of high nitrogen steels suggests that the fatigue performance of duplex steels can be improved substantially by nitrogen alloying, which causes preferential hardening of the austenitic phase.

Cyclic stress-strain curves published for duplex steels with nitrogen contents between 0.07 and 0.18 weight % were found to display significant scatter, which could not be attributed to the nitrogen content alone. The approximate level of scatter resulting from uncontrolled texture is well known from related studies of experimental model systems. It was concluded that uncontrolled textures alone could account for the magnitude of the scatter, but effects of differences in the contents of substitutional alloying elements and strain history also contribute.

An investigation of the plastic strain controlled fatigue of a duplex steel with 0.35 weight % nitrogen has shown that, although the austenitic phase is harder than the ferritic phase in this steel, the sites of fatigue damage at low plastic strain amplitudes are within the austenitic phase. Thus, the idea that the initially softer ferritic phase should be the site of cracking in low amplitude fatigue of high nitrogen steels cannot be defended. The possibility of including grain size, duplex phase morphology and nitrogen content as parameters in simulation of damage evolution and fatigue life was examined.

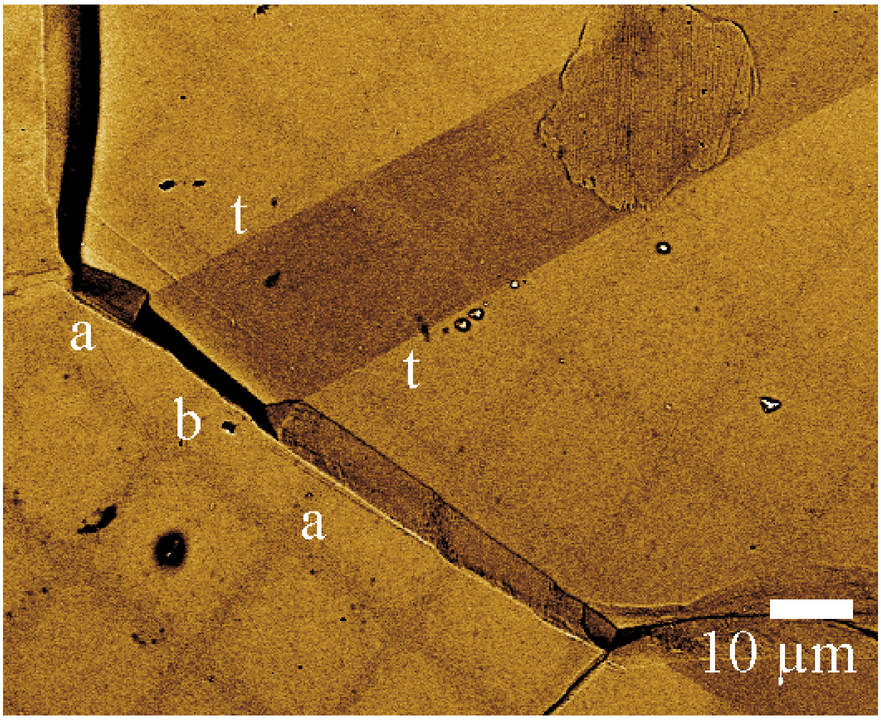
The steel displays pronounced cyclic softening of the austenitic phase. The ferritic phase must therefore carry a disproportionately large part of the plastic strain amplitude in the initial stage of fatigue. Electron microscopy confirmed this idea by revealing a predominance of misoriented cell structure at low amplitudes, where other structures would be expected in a single-phase ferritic steel. Neutron diffraction texture measurements showed that the ferritic phase is virtually texture-free. Thus the predominance of misoriented cells cannot be attributed to a preferred multislip orientation of the ferritic grains.

Experimental confirmation of grain boundary activity criterion during diffusional creep

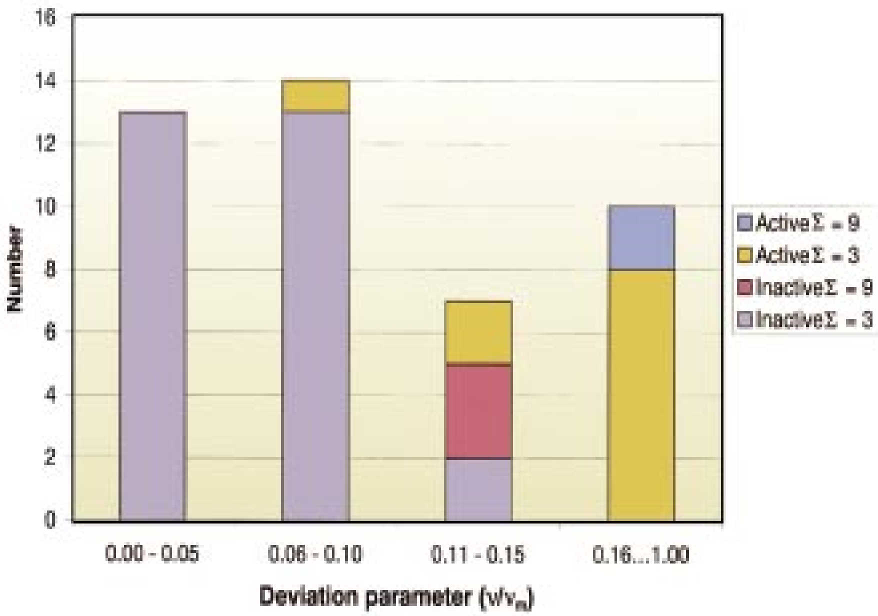
Diffusional creep is a deformation mode in which the deformation occurs by diffusion of material from grain boundaries in compression to grain boundaries in tension. The ability of a grain boundary to act as a source or sink for vacancies during diffusional creep depends on its structure.

An experimental study has been performed on Cu-2wt% Ni deformed in tension in the diffusional creep regime. A periodic fiducial grid was deposited on the specimens prior to creep, so that the local deposition (or removal) of material and the sliding at the individual boundaries could be measured. The misorientation across the boundaries was determined by electron back scattering patterns.

A number of special boundaries that can be described within the framework of the coincident site lattice (CSL) model have been examined. There is good reason to assume that vacancies can be absorbed or emitted only at grain boundary dislocations in these boundaries. It was therefore expected theoretically that only boundaries deviating more than a certain amount from the exact CSL misorientation will contain a suffi-



Scanning electron micrograph of Cu-2wt%Ni deformed in diffusional creep. The boundary segment 'b' deforms differently from the boundary segments 'a' because the misorientation across the boundary is changed where it is met by the twin boundaries 't'.



Histogram showing the number of active and inactive boundaries as a function of the deviation from an exact coincident site lattice misorientation. Inactive boundaries have small deviations.

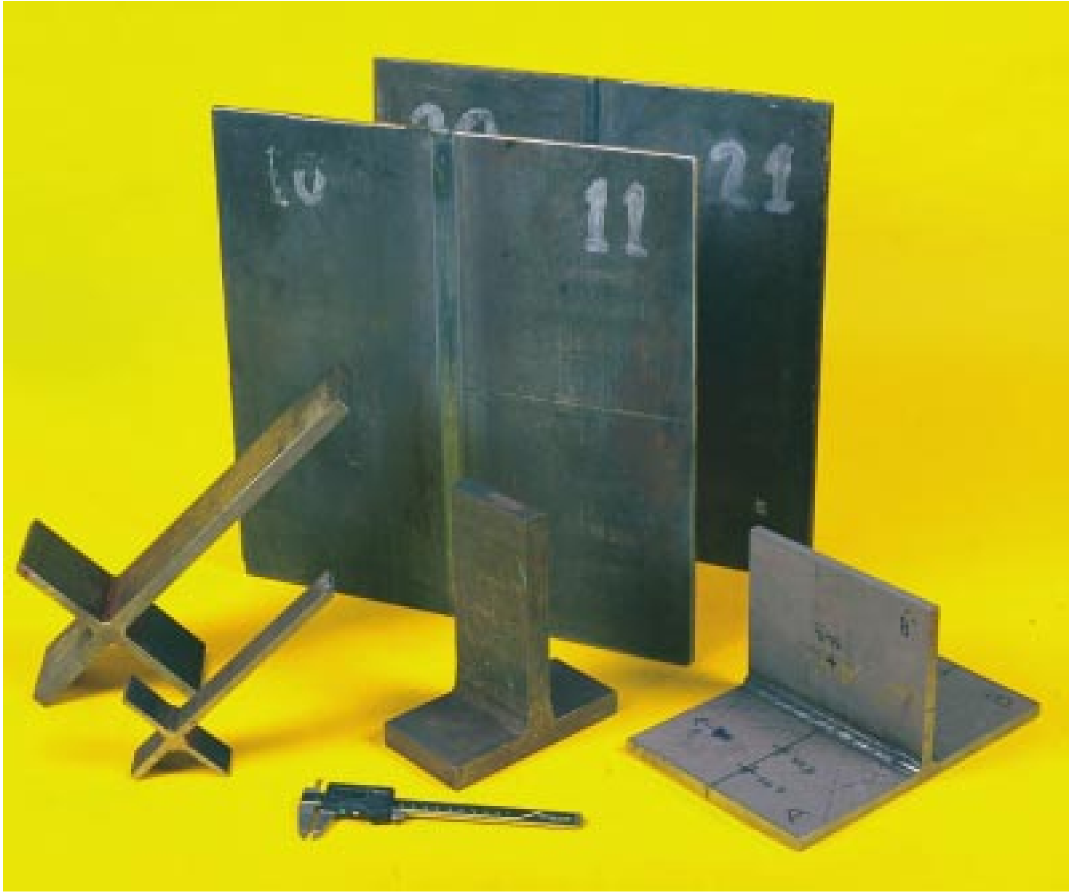
cient number of grain boundary dislocations to be efficient vacancy absorbers or emitters. The deviation was described by the ratio ν/ν_m, where $\nu_m = 15° \cdot \Sigma^{1/2}$ is the maximum deviation that can be accommodated by secondary grain boundary dislocations and ν is the measured deviation. Σ refers to the boundary geometry in the CSL model. For $\Sigma = 3$ and $\Sigma = 9$ boundaries, it was found that all inactive boundaries had values of ν/ν_m smaller than 0.15 and all active boundaries had values of ν/ν_m larger than 0.09. It was thus confirmed that all inactive boundaries have a misorientation close to the exact CSL misorientation.

Welding stresses: Modelling and experimental validation

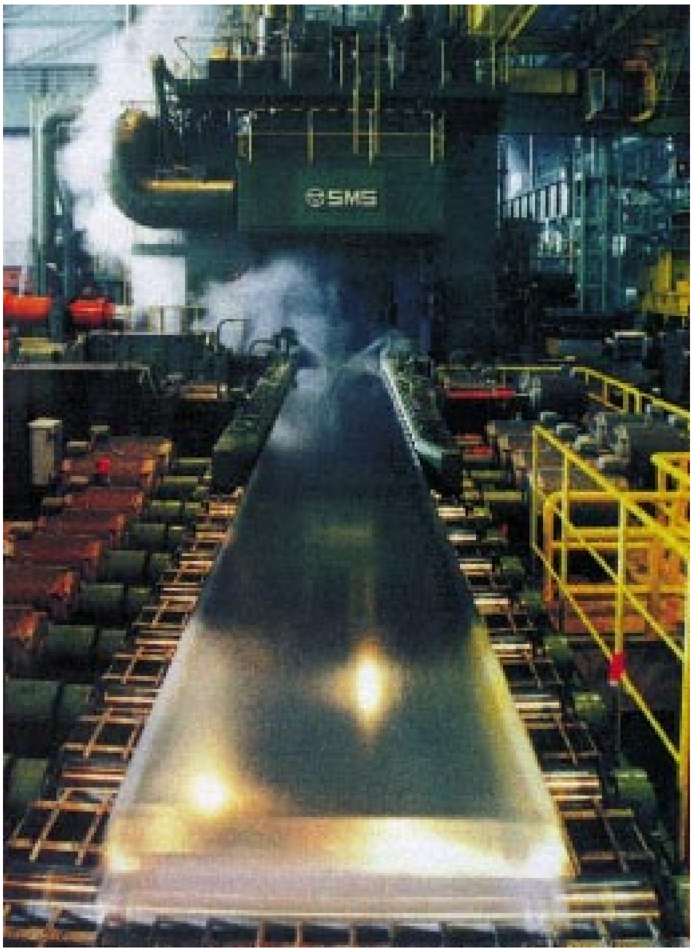
Residual stresses induced by welding processes have long been of concern to engineers. The potential detrimental consequences of welding stresses are well recognised, and often lead to conservative design, involving large safety factors and conservative life time estimates.

Few techniques are capable of non-destructive assessment of the size and distribution of these stresses, and theoretical predictions are non-trivial. The most promising approach to obtain numerical predictions of welding stresses is the Finite Element Method (FEM), where the solidification of the weld metal can be simulated by a so-called 'moving heat-source' method. By a consecutive solution of the heat transfer problem and the subsequent mechanical problem in terms of strains and stresses, it is today possible to estimate welding induced stresses. The numerical technique is still, however, in its infancy, and experimental validation of the numerical predictions is necessary to refine and optimise the calculation procedure.

In collaboration with the Department of Applied Engineering Design and Production at DTU, several test weldings have been modelled and experimentally investigated by neutron diffraction at Risø. The results were very promising and the described numerical procedure rendered an acceptable accuracy of the residual stress predictions. Through this combined numerical and experimental effort, one is rapidly approaching the situation where it will be common practice for industry to calculate welding stresses and thereby arrive at less conservative and more optimised structural design.



Residual stresses were measured by neutron diffraction in welded structures. The measured values agreed well with numerical predictions of welding induced residual stresses.



Industrial rolling mill: Alunorf which is jointly owned by Alcan and VAW.

Local structure and properties

The work in the programme is concentrated on quantitative characterisation of dislocation microstructure and local crystallographic orientations with the aim of understanding thermomechanical processes and mechanical/physical properties. For several years this work has been concerned mainly with characterisation of relatively simple pure or almost pure metals deformed at room temperature in the laboratory. This has led to establishment of a framework describing the microstructural and textural development in these metals. In 1998 the work was expanded along two paths: one being more applied, namely investigations of industrial processes and one being more fundamental, namely development of analytical models for the observed structure evolution. The more applied part of the work has been concentrated on rolling, one of the basic industrial processes in metal forming, and especially on hot rolling of commercial aluminium alloys. Key areas in the modelling have been slip pattern analysis and yield stress anisotropy.

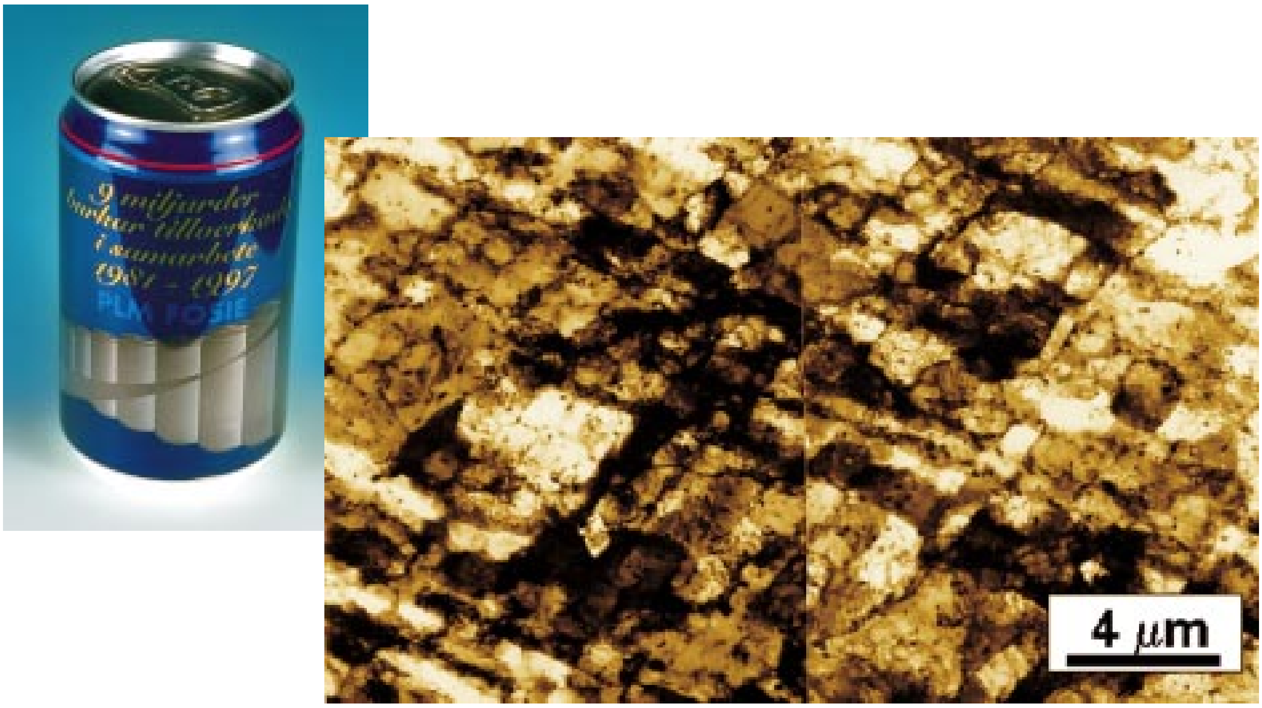
During 1998 the work on high-temperature superconductors focused on characterisation of microstructures and crystallographic orientations, with the aim of improving the physical properties of the superconductors. The present superconductor work thus follows the same approaches as those applied to the more traditional metals work in the programme and the main characterisation techniques are also the same. As a result the high-temperature superconductor project was moved from being a separate programme to being part of the present programme.

A necessity for the work in the programme and the Department is the use of, and maybe even more important, the development of advanced characterisation techniques. In 1998 most of the technique development effort was devoted to the so-called 3D X-ray microscope which is a high energy synchrotron radiation instrument being developed in collaboration with European Synchrotron Radiation Facility (ESRF) in France. This instrument will give new possibilities for *in-situ* characterisation in small selected volumes in the bulk of a sample during deformation or annealing.

Hot rolling textures and microstructures

While deformation microstructures at room temperature have been widely investigated, microstructures forming in materials deformed at elevated temperatures have been much less examined. Characterisation of hot deformation microstructures was carried out in collaboration with industrial partners in an EU-sponsored project to improve the quality of rolled and extruded aluminium products. Specimens from an aluminium alloy, AA3104, used primarily for beverage can applications were deformed at several temperatures by plane strain compression to several strains. Comparison of samples deformed to the same strain at different temperatures revealed several clear trends. For each strain investigated, the average size of the dislocation cells increased and the average misorientation angle between cells decreased slightly with increasing deformation temperature. Similarly, the strength of the texture components that characteristically form during deformation of AA3104 is less pronounced in the specimens deformed at higher temperatures.

Previously, the microstructure of aluminium and its alloys deformed at room temperature has been well characterised with three distinct microstructures observed dependent on the strain. For specimens deformed at high temperatures to low strains, the observed cell structures are similar to those that occur during room temperature defor-



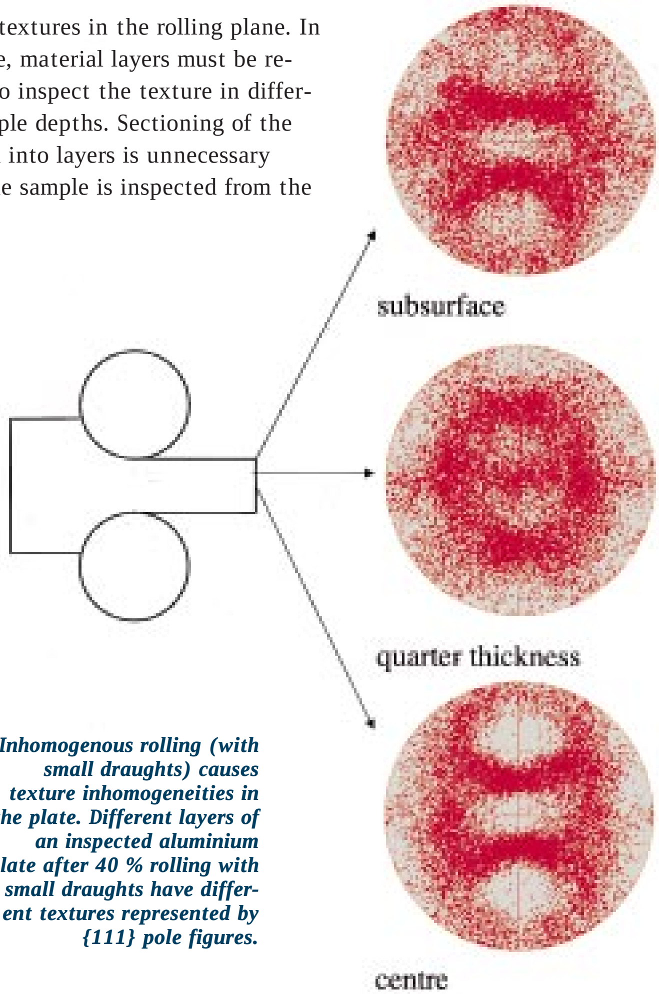
Transmission electron micrograph of an alloy used for beverage cans (AA3104) deformed by plane strain compression to a strain of 0.5 at a temperature of 350 °C. (Can made by Gränges).

mation. Deformation to high strains at elevated temperatures produces a microstructure consisting of equiaxed cells with little long range order. This microstructure is distinctly different from the long, linear boundaries that are observed in aluminium deformed at room temperature. This qualitative cataloguing of the principal microstructural features at a wide variety of strains and deformation temperatures enables a deeper understanding and better predictions of the effect of temperature and strain during deformation on the resulting microstructure.

Through-thickness texture variations in cold-rolled aluminium plates

The properties of rolled products depend on microstructure and crystallographic texture. It is known, however, that both deformation microstructure and texture may vary through the plate thickness. Parameters such as the geometry of the roll gap, friction, rolling sequence and temperature control the development of inhomogeneities in the rolled material.

For texture measurements different experimental techniques were applied. Traditional X-ray diffraction was used to inspect textures in the rolling plane. In this case, material layers must be removed to inspect the texture in different sample depths. Sectioning of the material into layers is unnecessary when the sample is inspected from the



Inhomogenous rolling (with small draughts) causes texture inhomogeneities in the plate. Different layers of an inspected aluminium plate after 40 % rolling with small draughts have different textures represented by {111} pole figures.

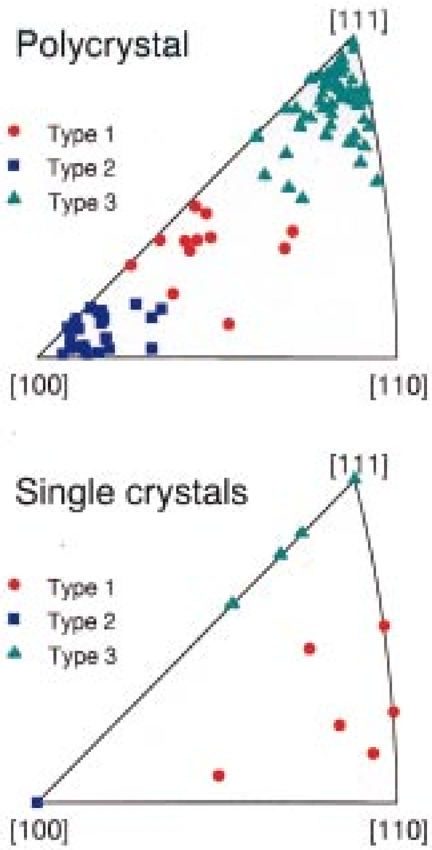
side (in the longitudinal plane) with the electron backscattering pattern (EBSP) technique. This technique measures local orientations and variation in the texture of the different layers can be characterised. However, the EBSP technique requires that the surface of the sample is polished. In synchrotron experiments, the high penetration power allows texture measurements in any selected layer of the test plate and thus eliminates the sectioning or polishing steps. A good qualitative agreement between the techniques was obtained.

Volume fractions of the rolling texture components and the shear texture components in eleven inspected layers were calculated from local orientations in aluminium plates ~ 40 % cold-rolled with different draughts on lubricated rolls. After rolling with small draughts, a pronounced shear texture was observed near the quarter thickness layer, while a typical rolling texture was formed in the centre and near the surface of the plate. Relatively homogeneous rolling textures were produced after rolling with intermediate draughts. Minor variations were consistent with the texture gradients in the initial undeformed sample.

Grain orientation effect on microstructure in tensile deformed copper

Microstructure observations of tensile deformed copper single crystals have shown that the crystal orientation has a strong effect on the deformation microstructure. In order to investigate whether the grain orientation in polycrystals has a similar effect, copper of medium grain size (50 µm) was deformed in tension. The deformation microstructure of individual grains in this material has been studied using transmission electron microscopy (TEM).

In total 92 grains were analysed in samples elongated up to 32 %. An inverse pole figure, i.e. a stereographic triangle with <100>, <110> and <111> at the corners, is usually used to show the tensile axis orientation of individual grains. When relating the microstructure to the orientation of the grains, the grains could be divided into three groups dividing the triangle into three regions. The first region includes grains in the middle of the triangle where perfect planar dislocation boundaries are formed on the {111} slip planes. In the second region in the <100> corner, cylindrical cells are developed. The third region is in the <111> corner where the grains develop planar dislocation boundaries that are less perfect and deviate from the slip planes by angles larger than 5°. Comparison between the present polycrystal observations and the reported single crystal structures showed that grains and single crystals of comparable orientations form the same type of microstructure. This suggests that grain orientation is the key factor controlling microstructure formation in polycrystals by determining the slip pattern and that there is at least some similarity between the slip pattern in single and polycrystals. The results also indicate that grain interaction plays a limited role.



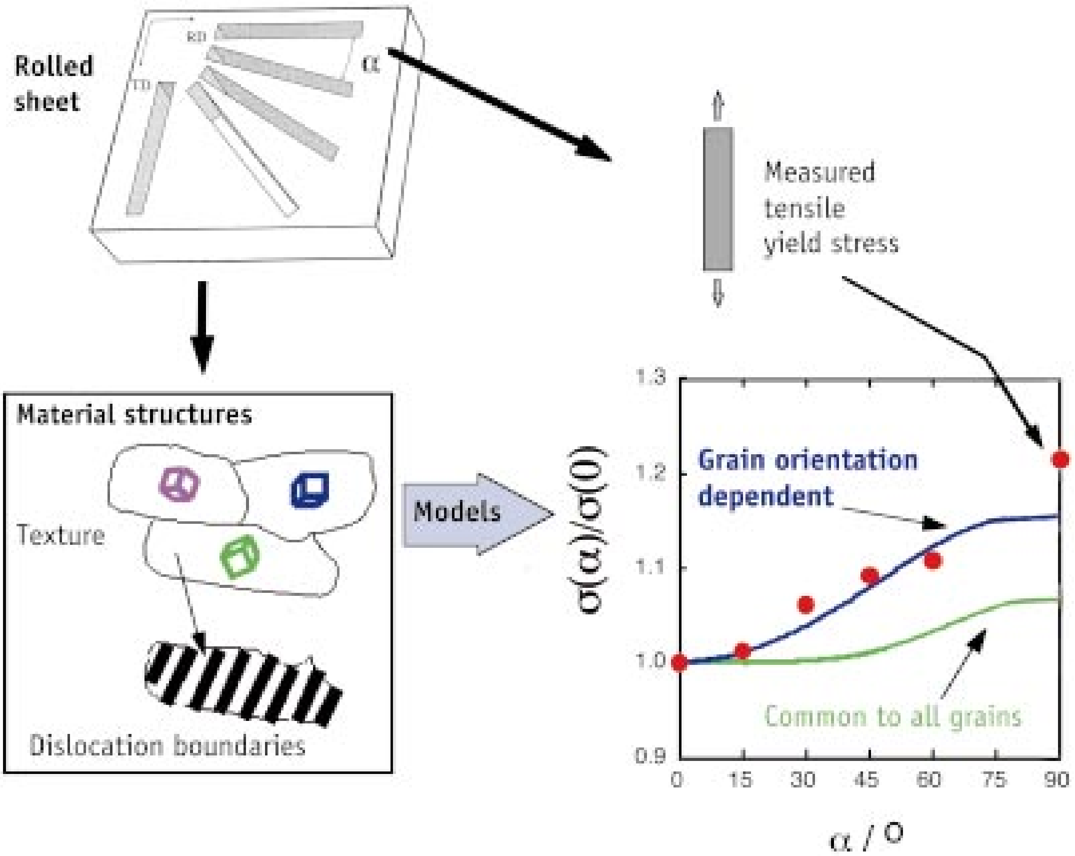
Dislocation microstructures in tensile deformed copper polycrystals and single crystals depend on the crystallographic orientation of the grain or single crystal. The structures in polycrystals resemble those in single crystals. Three types of microstructures have been found: Planar dislocation boundaries on slip planes (Type 1), dislocation cells (Type 2) and dislocation boundaries not on the slip planes (Type 3).

Improved prediction of yield stress anisotropy

The structures developed during deformation, i.e. texture and dislocation boundaries, influence the mechanical properties of the material. It is well known that texture gives rise to yield stress anisotropy because the yield stress of different grain orientations in different directions is different. Dislocation boundaries in metals rolled to low and intermediate strains have a common preferred orientation relative to the rolling direction, i.e. the structure is anisotropic. Dislocation boundaries therefore also contribute to the yield anisotropy.

A model of the combined effects of dislocation boundaries and texture on yield anisotropy has previously been developed. In this model all grains were assumed to have dislocation boundaries with the same preferred orientation relative to the macroscopic rolling direction. Discovery of the grain orientation effect on microstructure allows a more detailed and precise description of the boundaries with different boundary orientations in grains with different crystallographic orientations.

Incorporation of this grain orientation effect, and in particular the fact that some grain orientations have boundaries on {111} slip planes, in the yield anisotropy model lead to great improvements of the predicted yield anisotropy in rolled sheets. It is important to have good predictions to select the right materials for e.g. deep drawing processes where yield anisotropy controls the formation of ears and leads to inhomogenous material thickness in the final product.



The yield stress measured for samples cut out in different directions from a rolled sheet matches the yield anisotropy predicted from the texture and dislocation structure. Use of grain orientation dependent dislocation structures greatly improves the predictions compared to predictions assuming a common structure in all grains.

Modelling subdivision of single crystals

Previous analyses of single crystal subdivision during rolling focused on idealised rolling deformation conditions and included only a limited selection of slip systems. New investigation of the subdivision has incorporated the role of geometrical and frictional conditions present during the rolling, and has begun to incorporate new experimental information from marker wires embedded in the crystal prior to rolling. The shape of the wire is determined by metallographic sectioning after deformation. The marker offset information can be combined with the electron back scattering pattern (EBSP) lattice rotation measurements and transmission elecron microscopy (TEM) observations of the dislocation boundary structure to provide a more complete data set for an analysis of slip system activity. This technique has been applied to rolled single crystals of initial cube and rotated cube orientation.

The EBSP and marker experiments revealed that a single crystal subdivides on a macroscopic scale (of the order of 100 µm), in addition to a local scale (covering a few µm), and a new modelling effort addressed this larger scale subdivision. Frictional effects from the rolls and a geometrical effect from the roll gap configuration are thought to be the main factors promoting macroscopic crystal subdivision, and the factor opposing such subdivision is the orientation stability of the initial crystal orientation. Existing models of crystal subdivision do not include all of these factors, and can thus provide only an incomplete representation of the rolling process. A major goal is to formulate a comprehensive model including all of the primary factors that influence crystal subdivision at a macroscopic scale.

Modelling orientation stability of dislocation boundaries during deformation

Parallel extended dislocation boundaries with a spacing of a few micrometer are observed in single and polycrystals cold-rolled to low and intermediate strains. The boundaries are inclined approximately ± 40° to the rolling direction, an angle which decreases only slightly up to rolling reductions of 50 %. This is in strong contrast to the behaviour of ordinary grain boundaries, which are flattened during rolling.

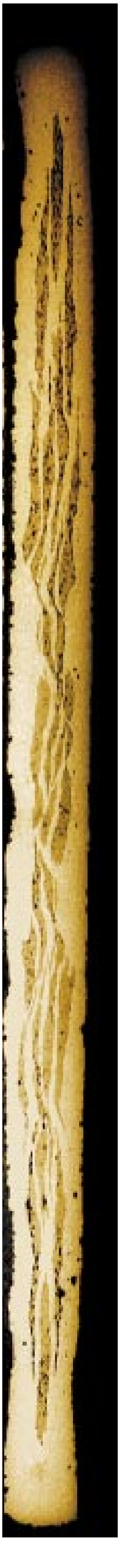
In case of grain boundaries, slip on unexpected slip systems may be activated in order to avoid incompatibilities during deformation or the occurring stresses may be relaxed by grain boundary sliding. Both mechanisms are not possible for dislocation boundaries and a different mechanism is proposed:

During deformation dislocation fluxes into the boundary from the active plastic slip on both sides lead to creation of boundary ledges and grain boundary dislocations, both associated with long range stress fields. If all dislocations in a dislocation boundary are assumed to be mobile in their respective slip planes, the dislocation boundary can rearrange to avoid long range stresses. In order to remain a low-energy configuration, the orientation of the boundary changes in the opposite direction of the flattening imposed by continuum mechanics.

With the proposed model the relative stability of the dislocation boundary orientation can be attributed to the specific dislocation content of the boundaries and their tendency of avoiding long range internal stresses.

Characterisation and modelling of the annealing behaviour of BiSCCO/Ag tapes

The Department is a participant in a Danish program for utilisation of high-temperature superconductors in the electric power sector. Cables for power transport are to be based on tapes containing the compound $Bi_2Sr_2Ca_2Cu_3O_x$ (Bi-2223). The tapes are produced at Nordic Superconductor Technologies by filling a precursor powder into silver tubes, drawing the tubes, rolling them into tapes and annealing the tapes several times at temperatures around 830 °C. The processing controls the phase purity, grain alignment and grain connectivity between the Bi-2223 grains – three factors that in turn determine the superconducting properties. The aim of the Department in close collaboration with Nordic Superconductor Technologies and the Department of Condensed Matter Physics and Chemistry at Risø is to understand the materials science and to optimise the annealing processes with respect to the superconducting critical current density J_c. Highlights for 1998 include: (i) the establishment of a model of the detrimental processes taking place during cooling of the tapes, (ii) clarification of the texture formation mechanisms, and (iii) the establishment of a test routine based on differential thermal analysis (DTA) measurements for control of the pre-cursor powder, which has been implemented by Nordic Superconductor Technologies.



Cross section (0.1 x 3 mm²) of superconducting tape. The current density decreases with the distance from the central part of the tape. This decrease is attributed to the non-uniform production technology.

Spatial variations in microstructure and current transport properties

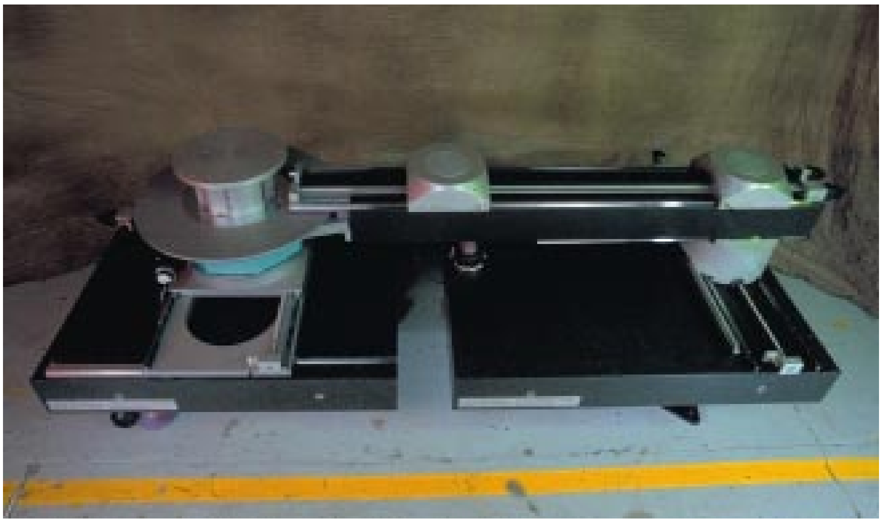
The superconducting tapes in use are multifilament tapes produced by bundling 19, 35 or more drawn tapes together and inserting them in a circular Ag tube. The assembly is then rolled until the cross section of the tape is approximately 0.1 x 3 mm^2. This deformation process is non-uniform. Microstructural characterisation by scanning electron microscopy reveals that the Bi-2223 density, phase purity and grain alignment near the edges are inferior to the centre part.

A cutting method based on the use of a diamond saw was developed, to prepare individual samples from the tape. This allows determination of the local superconducting critical current density, J_c, in the tape as function of the transverse position. The J_c of the individual filaments is found to vary between 0 and 70 kA/cm^2, with the homogeneously deformed centre in general being the optimum. These findings imply that the global J_c of the tapes may be doubled, provided a better design for the filament geometry and a more suitable deformation route can be found.

Construction of a 3D X-ray microscope

The Department has nearly completed the construction of a 3D X-ray microscope at the European Synchrotron Radiation Facility (ESRF) in Grenoble. The microscope will be dedicated to local structural characterisation of materials within millimetre thick specimens. Parameters such as strain, grain orientations, grain morphology and dislocation densities are to be investigated *in-situ* during annealing or deformation processes. The instrument is specified to operate with gauge volumes of order 5 x 5 x 50 μm^3. It is built in collaboration with ESRF, who has provided a 4 x 10 m^2 experimental site – a lead security hutch – as well as a control room. The first data set was acquired in December 1998 and the instrument is expected to become fully operational in 1999.

To meet specifications, novel types of X-ray optics have been designed and tested. These include a high-resolution two-dimensional detector operating in the relevant high energy X-ray range of 40 - 100 keV. The detector is based on three components: A fluorescence screen, which converts the X-rays into visual light, two optical mirrors for focusing the visual light and a CCD with a small pixel size. In total, an effective resolution of the order of 5 x 5 μm^2 is obtained. The detector may for instance be used to map out the contours of the grains within a polycrystal with medium to large grain sizes. In order to do



A vital part of the 3D X-ray microscope is a 3-axes diffractometer designed by JJ-Xray. The diffractometer is 3 m long. The table to the left is the sample stage, which can carry a load of 200 kg, allowing for the use of furnaces and stress-rigs. The two other tables will carry slits, detectors and focusing optics for the diffracted beam.

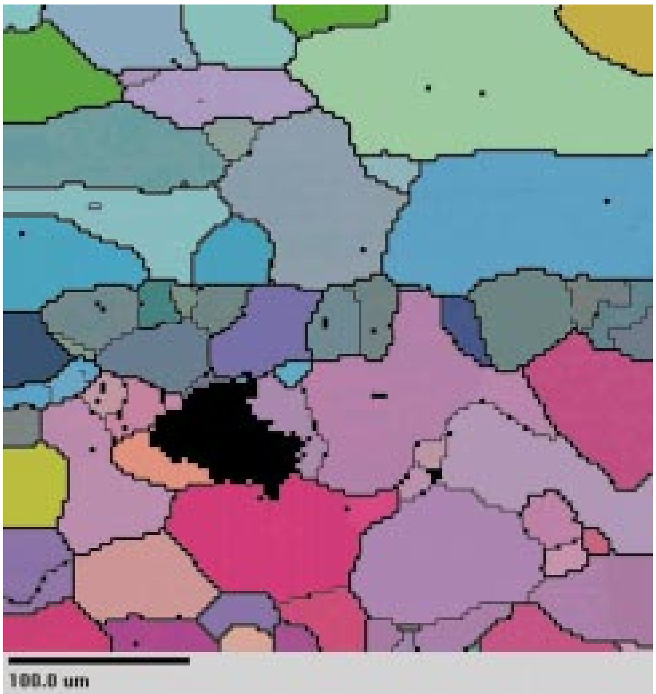
so the incoming monochromatic X-ray beam is focused in one direction in order to define a specific layer in the specimen. The high-resolution CCD is then inserted in the diffracted beam close to the specimen, and the various Bragg reflections brought into diffraction by rotating the specimen. By varying the distance between the specimen and the detector, the origin of the diffracted X-rays can be tracked. This procedure is repeated for the layers of interest until a full three-dimensional map is obtained.

Advances in crystal orientation microscopy

Crystal orientation microscopy (COM) is a unique technique for obtaining a precise image of how the crystal lattice is oriented on the surface of a crystalline material. The technique relies on a rigorous but fully automated analysis of so-called electron back-scattering patterns (EBSPs) which are collected by specialised attachments to the scanning electron microscope (SEM). COM and EBSP offer a high spatial resolution (100 - 500 nm), a high precision of the measured orientations (0.5 °), and rapid data collection.

Recent developments of our COM software have lead to significant improvements in both the precision and reliability of the collected crystal orientations, and furthermore the speed of data acquisition has been greatly increased. Employing a motorised stage, it is now possible to measure one point/orientation in 2 seconds. Further, with computer control of the scanning coils in the SEM, the speed should soon increase to around 5 points per second. However, even the current speed allows collection of significant amounts of data and thereby images of lattice orientations over fairly large areas at a fine resolution.

For visualisation of the sampled orientation data, new software was designed. This software offers several strategies for assigning colours to the crystal orientations so that a colour image of the microstructure can be created. Grain boundaries, or other boundaries associated with a disorientation, can easily be added to the image, and plots highlighting just disorientations (magnitude and axis) can be made. Finally, images of a pattern quality parameter can be created, illustrating local plastic strain over the sample surface.



Crystal orientation map of annealed aluminium. The map consists of 140 x 140 points and the distance between each sampled point is 3 μm. The mapping from orientation space to colour space is chosen so that the measure of distance in both spaces is well preserved. Black points are unmeasured, primarily due to holes and scratches on the material surface.

MATERIALS ENGINEERING
- modelling and performance

The activities within Materials Engineering are focused on characterisation and modelling of materials properties for design applications, component design and performance testing. The activities are carried out within two programmes: *Properties of Advanced Composite Materials*, which covers metallic, ceramic and polymer matrix composites reinforced with fibres or particles, and *Mechanical Characterisation and Design of Light Components*, which covers experimental and numerical methods that relate materials and components. The highlights from these activities are
(i) prediction of fatigue life time of polymer matrix composites under variable amplitude loading,
(ii) an improved materials model for tool-steels, which leads to a tenfold increase in tool-life-time,
(iii) technological development of an innovative flywheel for energy-storage,
(iv) a steady increase of commercial contracts concerning mechanical characterisation of materials.

Project Funded Research: Materials Engineering

Project type	Project name	Co-participants
Danish Research Councils	Natural Fibres for Polymeric Composites	• Royal Veterinary and Agricultural University, Denmark • Institute for Construction and Materials, DTU, Denmark
Ministry of Food, Agriculture and Fishery	Plantfibrebased 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
Ministry of Environment and Energy	Wood-fibres, their Structure and Properties with Reference to Composite Materials	• Royal Veterinary and Agricultural University, Denmark • Institute for Construction and Materials, DTU, Denmark
Ministry of Environment and Energy (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
Ministry of Environment and Energy (EFP)	Flywheel for Energy Storage-II	• TERMA Industries A/S, Denmark • NESA A/S, Denmark • DEMEX A/S, Denmark
EUCLID	Advanced Techniques for Add-on-Armour	• DEMEX A/S, Denmark • OTOBREDA, Italy • EN Santa Barbara, Spain • Sistema Compositi, Italy • TNO-PML, The Netherlands • Fokker SP, The Netherlands • Spav, The Netherlands • HB Consultancy, The Netherlands
EUCLID	Long Term Effects on Light Weight Add-on Armour	• DEMEX A/S, Denmark • DSM, The Netherlands • OTOBREDA, Italy • EN Santa Barbara, Spain • Sistema Compositi, Italy • TNO-PML, The Netherlands • Fokker SP, 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 • Ferriere e Fonderie di Dongo, Italy • Gussstahl, Germany • Aerospatiale, France • Magma, Germany • RWTH, GI, Germany
BRITE-EURAM	Assessment of Metal Matrix Composites for Innovations	• Technische Universität Wien, Austria • INTROSPACE GmbH, Germany • ISRIM, Italy • Austrian Research Center Seibersdorf GmbH, Austria • Aerospatiale, France • CSM Materialteknik AB, Sweden • Deutche Gesellschaft für Materialekunde, Germany • German Aerospace Center, Germany • Universidad Politècnica de Madrid, Spain • Swiss Fed. Inst. for Mat. Test. and Research, Switzerland • Ecole Polytechnique Fédérale de Lausanne, Switzerland • Institute National des Sciences Appliquées, France • University of Cambridge, UK • National Physical Laboratory, UK • VTT Manufact. Techn. Research, Finland

Project type	Project name	Co-participants
		• Katholieke Universiteit Leuven, Belgium • Daimler-Benz AG, Germany • Centro Ricerche FIAT, Italy • EA-Technology Ltd., 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 • Instituto de Soldadura e Qualidade, Portugal • Sauerwein System Technick GmbH, Germany • VIDROPOL S.A., Portugal • J. B. Plant Fibres Ltd., UK • Carl Bro A/S, Denmark • CEDIP S.A. France
BRITE-EURAM	Thixoforming of Advanced Light Metals for Automotive Components(TALMAC)	• Pechiney CRV SA, France • Norsk Hydro Aluminium a.s., Norway • Norsk Hydro (Mg Division) a.s., Norway • EFU GmbH, Germany • Stampal SpA, Italy • FIAT SpA, Italy • Volkswagen GmbH, Germany • INPG, Grenoble, France • University of Ancona, Italy • SINTEF, Norway
BRITE-EURAM	Design and Processing of Selectively Reinforced Magnesium-based Composites	• Daimler Benz GmbH, Germany • Aerospatiale SA, France • Blankguss, Germany • GF Automobilguss, Austria • Unitech, Austria • IMMG, Greece • Morgan MT, UK • EMPA, Switzerland • RWTH-Aachen, Germany • ILFB/TU Wien, Austria • LKR, Ranshofen, Austria
BRITE-EURAM	Hyper-Eutectic Alloys for Automobile Components (HAforAC)	• ISRIM, Italy • FIAT SpA, Italy • Stampal SpA, Italy • Pechiney CRV SA, France • Fagor Ederlan, S. Coop. Ltd., Spain • Bosch Systemes de Freinage, France • University of Sheffield, UK • Norsk Hydro Aluminium a.s., Norway • CEIT de Guipuzcoa, Spain • Allied Signal Bremsbelag GmbH, Germany
BRITE-EURAM	Development of a Portable Remote Controlled Real-Time Radioscopy System for Quantitative Industrial Inspection of large Thickness Steel Pipes and Weldings (RAYSQINS)	• Carl Bro A/S, Denmark • Photonic Science plc, UK • Thomsen Tubes Electroniques SA, France • Sauerwein System-Technik GmbH, Germany • Statoil a.s., Norway • Bundesanstalt für Materialprüfung, Germany • Instituto de Soldadura e Qualidade, Portugal • Institut de Soudure, France
BRITE-EURAM	Graintwist	• University of Cambridge, UK • Mitsui Babcock Energy Ltd., UK • Metall-SpecialRohr GmbH, Germany • University of Liverpool, UK • Plansee, Germany/Austria
EUREKA	EUROBOGIE	• EM-Fiberglas, Denmark • Sciotech, UK • Polymath, UK • English Welsh Scottish Railways, UK • Reading University, UK • Concargo/Polymer Engieneering, UK • Bombardier Talbot, Germany • SVUM, Prague, Czech Republic • Skoda Research, Pilzen, Czech Republic • IPM, Riga, Latvia
BRITE-EURAM	Action for low weight automobile technologies (FLOAT)	• Rover Group Ltd., UK • GIE Renault, France • FIAT SpA, Italy • Austrian Research Center Seibersdorf GmbH, Austria • Frauenhofer, Germany • Volvo AB, Sweden • SEPARIS, France • ISRIM, Italy • Daimler Benz AG, Germany
BRITE-EURAM	Plant Life Assessment Network (PLAN)	Thematic Network

Properties of advanced composite materials

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

Strain fields in metal matrix composites (MMCs)

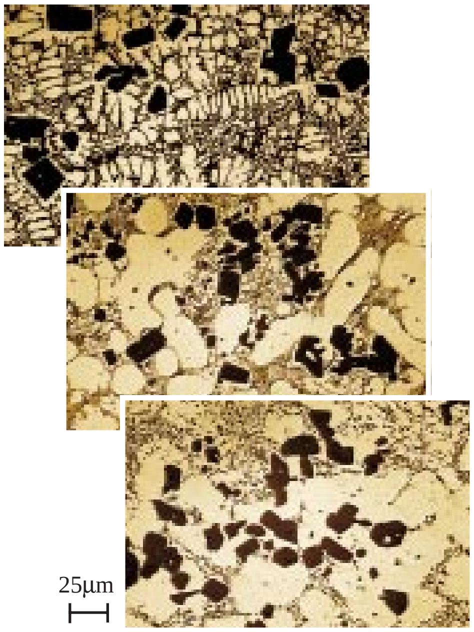
The strain fields around whiskers in aluminium-based composites following tensile deformation were determined experimentally by micro-diffraction studies of local orientations in a Transmission Electron Microscope (TEM). Discrepancies were found between the experimentally determined strain gradients and predictions from traditional finite element models (FEM) based on scale-independent continuum plasticity theory and the former are lower by a factor of 5 - 10 than the latter. However, the experimental results were in good agreement with a continuum mechanics model that incorporates a length-scale and strain-gradient plasticity theory. This leads to a better understanding of the deformation behaviour of MMCs: strain gradients exist over a characteristic scale, which in the whisker reinforced MMC may be of the order of the whisker size. The strain gradient causes elevated hardening. This effect reduces the strain localisation and leads to smoother strain fields.

So far the analysis has been concentrated on single whiskers with an aspect ratio of 5 - 6. But in the real material, whiskers are often present in clusters, and particles with smaller aspect ratio are also used as reinforcement. The TEM micro-diffraction technique was therefore applied to map the local misorientations of these cases. Strain fields were measured around whisker clusters having a simple geometry in an aluminium composite containing 2 % SiC whiskers and around equiaxed particles in an aluminium composite containing 2 % SiC particles with a size of ~ 1 µm. It was found that the local lattice rotation associated with clusters was larger than that around single whiskers. For example, at 10 % tensile deformation the maximum misorientation was ~ 5 ° for single whiskers, but 8 - 10 ° for clusters. This indicates a more intense strain localisation in the clusters. Also, an effect of the aspect ratio was observed: the local lattice rotation around particles was smaller than that around whiskers, indicating reduced strain localisation. These observations are qualitatively in agreement with the predictions by traditional FEM.

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

Light materials are of great importance for the transport sector and in particular for the automobile industry. A European project consortium, including car manufacturers, investigates light metals that are being processed into car components by thixoforming techniques. The focus is on minor components in personal cars, such as suspension devices and brake components. The typical weights of these components are about 5 kg and the goal is a reduction to 2 – 3 kg.

The materials considered are aluminium alloys, aluminium – metal matrix composites and magnesium alloys. Simulation of the thixoforming process is performed experimentally and theoretically to investigate the composition



Microstructures of hyper-eutectic silicon aluminium developed for automotive components. The three pictures show the alloy (i) before thixoforming, (ii) after thixoforming and (iii) after thixoforming and heat treatment.

and rheological behaviour of the semi-solid slurries. The practical experiments are performed on simple, generic shapes to gain experience under pilot plant/ industrial condition. The Department is responsible for quality control and mechanical characterisation of the thixoformed materials.

The quality was evaluated by X-radiography in terms of microstructure and defects. The quality of Al-alloys and Al-composites was good. However, after the first experimental thixoforming trials, the Mg-alloys showed evidence of minor voids and small cracks, preferentially distributed in the middle section of rod-shaped parts. Microscopy investigations of selected regions of the rod-shaped parts correlated well with the X-radiographic results for voids and cracks. Ultrasonic measurements of sound velocity agreed with mechanically measured stiffness values.

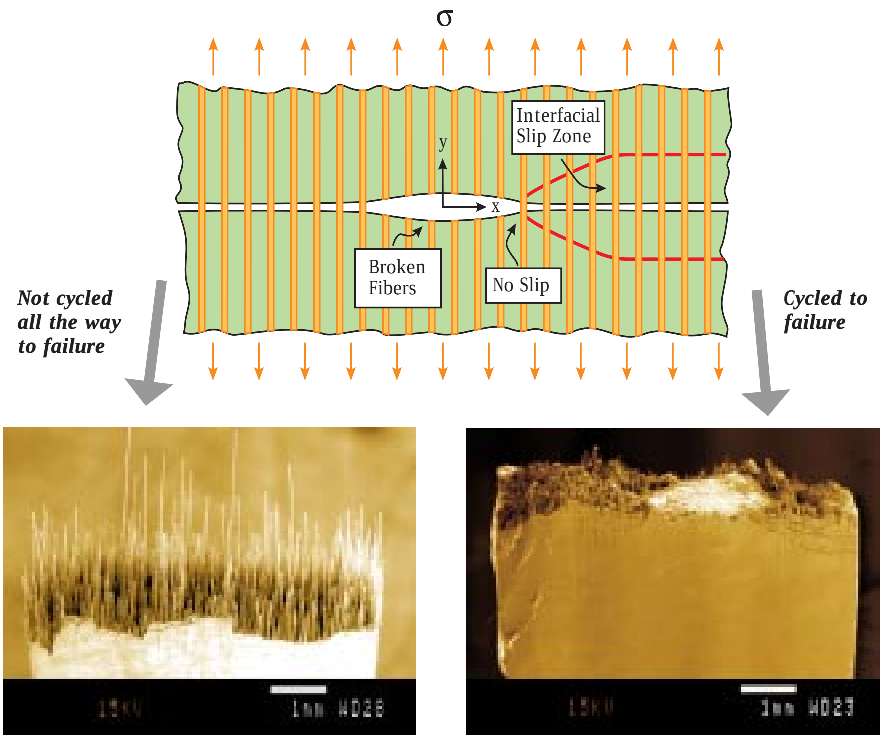
Mechanical characterisation was performed on all materials; tensile properties were recorded as basic reference values. Fatigue testing results were accumulated to allow fatigue diagrams to be established for use in design of the car components.

Fatigue of ceramic matrix composites

Ceramic matrix composites (CMCs) with weakly bonded fibre/matrix interfaces possess damage tolerance, i.e. crack insensitivity. Multiple matrix cracks can form without causing fibre fracture, since slip occurs along fibre/matrix interfaces. It is therefore of both practical and scientific interest to study the fatigue behaviour of material with frictional slipping interfaces.

Tensile specimens of a unidirectional SiC-fibre-reinforced calcium aluminosilicate matrix composite were cycled to failure or to a pre-selected number of cycles under identical loading histories. The residual strength of the specimens not cycled all the way to failure was found to be similar to that of virgin specimens. Microstructural investigations showed that the specimens not cycled to failure had uniform fibre pull out. In contrast, the fracture surfaces of the specimens cycled to failure had a central region where fibre pull out was negligible. This embrittlement of the centre is attributed to loss of interfacial sliding, i.e. rebonding so that the fibres cannot slip but will break.

On the microscale, interfacial temperatures may be very high at the local contact points between fibre and matrix due to frictional heating. High temperatures (> 600 °C) may cause oxidation of the carbon-interphase layer resulting in a strong SiO_2 bonding across the interface. This thermally induced rebonding is more likely to occur in the specimen centre where the heat generated by friction cannot escape by radiation or convection.



Fracture surfaces of ceramic matrix composites with weakly bonded fibre/matrix interfaces. Left: The fracture surface of a specimen not cycled all the way to failure displays long fibre pull out lengths. Right: The fracture surface of a composite cycled to failure has a central zone with no fibre pull out.

Model relating reference fatigue data to real service conditions for PMCs

Fatigue performance data on composites, in particular polymer matrix composites (PMCs), is often needed during design of components which must function in unattended service for long times and during numerous fatigue load cycles, for example rotorblades for very large wind turbines. Reference experiments are often made under constant amplitude fatigue loading, while service conditions are characterised by randomly variable amplitude fatigue loading. It is therefore of interest to compare these two types of fatigue loading, and to convert between the two types.

The concept of an equivalent stress has been developed starting with the calculation of the fractional life time sum (Miner's sum). This sum is based on the life times at constant amplitudes weighted with the number of cycles at each amplitude during the loading. The equivalent fatigue stress for variable amplitude loading is defined as the stress for constant amplitude loading which leads to failure after the same number of cycles.

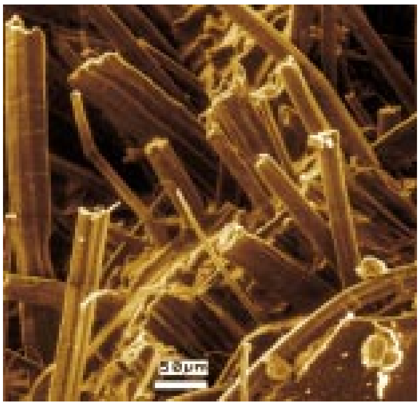
The fatigue data are plotted in a log-log-diagram of stress versus number of cycles to failure. In a reformulation of the equivalent stress calculation, the slope of this curve appears. In contrast to metals, the value of the slope parameter for PMCs is such that the calculated equivalent stress is almost independent of Miner's sum. This observation means that the equivalent stress can be used without prior knowledge of Miner's sum for the actual material and loading spectrum.

The equivalent stress obtained from variable amplitude fatigue data is in good agreement with the stress of constant amplitude data for several composites of glass fibre/polyester and for hybrid composites with glass- and carbon-fibres.

Mechanical properties of plant fibre composites

A long term aim is to fabricate and characterise composites based on natural fibres, i.e. plant fibres and wood fibres, bonded together by a polymeric matrix. The work is carried out in collaboration with the Plant Biology and Biogeochemistry Department at Risø. The matrix may be a traditional, synthetic polymer or a polymer derived from natural resources and potentially biodegradable and renewable.

The present series of composites are based on the simple approach of using



Tensile fracture region of a flax polypropylene composite observed by scanning electron microscopy. In the lower right corner the polypropylene matrix is seen between flax fibres and in the upper left corner smooth fibres are visible. The smoothness and the fact that many fibres have been pulled out of the matrix indicate a weak fibre/matrix interface.

fibres which are not surface treated, and matrices of conventional thermoplastic polymers, such as polypropylene. This will establish a reference for comparison against more advanced fibre treatments and highly developed fabrication processes.

The composites were made from fibre mats, with generally random fibre orientation distribution, and foils of polypropylene. The composites were laid up into film-stacking packages, which were either press-consolidated or vacuum-consolidated. The manufactured laminate plates were about 2 mm thick. Their density was around 1 g/cm^3 and they contain about 30 volume percent fibres and between 5 and 20 volume percent porosity.

The mechanical characterisation comprised tensile loading at room temperature under normally dry conditions. For both jute/polypropylene and flax/polypropylene composites the tensile stiffness was in the range 3 – 7 GPa, and the tensile strength was in the range 20 – 50 MPa. The mechanical properties are comparable to values predicted by composite theory. They are expected to be improved via the material (fibre and matrix) parameters and the processing conditions.

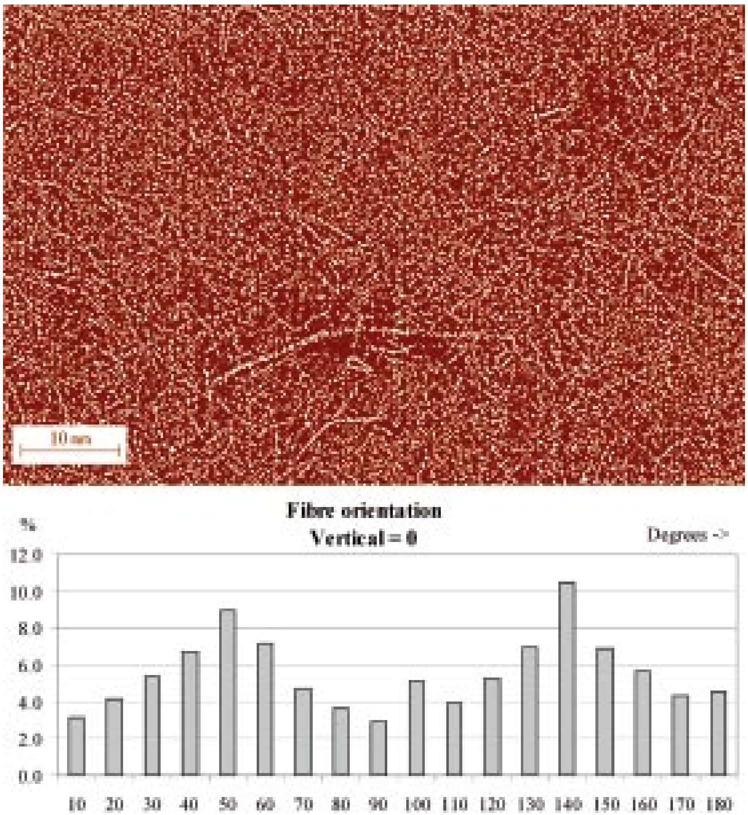
An extensive microscopical study was made of the morphology of several types of fibres, jute, flax, hemp, birch and wheat-straw. The data will be used to judge the suitability of the various fibres for e.g. mat-forming, and to evaluate fracture regions as observed on composite samples after mechanical loading to failure.

To optimise the fibre properties, the connection between chemical composition, molecular size, crystallinity and mechanical properties must be established.

Plant fibre composites: Non-destructive characterisation

Composites made of plant fibres offer a new supplement to traditional glass fibre composite materials. A major issue is the assessment of critical defects and development of industrial inspection methodologies and equipment. The work is directed towards the importance of the flaws for the mechanical properties, flaw detection and quantification by two different non-destructive characterisation (NDC) techniques: lock-in thermography and low-energy (dual energy) radioscopy. For the former technique, a close collaboration with the Optics and Fluid Dynamics Department at Risø has been initiated. NDC techniques may provide a variety of data relevant for evaluating the manufacturing processes involved in the production. As an example, the general fibre orientation can be determined by applying image processing techniques on digitised low-energy radiographic images. Apart from proper selection of the radiation energy, the quantification of the structural elements requires that the plant fibres are visualised. This is accomplished by applying a background correction procedure and a threshold technique based on maintaining the original dimensions of e.g. the plant fibres.

The industrial relevance also ex-



Digitised binary X-radiograph of a hemp fibre / polypropylene mat (50/50 volume %), in which the plant fibres have been visualised by quantitative image processing. In this case, the fibres are predominantly aligned at 45 ° and 135 ° .

tends to inspection of other fibre composites; in particular to the joining of e.g. fibre reinforced polyester pipes. In this respect, the collaboration with manufacturers (Vidropol S.A., Portugal and J.B. Plant Fibres, UK) and with inspection companies (such as Carl Bro A/S, DK) secures that methodological developments to a high degree are directed towards industrial demands on defect detection and quantitative characterisation.

Shape active composites

The work on advanced shape active composites (Smart Composites) was concentrated on composites based on reinforcement fibres of the shape memory alloy NiTi embedded in aluminium matrices. Polymer matrices have also been considered. Such systems based on NiTi or other shape memory alloys are part of a wider group of shape active systems, which also include reinforcements of piezo electric or magneto-strictive materials.

Shape active composites were successfully produced. Such materials can be tailored to have unusual properties. It has for instance been possible to produce a material with zero or even negative thermal expansion, which may be relevant in space applications.

The project has included theoretical modelling based on so-called Eshelby formulations, differential scanning calorimetry (DSC) for monitoring of phase transformations and traditional mechanical testing for characterisation of macroscopic properties. Furthermore, neutron diffraction has been utilised for *in-situ* monitoring of internal stresses and the progress of the phase transformation in the embedded fibres during thermo-mechanical loading.

Through this project the Department has pioneered the production of these novel composite systems. Plans are now being cast for the formulation of a larger European research proposal. This proposal will include processing, modelling and testing of the new materials, which have great potentials in the fields of sensors, actuators or even as candidates for artificial muscles.

Wind turbine rotorblades produced by LM Glasfiber A/S. Stronger polymer matrix composites reinforced with carbon fibres are considered for large rotorblades.

Mechanical characterisation and design of light components

The programme covers non-destructive and destructive techniques for characterisation of materials and components as well as design and development of specific components.

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

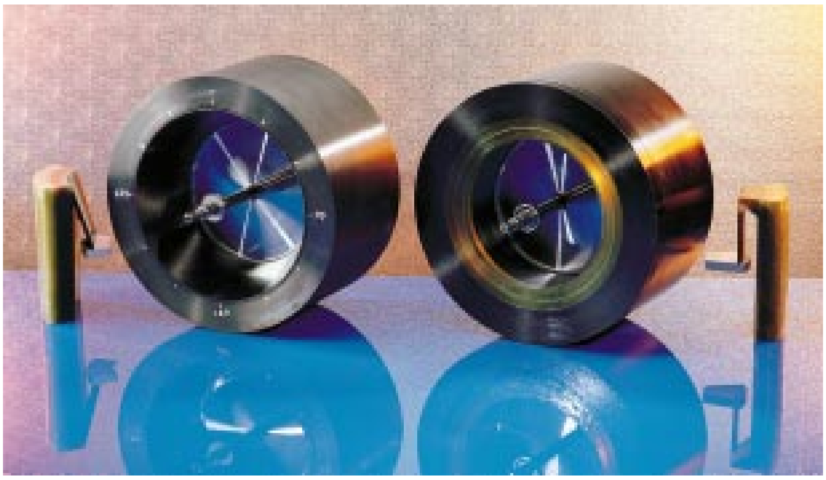
Mechanical testing of materials was performed on a commercial basis for several Danish and European companies in parallel with the testing performed within the Department's own project activities. The materials were both traditional metallic engineering materials (tooling materials, aluminium, steel), composite materials (metal matrix composites, polymer matrix composites, natural fibre composites) and rubber. The amount of commercial testing has increased significantly during 1998, presumably due to (i) the Department's unique testing facilities and expertise, (ii) a more active marketing effort in this area and (iii) an increase in the need within the industry to certify their engineering materials.

Projects on design of components involve rotorblades for wind turbines and a composite flywheel rotor for energy storage. Work on other components has been initiated.

Larger rotorblades for wind turbines require the use of carbon fibre composites

The cost efficiency of wind turbine generated power increases as the diameter of the rotor is increased. Today, the largest commercially available glass fibre-reinforced rotorblades are around 30 m long. It is foreseen that even larger rotorblades will become a market demand in the near future. Glass-fibre has a low specific stiffness, which for large blades results in a heavy blade. For rotorblades longer than 30 m the weight becomes important. The use of carbon fibres in the main structure are investigated because carbon fibres have a higher specific stiffness than glass fibres.

The scientific aim of the project is to develop and investigate experimental methods to characterise the strength of carbon fibre-reinforced composites. Focus is on methods to determine the compressive and interlaminar strength of thick polymer composites. Models based on both solid mechanics and fracture mechanics are being investigated. The industrial aim of the project is to apply the developed methods for optimisation of the structural laminate under various processing conditions. Simplicity of the experimental methods and an unambiguous interpretation based on models are important. The project is carried out in collaboration with LM Glasfiber A/S as an industrial post doc project.



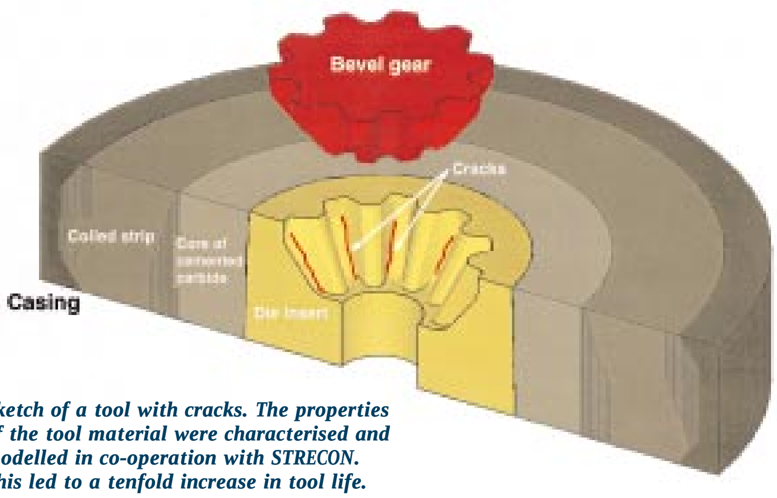
Two flywheel rotors with a diameter of 40 cm made of strong composites to increase the energy storage capacity of the flywheel. The left one has been successfully tested at an operating speed of 18000 rpm.

A flywheel rotor made of polymer matrix composite: Manufacturing and testing

The energy storage capacity of a flywheel depends on the strength-to-weight ratio of the material used for the flywheel. Strong and light materials have the highest capacity. Therefore, composites that utilise fibres such as glass and carbon are obvious candidates for this application.

A composite flywheel rotor with a novel rotor/shaft connection was designed and manufactured. The flywheel rotor outer diameter is 400 mm and the rotor mass is 20 kg. The flywheel was designed for a maximum operating speed of 36000 rpm and a usable storage capacity of 4 MJ (≅1 kWh). The prototype was designed as a testing unit for the flywheel as such, and the rotor is contained in a steel housing, intended to contain the rotor in case of rotor failure. The rim was manufactured by a filament winding technique using high strength carbon fibre epoxy composites. The thickness of the rim is large, 55 mm. Therefore, to reduce curing stresses, a low-temperature curing epoxy system was used. The connection element between the composite rotor and the steel shaft is a thin composite shell, allowing for the difference in deformations between shaft and flywheel rim while still being capable of transferring the power between rim and shaft. Two different designs of this element were studied: (i) a conical shell element was made in high strength carbon fibres where the variation in fibre angles allowed design of the correct stiffness, (ii) a cylindrical shell element with graduated material was obtained by using fibres of different stiffnesses (carbon and glass). The individual components of the rotor were connected to each other by adhesive bonding.

Static tests of the rotor connection demonstrated the very high torque transmission capabilities of the connection elements, resulting in power capacities of more than 1000 kW at the anticipated rotational speeds. The initial functional tests of the rotor in the evacuated safety housing - until now limited to 18000 rpm - were used for verification of the auxiliary systems: vacuum system, cooling and lubrication of the mechanical bearings. It also verified the rotor performance when running through the critical velocities.



Sketch of a tool with cracks. The properties of the tool material were characterised and modelled in co-operation with STRECON. This led to a tenfold increase in tool life.

Pressure die tools: materials characterisation and modelling improve lifetime

For industrial applications dies must be prestressed to prevent overload failures and improve die life. Strip wound tool elements which replace conventional multiple ring sets in tools for high pressure applications have been developed in collaboration with STRECON, which is a company in the Danfoss group.

The low cycle fatigue behaviour of cemented carbides used for dies in production of synthetic, industrial diamonds, and of conventional tool materials used for cold forging was studied. Materials were tested in uniaxial tension and compression. Static and cyclic tests were performed both at room temperature and at 250 °C on the very brittle cemented carbides and the conventional tool steels. The results were used to model and predict the behaviour of the tools. The material models were developed in an industrial PhD project in collaboration with the Department of Solid Mechanics at DTU and STRECON. The project resulted in a tenfold increase of tool life. Tool life improvements of the order of 100 may be gained during the continued optimisation work.

Detection of damage evolution by thermography

Thermography is an experimental technique for measuring temperature fields.

An infrared camera scans the surface of the specimen and an image of the temperature field is produced. In materials research, temperature fields can be used to detect damage propagation and localisation during cyclic loading. For instance, polymer matrix composites (PMCs) are used in wind turbine rotorblades, which are subjected to fatigue during service. During cyclic loading the fatigue damage causes heat dissipation, resulting in an increase in the local temperature. The temperature rise is a qualitative measure of the damage state. A quantitative interpretation of the temperature field is more complicated to achieve, since the field depends not only on the heat dissipation but also on the heat loss to the surroundings.

The evolution of the temperature field of a 0 ° glass/carbon fibre hybrid composite subjected to cyclic loading was investigated. During most of the fatigue life the temperature field was uniform. Just prior to failure a rapid temperature rise occurred locally, indicating a transition from distributed damage to localisation. Thus, a localisation of the surface temperature field is indicative of imminent fatigue failure in these materials.

Ultrasonic techniques used to measure aging of PMCs in humid environments

Polymer matrix composites (PMCs) can be made with desired stiffness and strength in the load-carrying directions. In addition, the composites have very large stiffness-to-weight and strength-to-weight ratios. However, knowledge of the material degradation due to environmental conditions is needed in order to predict the change in stiffness and strength of the material.

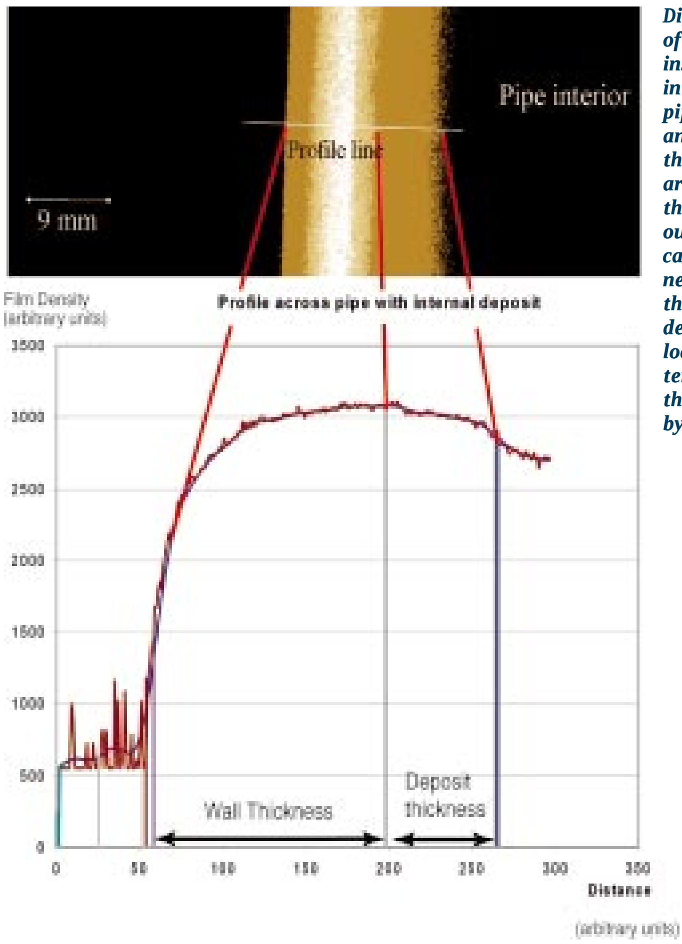
The effect of a humid environment on the in-plane and the out-of-plane stiffness (the elastic constants C_{11} and C_{33}) has been investigated for PE/kraton laminates of high density polyethylene fibres in a matrix of rubber compound (styrene-isoprene block copolymer).

Quantitative ultrasonic measurements of the elastic constants were compared for specimens with [0 °] and with [0 ° / 90 °] fibre orientations, respectively. The specimens were manufactured with different moisture resistant surfaces and immersed in water for 24 hours. The swelling coefficients of the materials were measured and used in the calculation of the elastic constants. The calculation of the elastic constants is further complicated by the viscoelastic dispersion of ultrasound in the anisotropic materials which changes the ultrasonic pulse form. To illustrate the effect of evaluation method the elastic constants were calculated using four different broadband methods.

Quantitative X-ray techniques for condition monitoring in industry

A system was developed for the high-energy radioscopic inspection of large structures, such as pipes, and the quantification of structural details in near real-time. The work was performed in close collaboration with the Danish industrial inspection company Carl Bro A/S. The tasks undertaken in the Department included: (i) characterisation of the detector, (ii) development of procedures for determining the remaining pipe wall thickness based on differences in attenuation and on tangential pipe wall thickness techniques and (iii) development of procedures for an expert system guiding the operator to the most efficient use of the equipment.

Digital image acquisition and processing procedures for quantification of pipe wall thickness were implemented as a routine method for industrial inspection applications. The latest developments incorporated into the system involve automatic quantitative assessment of digital X-ray images not only of the pipe wall thickness, but also of the thickness of external and internal deposits. This permits the pipe clearance to be assessed at proper time intervals. The results led to significant improvements in the efficiency of industrial inspection of pipes and similar structures.



Digitised X-radiograph of outer part of an insulated pipe with internal deposit. The pipe wall thickness and the thickness of the internal deposit are calculated from the profile across the outer wall. In this case, the pipe thickness is 12.3 mm and the thickness of the deposit 10.4 mm. The location of the characteristic points within the profile is indicated by the vertical lines.

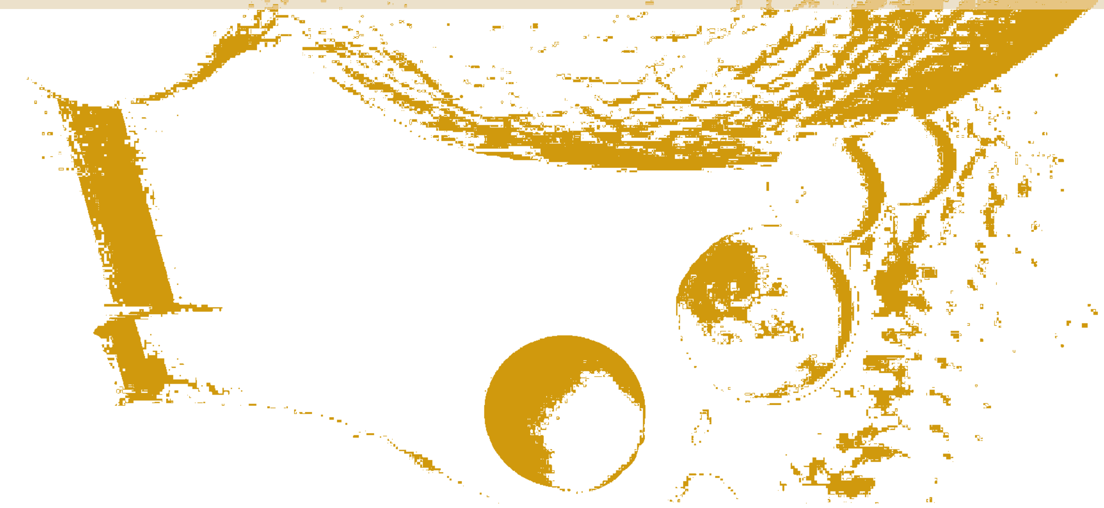
MATERIALS TECHNOLOGY
– synthesis, processing and product

Many projects described under this heading are conducted in collaboration with industrial enterprises in Denmark and abroad. Consequently, the majority of projects have goals not more than three years ahead. Very often activities are initiated by needs or problems encountered in industry, but conceived to be better solved in the research environment of the Department.

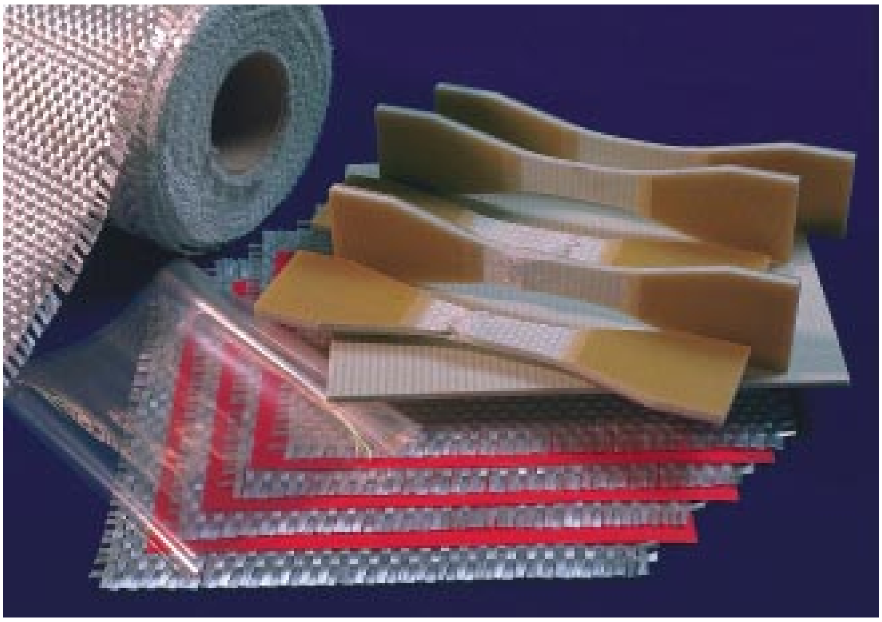
The area also covers more fundamental activities with the character of long term basic research. These projects are often carried out in collaboration with universities in Denmark, frequently as Ph.D. projects.
The activities in this area are described under the programmes *Manufacturing Technologies for Advanced Fibre Composites* and *Powder Technological Materials.*
The highlights in the area of Materials Technology are
(i) the demonstration that vacuum consolidated polymer matrix composites from layers of fibre fabrics and polymer films are feasible, but inferior to commingled fibres,
(ii) the development of a numerical model for simulation of temperature profiles in deposited materials produced by spray-forming,
(iii) the fabrication of bulk amorphous alloys in large (several mm) dimensions,
(iv) the development of a cheap technique for fabrication of ceramic electrical noise filters.

Project Funded Research: Materials Technology

Project type	Project name	Co-participants
JOULE-THERMIE	New generation Wind Turbine Blade	• Bonus Energy A/S, Denmark • Kemijoki Yo, Finland • VTT, Finland • Garrad Hassan & Partners, Ltd., UK
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
MUP	Electro-Ceramic Functional Graded Materials	• AMP, Denmark • Haldor Topsøe A/S, Denmark • PBI-Dansensor A/S, Denmark • Chemical Institute, KU, Denmark
Nordtest	Fractography of Cyclically Deformed Alumina Ceramics	• Swedish National Testing and Research Institute, Sweden • University of Tampere, Finland



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



Dog-bone fatigue test specimens cut from a 5 mm thick film stacked laminate. The film stacking lay up was established by alternate stacking of glass fibre fabrics and polypropylene (PP) polymer film. On the picture the PP polymer film is illustrated by the red layers between the glass fibre fabrics. A consolidation pressure of 0.1 MPa is easy and cheap to obtain by vacuum bagging the film stacked laminate, and a long time (hours) at the process temperature should make it possible for the melted PP polymer film to flow into the glass fibre bundles in the fabrics.

Evaluation of film stacked thermoplastic composites for wind turbine blades

The high melt viscosity of thermoplastic polymers means that it is difficult for the molten plastic to penetrate the fibre bundles and ensure a complete wetting of all individual fibres. The process technology must either apply a relatively high pressure to force the matrix material into the fibre bundles or start from pre-processed raw material where the polymer and the fibres are already thoroughly mixed.

Vacuum consolidation of film stacked thermoplastic fibre composite (alternating layers of polymer films and fibre fabrics) seems to be an attractive and possible process technology for wind turbine blades. A consolidation pressure of 0.1 MPa (1 bar) is easy and cheap to obtain by vacuum bagging the film stacked laminate, and a long time (several hours) at the process temperature should make it possible for the melted polymer film to flow into the fibre bundles in the fibre fabrics.

Ten different glass fibre fabrics and seven different polymer films for thermoplastic composites were tested and evaluated for the manufacturing of wind turbine blades. Heat treatment of the glass fibre fabrics under vacuum to three different process temperatures (200, 240 and 280 °C) was performed. Two different types of thermoplastic composites manufactured from a mixture of glass fibres and thermoplastic fibres (so-called commingled fibres) were also included in the investigation.

Evaluation of the laminate composites with respect to their fibre content, porosity and mechanical properties is in progress. Preliminary observations indicate that it is easier to obtain high materials quality by using commingled fibres instead of stacked polymer films and fibre fabrics. Only one combination of film stacked material matches the performance of commingled materials.

Polyethylene fibre PMCs for high-speed impact characterisation

Laminates of thermoplastic polymer matrix composites (PMCs) are used as backing plates for ceramic tiles in light weight armour for ballistic protection. Optimisation of the design and materials selection for such armour can be based on finite element simulations of high-speed impact. An adequate description of the mechanical properties of the PMC (a material model) is an important input to such a program.

For characterisation of the mechanical properties of high density polyethylene fibre composites, laminates and panels of 2 mm, 4 mm and 35 mm thickness were autoclave consolidated. Laminates with both [0 °] and [0 °/ 90 °] fibre orientations were manufactured from [0 °] PE/kraton prepreg. The prepreg consists of polyethylene fibre in a matrix of rubber compound (styrene-isoprene block copolymer). The laminates and panels were autoclave consolidated at a temperature of 127 °C

Three different types of semi raw materials based on natural fibres with the press consolidated laminate in between. Hemp is at the bottom, jute in the middle and wood fibres at the top. The hat shaped specimen at the very top is press consolidated from a jute fibre mat.

and a pressure of 1.9 MPa. In order to protect the polyethylene fibres from degradation and melting, it is important that the temperature in the laminate does not exceed 130 °C during the process.

Fast processing of PMCs with natural fibres

Industry is interested in natural fibres not only because they are a renewable resource, but also because they have good strength and weight properties. High performance bast fibres from flax, hemp and jute can offer a real alternative to glass and synthetic fibres in polymer matrix composites (PMCs).

Semi-products are an important means for efficient handling of fibres; mats of randomly oriented fibre configuration are typical semi-products for efficient fabrication of composites. Such mats influence not only the mechanical properties, but also the ease of handling of the material, the degree of compaction and thereby the maximum fibre content in the laminate and the material quality (low porosity).

Economical mass production of components requires short processing times at low process temperatures. Natural fibres have limited heat resistance. Use of thermoplastic polymer matrices with low melting temperature and low viscosity is therefore desirable. Some initial investigations were conducted on press consolidation of film stacked natural fibre composites. Alternate stacking of jute fibre mats and polypropylene (PP) foils constituted the lay-ups. The influence of the fibre content on the material quality was investigated at four levels by adding different numbers of PP foils (matrix material) between three layers of jute fibre mats. The lay-ups, with an area of 250 mm x 300 mm each, were heated at 220 °C for 15 minutes and subsequently press consolidated with a force of 50 kN for 30 seconds.

The four jute fibre weight contents of the laminates were 37 %, 43 %, 50 % and 55 % and the measured volume fractions of porosity were 6 %, 10 %, 18 % and 26 %, respectively. The result showed that it is difficult to obtain a good material quality with mats consisting of randomly oriented fibres if the jute fibre weight content is higher than approximately 40 %. The material quality for the press consolidated laminates with a weight content of 37- 43% jute fibres is similar to that achieved by a four hour autoclave consolidation process with a similar pressure of 0.6 MPa.

Powder Technology Materials

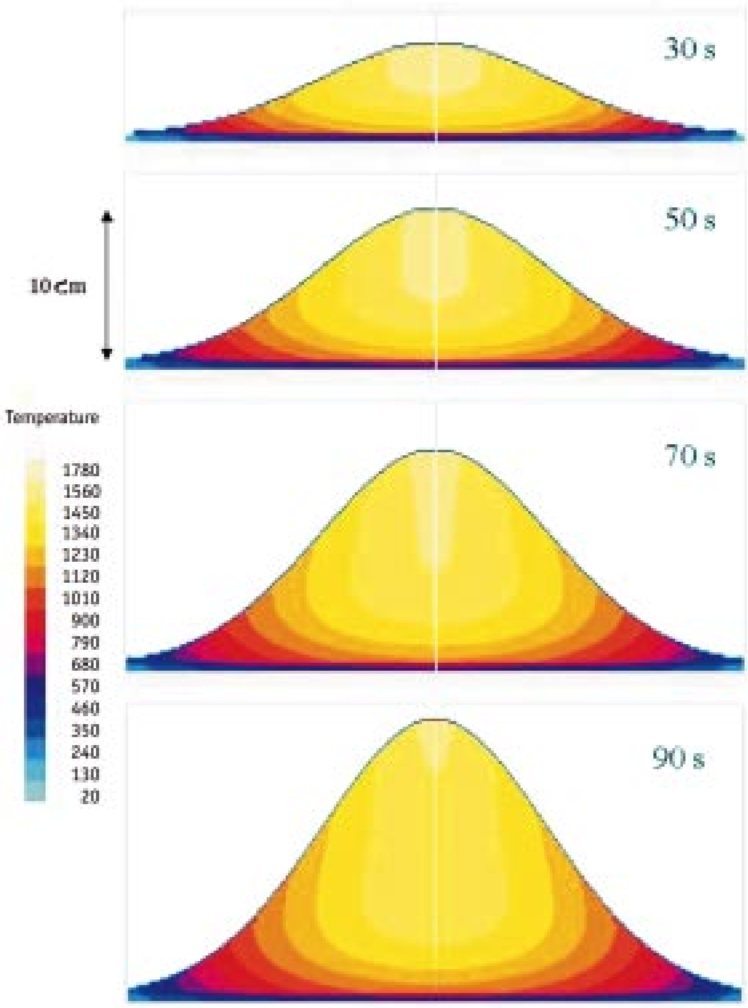
This programme is concerned with theoretical and experimental aspects of the production of metal powders and of materials from metallic and ceramic powders. Optimisation of the spray forming equipment in parallel with numerical simulation of the spray forming process has been an important task in 1998. The range of materials covered includes also metal matrix composites for applications where wear and friction resistance is critical.

The work on ceramics has been focussed on development of functionally graded electroceramics for two main applications: oxygen sensors (based on semiconducting oxides) and electronic noise filters that incorporate multilayer ceramics. A major problem in the second application is thermally induced delamination. A fracture mechanics model developed to overcome these problems has proved useful and versatile.

Work on rapid solidification via gas atomisation and spray forming has generated an important spin off aiming at the production of bulk metallic glasses. To date castings of dimensions in the range 3 x 40 x 60 mm^3 have been successfully produced. The perspectives for amorphous metals are very promising both technologically and scientifically.

Numerical simulation of the spray-forming process

A mathematical model has been developed for treating powder particles accelerated by a gas and for simulating their thermal behaviour during flight. In most existing models dealing with the solidification of metal droplets, the dynamic and the thermal behaviour of such droplets is only simulated for individual droplets. In the present study the interaction between an array of droplets and the surrounding gas was calculated numerically. This approach allows the effect of process parameters on the size distribution, temperature, velocity histories, solid fraction and cooling rate to be predicted, for droplet sizes covering the complete droplet size distribution. The validity of the model



Top: The simulated build up of the material and the deposit temperature during spray forming of a rounded structure. Bottom: Spray deposited cylindrical structure (Diameter 8 cm, height 20 cm). The different shapes are obtained by variation of the spraying parameters.

has been investigated by comparing experimental and calculated results for powder particles of 12Cr-Mo-V steel. The level of agreement obtained allows quantitative conclusions and guidelines for process optimisation to be drawn.

Current work is focussed on the computation of the temperature and the solidification rate in the growing deposit during and after deposition as well as the complex physical phenomena posed by the impact of a droplet onto a target. These phenomena take place simultaneously on a microscopic scale and involve fluid flow, heat transfer and rapid solidification. This study will provide a better understanding of the morphological characteristics and the adhesion of the deposit surface at impact, which largely determines the microstructure and mechanical properties of the spray-formed materials.

Spray-forming of steel-based alloys and composites

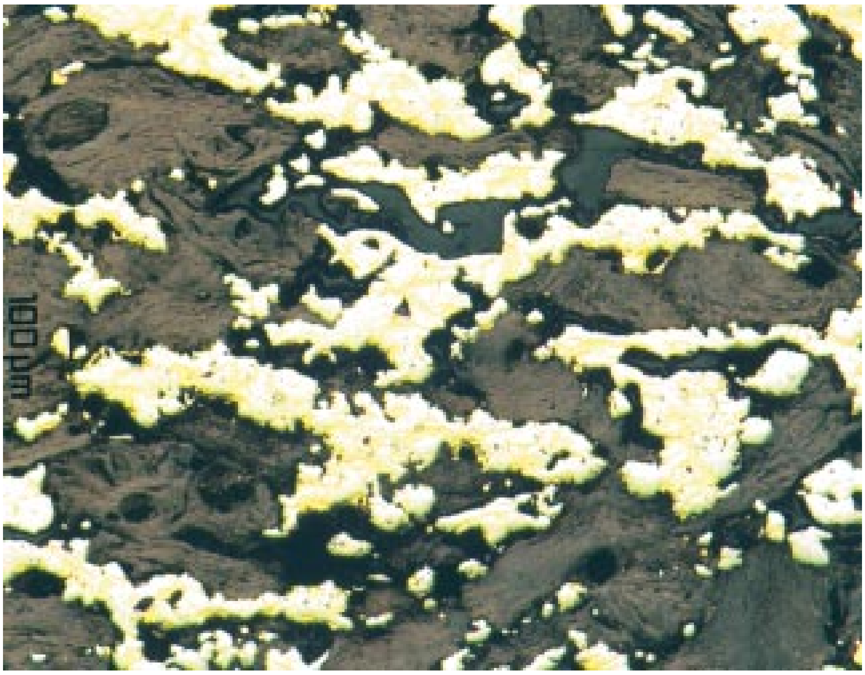
The existing spray-forming equipment in the Department has been optimised. The improved control of the process parameters has resulted in materials of a very good quality and appearance.

A series of chromium containing steel alloys (high as well as low alloyed) were prepared. These had fine, low porosities in the core areas, depending on melting interval and melt properties such as fluidity, surface tension etc. The products were characterised in terms of microstructure and hardness before and after heat treatment. The high chromium alloys could not be machined in the as-sprayed state, but careful heat treatment improved their properties. Much of the characterisation work was done by a master degree student as part of collaborative effort with the University of Bremen, Germany.

A crucial aspect of the spray-forming work is the addition of ceramic particles to the spray plume during processing. Little information on this subject is available in the open literature and this is certainly not conclusive. As a result, different methods for mixing gas and ceramic particles were explored in small experimental set-ups. The optimal method was selected and is now under construction for installation in the spray former. The system comprises a separate gas/solid mixing chamber with injection points midway between the atomising gas nozzle and the target.

Metal-matrix composites for friction applications

In pursuit of materials for heavy duty friction applications, such as brakepads for land transport vehicles, new metal-matrix composites (MMCs) have been developed. These materials are capable of working at higher temperatures and energy dissipation rates than the conventional polymer-based materials and are more environmentally acceptable. Typically, they are produced by sintering of metal, graphite, and ceramic



Optical micrograph of metal-matrix composite, showing regions of steel (white), bronze (yellow) and graphite (grey-green). The image covers an area of about 2 x 3 mm².

Electrical characteristics of a multilayer feed-through filter fabricated at Risø and measured at 50 Ω. These are comparable to those of an ideal filter at frequencies up to 1-2 GHz.

Parameter	Value
Capacitance	1.4 nF
Inductance	50 nH
Insertion loss	tan δ < 0.5 %
Resistance	> 5×10^4 MΩ

powder mixtures. At present, however, understanding of the correlations between the constituents, processing parameters and the materials properties is limited. The Department has been investigating the subject in collaboration with an industry and the Danish Technological Institute within the programme *COMPOMET*. The work has focussed on characterising the behaviour of single constituents and of composites, under conditions simulating processing and use, using techniques such as XRD, TGA/DTA, dilatometry, optical microscopy and high-temperature ESEM. The study has covered the sintering behaviour of these materials and revealed their interesting, anisotropic microstructures.

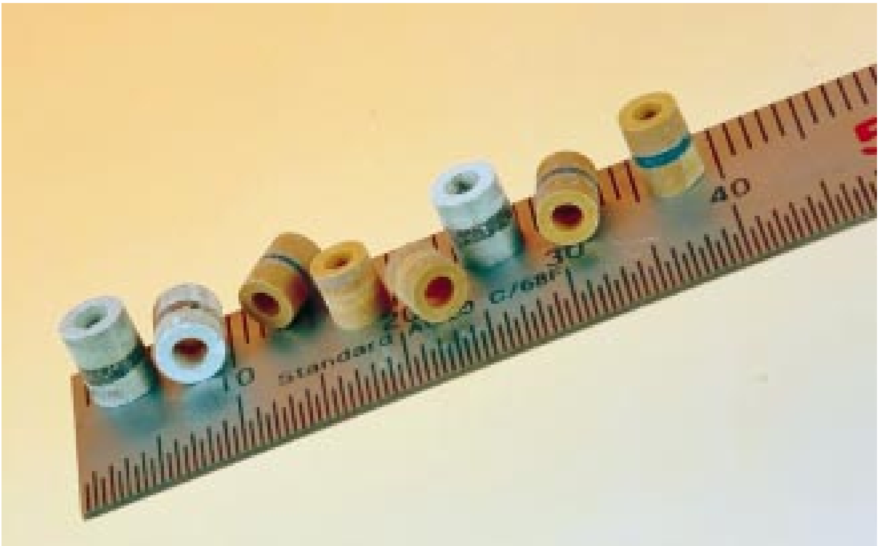
Fabrication of electronic noise filters

The main objective of this project is to develop a new process for fabricating low cost miniaturised electronic L- and Pi- noise filters. Functionally graded materials (FGM) have been used, as they allow the fabrication of components with a combination of properties that cannot be obtained in conventional, monolithic materials. In the present application, the FGM consists of layers of graded capacitive (dielectric) and inductive (ferrite) materials.

The fabrication of these structures presents problems of thermally induced delamination. A fracture mechanics model, developed within the project, showed that this problem could be solved only by using materials with a very small thermal expansion mismatch. Using a lead-based Pb $(Mg_{1/3}Nb_{2/3})O_3$ dielectric and a modified Ni/Zn ferrite, it was possible to reduce the thermal mismatch parameter to 0.3×10^{-6} K^{-1} and thus prevent thermally induced delamination. Prototype Pi-filters, consisting of two dielectrics and one ferrite, were fabricated and tested electrically. The physical properties of the symmetric multilayers match those of state-of-the-art commercial products. The uniaxial pressing used in this project is a very simple ceramic forming method and, hence, attractive for industrial use.

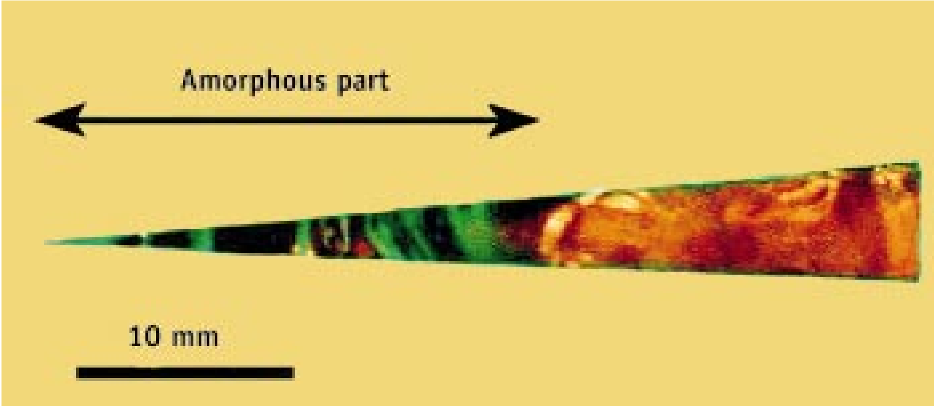
Ceramic oxygen sensors

The development of ceramic oxygen sensors based on Mg-doped $SrTiO_3$ was continued. This year saw further devel-



Prototype of electronic noise filter developed in collaboration with AMP Danmark. The filter consists of functionally graded ceramics and is produced by a cheap technique.

opment of the screen printing technique for the fabrication of sensors and of integrated sensors, which incorporate a heater. Tests showed that temperatures up to 650 °C could be obtained with a power consumption of only a few watt. A general problem with any oxygen sensor is the cross sensitivity toward other gases. As water is one of the most commonly encountered contaminants, the sensors were tested in humid atmospheres. The results showed that the resistance of the sensors generally decreased with water content in the carrier gas, indicating that partial proton conductivity was induced. However, the sensors' response toward changes in the oxygen partial pressure (log R ∝ $^1/_4$ log P_{02}) remained valid, demonstrating that they could be used in humid as well as dry atmospheres. Finally, a dipcoating technique was developed for the fabrication of yttria-doped zirconia tubes for oxygen sensors.



Mg-Cu-Y-Al bulk amorphous alloys were prepared by casting the material into a wedge copper mould. At the lower part of the cast material an amorphous part was often observed owing to the high cooling rate in this region.

Fabrication and properties of bulk Mg-Al-Cu-Y amorphous alloys

Mg-Cu-Y bulk amorphous alloys were discovered in 1993. These alloys show a large glass forming ability, so that mm sized specimens of the composition $Mg_{65}Cu_{25}Y_{10}$ have been obtained in amorphous, single phase form. Although Mg-based alloys generally have an inferior glass forming ability compared with Zr- and Pd-based ones, they have the advantages of low weight and cost.

A bulk amorphous alloy of composition $Mg_{60}Cu_{30}Y_{10}$ has been fabricated by chill-casting using a relatively simple experimental procedure, based on an induction furnace with control of the melt temperature. The melt was cast into a wedge shaped copper mould.

To ensure homogenisation of the master alloy while minimising the evaporation of magnesium, the products were melted and solidified several times. Finally, the melt was quenched into the mould from a temperature just above the liquidus. In this way a $Mg_{60}Cu_{30}Y_{10}$ bulk amorphous plate with a thickness of 2 mm was obtained. The Vicker´s hardness of the as-quenched alloy was found to be relatively high, about 290, and the material was rather brittle. To improve the mechanical properties while maintaining low specific gravity, small amounts of Al were added. This modified alloy, of composition $(Mg_{0.98}Al_{0.02})_{60}Cu_{30}Y_{10}$ could be cast into a plate of thickness 1.5 mm. Hardness measurements showed that the addition of Al improved both the ductility and the strength.

A series of alloys of composition $(Mg_{(1-x)}Al_x)_{60}Cu_{30}Y_{10}$ (x=0 to 0.07) was made in the wedge-shaped mould for further investigation of the effect of Al on the glass forming ability, thermal stability, mechanical properties and uniformity. This work has been done in collaboration with National Research Institute for Metals, Tsukuba, Japan.

Manufacturing of low enriched fuel elements

Fuel elements for the Danish research reactor DR3 at Risø have for almost 25 years been manufactured in a small production plant within the Department. A total of about 1000 elements have been produced. The yearly production is 30 - 40 elements, and for more than 10 years all have been made as LEU elements – containing Low Enriched Uranium, with less than 20 % U_{235}.

Each element consists of five concentric tubes. The tubes look alike and the wall thickness is 1.5 mm for all tubes. The outer tube is made of solid aluminium while the rest are made as sandwich plates so that only the surface is of aluminium. The core inside the tube wall contains the fuel - U_3Si_2. Each tube is made of three plates welded together. Each plate is a hot-rolled sandwich of three layers metallurgically bonded together. In the intermediate layer the sandwich holds a cold pressed brick made of a mixture of U_3Si_2 and Al powder.

Four LEU elements. Each tube has a diameter of 10 cm and is 65 cm high.

MATERIALS CHEMISTRY
- combined science and technology

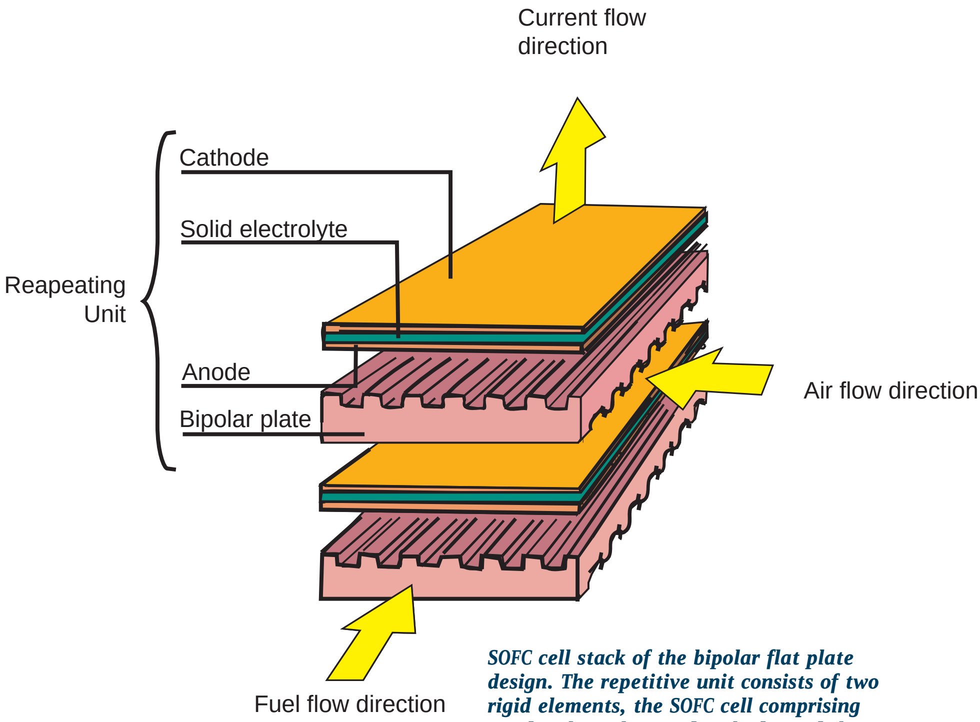
The majority of research in the Materials Chemistry area concerns electroceramics. The largest programme in the area is the fuel cell programme that aims at developing Solid Oxide Fuel Cells (SOFC). The programme spans all the way from synthesis and characterisation of cell materials over manufacture of cells to assembly and testing of small cell stacks. Fundamental research supporting the technological development is carried out in solid state chemistry and ceramic processing.
Highlights from the past year are
(i) the identification of an additive to the Ni/YSZ-anode that improves the electrochemical performance and in-plane conductivity, while reducing the steam reforming activity to methane,
(ii) the development and demonstration of a new type of cell based on an anode-supported thin electrolyte for operation at low temperature. A cell internal resistance of 0.4 $\Omega \cdot cm^2$ at 850 °C was achieved. The cell is produced by simple, scalable wet processes,
(iii) the development of cells based on anodes of doped ceria, that are tolerant to redox-cycling as well as thermal cycling,
(iv) improved understanding of the effects of impurities on the long term stability of the electrolyte.

Project Funded Research: Materials Chemistry

Project type	Project name	Co-participants
Ministry of Environment and Energy (EFP)	DK-SOFC 1996-1998	• Haldor Topsøe A/S, Denmark • IRD A/S, Denmark • Institute of Chemistry, OU, Denmark • Institute of Chemistry, DTU, Denmark
JOULE-THERMIE	Improving Durability of SOFC Stacks (IDUSOFC)	• Research Centre Jülich, Germany • ECN, Petten, The Netherlands • Imperial College, London, UK • University of Oslo, Norway • Siemens GmbH, Germany • Haldor Topsøe A/S, Denmark • Rolls-Royce, UK • Statoil a.s., Norway
BRITE-EURAM	Low Cost Fabrication and Improved Performance of SOFC Stack Components (LOCOSOFC)	• IRD A/S, Denmark • Rolls-Royce, UK • INPG, France • Gaz de France, France • NUVL, UK • EPFL, Switzerland
New Energy Development Organization, Japan (NEDO)	Advanced Ceramics for Protonics	• Nagoya University, Japan • Touhoku University, Japan • University of Pensylvania, USA • University of Missouri-Rolla, USA • Research Centre Jülich, Germany • TYK Corporation, Japan
Training and Mobility of Researchers Programme	Alternative Anodes for SOFC	• University of St. Andrews, UK • University of Aveiro, Portugal • University of Patras, Greece • British Gas, UK • University of Twente, The Netherlands • Research Centre Jülich, Germany

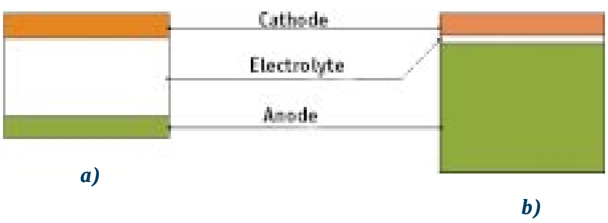
Solid oxide fuel cells are all-solid-state devices for generation of electric power by electrochemical conversion of a fuel, normally hydrogen or methane. The technology, which is currently at a pre-commercial level, has the potential of more efficient generation of energy with lower levels of pollution than traditional combustion technologies. The fuel cell programme has the aim to establish a base for a Danish production of SOFC components and consists of three parts: (i) Fundamental research in solid state chemistry, including crystallography, defect chemistry and electrochemistry. (ii) Fabrication of electroceramic components, using careful process control to obtain components with predetermined properties; only cheap fabrication routes with the possibility of scale up are under consideration.
(iii) Advanced characterisation, including determination of fundamental materials properties (of powders, ceramics and fabricated electrodes) and cell and stack performance (electrical and mechanical).

The expertise established in the fuel cell programme may find use also for development of other electroceramic components such as gas sensors and membranes permeable to oxygen or hydrogen. Materials of interest for electrochemical membrane reactors were prepared and characterised.



SOFC cell stack of the bipolar flat plate design. The repetitive unit consists of two rigid elements, the SOFC cell comprising anode, electrolyte and cathode, and the interconnect or bipolar plate, providing gas separation, gas channels and electrical contact between cells.

Newly developed anode supported cell with high flexibility, high strength and very low internal resistance (0.4 Ω·cm² at 850 °C). (Patent pending)



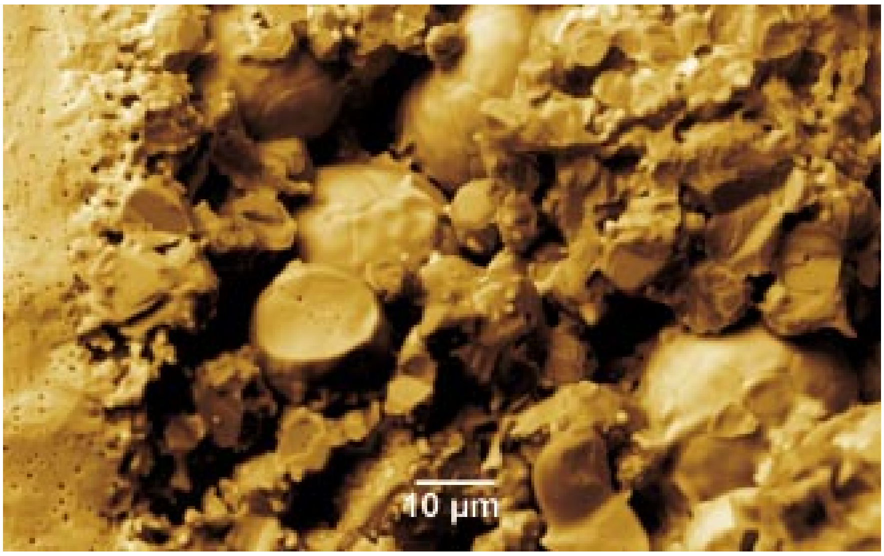
Schematic diagram of a cell (a) electrolyte supported (b) anode supported cell, for operation at lower temperatures.

Development of thin electrolyte solid oxide fuel cells

In the Danish SOFC programme, a bipolar flat plate design is being pursued. The key advantage of this design is a short current path with minimum electrical losses. The internal cell resistance comprises polarisation resistance in the electrodes and ohmic losses in the electrolyte.

The pressure of commercialisation demands a reduction in the cost of SOFC materials and an improvement in the durability of the assembled stacks. Lowering the operating temperature offers the possibility of using inexpensive, machinable, metallic interconnect materials as opposed to ceramics. Lower operating temperatures, however, cause an increase in the internal cell resistance and, therefore, require an improvement of the cell performance. With an electrolyte-supported cell design and the present electrodes, the electrolyte becomes the limiting factor for the cell performance at low operating temperatures. Therefore, a design with thin electrolyte is required and this electrolyte must be supported on one of the electrodes.

In 1998 an anode-supported cell has been developed for operation at 750 to 850 °C. It is produced by simple, scalable, wet ceramic processes. The cell



Low vacuum scanning electron micrograph of improved Ni/YSZ anode with YSZ electrolyte on left.

has higher mechanical strength and is more flexible than the previously produced electrolyte supported cells. Internal cell resistances as low as 0.4 Ω·cm² have been obtained on 25 cm² cells at 850 °C using hydrogen as the fuel. Further means to lower the internal cell resistance have been identified, and some are being tested at the moment. The possibility for patenting these cells is being investigated.

Anodes

For more than a decade the state-of-the-art SOFC anode has been a Ni/YSZ cermet which is characterised by low polarisation resistance and excellent chemical compatibility with other stack components.

Ni/YSZ cermet anodes have recently been further improved by addition of selected inexpensive transition elements. It was shown that by such additions to the Ni/YSZ cermet, the polarisation resistance can be reduced by a factor 2 to 3 and the resistivity of the current collecting layers of an electrode by a factor of 25 to 30. Exploiting parts of these results, anodes were fabricated that had a polarisation resistance of less than 60 mΩ·cm² at 850 °C. A patent on this modification is pending.

The major disadvantages of Ni-based anodes are the Ni-NiO phase transition upon redox cycling and the tendency towards carbon deposition during operation in methane. In the event of an accidental failure of the fuel supply to an SOFC or for operation in an electrolyser, the volume expansion due to Ni oxidation may cause disintegration of the electrode. Alternative anodes based on doped ceria and doped lanthanum chromites with good redox stability were developed and characterised. For the chromites, the polarisation resistance is about an order of magnitude higher than for state-of-the-art Ni/YSZ cermets; the ceria anodes have a performance similar to that of Ni/YSZ anodes. Both types of electrodes are relatively inactive in terms of carbon deposition during operation in methane.

Sealing materials

Gas seals are used to prevent mixing of the fuel gas with air and, therefore, are a vital issue in the development of SOFC stacks. A successful sealing material has to meet a number of tough chemical and physical requirements. In the Danish SOFC programme, glass or glass with ceramic fillers are the preferred sealing materials because of their softness at elevated temperatures. In the course of development it was found that systems having B_2O_3 or P_2O_5 as the main glass formers did not possess the required chemical stability and the work was thus concentrated on SiO_2 as glass former.

Compositions within the alkali-alumina-silicate system were found to provide excellent chemical stability as well as extremely low rates of crystalisation. Even after 6000 hours at 1000 °C under simulated anode conditions (i.e. in hydrogen containing atmospheres), no sign of crystallisation was observed. Furthermore, analysis of the interfaces between glass and other stack components did not reveal any evidence for solid state or vapour phase interaction.

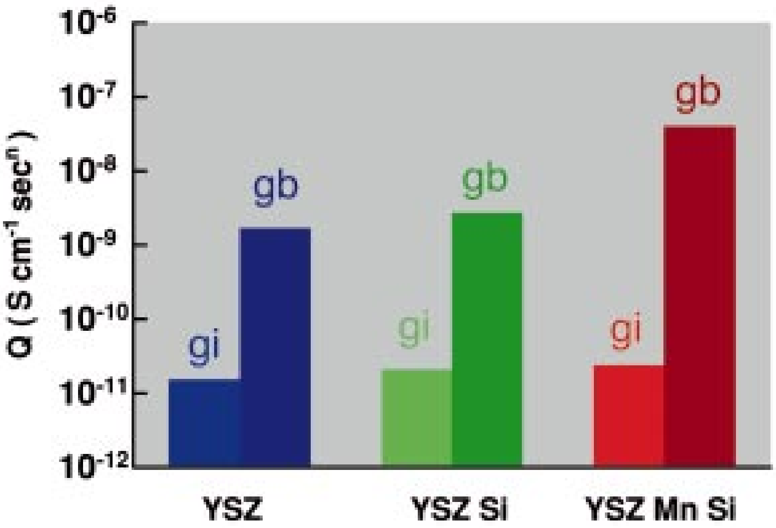
The properties of the sealing materials, e.g. thermal expansion coefficient, are easily tailored by changing the alkali concentration and/or by addition of ceramic filler materials, e.g. MgO, YSZ or Al_2O_3. Ceramic filler additions provide further benefits of an improved chemical stability and an increased viscosity of the sealant. The composite sealing materials developed have demonstrated excellent performance during stack testing. A patent application on these materials has been filed in the US.

Interactions of impurities in YSZ

As part of the effort to improve the durability in SOFCs, the effect of common contaminants of zirconia ceramic electrolytes has been investigated. Silicon has long been recognised as a particularly undesirable element in YSZ ceramics because it forms liquid phases at grain boundaries, thus blocking oxide ion transport.

SiO_2 was added to high purity YSZ at the level of 0.1 wt% and the material is sintered at the relatively low temperature of 1350 °C. The silica remained in the form of discrete crystalline particles and, therefore, did not degrade the ionic conductivity of the material. Additions of up to 2 atom % manganese also left the electrical properties unaffected.

However, the addition of SiO_2 and Mn together produced an amorphous phase completely covering the grain boundaries and causing electrical block-



Non-ideal capacitance elements, Q, measured for grain interior (gi) and grain boundaries (gb) on pure YSZ and on YSZ with deliberate additions of silica and manganese plus silica. The magnitude of Q_{gb} is increased by a factor of ten when both contaminants are present.

ing, as shown by an increased grain boundary resistance and capacitance. Understanding such impurity interactions can provide a basis for controlling degradation processes in SOFCs.

Sensors for flue gas analysis

Using techniques established within the SOFC programme, a ceramic electrode material was developed for zirconia-based oxygen sensors used for flue gas analysis in power plants. Unlike the metallic electrodes conventionally used in such sensors, this system is tolerant to CO and SO_2 contained in the flue gas. A sensor of this type was successfully tested at the Asnæs power station in Denmark.

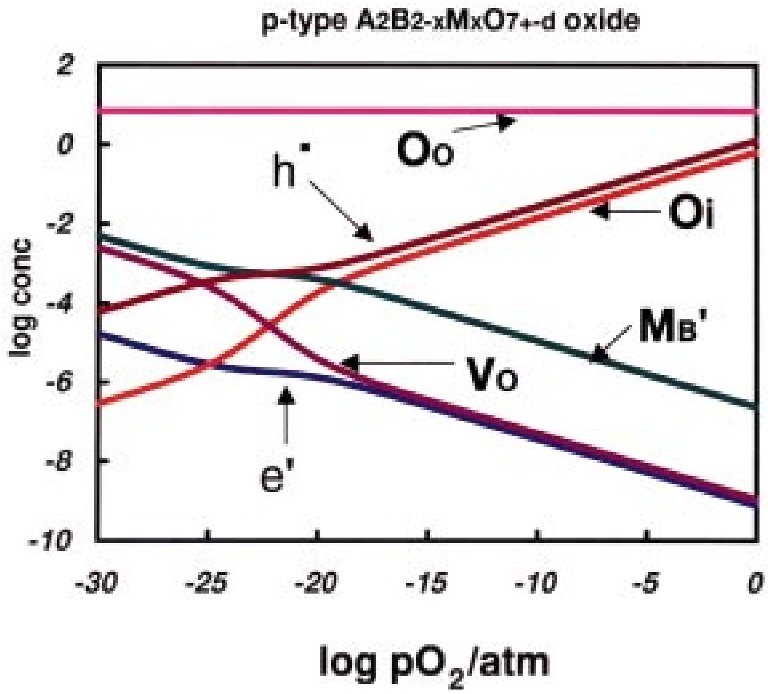
Materials exhibiting mixed ionic/electronic conduction were synthesised for possible use in membrane reactors and studied experimentally. Membrane reactors can be used for the production of a H_2/CO mixture, known as syn-gas. The prospects and problems involving mixed conductors in an intense gradient of oxygen activity were analysed using mathematical models developed for other aspects of SOFC work.

Modelling of electrical and chemical properties of oxide ceramics

Oxides in use at elevated temperatures will adjust their oxygen stoichiometry according to the oxygen pressure in the surrounding atmosphere and this affects their electrical, mechanical and dimensional properties. By understanding the underlying solid state defect chemistry these properties may be predicted. The use of oxides in solid oxide fuel cells and sensors depends on the selection and tailoring of these properties via doping.

The defect chemistry equations for five types of oxide structure, namely rocksalt, fluorite, perovskite, pyrochlore, and the so called Balachandran layered oxides were solved by a new method. The algorithm sequentially solves a set of linear equations, arising from charge and site balance, and non-linear ones arising from mass law expressions. Contrary to traditional defect chemistry methods, this numerical solution avoids the need to divide the calculation into sub-regions and use different approximations for each region.

The transition region between the substoichiometric and overstoichiometric regimes, which may extend over several decades of oxygen partial pressure, thus becomes the subject of analysis and prediction. The concentrations of oxide ion vacancies, electrons and holes is crucial in determining the diffusion rate of oxygen in the oxide, as well as the growth rate of oxide layers on metals.



Calculated concentrations of point defects in a B-site doped pyrochlore oxide as a function of oxygen partial pressure (pO_2). O_O: normal oxygen; V_O: Oxygen vacancy; O_i: oxygen interstitial; $h^\bullet$: electron hole; e': electron; M_B': dopant ion in reduced state.

FUSION MATERIALS
– defects and properties

The overall objective of the work in this area is to investigate (a) aspects of defect production and accumulation in metals and alloys irradiated with energetic particles under different conditions and (b) the impact of this radiation damage on the physical and mechanical properties of the materials. The activities cover a wide range of theoretical, simulation and experimental activities in a close international collaboration. The work is carried out within the Association Euratom – Risø National Laboratory.

Highlights from this year include
(i) the development of the production bias concept into a general theory to describe the damage accumulation during irradiation,
(ii) the establishment of void formation in pure iron already at an irradiation temperature of 100 °C and a dose level of ~ 0.2 displacement per atom.

Project Funded Research: Irradiated Materials

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

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

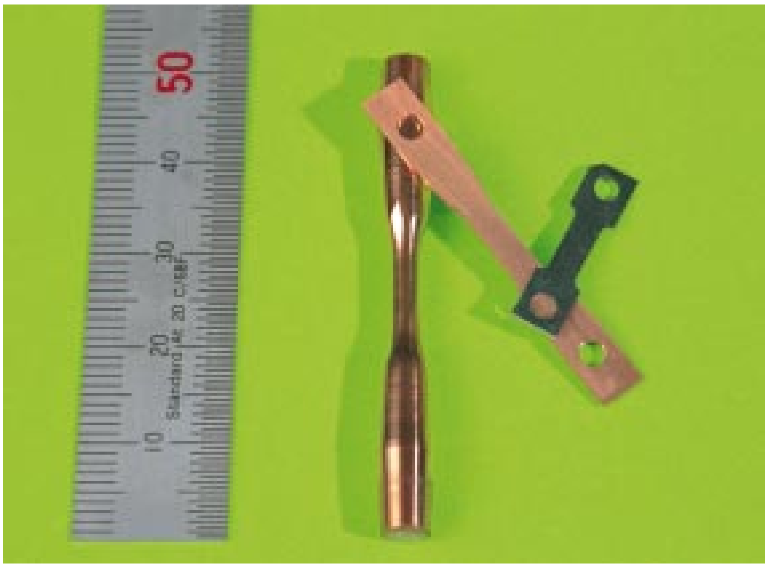
The investigations carried out in the field of the Next Step Technology constitute a part of the European contribution to the International Fusion Technology Programme aiming at design and construction of the International Thermonuclear Experimental Reactor (ITER). The activities in the Department in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

The Long Term Technology Programme covers all activities dealing with the problems related to design and construction of the DEMO reactor (a demonstration fusion reactor). At Risø, irradiation effects on iron and low activation steels, which are considered as construction materials, have been studied.

Finally, the programme has activities within the Underlying European Fusion Technology Programme, where more fundamental aspects of irradiated materials are investigated.

The programme covers both experimental work, e.g. irradiation experiments in the DR3 reactor at Risø, and theoretical and simulation studies that are carried out in collaboration with scientists from Europe, Russia, and USA.

In 1998, a workshop at Risø on 'Damage Production and Accumulation under Cascade Damage Conditions' and a workshop in Spain on 'Basic Aspects of Differences in Irradiation Effects between fcc, bcc and hcp Metals and Alloys' were organised. The programme was invited to participate in a collaboration programme with the newly established 'Academic Frontier Research Center for Ultra-High-Speed Deformation' in Hiroshima, Japan.



Specimens of irradiated and unirradiated copper and copper alloys for characterisation of mechanical properties.

Mechanical properties of pre- and post-irradiated copper alloys

The vacuum vessel components (e.g. first wall and divertor) in ITER will experience thermal-mechanical cycling as a result of the cyclic plasma burn operation of the system. Therefore the materials used must be able to withstand a large number of loading cycles. The tensile and fracture toughness properties are also important for the applicability of the copper alloys, in particular the effect of neutron irradiation on the fracture toughness.

In view of this, the mechanical properties of precipitation hardened CuCrZr and dispersion strengthened Cu-Al_2O_3 (both unirradiated and irradiated) have been investigated. The fatigue life was measured in vacuum (<10^{-5} torr) with a fully reversed loading frequency of 0.5 Hz in the strain-controlled mode at 250 °C. Irradiation causes a decrease of the fatigue life for all samples, even at a dose level of only 0.1 dpa. For low strain amplitudes (< 0.3 %) the fatigue life of CuCrZr was lower than for CuAl-25 (0.25 weight% Al) but this was reversed at higher amplitudes. An increase of the Al_2O_3 content to 0.6 % Al lead to significant fatigue life reduction.

Tensile and fracture toughness properties were determined in collaboration with VTT Manufacturing Technology in Finland. CuCrZr and CuAl-25 were irradiated with fission neutrons at ~ 50, 200 and 350 °C to a dose level of ~ 0.3 dpa. Tensile testing was performed in vacuum (~ 10^{-5} torr) in the Department. Both alloys exhibited irradiation-induced increase in the upper yield stress followed by plastic instability (i.e. negative work hardening) at irradiation temperatures up to 200 °C. At 350 °C, on the other hand, the deformation behaviour of both alloys in the irradiated condition was quite normal, but there was evidence of irradiation-induced softening.

Fracture resistance curves were determined at VTT using displacement controlled three-point bend testing. CuAl-25 exhibited stable crack growth at all temperatures, whereas extensive crack tip blunting in CuCrZr occurred and stable crack growth was observed only at 350 °C. Both the irradiation fracture toughness for stable crack growth and tearing resistance values were higher for CuCrZr than for CuAl-25.

The fracture toughness decreased with increasing irradiation and test temperature in both alloys. The decrease was more rapid for CuAl-25 which at all temperatures had substantially lower fracture toughness than

CuCrZr. It is interesting that the tensile elongation of CuCrZr irradiated and tested at 350 °C was higher than for the unirradiated CuCrZr. The fracture toughness exhibited the opposite tendency and was lower in the irradiated CuCrZr. At present, there is no explanation for this.

Electrical conductivity of irradiated copper and copper alloys

In studies of radiation damage, the electrical conductivity may be used to characterise the defect population, since the conductivity depends on the defect types and densities.

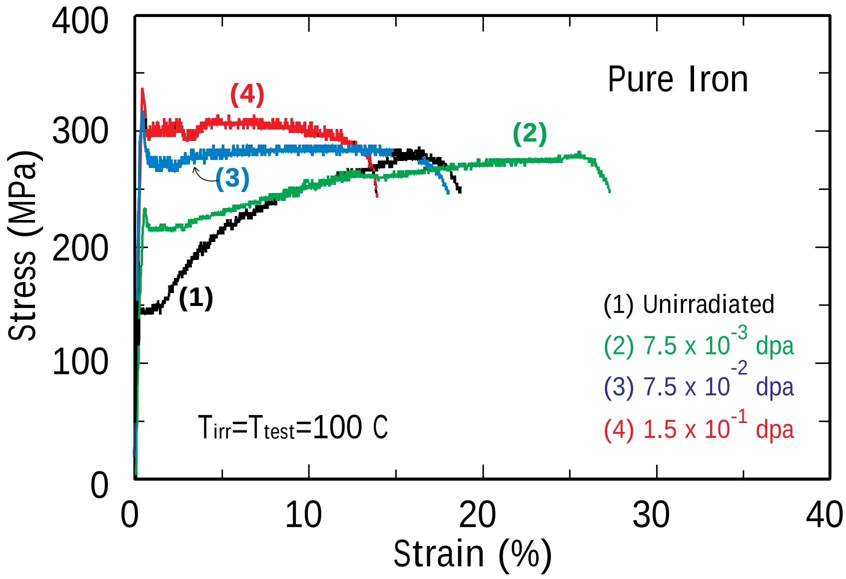
The electrical conductivity of high purity copper, neutron irradiated at different temperatures (100 – 350 °C) to a dose of 0.3 dpa turned out to be almost independent of the irradiation temperature, namely 90.5 ± 1.5 % of the conductivity of pure copper.

The sensitivity of the electrical conductivity to impurities or alloying elements was clearly demonstrated by results for copper-nickel alloys. For an alloy with 2 % Ni the conductivity was reduced to about 38 % of that for pure copper, while 5 % Ni reduced the conductivity to only 20 % of the copper value. Neither neutron irradiation (0.3 dpa) nor long-time post-irradiation annealing (300 °C for 50 hours) led to any change in these values. This suggests that the conductivity of copper would decrease during irradiation since Ni will be produced continuously by nuclear reactions during irradiation.

Radiation hardening and embrittlement in pure iron and low activation steels

Effects of irradiation on physical and mechanical properties of low activation steels (F82H and MANET-2), which may be used for components of DEMO, were studied as part of the European activities devoted to mechanistic studies of irradiation hardening and loss of ductility.

Pure iron and low activation steels were irradiated with fission neutrons at different temperatures in the range of 50 to 350 °C and to displacement doses between ~ 0.01 and ~ 0.2 dpa. Both



Stress-strain curves for pure iron irradiated and tensile tested at 100 °C. Note the increase in the upper yield stress and decrease in the work hardening rate with increasing dose. For doses higher than 0.15 dpa the upper yield stress decreases.

irradiated and unirradiated specimens were tensile tested in vacuum at the irradiation temperatures. Also, transmission electron microscopy was carried out and the fracture surfaces were examined by scanning electron microscopy. The irradiation-induced cluster density in pure iron irradiated at 50 °C was found to be considerably lower than that in pure copper irradiated under similar conditions. The increase in the upper yield stress due to irradiation was also smaller in iron than in copper. In pure iron irradiated at 100 °C, the upper yield stress was found to decrease above the dose level of ~ 0.15 dpa.

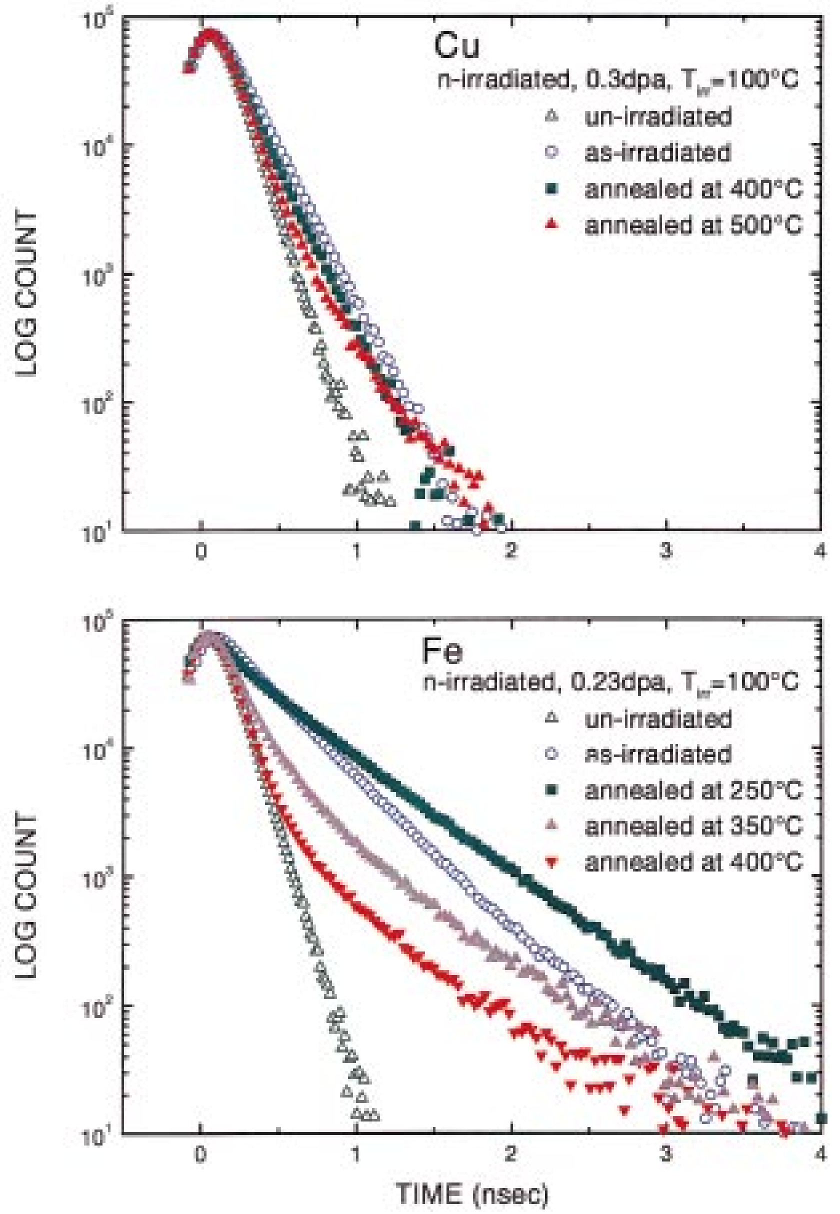
The effect of irradiation on microstructure and mechanical properties of F82H and MANET-2 steels was found to be rather small. In fact, the F82H steel irradiated at 350 °C was found to show irradiation-induced softening.

Comparison of irradiation created defects and their annealing behaviour in copper and iron

As a part of the ongoing efforts to obtain a detailed understanding of the defect creation and accumulation during irradiation, the annealing behaviour of copper (a face centred cubic metal) and iron (a body centred cubic metal) after neutron irradiation was compared. Specimens were neutron irradiated to a fluence of $1.5 \cdot 10^{24}$ neutrons/m^2, equivalent to doses of 0.3 displacements per atom (dpa) in copper and 0.23 dpa in iron. The irradiations were carried out at 100 °C which is above the annealing stage III (vacancy migration) and below the annealing stage V (vacancy evaporation from clusters) for both metals.

Positron annihilation spectroscopy (PAS) was used to characterise the defect populations. PAS is sensitive to defects such as vacancies, vacancy clusters, voids and gas bubbles. The lifetime of positrons that are trapped in three-dimensional vacancy clusters increases with cluster size in a size range of ~ 1 - 50 vacancies (i.e. sub-microscopic voids). The intensity of the lifetime increases with defect density (in a certain range). Positron lifetime measurements may therefore provide information about both cavity sizes and densities.

The measurements showed clear differences between the defect accumulation in copper and iron during neutron irradiation as well as in the annealing behaviour of the two metals. In copper the defect lifetime was ~ 180 ps, indicating that vacancy clusters had



Positron lifetime spectra for irradiated and annealed copper (fcc) and iron (bcc). The spectra show that in the irradiated specimens positron lifetimes are longer than in unirradiated ones. This is due to irradiation created defects that trap positrons. The spectra clearly show that in copper only rather short lifetimes are detected (indicative of vacancy clusters) while for iron much longer lifetimes show the presence of micro-voids and voids. On annealing of the iron, the average lifetime first increases (void coarsening) and then decreases (voids disappear).

formed on irradiation at 100 °C. These clusters annealed out at temperatures above ~ 250 °C (i.e. above Stage V) leaving a low density of cavities (probably helium bubbles).

In contrast to this, in iron a dominating lifetime of ~ 365 ps showed that mainly micro-voids (10 - 15 vacancies) were present in the as-irradiated specimens. On annealing, the micro-voids grow into voids that subsequently anneal out above ~ 300 °C.

In agreement with the PAS, electrical conductivity measurements showed that the recovery of the defect microstructure takes place in the temperature ranges 250 – 550 °C for copper and 150 – 400 °C for iron.

Analysis of defect accumulation in terms of the Production Bias Model

In recent years it was shown that defect accumulation under cascade damage conditions (e.g. during irradiation with fission or fusion neutrons) can be properly addressed within the framework of the production bias model (PBM). The model is based on intracascade clustering of point defects, differences in the thermal stability of the resulting clusters and one-dimensional (1D) diffusion of interstitial clusters. Furthermore, it has been demonstrated that aspects of the experimentally observed defect accumulation in face centred cubic (fcc) metals can be described quantitatively in terms of the PBM.

There is a very large difference in the defect accumulation behaviour between fcc and bcc (body centred cubic) metals under cascade damage conditions. Interpretation of this in terms of the PBM lead to the suggestion that the reaction kinetics of the 1D diffusing interstitial clusters is different in fcc than in bcc metals and that this difference may arise from rare changes in the direction of the diffusion. Preliminary analyses showed that this suggestion may lead to a better understanding of the defect accumulation in fcc as well as bcc metals and may also provide an explanation for the formation and stability of void superlattices.

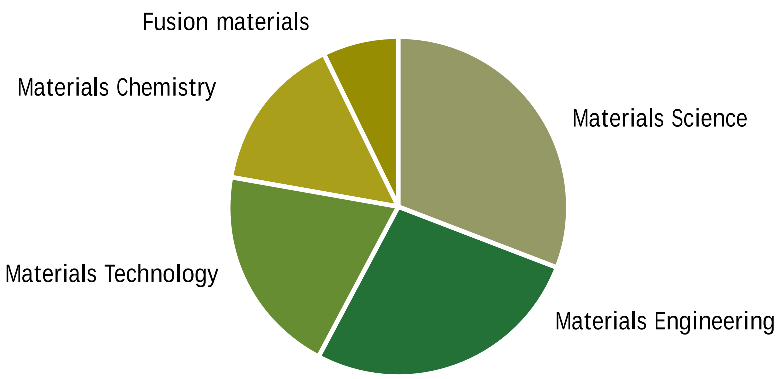
Monte Carlo simulations were carried out (in collaboration with Pacific Northwest National Laboratory, WA, USA) to test aspects of the above suggestion. The reaction kinetics of the 1D diffusing interstitial clusters were studied as a function of the frequency of direction changes and the size and density of sinks (absorbers) in a three-dimensional lattice. The results showed that even very infrequent changes in the direction of the transport of interstitial clusters modified their reaction kinetics. When the frequency of direction changes became so high that the mean transport distance between direction changes became of the order of the lattice parameter of the crystal, the reaction kinetics became very similar to the kinetics of pure 3D diffusional transport.

FINANCES 1998

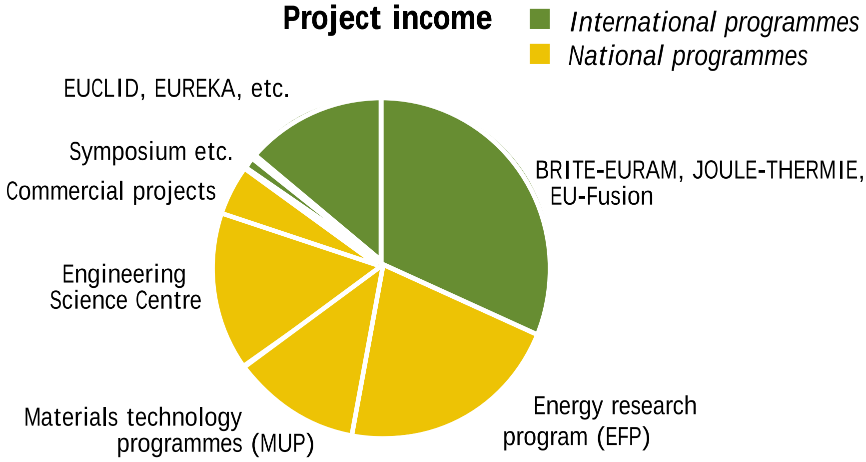
The activities of the Department are supported by a combination of direct government funding, focused project funds from national, international and EU programmes and fully commercial industrial contracts.

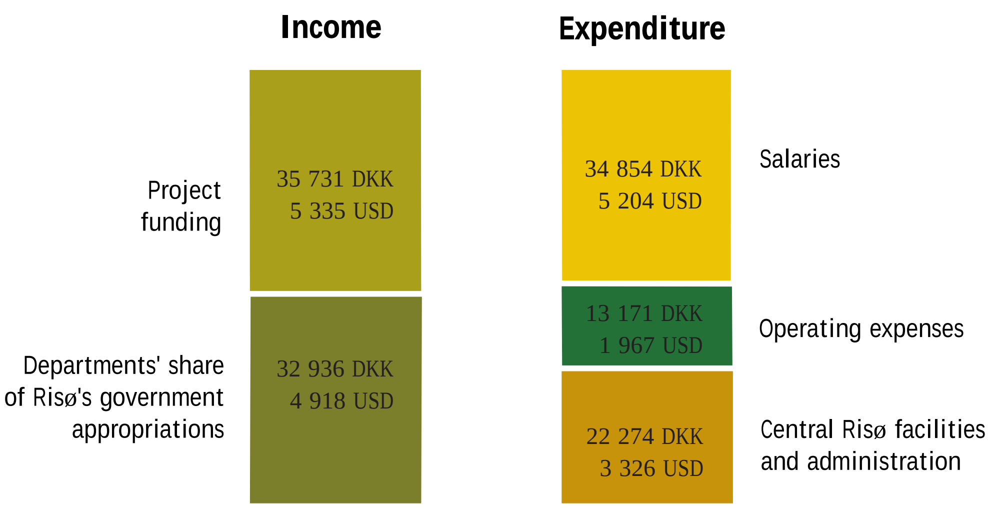
The numbers given in the tables are in units of 1000 Danish Kroner (DKK). The equivalent amount in US Dollars is also shown (DKK 1000 equal 149 USD, alternatively, 1 USD equals 6.70 DKK).

Research areas (hours)



Project income





Total 68 667 DKK
10 253 USD
(1997: 70 014 DKK)

Total 70 299 DKK
10 497 USD
(1997: 68 063 DKK)

PERSONNEL

Staff

In 1998 6 members of the permanent staff left the Department and 3 members joined.

Head of Department

Niels Hansen

Scientific staff

Adolph, Eivind
Andersen, Svend Ib
Appel, Charlotte C. *until 31 Oct.*
Bagger, Carsten
Bentzen, Janet J.
Bilde-Sørensen, Jørgen B.
Bonanos, Nikolaos
Borring, Jan
Borum, Kaj K.
Brøndsted, Povl
Debel, Christian P.
Eldrup, Morten
Gundtoft, Hans Erik
Hendriksen, Peter V.
Horsewell, Andy *until 31 Oct.*
Huang, Xiaoxu*
Johansen, Bjørn S.
Juul Jensen, Dorte
Jørgensen, Mette Juhl
Kindl, Bruno
Koch, Anita H. *until 17 May*
Krieger Lassen, Niels C.*
Larsen, Peter Halvor
Leffers, Torben
Lilholt, Hans
Linderoth, Søren
Liu, Qing
Liu, Yi Lin
Lorentzen, Torben
Lystrup, Aage S.
Løgstrup Andersen, Tom
Mogensen, Mogens
Nilsson, Tage M.
Pedersen, Allan Schrøder
Pedersen, Ole Bøcker
Poulsen, Finn Willy
Poulsen, Henning F.
Primdahl, Søren
Rheinländer, Jørgen
Singh, Bachu N.
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Toft, Palle
Toftegaard, Helmuth L.
Winther, Grethe

Postgraduate students

Andersen, Lotte Gottschalck*
Carstensen, Jesper Vejlø *until 30 Apr.*
Glerup, Marianne*
Hansen, Jesper Rømer
Lauridsen, Erik M.*
Lybye, Dorte
Mullit, Paw M. *until 28 Feb.*
Nielsen, Steen Arnfred *until 31 Aug.*
Nielsen, Søren Fæster
Nørbygaard, Thomas*
Vegge, Tejs*

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Carstensen, Jesper Vejlø*
Dam, Niels Ebbe *until 30 Sep.*
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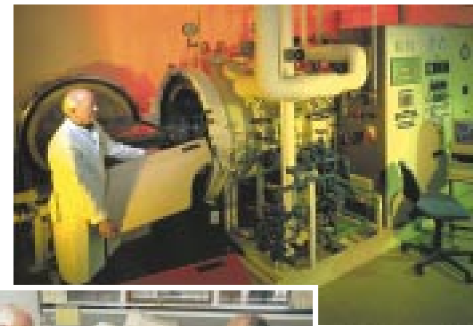
Grievel, Jean Claude*[Δ]
Holtappels, Peter W.
Huang, Xiaoxu *until 1 Sep.*
Jacobsen, Torben, K.[Δ]
Krieger Lassen, Niels C. *until 1 Sep.*
Laffargue, Denis
Lienert, Ulrich *until 31 Aug.*
Lyttle, Mark
Marina, Olga A. *until 31 Dec.*
Mishin, Oleg V.
Nielsen, Steen Arnfred *until 31 Dec.*
Pantleon, Wolfgang
Pryds, Nini H.
Rasmussen, Torben *until 31 Jul.*
Sarroute, Sabine *until 31 Jan.*
Schiøtz, Jakob*
Thorsen, Peter A. *until 30 Apr.*

Consultant

Waagepetersen, Gaston



Technical staff

Adrian, Frank
Borchsenius, Jens F.S.
Dreves Nielsen, Poul *until 30 Sep.*
Frederiksen, Henning
Gravesen, Niels Nørregaard
Hersbøll, Bent
Jensen, Knud
Jensen, Palle V.
Jespersen, John
Kjær, Anne-Mette Heie *until 31 Oct.*
Kjøller, John
Klitholm, Cliver
Larsen, Bent
Larsen, Birgit Herup Nygaard
Larsen, Jan
Larsen, Kjeld J. C.
Lillegaard, Keld *until 31 Mar.*
Lindbo, Jørgen
Mikkelsen, Claus
Nielsen, Birgitte
Nielsen, Palle H.
Nilsson, Helmer
Olesen, Preben B.
Olsen, Benny F.
Olsen, Henning
Olsen, Ole
Olsson, Jens O.
Paulsen, Henrik
Pedersen, Niels Jørgen
Sandsted, Kjeld
Strauss, Torben R.
Sørensen, Erling
Vogeley, Erik*
Aagesen, Sven

Office staff

Dreves Nielsen, Elsa
Hoffmann Nielsen, Lis
Lauritsen, Grethe Wengel
Mortensen, Jytte
Sørensen, Eva M.
Thomsen, Ann
Voss, Anita

Apprentices

Christensen, Lars F. *until 31 Jul.*
Hammershøj Olsen, Casper
Klein, Roland
Nilsson, Jesper

* joined the Department in 1998
Δ Industrial Post.Doc

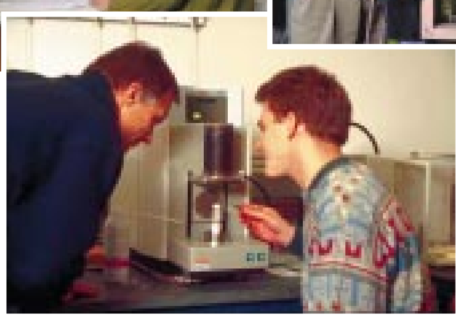
Staff members associated with industrial institutions/companies

Tage M. Nilsson
Consultant at Center for Advanced Technology, Roskilde, Denmark. 15 February - 31 December (part time).

Tom Løgstrup Andersen
Consultant at Trevira Neckelmann, Silkeborg, Denmark. 14 April - 14 July (part time).

Torben K. Jacobsen
Industrial Post. Doc.
LM Glasfiber A/S

Jean-Claude Grivel
Industrial Post. Doc.
Nordic Superconductor Technologies



Visiting scientists

An important ingredient in modern research is international collaboration. In 1998 many guests visited or worked in the Department for a shorter or longer period. Similarly members of the Department's staff worked at other institutions.

Scientists visiting the Department

Only visits of three of more days are listed.

Dr. G. Abertinni
University of Ancona, Italy.
10 - 13 December.

Dr. Dorota Artymowicz
Swedish Institute for Metals Research, Stockholm, Sweden. 9 - 15 November.

Dr. Claire Y. Barlow
University of Cambridge, UK.
23 August - 11 September.

Dr. Holger Bausinger
Dornier, Friedrichshafen, Germany.
5 - 12 August.

D.W. Brown
Los Alamos National Laboratory, USA. 11 - 21 April.

Dr. R. Burguete
British Aerospace, UK.
16 - 19 November.

Dr. Bjørn Clausen
Los Alamos National Laboratory, USA. 11 - 21 April.

David Coimbra
Ecole Nationale Supérieure de Céramique Industrielle, Limoges, France.
1 January - 15 March.

Dr. Dan J. Edwards
Pacific Northwest National Laboratory, Richland, WA, USA.
15 - 23 May.

Dr. E. Girardin
University of Ancona, Italy.
29 November - 13 December.

Dr. A. Giuliani
University of Ancona, Italy.
29 November - 9 December.

Dr. Stanislav I. Golubov
Institute of Physics and Power Engineering, Obninsk, Russia.
15 May - 30 November.

Dr. Howard L. Heinisch
Pacific Northwest National Laboratory, Richland, WA, USA.
29 June - 11 July.

Dr. Huang Yanding
University of Science and Technology, Beijing, China.
18 February - 19 December.

Dr. Darcy A. Hughes
Sandia National Laboratory, Livermore, CA, USA.
18 July - 15 August.

Prof. Bevis Hutchinson
Swedish Institute for Metals Research, Stockholm, Sweden.
9 - 11 November.

Dr. A. Korsunsky
University of Newcastle, UK.
26 April - 4 May.

Dr. Liu Wenchang
Yanshan University, Qinghuandao, China.
18 February - 19 December.

A. Madgwick
University of Cambridge, UK.
23 January - 3 February.

F. Maillart
Université Catholique de Louvain, Belgium.
24 - 30 August.

Prof. Frans H. J. Maurer
Chalmers Technical University, Gothenberg, Sweden.
16 March - 9 April.

Dr. E. O´Brien
British Aerospace, UK.
18 - 20 November.

Dr. Masato Ohnuma
National Research Institute for Metals,
Tsukuba, Japan.
1 March - 31 December.

Dr. N. Plouzenec
British Aerospace, UK.
18 - 20 November.

Dr. H.G. Priesmeyer
University of Kiel, Germany.
2 - 7 July.

Dr. Thomaz Augusto Guisard Restivo
Centro Tecnologico da Marine em Sao Paolo,
Brazil.
22 October - 22 December.

Dr. U. Rodrian
Daimler Benz, Germany.
3 - 6 June.

L. Ryelandt
Université Catholique de Louvain,
Belgium.
24 - 30 August.

C. Salmon
Université Catholique de Louvain,
Belgium.
24 - 30 August.

Prof. Nigel Sammes
University of Waikato, New Zealand.
1 - 30 March.

J. Sarkar
University of Cambridge, UK.
23 January - 3 February.

M. Sinnaeve
Université Catholique de Louvain,
Belgium.
24 - 30 August.

A. Steuwer
University of Manchester, UK.
15 - 23 November.

Ursula Tietze
University of Kiel, Germany.
2 - 7 July.

J. R. Tuck
University of Newcastle, UK.
26 April - 4 May.

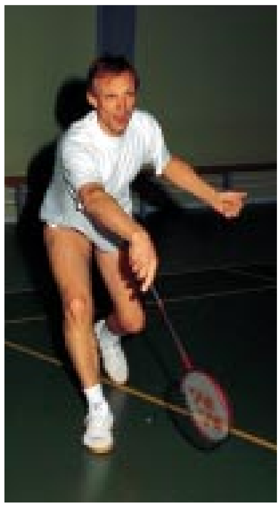
Dr. Roy Vandermeer
Alexandria, Virginia, USA.
4 September - 2 October.

K. E. Wells
University of Newcastle, UK.
26 April - 4 May.

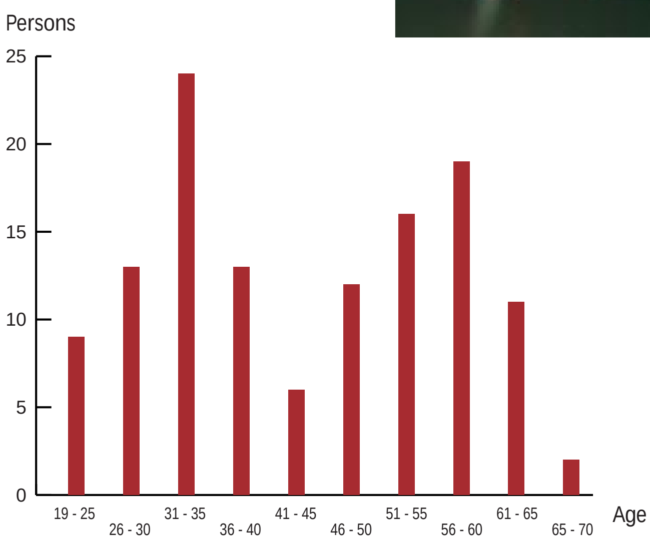
Prof. John A. Wert
University of Virginia,
Charlottesville, VA, USA.
10 August - 31 December.

Dr. Hong Zheng
University of Science and Technology of
China, Hefei, China.
1 January - 1 July.

The Department sailed to Sweden for the annual summer picnic.



Members of the Department are active in Risø's sports clubs.



The age profile of the staff.

Staff members visiting universities

Only visits of three or more days are listed.

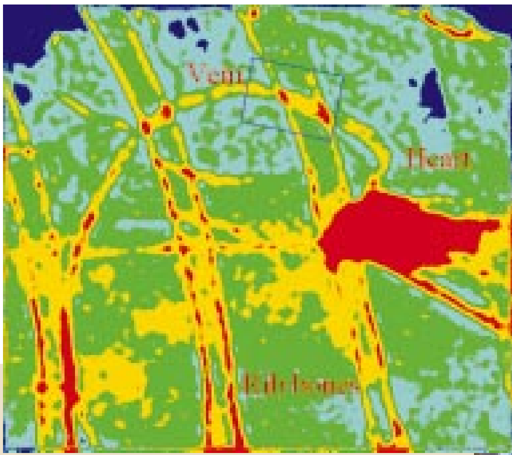
Dorte Juul Jensen
Carnegie Mellon University,
Pittsburgh, USA.
10 - 20 June.

Liu Qing
University of Science and Technology,
Beijing, China.
15 April - 16 May.

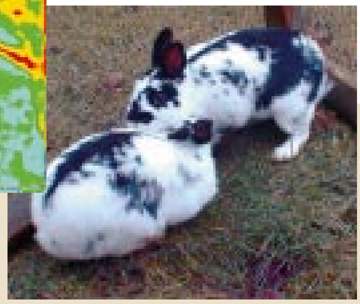
Mogens Mogensen
University of Waikato, New Zealand.
14 - 23 December.

Bent F. Sørensen
University of Michigan, Ann Arbor,
MI, USA.
25 May - 1 June.

In 1998, the Department assisted about 20 predominantly Danish industrial companies with specific problems (mostly in confidentiality) and collaborated with about 20 Danish and 60 foreign companies in programmes partly funded by the Danish government and the European Union.



Imaging techniques and analysis are developed for industrial quality control and are also applied in areas other than Materials Research. One example is the diagnosis of arteriosclerosis where the vein diameter is calculated from a radiograph of a living rabbit. Even veins covered by rib bones can be measured.



Imaging techniques (non-destructive characterisation)

Specimens are 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 upon testing. Typical areas of application include determination of

- Dimensions of flaws
- Dimensional variations which characterise the integrity of hollow or hidden structures
- Distribution of the structural elements of the specimens

Measurements of residual stresses in industrial components include: Car engine crank shafts (Mercedes Benz, Germany), welded aluminium structures (British Aerospace Airbus Ltd., UK), Nickel alloy weldings for aircraft turbines (Rolls Royce plc., UK) and specimens produced by sheet metal forming operations (Centro Ricerche Fiat, Italy).

Scanning electron micrograph of copper surface

Neutron diffraction

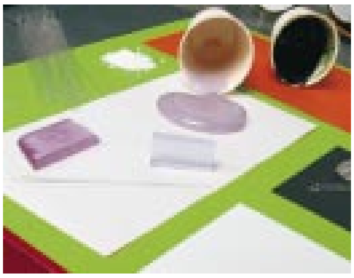
Neutron diffraction provides information about crystal orientation and crystal lattice parameters:

- Residual stresses and strains can be monitored non-destructively inside large engineering components. Strains are typically monitored to an accuracy of $\pm 10^{-4}$, in gauge volumes down to 10 mm^3, at freely selected positions in the sample. The penetration depth is several centimetres, depending on the material. Phase specific stresses in multi-phase systems and composite materials can also be determined.
- Bulk textures, i.e. the crystallographic orientations in the sample, are measured. Typical sample sizes are in the range 0.5 - 10 cm^3. *In-situ* measurements during annealing are performed at temperatures up to 1500 °C. Standard software calculates three-dimensional orientation distribution functions and parameters related to the mechanical properties.

Textures are measured in metals and alloys.

Electron microscopy and microanalysis

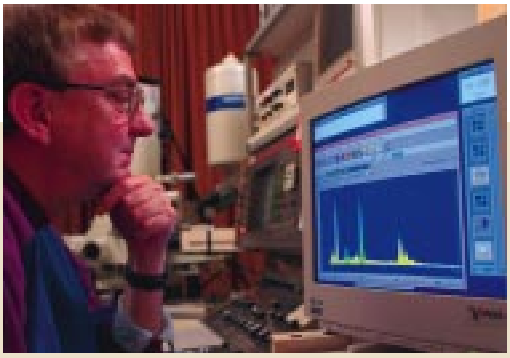
Two transmission electron microscopes (TEM) and three scanning electron microscopes (SEM) are operated by the Department. Attachments for TEM include energy-dispersive X-ray spectrometer (EDS), scanning transmission attachment, serial electron energy loss spectrometer and hollow cone illumination. In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



Thermal analysis

Differential thermal analysis (DTA) is used to characterise endothermic or exothermic reactions between -70 °C to 1300 °C. Thermogravimetric analysis (TGA) registers weight changes during heating up to 1500 °C. Dilatometry involves measurement of dimensional changes as a function of temperature or oxidising conditions. Amorphous as well as crystalline materials (polymers, alloys, glasses and ceramics) are 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 and sintering profiles (dilatometry)



tering diffraction (EBSP) equipment are available. SEM can be performed at various pressures in different atmospheres (LVSEM, ESEM). Heating and cooling stages, micromanipulator, micro-injector and deformation stages are available for *in-situ* observations in ESEM. Examples are investigations of

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

Technical innovations incorporated in commercial instruments

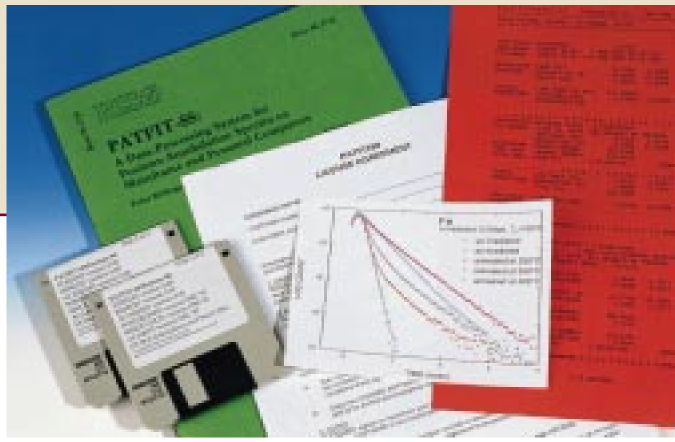
The techniques and analysis algorithms used in the Department are continuously developed for new research applications. However, some of the innovations are later implemented in instruments made by commercial companies.

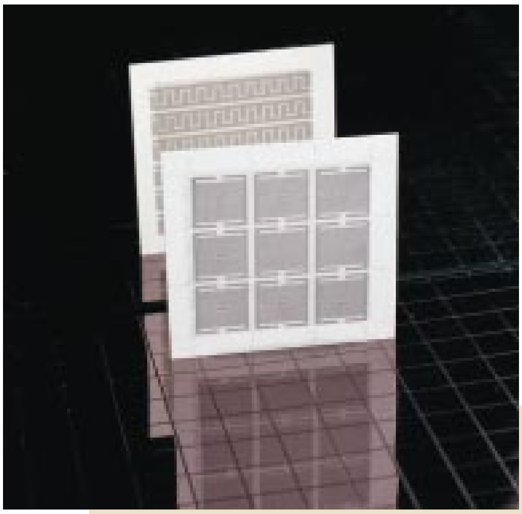
X-ray spectrometry in environmental and low vacuum SEMs

The presence of gas in the specimen chamber of environmental or low vacuum scanning electron microscopes causes the spatial resolution for X-ray spectrometry to deteriorate. The reason is that some of the primary electrons are scattered by the gas, giving rise to a skirt of scattered electrons, and these scattered electrons may excite X-rays far from the target point for the analysis. As described in previous annual reports, the Department developed a method by which the results from spectral analysis in the same point at various pressures can be extrapolated to the result that would have been obtained without the beam skirt effects. The spectrometer producer EDAX now offers a computer software option for their spectrometer systems that utilizes this pressure variation method in an automated analysis routine to increase the spatial resolution.

Analysis of positron annihilation spectra

For a number of years positron annihilation spectroscopy has been used in the Department for the investigation of defects in metallic and insulating materials. Early on a need developed for computer programs that could analyse experimental positron annihilation spectra. In collaboration with the Risø Computer Section such programs were developed for main frame computers. Over the years the programs have been updated and expanded several times. A PC version, called PATFIT-88, was made about ten years ago. This program package has attracted interest from other researchers in the same field and has until now been distributed on a commercial basis to more than 90 laboratories in 30 different countries around the world. In 1998 the most recent, updated version was sold to a major American nuclear electronics company for incorporation into one of their products.





Ceramic oxygen sensor developed in collaboration with PBI-Dansensor A/S.

Ceramic synthesis and powder characterisation

Cost-effective syntheses of complex oxide-ceramic compounds, usually electroceramics are developed. Control of dopant elements allows materials properties to be tailored. High phase purity and nanocrystallite size are obtained with powder in amounts up to multi-kilogram scale. Subsequent high temperature calcining processes produce desired particle size distributions to suit demands for porosity size distributions and density in the final components.

X-ray at high temperatures and in different atmospheres as well as other techniques provide information on

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

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

Solid oxide fuel cell developed in co-operation with IRD A/S and Haldor Topsøe A/S.

A cheap manufacturing method for electronic noise filters of functionally graded ceramics was developed in collaboration with AMP Danmark.

Ceramic processing

Several shaping techniques are available:

- Biaxial and isostatic pressing
- Tape casting (thin sheets (50 - 1000 µm) for dense or porous flat membranes)
- Ceramic spray painting and dip coating produces thin (5 - 100 µm) layers. Multiple layers create material gradients (chemical composition, porosity and properties).
- Viscous processing produces planar or profiled ceramic sheets

A flywheel for energy storage made of polymer matrix composites was designed, manufactured and successfully tested (patented by Risø, developed in collaboration with DEMEX A/S, NESA A/S, TERMA Industries A/S and Aalborg University).

Characterisation and modelling of mechanical material properties resulted in a tenfold increase of tool life (STRECON (Danfoss A/S))

A car door-post in thermoplastic composites. The process technology is developed in collaboration with Trevira Neckelmann and Komposit Procesteknik.

Processing of polymer matrix composites

The Department has equipment and qualified personnel for optimisation of process parameters for production of thermoplastic and thermosetting composite components. Quality control of porosity, fibre content and fibre orientation ensures high reproducibility. Specimens for mechanical characterisation are also produced. Processes include:

- Autoclave and vacuum consolidation and curing (530 °C, 2 MPa)
- Pressing (200 kN, preheating in vacuum, fast processing)
- Filament winding (computer aided design, engineering and manufacturing by use of a developed PC program - freely available)

Mechanical characterisation

The Department is accredited by DANAK (Danish Accreditation) to carry out mechanical testing. Static and dynamic tests are 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 universal test machines, including a combined tension-torsion machine, two static test machines and a resonance test machine for higher frequency testing.

Testing is carried out in - or in combinations of - the following modes:

- Torsion
- Tension
- Compression
- Bending
- Shear

Mechanical properties of polymer matrix composites and process technologies for wind turbine rotorblades are investigated. Optimisation based on modelling is in progress. New recyclable materials are considered. Some of the work is in co-operation with Bonus Energy A/S and LM Glasfiber A/S.

Brazing

Dipbrazing is carried out in a furnace containing a crucible with molten salt. The subject to be brazed is assembled from more pieces as sheets or as machined parts. Filler material is added at the joints, and the whole assembly is immersed in the salt. For vacuum brazing, the Department is equipped with several programmable furnaces heating to 1300 °C in a vacuum better than $5 \cdot 10^{-5}$ torr. Maximum specimen dimensions are 20 x 20 x 45 cm^3. Heating in a constant flow of inert gas is also possible. Brazing of metals, ceramics and ceramics to metals is performed.

The Department is strongly involved in the education of students at different levels. The involvement ranges from postgraduate and undergraduate research projects in collaboration with universities, to summer jobs for university students and one-day courses for high school students. Several of the projects are carried out in collaboration with industrial companies, through Industrial PhD projects, in which students work an apreciable part of their time in the company. Alternatively, university students on summer jobs work on joint projects between the Department and industrial companies, spending time at both places. In addition, many staff members of the Department act as external university teachers and examiners.

Postgraduate (PhD) projects

PhD Projects finished during 1998

Jesper Vejlø Carstensen
'Structure evolution and mechanisms of fatigue in polycrystalline brass'.
The Technical University of Denmark, Lyngby.
Supervisors: Viggo Tvergaard (DTU), *Ole Bøcker Pedersen*

Darja Kek
'Electrochemical properties of interfaces between metals and solid state ion conductors'.
National Institute of Chemistry/University of Ljubljana, Slovenia.
Supervisors: Stane Pejovnik (UL), *Mogens Mogensen*

Steen Arnfred Nielsen
'Ultrasonic characterization of materials using tomographical methods'.
The Technical University of Denmark, Lyngby.
Supervisors: Leif Bjørnø (DTU), *Hans Erik Gundtoft, Svend Ib Andersen, Jørgen T. Rheinländer*

Ongoing Industrial PhD Projects

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

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

Kenneth Petersen
'Development of the spray-forming process for production of steel-based composites'.
The aim of the project is to identify the correlation of the applied, experimental parameters and the properties of the resulting material. This is done by studying the effect of parameter variations and by investigation of the alloy solidification in the presence of ceramic particles and the final microstructures. The project focuses on wear properties, strength and toughness.
The Technical University of Denmark, Lyngby.
Supervisors: Jan Laurberg List (Danish Steel Works), Knud Strande (DTI), K. Aa. Thorsen (DTU), *Allan Schrøder Pedersen*

Ongoing PhD Projects

Lotte Gottschalck Andersen
'Structural properties of superconducting BSCCO/Ag tapes during cooling'.
Tapes containing the high temperature superconductor Bi-2223 will be characterized by a combination of SEM, synchrotron XRD and magneto-optic measurements. The goal is to identify detrimental effects on the superconducting critical current during cooling of the tapes and to optimise the cooling procedure.
The Technical University of Denmark, Lyngby.
Supervisors: Jørgen Bindslev Hansen (DTU), *Henning Friis Poulsen*

Michael S. Brown
'Anodes for solid oxide fuel cells'.
Technological types of Ni-YSZ-cermet anodes are studied, in particular features like the correlations between the performance and Ni/YSZ-ratio, particle size of Ni and YSZ, porosity and electrode thickness.
University of Waikato, Hamilton, New Zealand.
Supervisors: Nigel Sammes (UW), *Mogens Mogensen*

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

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

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

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

Paw Mullit
'Quantification of the pore structure in porous materials'.
The mechanical and physical behaviour of porous materials, such as cement, concrete and brick, is strongly influenced by the shape, size and amount of pores in the material. Although understood qualitatively, quantitative treatments of the relationship between pore stereology and properties need to be developed further.
The Technical University of Denmark, Lyngby.
Supervisors: Lauge Fuglsang Nielsen (DTU), *Andy Horsewell*

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

Thomas Nørbygaard
'Structure and characteristics of general and special grain boundaries'.
The aim of the project is to achieve an understanding of the connection between grain boundary structure and boundary properties. Cu-Ni(2%) samples with inactive surface markers (a fine-meshed grid) are tested under diffusional creep conditions and studied by SEM and TEM. Bubble formation behavior in copper implanted with helium at elevated temperatures is investigated with TEM.
University of Copenhagen, Denmark
Supervisors: Erik Johnson (KU), *Jørgen B. Bilde-Sørensen*

Bent Tveten
'High temperature oxidation of metals'.
Studies of the kinetics of formation and nature of the oxidation products on Cr-Fe alloys in moist air up to 1000 °C are carried out.
Thermogravimetry, electron microscopy and X-ray diffraction are used as major tools of characterization.
The University of Oslo, Norway.
Supervisors: Truls Norby (UO), *Mogens Mogensen, Finn Willy Poulsen*

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

Rolf Jarle Åberg
'Studies of kinetics and reaction mechanisms on Ni-YSZ anodes for solid oxide fuel cells (SOFC)'.
Ni-YSZ cermets are used as anodes for oxidation of hydrogen and CO in SOFC. Several rate limiting steps in the electrode mechanism have been suggested. Experiments with Ni-electrodes with well defined geometry are used to elucidate the mechanisms.
Norwegian University of Technology and Science, Trondheim, Norway.
Supervisor: Reidar Tunald (NTNU), *Mogens Mogensen*

Undergraduate Projects

Helen Bee
'Characterisation by scanning electron microscopy of the interface between zirconia and rare earth element based manganites'.
University of Cambridge, UK.
Supervisors: *Charlotte C. Appel, Andy Horsewell*

Alexander Godfrey
'The role and nature of bridging in the fracture of unidirectional fibre composites'.
University of Cambridge, UK.
Supervisors: *Bent F. Sørensen, Andy Horsewell*

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

Trine Bjerre Pedersen
'Numerical modelling of spray-forming'.
The Technical University of Denmark, Lyngby.
Supervisors: Jesper Hattel (DTU), *Nini H. Pryds*

Kai Schimanski
'Microstructural investigation of spray-formed 100Cr6 steel'.
University of Bremen, Germany.
Supervisor: Sabine Spangel (UB), *Allan Schrøder Pedersen*

External lecture courses

Svend Ib Andersen
Jørgen B. Bilde-Sørensen
Morten Eldrup
Mark Lyttle
Allan Schrøder Pedersen
Henning Friis Poulsen
Bent F. Sørensen
Ole Toft Sørensen
'Introduction to New Materials'. University of Aarhus and Aalborg University, Denmark.
4 - 20 May. *(Distance learning course)*

Charlotte C. Appel
'Electron microscopical characterisation of zirconia with manganese and yttrium in solid solution'. Mini symposium on electron microscopy at the University of Copenhagen. 17 September.

Jørgen B. Bilde-Sørensen
'Energy dispersive X-ray spectroscopy in the low vacuum and environmental scanning electron microscopes';
'Applications in low vacuum and environmental scanning electron microscopy at Risø';
'Low vacuum and environmental scanning electron microscopy. Basic principles'.
Course arranged by Chalmers University, JEOL, Philips, Hitachi and Leo on 'Low Vacuum and Environmental Scanning Electron Microscopy', Gothenburg, Sweden. 17-19 November.

Yi-lin Liu
'Phase conversion and texturing in BSCCO tapes'. Nordic Ph.D Summer School, 'Power Applications for Superconductivity'. Lyngby, Denmark. 9 - 14 August.

Wolfgang Pantleon
'Physics of plastic deformation'. Lectures within course on modern physics. The Technical University of Denmark, Lyngby. November. (DANVIS-Lectures)

'Konstitutive Materialgesetze und Modellierung der plastischen Verformung'. Summer school on 'Continuum theory of plastic deformation'. Freiberg University of Mining and Technology, Germany. 6 - 10 July.

Bachu N. Singh
'Materials problems in fusion devices'. Fusion Energy. A course for high school teachers. The Danish Museum of Electricity, Bjerringbro, Denmark. 2 - 4 September.

Ole Toft Sørensen
'Defect chemistry'. Course no. 3525. The Technical University of Denmark, Lyngby. March.

External Examiners

Svend Ib Andersen
Jørgen B. Bilde-Sørensen (until 31/3-1998)
Povl Brøndsted
Christian P. Debel
Morten Eldrup
Andy Horsewell
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.

Nikolaos Bonanos
PhD Examiner. University of Oslo, Norway.

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

Andy Horsewell
PhD examiner. University of Copenhagen, Denmark.

Dorte Juul Jensen
PhD examiner. University of Cape Town, South Africa.

Torben Leffers
PhD examiner. University of Science and Technology of Lille, France.

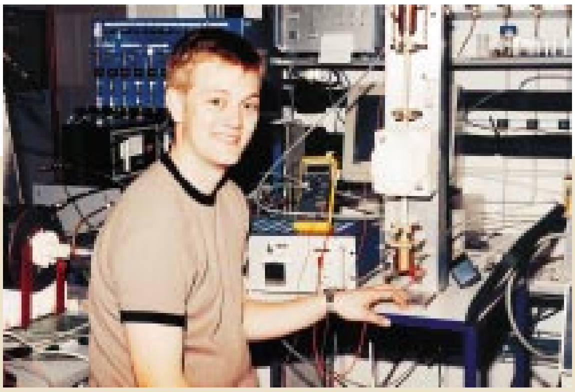
Hans Lilholt
PhD examiner. University of Aalborg, Denmark.
Techn. Dr. Faculty examiner. Technical University of Luleå, Sweden.

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

Mogens Mogensen
PhD examiner. University of Twente, Enschede, The Netherlands.

Bent F. Sørensen
PhD examiner. University of Aalborg, Denmark.

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



Motivating youth for science and technology

The number of students who take a technical or scientific education at a Danish university has been declining and it is foreseen that Danish industry and research institutions will have difficulties finding qualified employees in the future. In an effort to help remedy this situation, the Department has initiated a number of activities to stimulate the interest of young people in scientific subjects.

One branch of the activities is aimed at high school students who are invited to come to Risø on a one day course covering theory and practice within the field of materials science, more specifically: polymer chemistry and polymer based fiber composites. This provides the students with some knowledge about the production and the properties of materials along with the experience that science can be fascinating and exciting. This year 86 students participated in the courses. They came from the following schools: Himmelev Gymnasium, Svendborg Gymnasium, Espergærde Gymnasium, Helsingør Gymnasium, Nordfyns Gymnasium, Esbjerg Gymnasium, Århus Statsgymnasium, Køge Tekniske Skole (HTX) and Marselisborg Gymnasium.

'Exciting not to know the result in advance...'

'Interesting exercises that we could not do at our own school.'

High school students may also work for a number of days in the Department on specific projects. In 1998 two students did so:

Kristoffer Barfod
'Selected area channeling patterns' .
Næstved Gymnasium, Næstved, Denmark.
Supervisor: *Jørgen B. Bilde-Sørensen*

Melissa Sharp
'Investigation of aluminium single crystals'.
Seven Oaks School, Seven Oaks, UK.
Supervisor: *Dorte Juul Jensen*

At the more advanced level, the Department offers summer jobs to university students. During their vacation the students work on joint projects between the Department and industrial companies, spending time at both places. The aim is to present the students with both scientific and industrial problems and motivate them to take more advanced courses in materials science. The first such projects were completed in 1998 with great success as judged from the reports from both students and companies. The students generally found the organisation and interdisciplinary work of 'real projects' challenging and they felt accepted as members of the Department. One of the projects is even being continued in a contract between the Department and Asnæs Power Station.
The projects are listed below.

'It has been exciting to get an impression of the daily work of a scientist in real life (outside the University) .. and of an engineer in industrial companies.'

'.. an experience to work with something interdisciplinary ...'

Michael H. Iversen
'Mechanical testing'.
Raufoss AS

Peter Jans
'Oxygen sensor for flue gas'.
Asnæs Power Station

Pernille Schiøtz Larsen
'Investigation of texture in stainless steel'.
Grundfos A/S

Peter Poulsen
'Long term stability of Mg-doped $SrTiO_3$ sensors'.
PBI-Dansensor A/S

Eugen Zoica
'Texture development and phase transformation in BiSCCO/Ag tapes'.
Nordic Superconductor Technologies A/S

19th Risø International Symposium

The title of the 19th Risø International Symposium on Materials Science was '**Modelling of Structure and Mechanics of Materials from Microscale to Product**'. The Symposium was held at Risø 7 - 11 September with 81 participants from industry, research institutes and universities in 21 countries. The theme of the Symposium was the relation between the processing, structure and mechanical behaviour of materials and its expression in analytical or numerical models. The length scales involved in the models range from macroscopic dimensions to the scale of atoms. The symposium focused on attempts to bridge the various length scales. The 588 page symposium proceedings containing 14 invited papers and 58 contributed papers were published and are available upon request.

The Symposium was organised by the Department in collaboration with the Engineering Science Centre for Structural Characterisation and Modelling of Materials and the Centre for Materials Processing, Properties and Modelling within the Danish Materials Technology Development Programme. The latter centre includes institutes at the Technical University of Denmark and Aalborg University together with the Department.

Organisers: *Jesper Vejlø Carstensen, Niels Hansen, Dorte Juul Jensen, Grethe W. Lauritsen, Torben Leffers, Torben Lorentzen, Ole Bøcker Pedersen, Bent F. Sørensen and Grethe Winther.*

The symposium excursion went to the Open Air Museum.

'Damage Production and Accumulation under Cascade Damage Conditions'

A small workshop, organised at Risø 2 - 4 July 1998, was dedicated to in-depth discussions of basic problems in the understanding of the production and accumulation of radiation damage under cascade damage conditions. Apart from Denmark, the workshop gathered participants from the UK, Russia, China, Spain and USA.

Organisers: *Bachu N. Singh*, *Stanislav Golubov*.

'Basic Aspects of Differences in Irradiation Effects between FCC, BCC and HCP Metals and Alloys'

An international Workshop on 'Basic Aspects of Differences in Irradiation Effects between fcc, bcc and hcp Metals and Alloys' was held at Canga de Onis in Spain during 15 - 20 October 1998. It was the 7'th Workshop in a series which was initiated jointly by the Paul Scherrer Institute, Switzerland and Risø in 1983. The workshop attracted about 50 participants from 12 countries.

Organisers: Tomas Diaz de la Rubia, *Bachu N. Singh*, Maximo Victoria and Abderrahim Almazouzi.

Colloquia

A number of scientists visiting the Department presented their work in colloquia.

Prof. Terrence E. Warner
University of Cambridge, UK.
'The electrochemical properties of $LaAl_{12}O_{18}N$'.
6 January.

Prof. Jean-Bernard Vogt
Universite des Sciences et Technologies de Lille, France.
'Fatique of high nitrogen stainless steels'.
13 January.

Dr. H. J. M. Bouwmeester
University of Twente, Enschede, The Netherlands.
'Dense ceramic membranes for oxygen separation'.
28 January.

Prof. M. K. Surappa
Indian Institute of Science, Bangalore, India.
'Processing and tribological characteristics of aluminium matrix SiC particle composites'.
16 March.

Dr. Baldev Raj
Indira Ghandi Centre for Atomic Research, Kalpakkam, India.
'Nuclear and non-nuclear energy research in India'.
20 May.

Dr. T. R. Armstrong
Pacific Northwest National Laboratory, Richland, WA, USA.
'Properties of A-site deficient lanthanum gallate'.
27 May.

Dr. Geoff Tompsett
University of Waikato, New Zealand.
'Raman spectroscopy on ceramics'.
8 June.

Dr. A. K. Vasudevan
Office of Naval Research, Arlington, VA, USA.
'Role of internal stresses and K_{max} on long and short fatigue crack growth'.
22 June.

Prof. D. H. Sastry
Indian Institute of Science, Bangalore, India.
'Effect of alloying on the creep behavior of Fe_3Al'.
3 July.

Prof. Akihisa Inoue
Tohoku University, Japan.
'High-mechanical strength of bulk nanostructure alloys consisting of compound and amourphous phases'.
28 August.

Dr. Masato Ohnuma
National Research Institute for Metals, Tsukuba, Japan.
'Microstructure of sputter deposited Co-Al-O granular films'.
3 November.

Dr. Kunie Ishioka
National Research Institute for Metals, Tsukuba, Japan.
'Phonon localization in ion-irradiated crystals'.
8 December.

PARTICIPATION IN COMMITTEES

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

Research Programme Committees

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

The Steering Comittee for the Danish Superconductor Programme.

The Steering Committee for the MUP 2.2 Programme: Electroceramic Functionally Graded Materials.

Niels Hansen
The Steering Committee for the Danish Solid Oxide Fuel Cell Programme. Risø.

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

Dorte Juul Jensen
Board of the Danish Research Councils. Copenhagen. (*Until 26. August, Chairman*).

The Programme Committee of FREJA (Female Researchers in Joint Action).

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

The Technical Subcommittee for the Danish Materials Technology Development Programme. Copenhagen. *(Chairman)*.

The Coordination Committee for the Danish Materials Technology Development Programme. Copenhagen.

The Board of Ingeniørvidenskabelig Fond og G. A. Hagemanns Mindefond. Copenhagen.

Hans Lilholt
Project Management Committee of the EU BRITE Project: 'Thixoforming of Advanced Light Metals for Automotive Components (TALMAC)'. *(Chairman)*.

Søren Linderoth
The Steering Committee for the Danish Solid Oxide Fuel Cell Programme.

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

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

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

The Project Management Committee of the EU JOULE 3 project 'Improving Durability of Solid Oxide Fuel Cell Stacks'. *(Chairman)*.

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

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

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

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

Jørgen T. Rheinländer
Project Management Committee of the 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)*.

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

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

Steering Committee of the EU-funded thematic network PLAN: Plant Life Assessment Network *(Cluster Co-ordinator)*.

Advisory Committees

Eivind Adolph
Technical Assessor, DANAK. Copenhagen.

Niels Hansen
Advisory Group for Advanced Energy Technologies. The Danish Ministry of Environment and Energy. Copenhagen.

Technical Assessor, DANAK. Copenhagen.

Reference Group for the BRITE/EURAM Programme, The Danish Ministry of Industry. Copenhagen.

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

The COST Technical Committee on Materials. Brussels, Belgium.

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

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

Torben Leffers
Working Group for the Formulation of a National Strategy for Materials under the Ministry of Research. Copenhagen.

Task Force for the EU Programme on Standards, Measurements and Testing. Copenhagen.

Torben Lorentzen
VAMAS committee, Technical Working Area TWA20, Measurement of Residual Stresses.

External review Committee for Powder Diffraction Instruments, LANSCE Short Pulse Spallation Source Spectrometer Development Project, Los Alamos, USA.

ISP-7 Scheduling Panel ; ISIS-facility, Rutherford Appleton Laboratory, UK.

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

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

Jørgen T. Rheinländer
Technical Assessor, DANAK. Copenhagen.

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

Task Force Materials, EU Fusion Technology Programme. Brussels, Belgium.

Ole Toft Sørensen
ICTAC Standardization Committee.

Editorial Committees

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

Povl Brøndsted
Editorial Board of 'Advanced Composites Letters'.

Morten Eldrup
Advisory Board of 'Materials Science Forum'.

Niels Hansen
Editorial Board of 'Revue de Metallurgie'.

Editorial Board of 'Monographs in Materials Science'.

Dorte Juul Jensen
Advisory Board of 'Zeitschrift für Metallkunde'.

Associate editor of 'Acta Materialia' and 'Scripta Materialia'.

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'.

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

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

Allan Schrøder Pedersen
Editorial Panel of 'Powder Metallurgy'.

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

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

Ole Toft Sørensen
Editorial Board of 'Journal of Thermal Analysis'.

Editorial Board of 'Journal of the European Ceramic Society'.

Conference Committees

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

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

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

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

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

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

Hans Lilholt
International Advisory Committee for the Second International Symposium on Engineering Ceramics and Third International Symposium on High Temperature Ceramic Matrix Composites. September 1998. Osaka, Japan.

International Advisory Board for the Second International Conference on Composites and Ceramics. May 1998. Moscow, Russia.

International Advisory Committee for the Topical Symposium V: 'Advanced Structural Fibre Composites' of World Forum on New Materials. June 1998. Florence, Italy.

Programme Committee for the Dedicated Conference on Materials for Energy-Efficient Vehicles, (International Symposium on Automotive Technology and Automation, ISATA). June 1998. Florence, Italy.

Executive Committee for the 8th European Conference on Composite Materials. (ECCM-8). June 1998. Naples, Italy.

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

Finn Willy Poulsen
Organising Committee for the Third European Solid Oxide Fuel Cell Forum. June 1998. Nantes, France.

Ole Bøcker Pedersen
Advisory Board for the International Symposium on Materials Ageing and Life Management. October 2000. Kalpakkam, India.

Scientific Committee for the 2nd International Conference on Fatigue of Composites. June 2000. Atlanta, USA.

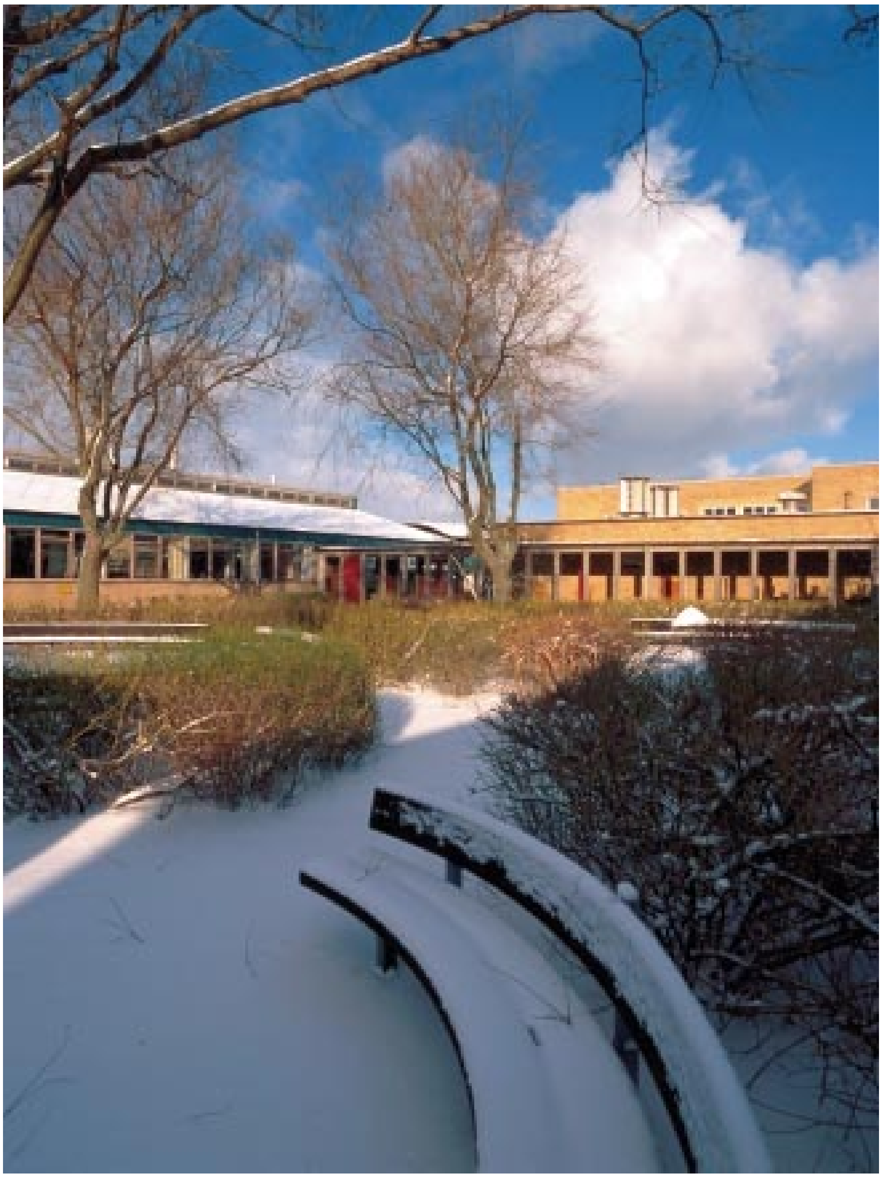
Henning Friis Poulsen
International Organising Committee of Euroseminar 'Integrated Analysis of Defect Structures'. November 1998. Freiberg, Germany.

Bachu N. Singh
Organising Committee for 19th International ASTM Symposium on the Effect of Radiation on Materials. June 1998. Seattle, WA, USA.

Organising Committee for the International Workshop on Basic Aspects of Differences in Irradiation Effects Between FCC, BCC and HCP Metals and Alloys. October 1998. Cangas de Onis, Spain.

Technical Programme Committee for 9'th International Conference on Fusion Reactor Materials, Colorado Springs. October 1999. Colorado, USA.

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



Professional Societies

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

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

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

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

Peter Halvor Larsen
Danish Ceramic Society. *(Chairman)*.

Nordic Society for Thermal Analysis and Calorimetry.

Council of the European Ceramic Society.

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

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

Mogens Mogensen
The Executive Committee of the Danish Electrochemical Society.

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

Søren Primdahl
The Executive Committee of the Danish Electrochemical Society.

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

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

Ole Toft Sørensen
Nordic Society for Thermal Analysis and Calorimetry.

For a public research centre the most important way of communicating its scientific results is to publish them in the scientific literature. The number of publications in 1998 was 165, very similar to that of 1997. In addition to the publications listed below, members of the Department delivered about 90 presentations at conferences and meetings where no proceedings were subsequently published, and wrote 20 internal reports.

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

International publications

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

1. Aaberg, R. J.; Tunold, R.; *Mogensen, M.*; Berg, R. W.; Odegard, R., Morphological changes at the interface of the nickel-yttria stabilized zirconia point electrode. J. Electrochem. Soc. (1998) v. 145 p. 2244-2252

2. Adams, B. L.; *Juul Jensen, D.*; *Poulsen, H. F.*; Suter, R., Future trends: Texture analysis for structure-sensitive properties. Mater. Sci. Forum (1998) v. 273-275 p. 29-39

3. *Appel, C. C.*; *Bilde-Sørensen, J.B.*; *Horsewell, A.*, Characterisation of materials by environmental SEM. Proc. R. Microsc. Soc. (1998) v. 33 p. 99

4. Arentoft, M.; *Bjerregaard, H.*; Andersen, C. B.; Wanheim, T., The influence of the constitutive behaviour of materials on the formability in radial extrusion of tubular components. J. Mater. Process. Technol. (1998) v. 75 p. 122-126

5. *Armstrong, W. D.*, Stress-dependent dynamic compliance spectra approach to the nonlinear viscoelastic response of polymers. J. Polym. Sci. B (1998) v. 36 p. 2301-2309

6. *Armstrong, W. D.*; *Lorentzen, T.*; *Brøndsted, P.*; *Larsen, P. H.*, An experimental and modeling investigation of the external strain, internal stress and fiber phase transformation behavior of a NiTi actuated aluminum metal matrix composite. Acta Mater. (1998) v. 46 p. 3455-3466

7. Barlow, C. Y.; *Liu, Y. L.*, Microstructure, strain fields and flow stress in deformed metal matrix composites. Acta Mater. (1998) v. 46 p. 5807-5817

8. Bouchard, R.; Hupfeld, D.; Lippmann, T.; Neuefeind, J.; Neumann, H. B.; *Poulsen, H. F.*; Rutt, U.; Schmidt, T.; Schneider, J. R.; Sussenbach, J.; Zimmermann, M. von, A triple-crystal diffractometer for high-energy synchrotron radiation at the HASYLAB high-field wiggler beamline BW5. J. Synchrotron Radiat. (1998) v. 5 p. 90-101

9. *Brøndsted, P.*; Skov-Hansen, P., Fatigue properties of high-strength materials used in cold-forging tools. Int. J. Fatigue (1998) v. 20 p. 373-381

10. Chen, F.; *Toft Sørensen, O.*; Meng, G.; Peng, D., Thermal decomposition of $BaC_2O_4 \cdot 0.5H_2O$ studied by stepwise isothermal analysis and non-isothermal thermogravimetry. J. Thermal Anal. (1998) v. 53 p. 397-410

11. Chen, F.; *Toft Sørensen, O.*; Meng, G.; Peng, D., Preparation of Nd-doped $BaCeO_3$ proton-conducting ceramic and its electrical properties in different atmospheres. J. Eur. Ceram. Soc. (1998) v. 18 p. 1389-1395

12. *Christoffersen, H.*; *Leffers, T.*, The orientation of dislocation walls in rolled copper relative to the sample coordinate system. Acta Mater. (1998) v. 46 p. 4093-4102

13. *Clausen, B.*; *Lorentzen, T.*; *Leffers, T.*, Self-consistent modelling of the plastic deformation of FCC polycrystals and its implications for diffraction measurements of internal stresses. Acta Mater. (1998) v. 46 p. 3087-3098

14. Doherty, R. D.; Hughes, D. A.; Humphreys, F. J.; Jonas, J. J.; *Juul Jensen, D.*; Kassner, M. E.; King, W. E.; McNelley, T. R.; McQueen, H. J.; Rollett, A. D., Current issues in recrystallization: A review. Mater. Sci. Eng. A. (1997) v. 238 p. 219-274

15. Edwards, D. J.; *Singh, B. N.*; *Toft, P.*; *Eldrup, M.*, The effect of bonding and bakeout thermal cycles on the properties of copper alloys irradiated at 100 degrees C. J. Nucl. Mater. (1998) v. 258-263 p. 978-984

16. Edwards, D. J.; *Singh, B. N.*; *Toft, P.*; *Eldrup, M.*, Comparison of properties and microstructures of Tréfimétaux and Hycon 3HP ™ after neutron irradiation. In: Fusion materials. Semiannual progress report for the period ending June 30, 1998. DOE/ER-0313/24 (1998) p. 183-188

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19. Fisker, R.; *Poulsen, H. F.*; Schou, J.; Carstensen, J. M.; *Garbe, S.*, Use of image-processing tools for texture analysis of high-energy X-ray synchrotron data. J. Appl. Cryst. (1998) v. 31 p. 647-653

20. *Garbe, S.*; *Juul Jensen, D.*; *Poulsen, H. F.*; *Krieger Lassen, N. C.*; Raabe, D., Through-thickness texture variations determined non-destructively by high energy synchrotron radiation. Mater. Sci. Forum (1998) v. 273-275 p. 271-276

21. *Godfrey, A.*; *Juul Jensen, D.*; *Hansen, N.*, Slip pattern, microstructure and local crystallography in an aluminium single crystal of copper orientation {112}<111>. Acta Mater. (1998) v. 46 p. 835-848

22. *Godfrey, A.*; *Juul Jensen, D.*; *Hansen, N.*, Slip pattern, microstructure and local crystallography in an aluminium single crystal of brass orientation {110}<112>. Acta Mater. (1998) v. 46 p. 823-833

23. Grabis, J.; Kuzjukevics, A.; Rasmane, D.; *Mogensen, M.*; *Linderoth, S.*, Preparation of nanocrystalline YSZ powders by the plasma technique. J. Mater. Sci. (1998) v. 33 p. 723-728

24. *Hansen, N.*; *Huang, X.*, Microstructure and flow stress of polycrystals and single crystals. Acta Mater. (1998) v. 46 p. 1827-1836

25. *Hansen, U.*, Compression behavior of FRP sandwich specimens with interface debonds. J. Compos. Mater. (1998) v. 32 p. 335-360

26. Hattel, J.H.; *Pryds, N. H.*, On the lack of dependence of extent of columnar growth on wheel speed for melt-spun 12Cr-Mo-V steel. Scr. Mater. (1998) v. 38 p. 723-727

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29. *Huang, X.*; *Liu, Q.*, Determination of crystallographic and macroscopic orientation of planar structures in TEM. Ultramicroscopy (1998) v. 74 p. 123-130

30. Hughes, D. A.; Chrzan, D. C.; *Liu, Q.*; *Hansen, N.*, Scaling of misorientation angle distributions. Phys. Rev. Lett. (1998) v. 81 p. 4664-4667

31. *Jacobsen, T. K.*; *Sørensen, B. F.*; *Brøndsted, P.*, Measurement of uniform and localized heat dissipation induced by cyclic loading. Exp. Mech. (1998) v. 38 p. 289-294

32. *Johannesson, B.*; *Pedersen, O. B.*, Analytical determination of the average Eshelby tensor for transversely isotropic fiber orientation distributions. Acta Mater. (1998) v. 46 p. 3165-3173

33. *Johansen, B. S.*; *Lystrup, Aa.*; Jensen, M. T., CADPATH: A complete program for the CAD-CAE- and CAM-winding of advanced fibre composites. J. Mater. Process. Technol. (1998) v. 77 p. 194-200

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35. *Kindermann, L.*; Das, D.; Bahadur, D.; Nickel, H., Influence of iridium on the reactivity of $LaFeO_3$ base perovskites. Solid State Ionics (1998) v. 106 p. 165-172

36. *Krieger Lassen, N. C.*, A new procedure for automatic high precision measurements of the position and width of bands in backscatter Kikuchi patterns. Mater. Sci. Forum (1998) v. 273-275 p. 201-208

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38. Larsen, J. G.; *Larsen, P. H.*; *Bagger, C.*, High temperature sealing materials. US provisional patent application 60/112039

39. *Larsen, P. H.*; James, P. F., Chemical stability of $MgO/CaO/Cr_2O_3$-Al_2O_3-B_2O_3-phosphate glasses in solid oxide fuel cell environment. J. Mater. Sci. (1998) v. 33 p. 2499-2507

40. *Leffers, T.*; *Christoffersen, H.*, The two-dimensional orientation distribution of planar microstructural features. Mater. Sci. Forum (1998) v. 273-275 p. 77-85

41. *Lienert, U.*; Schulze, C.; Honkimäki, V.; Tschentscher, T.; *Garbe, S.*; Hignette, O.; *Horsewell, A.*; Lingham, M.; *Poulsen, H. F.*; *Thomsen, N. B.*; Ziegler, E., Focusing optics for high-energy X-ray diffraction. J. Synchrotron Radiat. (1998) v. 5 p. 226-231

42. *Linderoth, S.*, Oxidation of $Cr_{94}Fe_5(Y_2O_3)_1$ at high temperatures. High Temp. Mater. Process. (1998) v. 17 p. 217-222

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48. *Lorentzen, T.*; Faurholdt, T.; *Clausen, B.*; Danckert, J., Characterization of residual stresses generated during inhomogeneous plastic deformation. J. Strain Anal. Eng. Des. (1998) v. 33 p. 243-252

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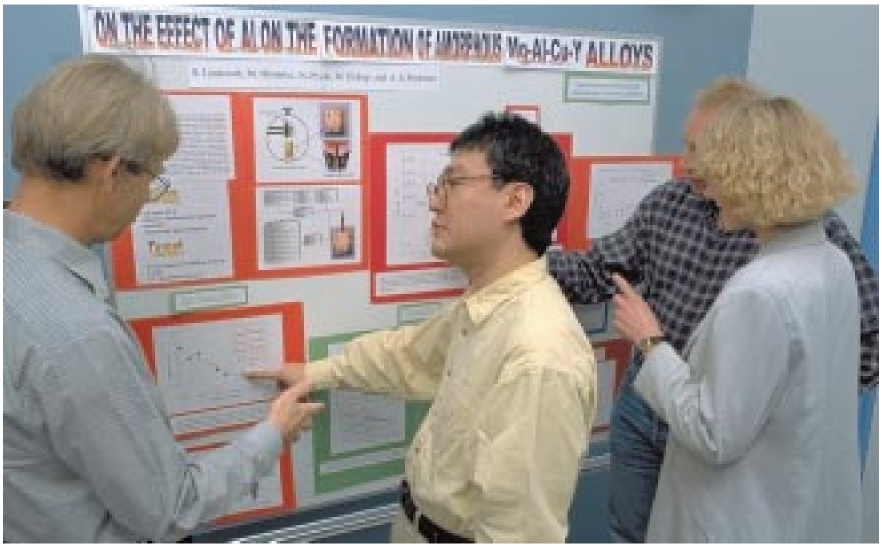

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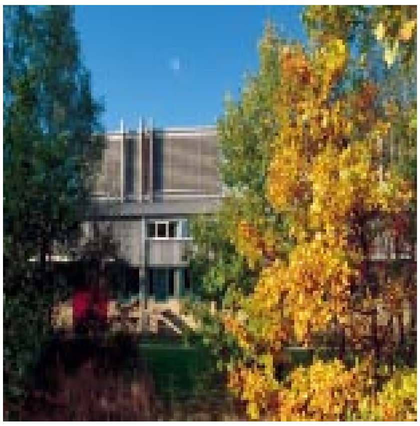

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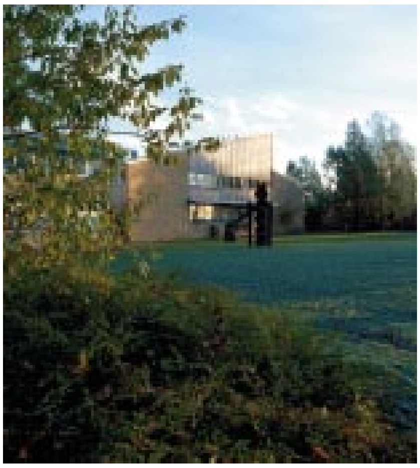


ACRONYMS AND ABBREVIATIONS

AAU Aalborg University

AU Aarhus University

BCC Body centred cubic. A crystal structure

BET A method for measuring surface area of powder named after Brunauer, Emmett and Teller

BiSCCO $Bi_2Sr_2Ca_2Cu_3O_x$. A material with superconducting properties

BRITE- EURAM An EU programme, Basic Research in Industrial Technology for Europe

CCD Charge coupled devices

CMC Ceramic matrix composites

COM Crystal orientation microscopy

CSL Coincident site lattice

dpa Displacements per atom

DR3 Danish Reactor 3. A nuclear research reactor at Risø

DSC Differential scanning calorimetry

DTA Differential thermal analysis

DTU The Technical University of Denmark

EBSP Electron back-scattering patterns

EDS Energy dispersive (X-ray) spectrometry

EELS Electron energy loss spectrometry

EFP The Energy Research Programme of the Danish Ministry of Environment and Energy

ELKRAFT The electrical utility group of the Danish island Zealand

ELSAM The electrical utility group of the Danish mainland Jutland and the island Funen

ESEM Environmental scanning electron microscope

ESRF European Synchrotron Radiation Facility in Grenoble, France

EUCLID European cooperation for the long term in defense

EUREKA Association for European Market oriented R&D

FCC Face centered cubic. A crystal structure

FEM The finite element method

HASYLAB A synchrotron radiation laboratory in Hamburg, Germany

ITER International Thermo-nuclear Experimental Reactor

IVC Engineering Science Centre. A programme under the Danish Technical Research Council

JOULE-THERMIE An EU programme on non-nuclear energy and rational exploitation of energy

KU University of Copenhagen

LEU Low enriched uranium. Fuel for nuclear test reactors

LIP Large Installation Programme An EU programme

LSC Lanthanum strontium chromite. A ceramic material used as interconnect in SOFCs.

LSM Lanthanum strontium manganite. A porous ceramic material used in SOFCs

LVSEM Low vacuum scanning electron microscopy

MMC Metal matrix composites

MUP The Danish Materials Technology Programme

NDC Non-destructive characterisation

NEB Nudged elastic band. A simulation method

NORFA Nordic Academy for Advanced Study. An organization set up by the Nordic Council to promote educational activities for research students

OU University of Odense

PAS Positron annihilation spectroscopy

PBM Production bias model

PMC Polymer matrix composites

PP Polypropylene

SEM Scanning electron microscope

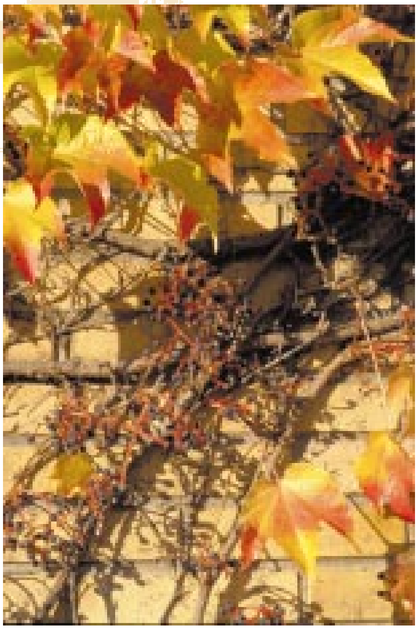
SOFC Solid oxide fuel cell.

TEM Transmission electron microscopy

TGA Thermogravimetric analysis

XRD X-ray diffraction

YSZ Yttria stabilized zirconia



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ISBN 87-550-2506-4
ISBN 550-2507-2 (internet)
ISSN 0106-2840
ISSN 1397-8071

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