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Materials Research Department

Annual Report 1997

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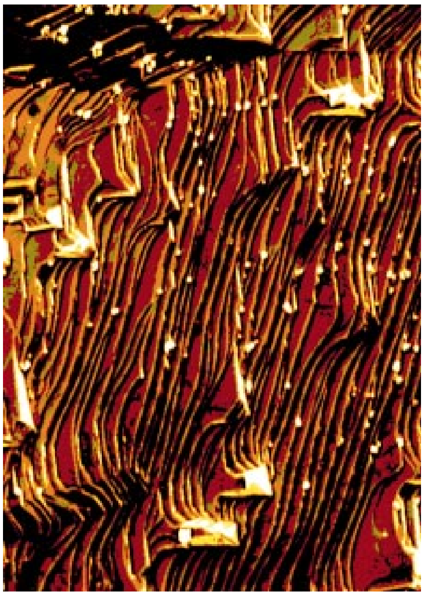

Materials Research Department

Annual Report 1997

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ABSTRACT

Selected activities of the Materials Research Department at Risø National Laboratory during 1997 are described. The scientific work is presented in four chapters: Materials Science, Materials Chemistry, Materials Engineering and Materials Technology. A survey is given of the Department's participation in international collaboration and of its activities within education and training. Furthermore, the main figures outlining the funding and expenditure of the Department are given. Lists of staff members, visiting scientists, publications and other activities are included.

Risø-R-1013(EN)

Published by the Materials Research Department, Risø National Laboratory
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Cover composed of scanning electron microscope images of microstructures of materials having been subjected to high temperatures.

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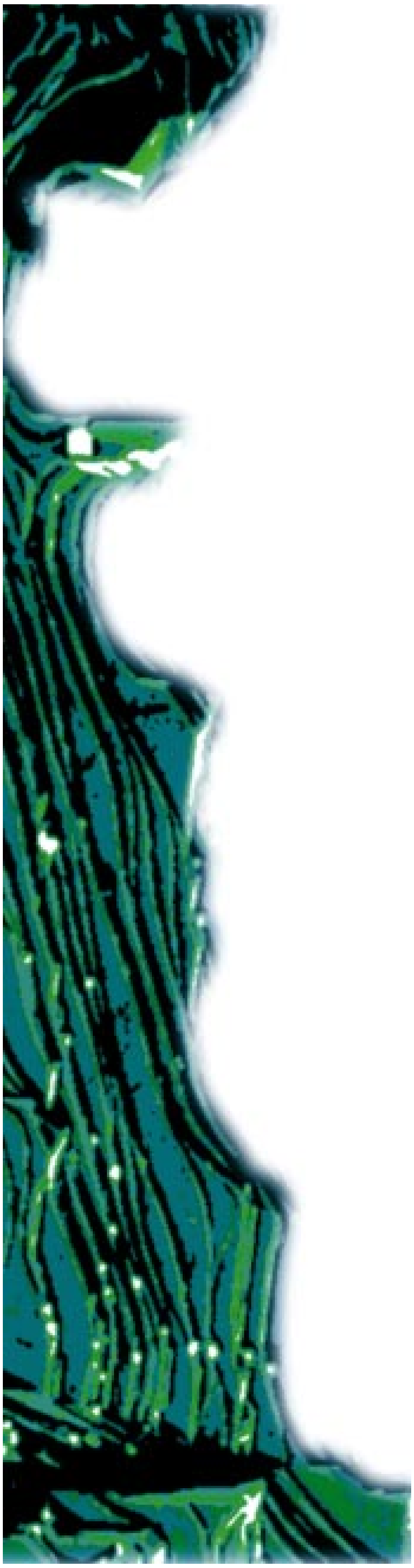

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INTRODUCTION
– Materials Research Department 1997

The international evaluation of Risø and of the Department in 1996/97 was very positive with respect to the quality of our scientific and technological work. On this basis, the evaluation panel suggested that Risø should further strengthen its many links to society. It was emphasized that collaboration with universities and industry should be increased to make better use of Risø resources. This means that future activities must demonstrate solid links between research and teaching and between basic work and industrial applications. Guidelines for these efforts were later laid down in the form of specific milestones in a 4 year contract between Risø and the Ministry of Research. This contract also contained the guarantee that the financial contribution from the Government to Risø's activities will remain unchanged until the end of year 2001. Thus, to maintain the current activity level, external funding (which supplements the Government funding) must be maintained at least at present levels. Fortunately, external funding has shown a slight increase in 1997, with a further increase expected in 1998. This positive development, however, just balances increases in salaries and operating expenses and thereby precludes any substantial new investments. We are, however, glad that joint funding from the Danish Technical Research Council and from Risø's central fund has allowed us to enter into a collaborative agreement with the European Synchrotron Radiation Facility (ESRF) in Grenoble, France. The aim of this collaboration is the construction of a high energy X-ray diffractometer. This instrument, to be operative in 1998, will provide breakthroughs in materials research by allowing in-depth structural analysis with high spatial resolution; a radically new technique, dubbed three dimensional X-ray microscopy.

Achievements

Many years of research was concluded when Dorte Juul Jensen defended her Dr. technices thesis entitled 'Orientation Aspects of Growth During Recrystallization'. The defence took place at the Technical University of Denmark and the event was given more than usual attention as it was the first time that a woman obtained this degree. The staff of the Department now includes four scientists with this title.

As a further recognition of the quality of basic research in the Department, The Engineering Science Centre for Structural Characterization and Modelling, financed jointly by The Danish Technical Research Council and Risø, was extended by a further 5 years until 2002. Also to be mentioned is that electron microscopy has provided important new information on the microstructure and the chemical composition of materials ranging from conventional metals like aluminium, copper and nickel to complex ceramic materials used in high temperature superconductors and in solid oxide fuel cells. Measurements of internal stresses in polycrystals, thin layered structures and composites have also progressed well using neutron, electron and X-ray diffraction.

Other achievements concern the research and development carried out in collaboration with industry. An important theme has been composite materials, especially in the development and optimisation of thermoplastic composites and associated process technologies. There is a strong industrial interest in this type of materials since reduced occupational health hazards can be combined with improved properties of products, for example for windmills and automobiles. This potential has led to the formation of a consortium of 6 Danish industries which, together with the Department and the Danish Technological Institute, has formulated a 4-year research, development and demonstration programme.

Also in the composites area, materials with good strength and stiffness have been produced using natural fibres from trees and plants. One aim of this programme is to improve recyclability of products in the automotive and packaging industries. Finally, metal matrix composites with extremely high wear resistance have been developed in collaboration with Danish and foreign industries.

Dorte Juul Jensen (centre) defended her Dr. technices thesis in the presence of the the Minister of Research (left) and the director of the Technical University of Denmark (right).

Good progress has also been achieved in the energy programmes carried out mainly under the sponsorship of the Danish Ministry of Environment and Energy. The aim here is to improve efficiency and reduce detrimental environmental effects of energy production, transport and usage. These programmes are (i) Solid Oxide Fuel Cells, (ii) High Temperature Superconductors, (iii) Windmill Wings, and (iv) Flywheels. In these programmes, large and medium size Danish firms are the industrial partners. Staff from these companies participate actively in the work in the Department with the aim of integrating our joint research and development activities.

On the European level, the Department has participated in a number of programmes within EU's 4th Framework programme, mainly BRITE-EURAM and JOULE-THERMIE. In 1997, the Department has co-ordinated three BRITE-EURAM proposals and participated in two. Three of these five proposals have now been approved covering such activities as forming, non-destructive testing and high temperature materials. Danish industry participates in two of these programmes. These new contracts will bring the Department's annual income from EU's research programmes up to about 9 mill. Danish kroner (DKK). This is about 25 % of the total external funding; this was the target that was set in the beginning of the 4th framework programme about 4 years ago.

Also, educational activities may be mentioned. Teaching activities range from teaching high school students and high school teachers at Risø to external lecturing of undergraduate and graduate courses at the Technical University of Denmark and Aarhus University. To strengthen our contact with Danish universities, the Department has established facilities for distance learning and video conferencing in a centre initiative together with 7 universities. Funding was partly provided by the Ministry of Education; the centre was inaugurated by the Minister in May.

Video conferencing was used for teaching university students using two way video links (distance learning) and for video conferences.

Finally, we held the 18th International Risø Symposium on Materials Science in September. The title was 'Polymeric Composites - Expanding the Limits'. The 19th symposium, to be held in September 1998, has the title 'Modelling of Structure and Mechanics of Materials from Microscale to Product'.

The coming years

With the present portfolio of programmes, most of the activities in the coming year or two will be along lines known today. However, as more than 50 % of the funding of the Department's activities comes from external sources, changes around us will have a great impact on the future directions of our research. An example is EU's Framework programme, planned to start in 1999. The programme will, without doubt, be agreed upon and will almost certainly contain an increase in budget compared with the present programme. A change in priorities can also be predicted, but the funding of specific areas is at present unknown. It is therefore mandatory for the Department to be proactive as before in the formulation of European and Danish research programmes and this will require great flexibility. In this situation we are in the fortunate position that the many large and complex programmes that we have completed over the years may also be seen as having provided good training. Such programmes have shown us the benefit of a good and friendly collaboration within the whole Department. We are therefore confident that we both have the competence and the spirit to meet the changes occurring around us.

To deal with developments taking place in society, a broader customer base must be aimed at. Here, it is very positive that the main requirement in the new contract between Risø and the Ministry of Research is specifically that we expand our contact with industry and universities. Thus, what looks like an imposed political demand is in fact fully in accord with our visions for the future. Many new activities are planned in order to follow the intentions in this contract. Both for these new activities and for the old ones it is a requirement that the research and development must be both focused and on an international level. It is only by maintaining these research standards that we can be qualified as partners for Danish and international industry, be inspiring teachers at the university level and provide solutions to problems arising in our society within sectors where materials are developed, produced and utilized.

MATERIALS SCIENCE
– theory and characterization

The research activities to be described within this programme area cover a very wide range of subjects. The emphasis is on basic and strategic research, but all projects have a clear relation to potential technological applications.

The Engineering Science Centre for Structural Characterization and Modelling of Materials, financed jointly by the Danish Technical Research Council and Risø, is managed within the programme area, but the centre also includes many activities from other programme areas (and from the Condensed Matter Physics and Chemistry Department at Risø). The establishment of this centre, in 1993, has contributed very significantly to the programme area and to its interaction with the other programme areas.

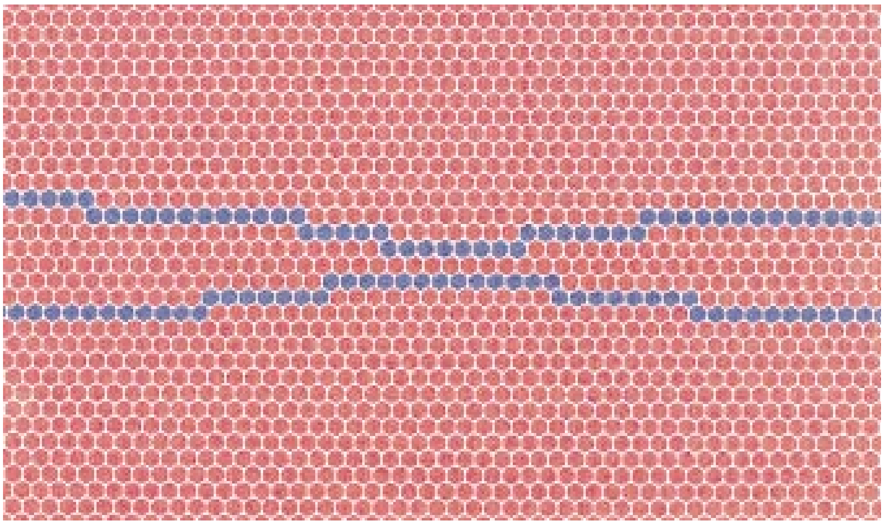
Among the highlights one may point to (i) identification of the cross-slip mechanism in FCC metals by modelling at the atomic scale, (ii) experimental verification of diffusional creep via microstructural observations, (iii) demonstration of a clear relation between the microstructure and the crystallographic orientation of grains in tensile deformed polycrystals, (iv) experimental confirmation of the production bias model for defect accumulation during irradiation, (v) final decision by the European Synchrotron Radiation Facility to establish a dedicated beam line for 3D X-ray microscopy in collaboration with Risø and (vi) improvement of the methods for correction of beam-skirt effects in X-ray spectrometry in poor-vacuum scanning electron microscopes.

Project Funded Research: Materials Science

Project type	Project name	Co-participants
Danish Technical Research Council, Engineering Science Centre (IVC)	Structural Characterization and Modelling of Materials	• Condensed Matter Physics and Chemistry Department, Risø
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 Labaratory, NBI, 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)	• Inst. of Manufacturing Eng., 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 University of Science and Technology, NTNU, Norway • Ecoles des Mines de Saint-Etienne, France • Swedish Institute for Metal Research, Sweden • Norwegian Inst. 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, 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
EU-Fusion Materials Technology Programme (EU-FTP) Long Term Materials Programme	Effects of Irradiation on Deformation Behaviour of Iron and Low Activation Steels	• EU-sponsor, in collaboration with: - CRRP, Lausanne, Switzerland - Research Centre Jülich, Germany - AEA Technology, Harwell, UK
EU-FTP Underlying Technology	Effects of Irradiation on Physical and Mechanical Properites of Metals	• EU-sponsor, in collaboration with: - Oak Ridge National Laboratory, USA - London University, UK - Research Centre Jülich, Germany - Inst. of Physics and Power Engr., Obninsk, Russia - Pacific Northwest National Laboratory, USA
EU-FTP International Thermonuclear Experimental Reactor R & D (ITER)	Copper and Copper Alloys Irradiation Testing for First Wall and Divertor	• EU-sponsor, in collaboration with: - Pacific Northwest National Laboratory, USA - University of Illinois, USA - VTT Manufacturing Technology, Finland

Materials models and materials structures

This programme concerns the development of micromechanical models for the mechanical behaviour of single- and multi-phase materials, including the (multi-phase) microstructure. The models are related to micromechanics and the basic physics as reflected in dislocation processes and, in certain contexts, processes at the atomic level.



The new method of simulation, applied to a computer crystal of copper with 3.4 million atoms, identifies the lowest-energy cross-slip path through configuration space. The path corresponds to the mechanism proposed by Friedel and Escaig. The blue-coloured atoms show a configuration close to constriction.

Cross-slip of dislocations studied by a new atomistic method

Among the many different dislocation reactions taking place during plastic deformation of materials, cross-slip is of special interest, because the cross-slip frequency seems to control the evolution of the deformation induced dislocation microstructures as well as the growth of short fatigue cracks. Cross-slip is the mechanism by which a dissociated screw dislocation changes its glide plane.

Different models for the cross-slip mechanism have been proposed, but it has not been possible so far to determine the exact mechanism by simulation or experiment. The reason lies in the complexity of the problem; a proper treatment of cross slip of a dissociated screw dislocation from the primary glide plane to the cross-slip plane requires atomistic three dimensional simulations, which have only recently become feasible.

In the present work (in collaboration with the Institute of Physics, the Technical University of Denmark, where a model technique has been developed) the problem of determining the cross-slip mechanism was addressed with the 'nudged elastic band' method. This method is an atomistic method particularly well suited to find optimal transition paths through configuration space in cases where simpler methods are not adequate. To find the optimal transition path, i.e. to determine how the dissociated screw dislocation changes its glide plane, the method needs only the initial and final state of the dislocation, but, importantly, does not include any information about the transition path itself. The simulations were carried out on a parallel computer, and each simulation modelled roughly 3.4 x 10^6 atoms.

The cross-slip process was found to be initiated by the formation of a constriction of the dislocation in the primary glide plane. The short segment of perfect screw dislocation then redissociates into the cross-slip plane, thereby taking the dislocation from the primary to the cross-slip plane via a transition state with two twisted dislocation constrictions. This cross-slip mechanism is the mechanism by Friedel and Escaig.

Grain shape and lattice rotation during plastic deformation

In theories for the formation of deformation texture one normally focuses on the slip pattern - with the assumption that a specific combination of slip systems leads to a specific texture. However, this assumption is not correct. A given slip pattern may theoretically lead to quite different textures depending on the rules used for the calculation of the lattice rotations producing the texture.

For rolling deformation (or plane-strain deformation) one may use two different sets of rules for the lattice rotations: 'mathematical analysis' (MA) and 'plane strain analysis' (PSA). It was demonstrated (in collaboration with Universidad Nacional de Rosario, Argentina) that an advanced self-consistent model for the plastic deformation of polycrystals includes a systematic selection of the appropriate rotation rule depending on the shape of the grains. Equiaxed grains lead to MA, and flattened and elongated grains lead to PSA. For low strains (with equiaxed grains) the model selects MA. Increasing strain (with increasing flattening and elongation of the grains) leads to a continuous transition towards PSA. The theoretically predicted transition to PSA is based on the assumption that a flattened and elongated grain can be considered to be a homogeneously deforming entity, which is not necessarily correct. This is a question which is rather fundamental for the modelling of polycrystal deformation at higher strains altogether.

The two-dimensional orientation distribution of planar microstructural features

The orientation distribution of planar microstructural features like dislocation walls, twin lamellae and plate-shaped precipitates is expressed in a two-dimensional pole figure (expressing the distribution of the normals or the poles of the planar features). The experimental observations are one-dimensional orientation distributions of the traces of the planar features on different planar sections as observed by microscopical techniques (optical microscopy or electron microscopy).

The synthesis of the two-dimensional orientation distribution from the one-dimensional trace distributions corresponds to the fundamental procedure in the analysis of crystallographic texture, viz. the synthesis of the three-dimensional orientation distribution function (ODF) from the two-dimensional pole figures. And the basic problem is the same: the experimental information is insufficient for a unique solution.

It has been shown that there is an analytical solution for the synthesis of the two-dimensional orientation distribution of planar features from observation of the trace distributions in two planar sections. Depending on various conditions this analytical solution may or may not come close to the 'proper' solution.

On the basis of the present work, new and more sophisticated procedures are suggested. The applicability of these procedures remains to be tested in subsequent theoretical and experimental work.

Dislocation processes in cyclic plasticity of copper-zinc polycrystals

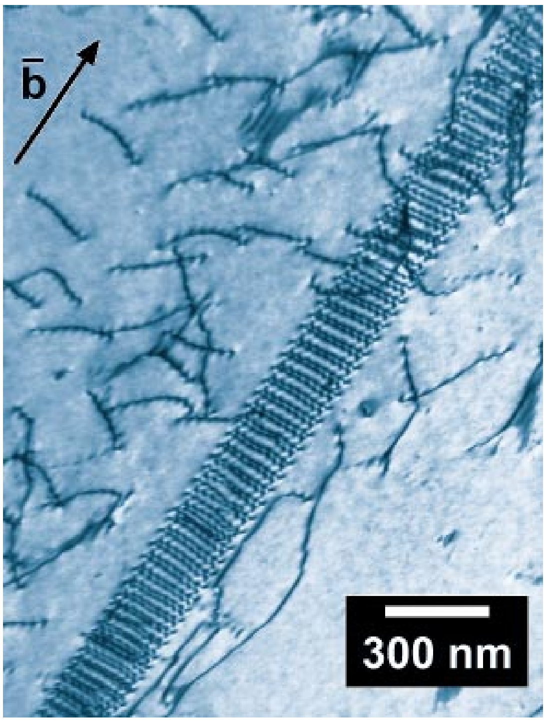
Cyclic plastic deformation of pure metals involves frequent cross-slip events (wavy slip). When alloying elements are added there is usually a transition to planar slip, which apparently involves a lower cross-slip frequency. This type of behaviour is observed when zinc is added to copper. The usual explanation is that reduced stacking fault energy (SFE) causes reduced cross-slip frequency. However, experiments show that planar slip cannot always be correlated with a low SFE. Therefore, short range ordering (SRO) of the alloying atoms into submicroscopic clusters has been suggested to provide an alternative explanation of planar slip.

The roles of SFE and SRO were both considered in the interpretation of a transmission electron microscope (TEM) study of the recently observed stages of primary hardening, softening and secondary hardening during cyclic plasticity of polycrystalline Cu-30%Zn. It was found that specimens strained into the primary hardening stage contained arrays of dislocation dipoles in comparatively undislocated crystal. The onset of softening was accompanied by formation of intense shear bands (ISBs) forming preferentially at grain boundaries and destroying the initial dipole arrays. In the stage of secondary cyclic hardening the polycrystal was found to contain a fairly uniform structure of planar dislocation arrays.

These observations appear to be explained equally well in terms of SFE and SRO: During primary hardening statistically 'softer' regions of the solute hardened crystal undergo localized plastic shear with formation of dipole arrays, possibly by a cross-slip mechanism. The local dislocation hardening by the dipole arrays shifts the localized shear to less soft regions, thus causing overall primary hardening. Softening is initiated at a stress amplitude sufficient to trigger formation of ISBs, which destroy the dipole arrays or the hardening by SRO. Some planar slip materials, such as nitrogen alloyed austenitic steels, display very short primary hardening stages, suggesting that their cyclic softening is due mainly to destruction of SRO.

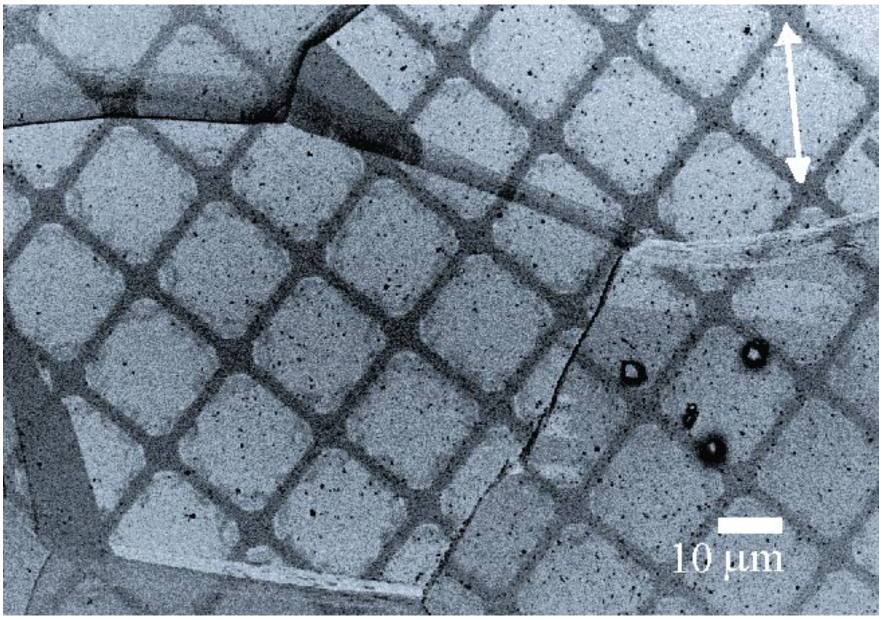
Modelling scale-dependent work hardening in copper-tungsten composites

The prediction and optimization of metal matrix composite behaviour necessitates an understanding of the influence exerted by the reinforcement on plastic deformation of the matrix. Tensile experiments show that this



TEM micrograph of polycrystalline Cu-30%Zn cyclically strained into the primary hardening stage at a plastic strain amplitude of ϵ_p = 0.0034 (the arrow indicates the primary slip direction). A regular array of dislocation dipoles is em-bedded in comparatively undislocated crystal.

influence can be substantial in the model system of copper reinforced with a volume fraction V_f of thin parallel tungsten fibres. Thus the *in-situ* matrix hardening rate is found to be approximately equal to the matrix elastic shear modulus times $V_f/(1-V_f)$. A new model for this striking effect was proposed (in collaboration with MIT, USA) on the basis of two observations. The first observation is that during fabrication of the composites by liquid infiltration the fibres tend to cluster into close packed bundles. The second observation is that there is a critical value of V_f of approximately 0.5, above which the matrix remains non-yielding during a tensile experiment. A comparison between predictions from this new fibre cluster model and predictions from an earlier elastic friction model shows that both models provide quite acceptable agreement with the available experimental observations. The two models are comparable in their basic hardening mechanism, since both invoke dislocation mechanisms for the occurrence of non-yielding regions of matrix. The models therefore both capture the dependence of the *in-situ* matrix hardening rate on V_f, but they differ somewhat in regard to the observed dependence of work hardening on fibre diameter. In the elastic friction model the dependence is explained entirely as a scale-dependent stress relaxation rate, which subtracts from the matrix hardening rate. In the clustering model a possible source of scale-dependence can be found in the expected increase in punched dislocation density with decreasing fibre diameter and in the dependence on the extent of clustering. Thus, the clustering model adds the new possibility that the scale-dependence of work hardening arises from a scale-dependent production and storage of dislocations inside the fibre clusters during processing of the composite.



Scanning electron micrograph of a sample of Cu-2wt%Ni deformed 2.4 % in the diffusional creep regime. The sample is covered with a grid of alumina so that the deformation at the individual boundaries can be measured from the displacement of the grid. The arrow indicates the tensile direction.

Influence of grain boundary structure on diffusional creep

A fine-grained material may under certain conditions deform by diffusion of material from grain boundaries in compression to grain boundaries in tension. This deformation mode is called diffusional creep.

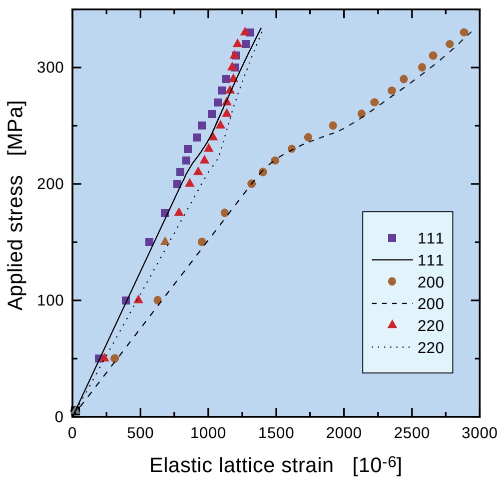
In order to achieve a better understanding of the processes occurring at the individual boundaries, a tensile specimen of Cu with 2 wt % Ni was deformed in the diffusional creep regime. A periodic fiducial grid of alumina was deposited on the specimen prior to creep. After creep the magnitude of deposition (or removal) of material and the magnitude of grain boundary sliding could then be measured at individual boundaries from the displacements of the grid combined with a knowledge of the orientation of the grain boundary plane. The misorientation across the grain boundaries was determined from electron back scattering patterns.

The experiment unambiguously demonstrated that material was deposited on boundaries in tension during creep and that material was removed from some of the longitudinal boundaries. In a model which we have previously developed it is assumed that vacancies can be absorbed or emitted only at grain boundary dislocations. A number of the predictions derived from this model have been corroborated by the experiment, e.g. (i) deposition (or removal) of material at grain boundaries is coupled to grain boundary sliding and migration (the coupling depends on the grain boundary structure, and therefore the climb/glide ratio varies from boundary to boundary), (ii) negative sliding may occur at some transverse boundaries, (iii) deposition of material may occur at some longitudinal boundaries, and (iv) boundaries close to an exact coincident site lattice misorientation are inactive during the deformation.

Evaluation of polycrystalline modelling scheme by neutron diffraction

Modelling the mechanics of polycrystal deformation has been a central issue in materials science since the early attempts by Taylor and Sachs 60 - 70 years ago. The state of the art in terms of theoretical analytical modelling schemes is the so-called 'self-consistent' approach. Such a modelling scheme has now been fully implemented and with the aim to perfect this approach it is now undergoing experimental evaluation by neutron diffraction. Whereas earlier work on evaluating such modelling schemes has focused on evaluation of the models ability to predict texture evolution, the current evaluation of the evolution of lattice strains and stresses is more specifically addressing the mechanics of deformation at a grain size scale. By performing *in-situ* loading experiments on the neutron diffraction spectrometer, we can selectively choose to monitor the lattice strain evolution in specific families of grains all being characterized by having a specific crystallographic orientation along a specific direction in the sample. Such experimental observations of lattice strain evolution are well matched to the level of detail in the modelling scheme. We have, as such, a unique opportunity to evaluate the model predictions based on direct experimental observations.

An example is the evolution of lattice strains in selected families of grains in a uniaxially loaded stainless steel specimen. Plasticity was found to develop much earlier on a grain size scale than as defined macroscopically for engineering purposes. This early onset of plasticity can furthermore be quantified by focusing on the crystallographic slip pattern predicted by the model. Plasticity initiates in grains with a <531> or a <331> orientation along the macroscopic deformation axis. With increasing stress, plasticity subsequently spreads to those grains with a <100> orientation along the deformation axis.



Comparison of lattice strain evolution in selected grain orientations in a uniaxially loaded stainless steel specimen. Symbols indicate experimental observations by neutron diffraction, while the lines show model predictions based on a self-consistent modelling scheme. The non-linearities develop before reaching the stress level which, in an engineering sense, would be defined as the elastic limit (275 MPa).

Local deformation monitored by synchrotron radiation

The next generation of finite element methods (FEM) for polycrystal deformation operates at a higher level of detail encountering specific grain-to-grain interactions. This calls for novel experimental techniques providing a bulk spatial resolution comparable with the grain size; typically an order of magnitude better than achievable by neutron diffraction. During 1997 the Department has been engaged in pioneering X-ray diffraction experiments at the European Synchrotron Radiation Facility (ESRF) in Grenoble to demonstrate the potential for investigating single grains in bulk material.

Pre-strained stainless steel samples were investigated using an incident beam cross section of merely 0.4 x 0.4 mm^2 and a photon energy of 68 keV. This allowed full penetration of the Ø 4.5 mm steel specimens while monitoring diffraction peaks for single grains along the beam path through the specimen. For each local volume combined $\omega/2\theta$-scans were performed to assure full 2D-mapping of the diffraction peaks. For specimens with different plastic strain a dramatic broadening of the diffraction peaks was found in the ω-direction as a function of the plastic deformation history.

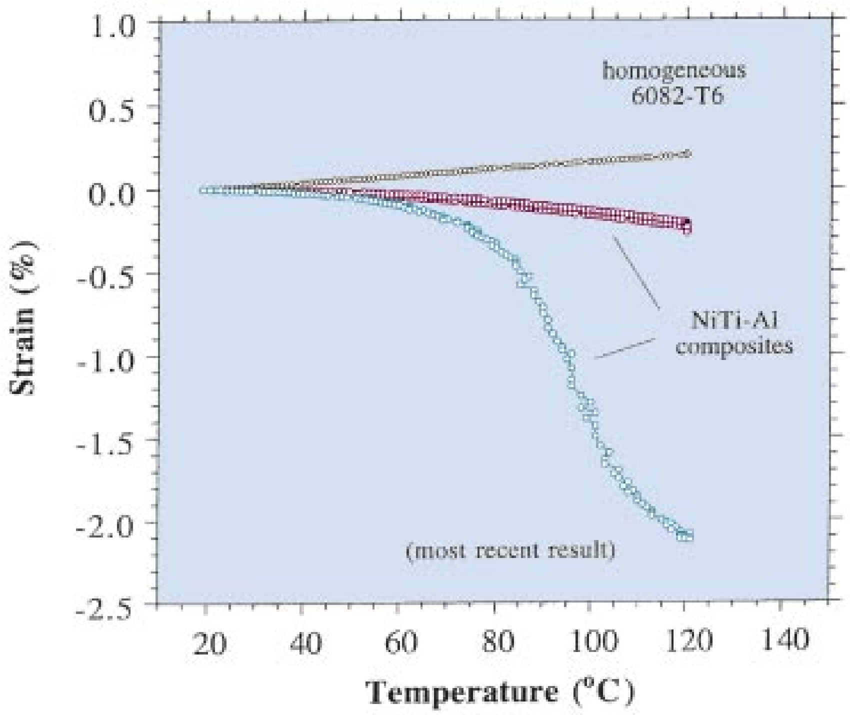
It was demonstrated that high energy X-ray diffraction is a very powerful tool for studying local aspects of deformation in polycrystalline samples. Thus, as a complimentary technique to neutron diffraction it is becoming a valuable and necessary tool for the evaluation of the detailed micro-mechanics encountered by the next generation of models for polycrystal deformation.

Thermomechanical response of shape active composites

Smart materials or structures have been a challenge to materials scientists for many years. One of the most attractive metallic candidates for such materials systems is the shape memory alloys of which most work has focussed on the nickel titanium system (NiTi). The attractive characteristics of these alloys are the stress and temperature driven crystallographic transformations these alloys can undergo. This phase transformation is accompanied by unusual changes in mechanical properties and crystallographic identity, and holds opportunities for tailor-making structures with properties which would be impossible to obtain in traditional alloy systems. These special properties of shape memory alloys also make them attractive candidates for reinforcements in composite systems, and for two years the Department has been engaged in the challenge to produce metal based composites with shape memory alloy fibre reinforcements.

The phase transformation characteristics of the shape memory alloys are well documented, and found to be extremely sensitive to the thermomechanical treatment of the materials. It is hence a challenge to manufacture composites through a thermomechanical processing route while retaining the phase transformation characteristics of embedded fibres of shape memory alloys.

As an illustration such a system has been brought to a state where it shows *thermal contraction* during heating in contrast to the regular thermal expansion of metals. The achieved 2.2 % thermal contraction is quite astonishing and points towards the prospects of



Thermal contraction of aluminium based composites with fibres of a shape memory alloy. During heating the fibres undergo reverse transformation from martensite to austenite resulting in a thermal contraction of the composite, in contrast to the usual thermal expansion of metals, as shown for the unreinforced control specimen.

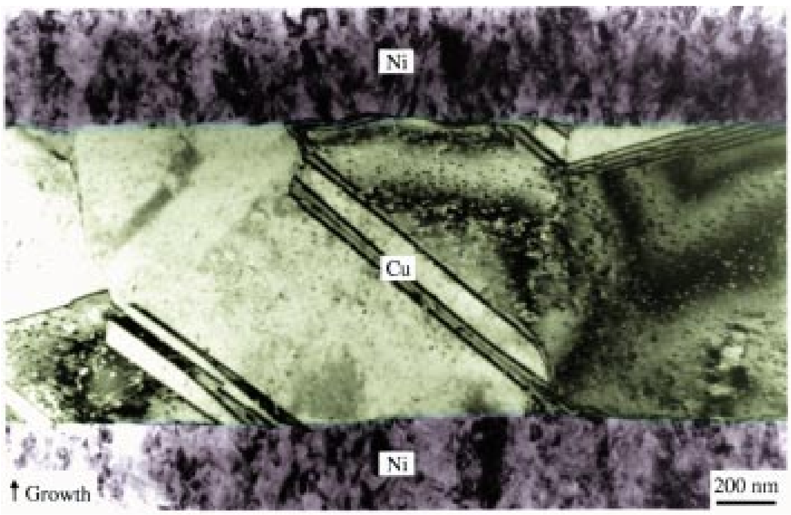
developing structures e.g. with zero thermal expansion or tailor-made thermal expansion (or contraction). The measurements were done *in-situ* on the neutron diffraction spectrometer while continuously monitoring the ongoing interior phase transformation of the embedded fibres. The spectacular macroscopic thermal contraction has hence been seen to be directly associated with the reverse transformation of the NiTi-fibres from martensite to austenite.

Microstructure and mechanical properties of copper-nickel multilayers

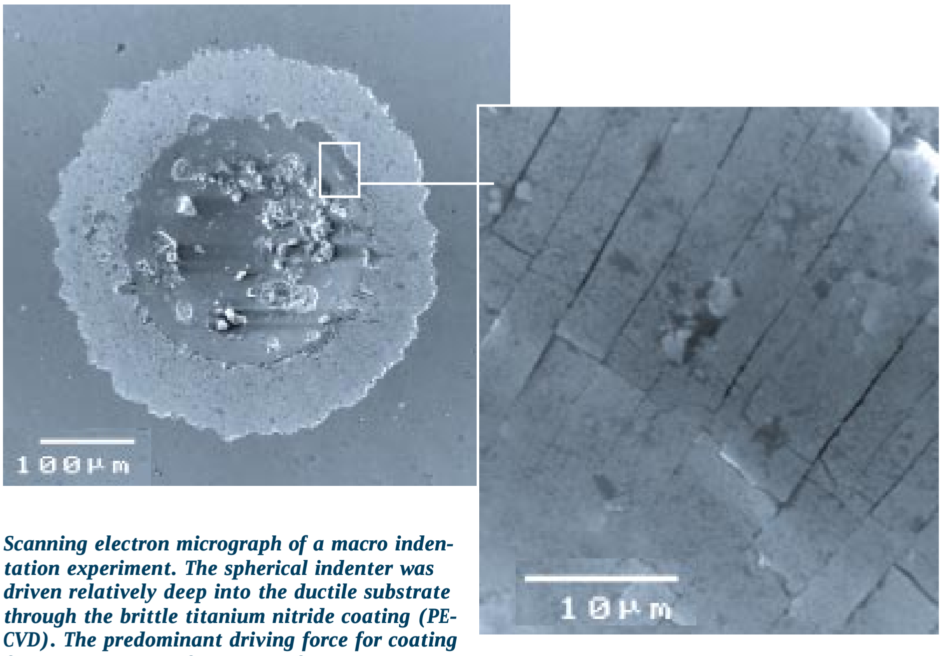
The mechanical strength of both single- and dual-bath electrodeposited Cu-Ni multilayers are known to vary with deposition wavelength. Since we know that layered coatings are harder and more resistant to wear and abrasion than non-layered coatings, this technique is of industrial interest. Optimization of the process requires a better understanding of the strengthening mechanisms and the microstructural changes which affect such mechanisms. To this end we have characterized a series of Cu-Ni multilayers, covering a wide range of thicknesses of the individual layers in the multilayer, using X-ray diffraction, cross-section TEM, hardness testing and tensile testing.

It was found that the copper and nickel layers were strongly textured. Grains of copper traversed the layer thicknesses. The nickel layers were fine grained. This is broadly in agreement with that reported in the literature. In addition, we also discovered that a changeover between epitaxial and non-epitaxial layer deposition occurs at a wavelength of 0.4 microns; this changeover may be attributed to changes in current density.

Hardness measurements suggested that the strength of the multilayers was in accordance with the observed grain size of nickel (Hall-Petch relationship) within each structure. Rather controversially therefore, we have concluded that the multilayers show no enhanced strength due to the layered morphology *per se*. Previous assertions were that a copper-nickel multilayer with a wavelength of less than 0.4 microns is harder than fine-grained continuous nickel plating. The hardening is now realized to be due to the change to columnar growth in thick nickel coatings. In other words, small wavelength Cu-Ni multilayers are hard because interlayers of copper interrupt the changeover to columnar growth of nickel and allow the nickel deposit to remain extremely fine grained and equiaxed.



Cross-section transmission electron micrograph of an electrodeposited copper-nickel multilayer. The copper and nickel layers are strongly textured, with grains of copper traversing the layer thicknesses, while the nickel layers are fine grained. In thick coatings of nickel there are much larger, columnar grains. Layering of the microstructure on a fine scale thereby allows us to keep the nickel extremely fine grained with a resulting high hardness.



Scanning electron micrograph of a macro indentation experiment. The spherical indenter was driven relatively deep into the ductile substrate through the brittle titanium nitride coating (PE-CVD). The predominant driving force for coating failure is plastic deformation of the substrate. The aim of this work is to relate both microscopic observations and depth sensing data to fracture mechanical analysis.

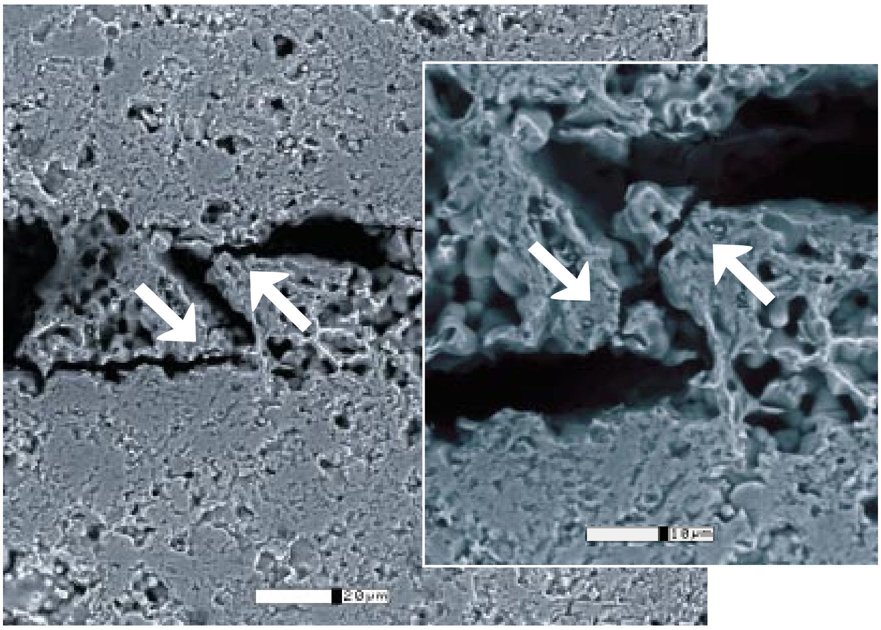
Residual stress in TiN coatings for wear resistance

The main objective of this work is to study the residual macroscopic stress in titanium nitride, TiN, coatings deposited onto a tool steel substrate. The measurements were performed with a θ-θ decoupled X-ray diffractometer. The coatings were manufactured using an industrial pulsed-DC plasma-enhanced chemical vapour deposition (PE-CVD) technique. The coatings were characterized in terms of microstructure, mechanical and tribological properties.

A parametric study of the deposition parameters was performed. Process pressure, bias voltage, temperature and partial gas flow (argon, hydrogen, nitrogen and titanium tetrachloride) were varied in an effort to obtain optimal coating properties. Besides the biaxial stress, the stress free lattice constant, d_0, was determined. Also, we obtained an indication of the changes in texture as a function of process parameters. Total macroscopic stress values were found to range from -1.5 to 1.5 GPa. The intrinsic stresses for the major part of the coatings were close to zero leading to low intrinsic strain energies. This favours a preferred orientation of the coating corresponding to the plane with the lowest surface energy which is (100).

Interfacial crack growth in layered porous ceramic materials

Thin layers of porous ceramic materials are used in solid oxide fuel cells. In order to design components against delamination it is necessary to measure the fracture energy of the relevant interfaces. Sandwich specimens consisting of a thin porous layer of lanthanum-strontium-manganite in between two beams of fully dense lanthanum-strontium-chromite were studied. The porous layers were created by air-brushing a suspension of the powder in liquid onto one of the beams before sintering. A cross-section of the sandwich specimen was polished. Crack growth experiments were conducted inside the environmental scanning electron microscope (ESEM), using a special fixture that applies pure bending moments to the double cantilever beam specimen. It was difficult to initiate a sharp crack without overloading the specimen. But, in specimens where crack initiation was successfully obtained, the subsequent crack growth could easily be controlled. The cracks grew nearly always along an interface. *In-situ* observations revealed that the presence of pores, however, did affect the crack propagation. Sometimes the crack would jump from one interface to the other. In other cases a new crack initiated ahead of the main crack. The fracture energy was measured to be 1.5 - 2.4 J/m^2, which is only about one tenth of the fracture energy of glass.



The interfacial crack growth in porous ceramic materials is clearly influenced by the presence of pores. The crack, growing from left to right, reaches a large pore, leaves the lower interface and grows along the upper interface for a while. As the crack tip extends further to the right, the crack opening increases, the two crack faces come into contact and two parts break off.

Local structure and properties

This programme concerns quantitative characterization of microstructures and local crystallographic orientations with the aim of understanding mechanical properties and recrystallization. This includes development and automation of advanced experimental techniques.

Macroscopic and microscopic subdivision of cube oriented aluminium single crystals during rolling and channel die compression

Grain orientation is known to have a strong influence on deformation behaviour of metals, especially on dislocation structure evolution, work hardening rates, stored energy and subsequent recrystallization mechanisms. The behaviour of the cube orientation in rolling or plane strain compression is of major interest because of its industrial importance for texture control in rolled and recrystallized FCC metals. Both rolled and channel die compressed aluminium single crystals of cube orientation were chosen for a systematic study of their macroscopic and microscopic subdivision.

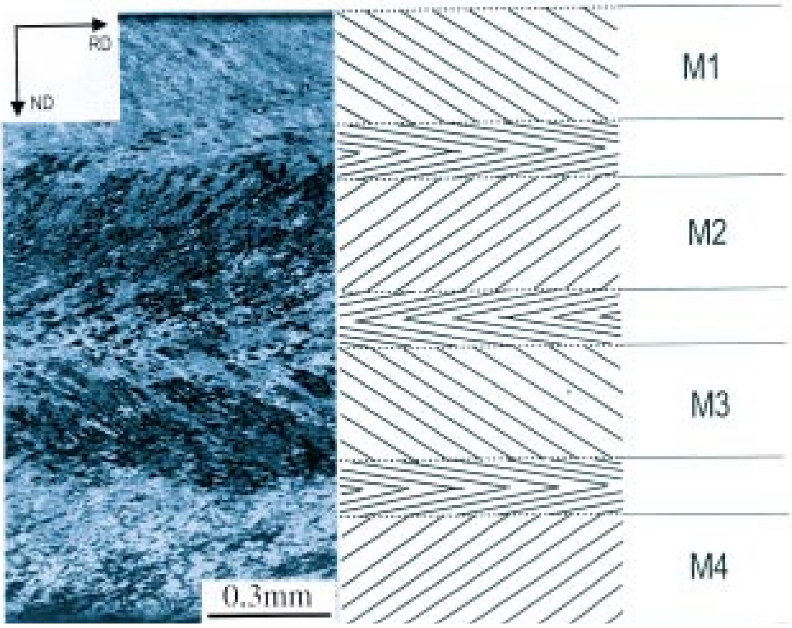
For both deformation modes, the crystals are split into deformation bands which show rotations mainly around the transverse direction (TD). But the orientation and the size of the deformation bands are very different for the two deformation conditions. During rolling, the crystal is symmetrically subdivided into four matrix bands which are parallel to the rolling plane. Between the four matrix bands there are three transition bands with a width of about 100 μm after 50 % rolling, in which the orientation changes continuously from that of one matrix band to that of the adjoining one. A model based on the idea of location-dependent shear strain caused by rolling geometry and friction effects together with plasticity analysis has been proposed to explain the macroscopic subdivision of the crystal.

During the channel die compression, the size and the orientation of the macroscopic bands are very heterogeneous although they are roughly along the elongation direction. Between the macroscopic bands there are narrow transition regions with a width of about 20 μm after a strain of 1.0, which accommodate continuous orientation gradients. The difference of the macroscopic subdivision between rolling and channel die compression is considered to be due to the high sensitivity of the cube orientation to small variations in the macroscopic stress state during the channel die compression.

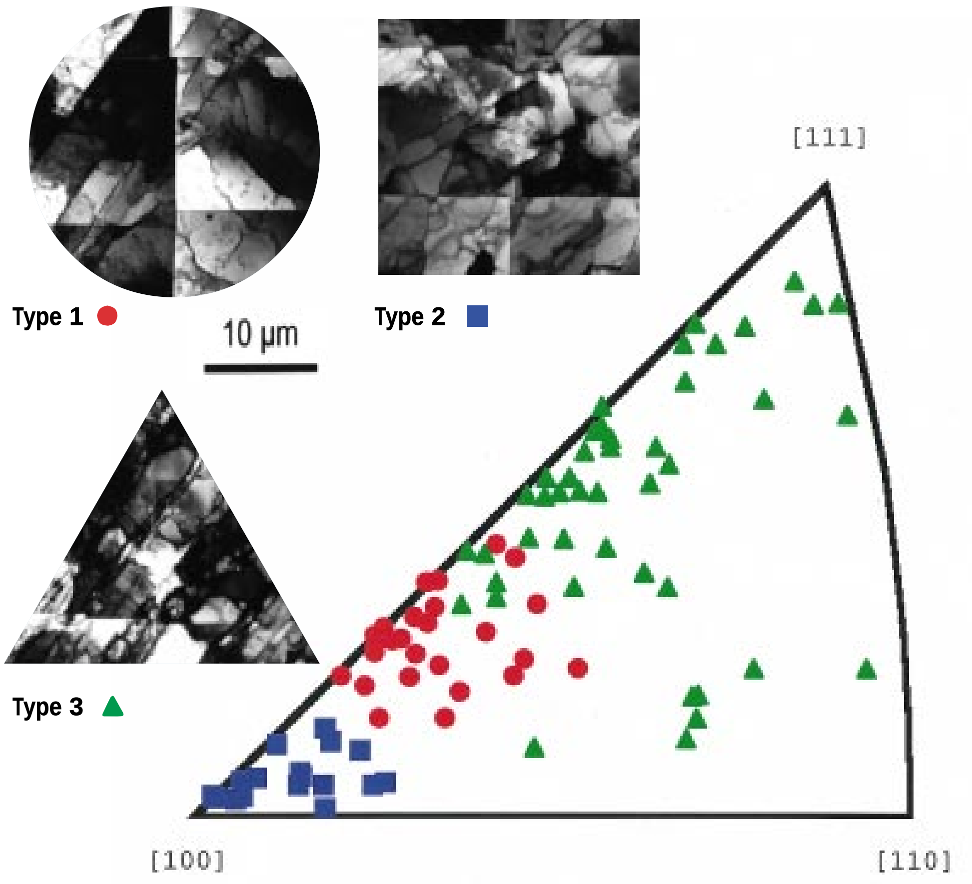
In addition to the macroscopic subdivision, for both deformation modes, a microscopic subdivision by the formation of cell-blocks within the matrix bands and an equiaxed cell structure within transition bands (regions) has been observed. The differences of the dislocation structure in different regions also resulted in different dislocation density and stored energy. These were analyzed by considering the active slip systems in different regions and the interactions among the dislocations.

Microstructure and grain orientation relations in tensile deformed aluminium

High purity polycrystalline aluminium (99.996 %) was deformed in tension at room temperature. The evolution of the deformation microstructure was characterized by a microstructural analysis of individual grains by TEM. It was observed that the deformation microstructure was never uniform in the whole sample but varied from grain to grain. This observation suggests that grain to grain variations in the slip pattern determine the observed microstructures in the grains. Under a certain deformation condition, the slip pattern in a grain is considered to be mainly determined by the orientation of that grain. For tensile deformation, this grain orientation was characterized by only a single reference direction, the crystallographic direction being parallel to the tensile axis of the sample. In this study, the tensile axis orientation of individual grains was determined in the TEM and then related to the observed microstructure.



SEM micrograph taken using channelling contrast of the ND/RD plane of a 50 % cold-rolled cube oriented single crystal. The changes of the sign of the angle between the long lines and RD along ND define four matrix bands, marked M1, M2, M3 and M4, which are parallel with RD. Between the four matrix bands are the three transition bands.



Relationship between the microstructure type and the tensile axis orientation of grains embedded in polycrystalline aluminium for specimens strained in the range 0.05 to 0.34. Type 1: Grains with dislocation boundaries on the slip plane. Type 2: Grains containing equiaxed cells. Type 3: Grains with dislocation boundaries not on the slip plane.

In total 90 grains were examined in four samples of different strains, which have orientations distributed over most of the inverse pole figure with <100>, <110> and <111> poles at the corners of the standard triangle. According to the microstructural characteristics, the grains can be grouped into three types showing different microstructures. In grains having the <100> direction within 15 ° from the tensile direction, randomly oriented dislocation boundaries are formed, which define a three-dimensional cell structure. In grains next to the <100> group, a cell block structure is developed, which is delineated by long and straight dislocation boundaries that are on or within 5° of parallel to the {111} slip planes. Close to the <111>, the cell block boundaries become less straight and deviate from the slip planes by increasing angles. The grains with deviation angles larger than 5° constitute the third group. Slip pattern analysis in terms of a Schmid factor calculation or a Taylor model indicates that the formation of either a three-dimensional cell structure or a cell block structure depends on the multiplicity of slip. Fewer dominant active slip systems favour the development of the cell block structure with dislocation boundaries on the slip planes. With increasing multiplicity of slip, the deviations of the dislocation boundaries from the slip planes increase and finally form randomly oriented dislocation boundaries.

Flow stress, single crystals/polycrystals

Pure polycrystalline aluminium (99.996 %) having an average grain size of 300 µm was strained in tension at room temperature. The flow stress was determined at four different strains (0.05, 0.14, 0.22 and 0.34). Deformation microstructures were characterized qualitatively and quantitatively by TEM. A classification of the deformation microstructures into three different types has shown a correlation between the grain orientation and the type of deformation microstructure which develops during straining, as described in the preceeding subsection. The dislocation density was calculated at each strain. The shear stress-strain relationship was derived for each of the three groups of grains showing different deformation microstructures, by assuming that the shear stress is proportional to the square root of the dislocation density. The stress-strain curves show a strain hardening behaviour which depends on the orientation of the grain. The behaviour of the grains embedded in the polycrystal was compared with the behaviour of single crystals. The stress-strain curve of the polycrystal was estimated with good accuracy from single crystal data, which were weighted based on a quantitative texture analysis of the polycrystal.

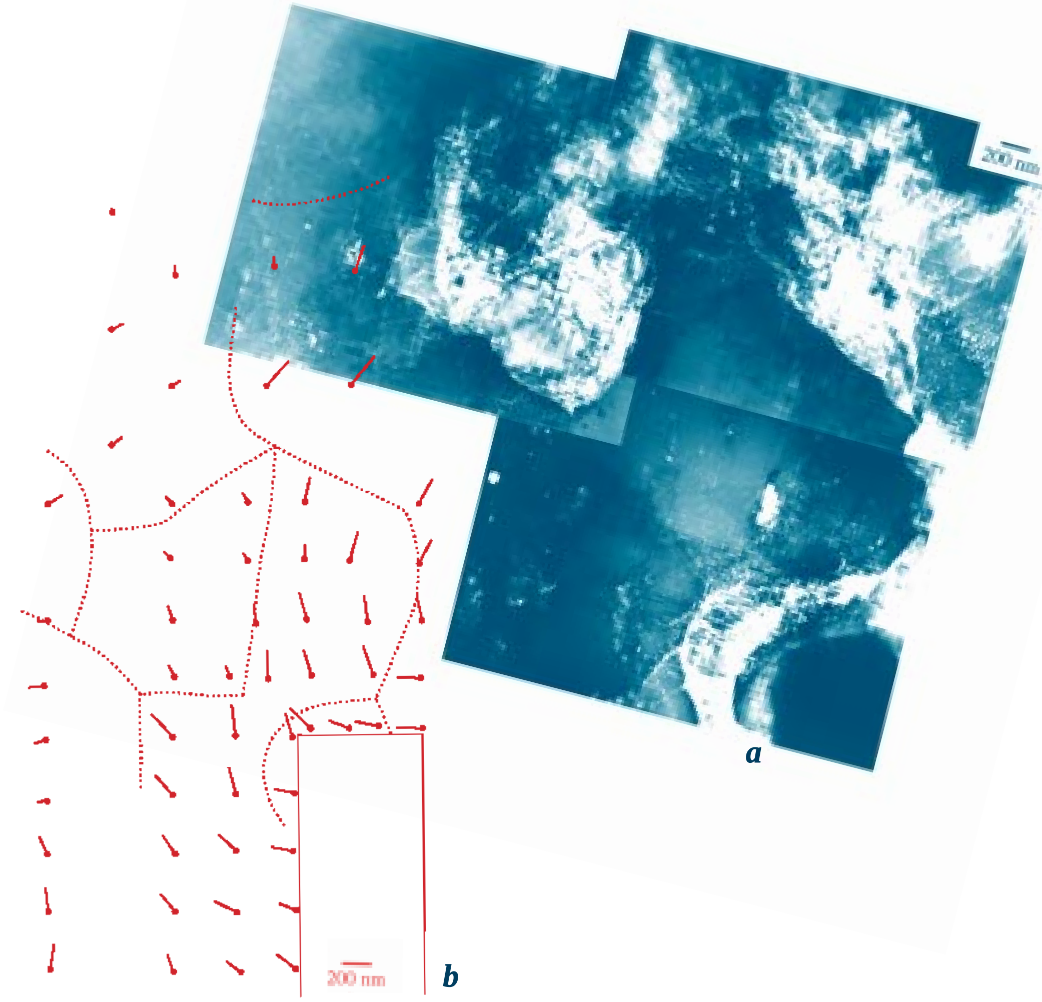
Scaling in polycrystals

During plastic deformation, dislocation structures are developed. They can be characterized by several representative lengths: the overall average distance between dislocations related to the dislocation density, the distance between dislocation boundaries and, finally, the distance between dislocations in a boundary connected with the disorientation angle between the adjacent regions. All these characteristic sizes shrink with proceeding deformation, but empirically a strong proportionality between the first two and the inverse flow stress is well established. Consequently, both lengths are also intimately related, reflecting some similitude inherent in the structures. If properly defined, the proportionality coefficients in these scaling laws even become independent of the material or the deformation mode.

Recently, after a distinction between several types of boundaries, an additional scaling law was discovered for cold-rolled aluminium polycrystals. For each individual boundary type, the average disorientation angle is inversely proportional to the boundary distance, but with different coefficients for different types. Future investigations have to test if the proportionality factors are universal or depend on, for example the orientation, the deformation conditions or the material.

Furthermore, another type of scaling was found for the distribution functions of the disorientation angles of each kind of boundaries. The measured distributions for several plastic strains collapse into a single master curve after normalization by their respective mean disorientation angle. According to a recent proposal the existence of more than one set of dislocations in each boundary can explain the observed scaling behaviour. The (modified) Rayleigh-distributions resulting from the model are in good agreement with the experimental data on aluminium. Distributions of similar shape are also found after hot-rolling of steels and hot-compression of copper single crystals. Here, the universal behaviour of the distribution functions is caused directly by the geometrical arrangement of the dislocations in the boundary.

Establishing such scaling laws is an important guide to distinguish between properties which are material dependent or specific for the deformation mode and classes of universal behaviour. Simple structural arguments as similitude or geometrical requirements are sufficient to explain the universality of some properties, whereas other features like work-hardening require a more detailed understanding of the underlying processes.



(a) TEM dark field micrograph of a Al-2%SiC composite strained to 0.1. (b) Schematic of the area in (a) showing the principal dislocation features. The misorientations of the matrix are indicated by 'vector markers', with length proportional to the rotation angle and direction related to the rotation direction.

Characterization of local strain fields in metal matrix composites

From TEM micro-diffraction studies of local orientations in an aluminium composite containing 2 % SiC whiskers deformed by tension, it was possible to determine the local misorientation map and local strain map around individual whiskers. A novel approach to representing misorientations was applied using a 'vector marker', i.e. the misorientation vectors show orientation changes not only in magnitude but also in direction. This technique allows more information to be presented in a way that provides an intuitively satisfying indication of flow patterns in the matrix. In the local strain map, the strain gradients existing in the matrix following tensile deformation was determined. The experimentally derived strain gradients were lower by a factor of 5 - 10 than values estimated by FEM. The conclusion is that high dislocation mobility results in (i) dislocation structures with more relaxation, and (ii) strain fields which are more uniform than those predicted by FEM.

By using the object grating technique (in collaboration with Dortmund University), the local strain fields around large particles (> 10 µm) in an aluminium composite containing 10 % Al_2O_3 were characterized on a surface of a bulk specimen in a scanning electron microscope (SEM) *in-situ* during tensile deformation. Effects of particle shape on the constrained plastic flow in the associated matrix were observed. For the aligned cylinder-shaped particles the strain in the matrix tends to be largest at the end of the particle and the plastic flow bands oriented at 45 ° to the tensile axis are often interrupted. For the ~ 45 ° rotated equiaxed particle the development of such bands is favoured. These observations agree qualitatively well with the constraint effects of particle shape predicted by FEM.

Modelling of anisotropy

The mechanical anisotropy of metals is critical for the formability. For example, cups drawn from mechanically anisotropic materials form ears and develop an inhomogenous thickness. Understanding the origin of anisotropy is therefore important in order to select suitable production methods. It is well known that the texture of a material causes mechanical anisotropy but the observed flow stress anisotropy of a rolled metal sheet cannot be fully explained by texture. Other causes must therefore exist. The deformation induced dislocation walls in rolled materials typically have a preferred orientation with respect to the rolling coordinate system. The dislocation structure is hence anisotropic and may therefore also lead to mechanical anisotropy. The dislocation walls are expected to resist deformation by slip in a way similar to grain boundaries. Their contribution to the flow stress was modelled by a Hall-Petch law where the grain size was replaced by the distance between the dislocation walls. This distance is different for the different slip systems because it depends on the relative orientation of the slip system and the dislocation walls.

The texture contributions are usually calculated by the Taylor or Sachs models. The contributions from the dislocation walls were incorporated in these models through an anisotropic critical shear stress given by a Hall-Petch law for each slip system.

Depending on model parameters the flow stress in different directions of a rolled sheet was predicted to vary with up to 18 % for a random texture. The model predictions agree well with experimental flow stress data.

Grain boundary mobility during recrystallization of copper

The migration of grain boundaries is a vital feature of the industrially important annealing processes of recrystallization and grain growth. The velocity of grain boundary migration depends on the driving force acting on the boundary and on the mobility of the boundary into the surrounding material. For recrystallization, there is a paucity of work on the velocity versus driving force and mobility relationship. Knowledge about this relationship is required as input for physically based modelling of recrystallization kinetics.

In an investigation of the recrystallization of copper, cold rolled 92 %, the driving force and the grain boundary velocity was determined by performing *both* calorimetric measurements and stereological metallographic measurements in the *same* samples annealed for selected times at 121 °C. It was found that there was a linear dependence of the average grain boundary velocity on the driving force. The mobility of the migrating grain boundaries was determined to be 6.3 x 10^{-10} $m^4 s^{-1} MJ^{-1}$. This mobility value is close to that of grain boundary self-diffusivity in pure copper.

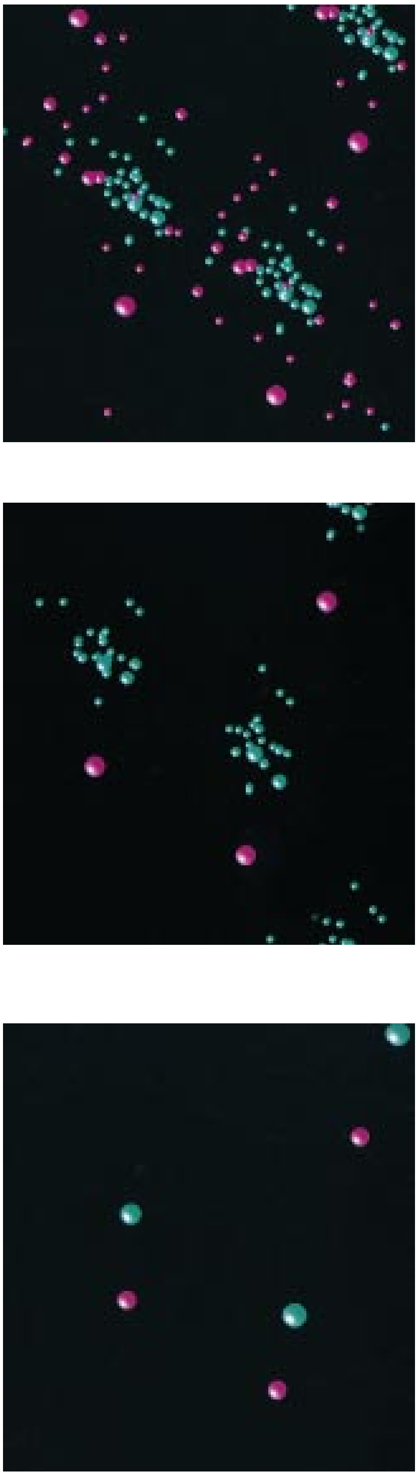
Irradiation damage, defects and fusion materials

This programme concerns the experimental determination and theoretical evaluation of the parameters which characterize the accumulation of defects and their effect on the physical and mechanical properties of the materials to be used in fusion reactors, such as ITER (International Thermonuclear Experimental Reactor).

Kinetic Monte Carlo simulation of damage accumulation

In recent years a considerable amount of effort has been made to understand various aspects of production of defects and their clusters in multi-displacement cascades using molecular dynamics (MD) simulations. These studies have provided some very valuable information regarding intracascade events and the surviving defects, their clusters and morphology at the end of the cooling down phase of isolated individual cascades. However, global accumulation of defects on a macroscopic scale (where inter-cascade defect interactions and temporal and spatial fluctuations in defect size and concentration have to be taken into account) cannot be described at present by MD simulations. Instead, the stochastic annealing simulation technique (which has been developed for describing the short-term annealing stages of defect evolution within an individual or a group of cascades) can be used. With this technique, the effects of cascade production on defect accumulation at macroscopic scales can be studied as a function of irradiation time. This type of simulation technique is commonly known as kinetic Monte Carlo.

This technique was used (in collaboration with Pacific Northwest National Laboratory, USA) to simulate the evolution of defects produced in copper during continuous irradiation with 14 MeV neutrons at 300 K. The irradiation was simulated by successive introduction of collections of defects and defect clusters representing the primary damage state of individual cascades (as determined by MD simulation) placed in the annealing volume randomly in time



An assembly of five 25 keV cascades containing clusters of vacancies (green) and SIAs (red) 10 picoseconds after the collision event during MD simulations (top picture). After stochastic annealing at 450 K for one nanosecond, the SIA population consists of only sessile clusters (middle picture) whereas after annealing for 100 seconds (bottom picture) only large clusters of SIA and vacancies remain.

and space. Cascade energies and the rate of cascade generation were chosen to approximate the damage due to the neutron flux of the 14 MeV neutron source RTNS-II (Rotating Target Neutron Source). The cascades were chosen from a library of cascades generated in MD simulations for recoil energies in the range 5 - 25 keV. The numbers of each type of defect, defect cluster size distributions, as well as the positions of defects within the crystal were monitored as a function of irradiation time (displacement dose). The highest dose of slightly more than 0.1 dpa (displacements per atom) was obtained by introducing approximately 12,000 cascades into the annealing volume. The calculated dose dependence of cluster density was found to be in good agreement with the experimental results.

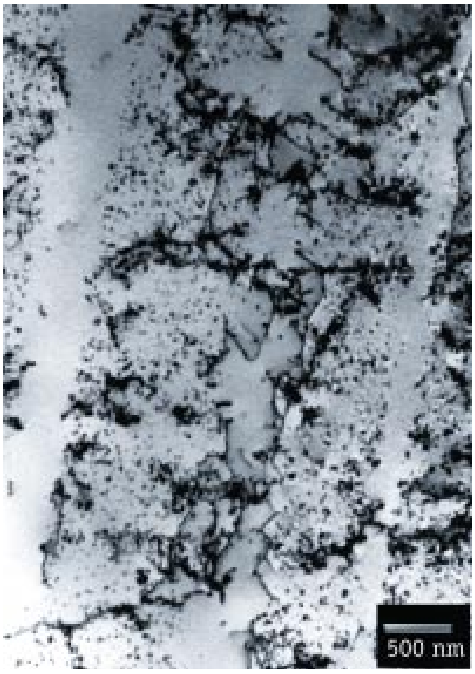
Effects of recoil energy on damage accumulation calculated using the production bias model

In the past, the effect of recoil energy on defect accumulation in the form, for example, of voids has not been treated explicitly. An examination of the literature reveals two main reasons for this limitation. First, the vital information regarding the details (e.g. nature, efficiency and morphology) of defect production as a function of recoil energy did not become available until rather recently. The second reason is related to the fact that the earlier theories were based on homogeneous kinetics and mean field approach and were unable to treat the problem of defect accumulation under the condition when both glissile and sessile clusters of self-interstitial atoms (SIAs) were generated at higher recoil energies. Furthermore, up to a certain level of recoil energy, both the damage efficiency and the amount of SIA clusters produced during irradiation depends strongly on the damage energy.

The recently proposed 'production bias model' (PBM), on the other hand, is fully capable of treating the problem of intracascade clustering of SIAs and the glide of SIA clusters. In fact, one of the major predictions of the PBM is that the defect accumulation above stage V should be recoil energy dependent. In the present work (in collaboration with IPPE, Russia), the effect of recoil energy on defect accumulation under 2.5 MeV electron, 3 MeV proton and fission neutron irradiation has been investigated. Calculations were carried out in terms of PBM using one-dimensional glide of SIA clusters, sessile-glissile loop transformation and size distribution function. The calculated components of the irradiation-induced microstructure (i.e. size and density of SFT (stacking fault tetrahedra), SIA clusters and voids) and their dose dependence for copper irradiated with 3 MeV protons and fission neutrons were found to be in very good agreement with the experimental results. The void swelling behaviour observed under 2.5 MeV electron irradiation where defects are produced in the form of Frenkel pairs (i.e. no clusters of SIAs) can be clearly understood in terms of the standard rate theory and dislocation bias. The analysis of these results yields a dislocation bias of ~ 2 %.

Effects of various heat-treatments and irradiation on mechanical properties of copper alloys

Precipitation and dispersion strengthened copper alloys (CuCrZr, CuNiBe and $CuAl_2O_3$) are being considered as heat sink materials in the first wall and divertor assemblies of ITER because of their high thermal conductivity and high strength. The joining procedures employed to fabricate the structural components of the first wall and the divertor may influence the performance of these materials during irradiation. In order to evaluate the effects of bonding and bakeout thermal treatments on pre- and post-irradiation properties of these alloys, a series of screening experiments were carried out. A number of tensile specimens of CuCrZr and CuNiBe alloys were given different heat treatments to simulate the effect of joining and bakeout thermal treatments. The heat treated specimens were irradiated in the DR3



Post-deformation microstructure of monocrystalline molybdenum irradiated with fission neutrons at 320 K to a dose level of 5.4 x 10^{-3} dpa. Note the evidence of localized deformation in the form of 'cleared' channels.

reactor at Risø at 100 °C to a displacement dose level of ~ 0.3 dpa. Both unirradiated and irradiated specimens were tensile tested in vacuum at 100 °C.

The irradiation of CuCrZr at 100 °C caused a drastic reduction in the uniform elongation and led to plastic instability (localization) almost immediately after the initiation of plastic deformation. The CuNiBe and the $CuAl_2O_3$ specimens suffered too from an irradiation-induced reduction in the uniform elongation, but to a lesser degree than the CuCrZr specimens at the same dose level. The prior irradiation treatments corresponding to the joining and the bakeout thermal treatments did not lead to any improvement in the mechanical and physical properties after irradiation.

Effect of irradiation on deformation behaviour of metals and alloys

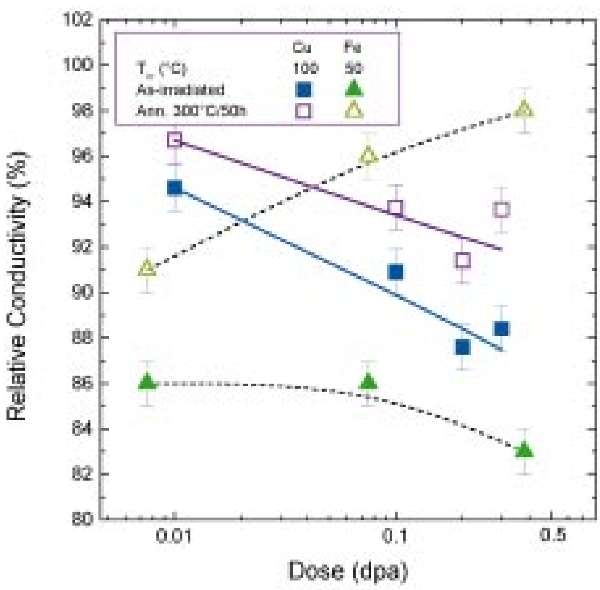
It is well known that metals and alloys exposed to radiation with energetic particles (e.g. fission or fusion neutrons) at relatively low temperatures become significantly harder, suffer from plastic instability and lose ductility. It is the loss of ductility, commonly known as embrittlement, which is a matter of serious concern from the point of view of the lifetime of structural components in fission or fusion reactors. Because of this concern, the effects of irradiation on deformation behaviour of metals and alloys are being studied internationally.

Tensile specimens and 3 mm diameter discs of monocrystals of pure molybdenum and Mo-5%Re alloy were irradiated in helium-filled capsules with fission neutrons in the DR3 reactor at Risø. Irradiations were carried out at ~ 50 °C to displacement doses in the range 5.4 x 10^{-4} to 1.6 x 10^{-1} dpa. For comparison, polycrystalline specimens were also irradiated. Post-irradiation microstructures were quantitatively characterized using TEM. Both unirradiated and irradiated specimens were tensile tested at ambient temperature. The microstructure of the irradiated and deformed specimens was also examined using TEM.

Monocrystals of both pure Mo and Mo-5%Re alloy suffer from plastic instability in the same way as the polycrystals. The occurrence of plastic instability suggests that up to a certain stress level dislocation sources remain locked. At the upper yield stress, a number of dislocation sources begin to operate and form 'cleared' channels. In other words, the plastic deformation is localized and occurs only in these cleared channels. The analysis of the present results suggests that during irradiation under cascade damage conditions, gliding SIA clusters block the dislocation sources and thus prevent homogeneous plastic deformation. Consequently, the crack nucleation is no longer caused by plastic deformation. Instead, the crack must initiate at internal and/or external flaws in the material. The interaction of these cleared channels with external surfaces or grain boundaries or intersections of these channels with each other may initiate cracks. Once initiated, the crack is likely to propagate rapidly through the material since the irradiated material is unable to deform plastically in a homogeneous fashion.

Electrical conductivity of iron, copper and copper alloys

Cu-Al_2O_3 (with 0.25 % Al, CuAl25) has been chosen as prime candidate for the first wall and divertor material in ITER and CuNiBe and CuCrZr as potential backup materials. Electrical conductivity



The electrical conductivities of neutron irradiated copper and iron, relative to the conductivities for the un-irradiated materials as a function of irradiation dose. Data are shown both for the as-irradiated state and after annealing at 300 °C for 50 hours. The clearly different behaviour of the two materials is ascribed to differences in crystal structure and impurity content.

measurements were carried out on these alloys and pure copper after different initial heat treatments, fission neutron irradiation at different irradiation temperatures, and post irradiation annealing. The treatments simulate the possible histories of the material in ITER.

In CuAl25, the conductivity was found to be 88 % of that of pure copper. Irradiation to 0.2 - 0.3 dpa reduces this number to 81 %, irrespective of irradiation temperature. Annealing at 300 ºC changed these values only slightly. For Cu-Al_2O_3 (0.6 % Al) the reduction due to the alloying was down to 79 %, but the effect of irradiation was smaller.

The electrical conductivities of the unirradiated and irradiated (0.3 dpa) CuCrZr and CuNiBe alloys fell in the range of 50 - 80 % and below 55 %, respectively, of the conductivity of pure copper. Generally, irradiation was found to cause an increase of the conductivity of CuCrZr. For CuNiBe it increased at 350 ºC, but decreased for lower irradiation temperatures. By subsequent annealing the conductivity always increases. The results correlate well with data obtained by electron microscopy and mechanical testing and can be ascribed to effects of (i) cascade-induced dissolution of precipitates by cascade impingement, (ii) segregation of alloying elements and (iii) reprecipitation. In pure copper the conductivity decreased with irradition dose, and annealing at 300 ºC for 50 hours led only to a partial recovery of the radiation created defects.

Iron irradiated at 50 ºC showed a decrease in conductivity to 86 % of the value for un-irradiated iron even at a low dose. Only a small further decrease to 83 % was observed up to 0.23 dpa. For irradiation at 100 ºC, the conductivity decreased from 99 % to 92 % in the same dose range. This large difference in behaviour probably reflects the migration of carbon impurities between 50 ºC and 100 ºC and illustrates the sensitivity of the electrical conductivity to impurities and the defect microstructure.

Techniques

The success of the projects within the programme area depends on the efficient use of a number of different experimental and modelling techniques. The modelling techniques are rather diversified with specific techniques used in the individual projects, ranging from closed-form matematics to advanced numerical methods (e.g. for solving non-linear problems), which require extensive computer power. For instance, micromechanical models are used for providing a direct relationship between the geometry of the microstructure and the macroscopic mechanical behaviour of materials.

The experimental techniques managed within the programme are summarized in the following table. Many of the techniques are also used in a number of projects from other programme areas. The techniques are not just brought in. The development of methods and equipment is a research topic in its own right, as described below in separate sections.

The Department has two transmission electron microscopes and three scanning electron microscopes. Neutron diffraction experiments are conducted at the DR3 research reactor at Risø, while synchrotron radiation experiments are conducted at HASYLAB, Hamburg and ESRF, Grenoble.

The programme area also utilizes a number of experimental techniques managed by other programme areas, particularly mechanical testing and X-ray diffraction.

X-ray spectrometry in the environmental scanning electron microscope

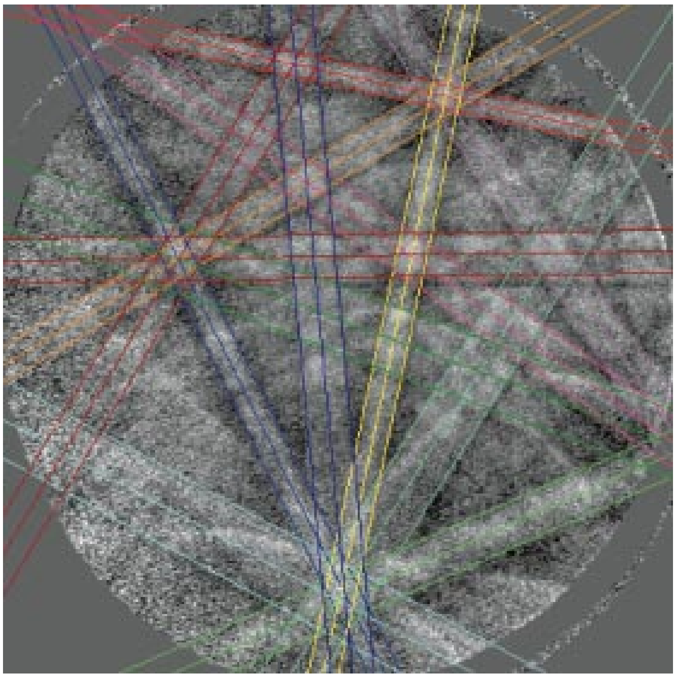
The presence of gas in the specimen chamber of the ESEM causes the primary electrons to be scattered on their way towards the specimen. The skirt of scattered electrons generate X-rays far from the electron beam target. This leads to a deterioration of the spatial resolution for chemical analysis by X-ray spectrometry. Two methods to overcome this problem were described in the previous annual report.

The pressure variation method is based on spectra being measured at the same beam target at a number of different pressures. These measurements are then used to extrapolate to the measurement that would have been obtained under high vacuum conditions. The method was improved by the introduc-

	Method	**Objective**
Characterization Techniques	Transmission electron microscopy (TEM); EDS and EELS	Microstructure, local crystallographic orientation, local chemical composition
	Scanning electron microscopy (SEM); EBSP, COM, EDS	Microstructure, local crystallographic orientation, orientation maps, local chemical composition
	Environmental scanning electron microscopy (ESEM); EDS; high temperature, mechanical testing (tension/compression/bending/controlled crack growth)	Microstructure, local chemical composition, *in-situ* investigations of insulators and wet materials
	Neutron diffraction; elevated temperature tensile testing	*In-situ* measurements of texture and internal strains
	Synchrotron radiation; ambient and high temperature	Local crystallographic orientation (3D), local strain state
	Positron annihilation spectroscopy (PAS)	Point defects, line defects, free volume, interfaces and gas bubbles

tion of a new extrapolation procedure which accounts for the uncertainty of the individual measurements. It has been shown experimentally that the correct result in most cases lies within one standard deviation from the extrapolated result when the new extrapolation procedure is used.

The beam stop method was developed for use with line scans. The specimen is covered with a foil along the line where a line scan is desired. The spectral measurement in a point close to the foil is corrected for beam skirt effects by a corresponding measurement at a point on the foil close to the first point. It was shown experimentally that the spatial resolution in the line scan can be restored to about 10 μm by this method. The reason that the spatial resolution cannot be completely restored to the high vacuum value is that there is an intense skirt component of inelastically scattered electrons just around the unscattered beam, and this component is sensitive to a movement of the beam by a few micrometers.

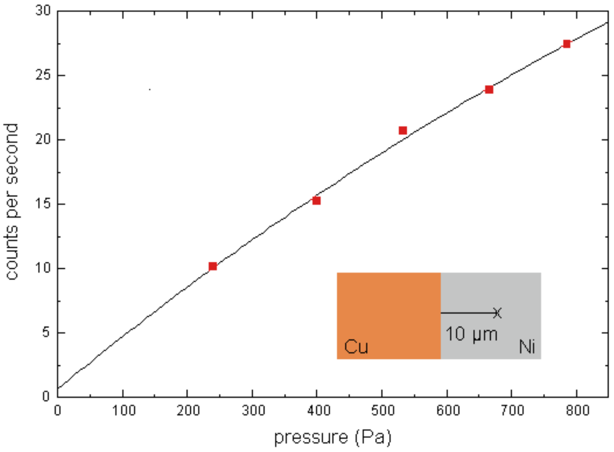


An EBSP from recrystallized pure copper. The 15 triplets of coloured lines illustrate the position and width of Kikuchi bands which were detected automatically by a novel pattern recognition procedure. A high precision of the band positions and of the measured band widths is observed.

Advancements of the EBSP technique

Electron backscattering patterns (EBSPs), also known as backscatter Kikuchi patterns, are electron diffraction patterns from which crystal orientations can be measured with a spatial resolution of around 0.5 μm in the SEM. The recent development of specialized image analysis procedures for automatic recognition of linear features in the patterns, the Kikuchi bands, has led to a complete automization of the technique and made it an extremely powerful tool in studies of the orientational aspects of microstructures in crystalline materials.

The reliability and accuracy of the EBSP technique are strongly dependent on the reliability and precision by which the Kikuchi bands are found by the image analysis procedure. In an effort to improve the quality of the automatically collected crystal orientation data a new procedure was developed for this band detection task. This novel procedure provides significant improvements to the precision by which the bands are localized, and is generally capable of identifying a larger number of bands than the traditional approach. Additionally, the new procedure also provides reasonably precise measurements of the width of the band which may be utilized for improving the reliability of the measurements even further. We have shown that the new procedure leads to significant improvements in both the reliability and precision of the automatically measured crystal orientations. The best obtainable precision is around 0.7 ° with the traditional procedure whereas 0.4 ° can be achieved with the new procedure.



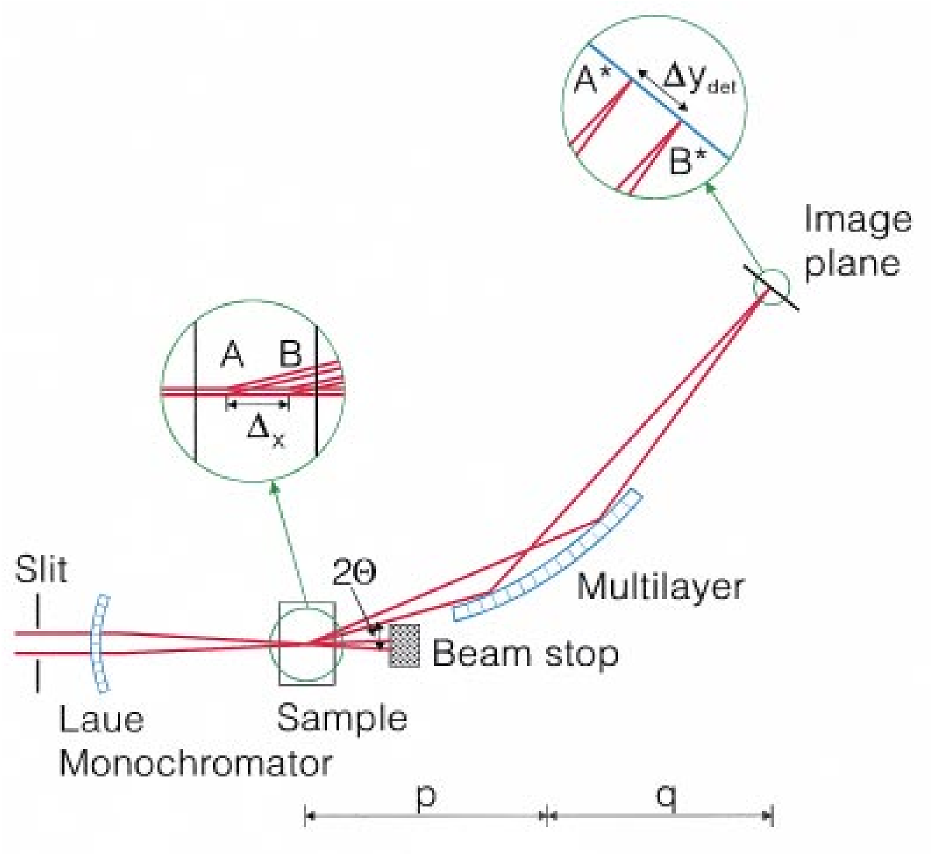
Extrapolation by the pressure variation method. The counts per second for CuKα were measured at various pressures in a point in pure Ni 10 μm from an interface to pure Cu. The extrapolation yields 0.64 ± 0.71.

Optics for a three dimensional X-ray microscope

The Department is collaborating with ESRF on the installation of a three-dimensional X-ray microscope (3DXRD) at the synchrotron. The microscope will be dedicated to characterization of materials locally within millimetre thick specimens. Parameters such as strain, grain orientations, grain morphology and dislocation densities are to be investigated *in-situ*, that is during an annealing or deformation process. The instrument is to operate in the energy range 40 - 100 keV with gauge volumes of order 5 x 5 x 50 μm^3.

To meet specifications, novel types of X-ray optics must be designed. In particular it is necessary to reconsider the way depth-information is obtained. This is conventionally done by means of cross-beam techniques, where both the incoming and the diffracted beams are confined, typically by collimators. Unfortunately, collimators are very difficult to manufacture for the 3DXRD case and they do not allow an efficient focusing of the incoming beam.

An alternative set-up, giving superior depth-resolution without any of the problems associated with the use of collimators, was developed. The properties of this set-up were tested - by a Risø/ESRF/SLS (Swiss Light Source) team - at 30 keV at beamline D5 at ESRF. Results showed that theoretical predictions for resolution, magnification etc. were matched by experiment.



The incoming beam is focused by a Laue monochromator - a bent perfect crystal. Next, a bent multilayer with a lateral grading is used to also focus the diffracted beam. The multilayer has the property that diffracted rays from one point A in the sample will all converge in a spot A*, while rays from another point B will converge in B*. In short, a line through the depth of the sample is projected onto an image plane transverse to the diffracted beam. By changing distances p and q, the magnification ratio of q/p may be optimized.

MATERIALS CHEMISTRY
– combining science and technology

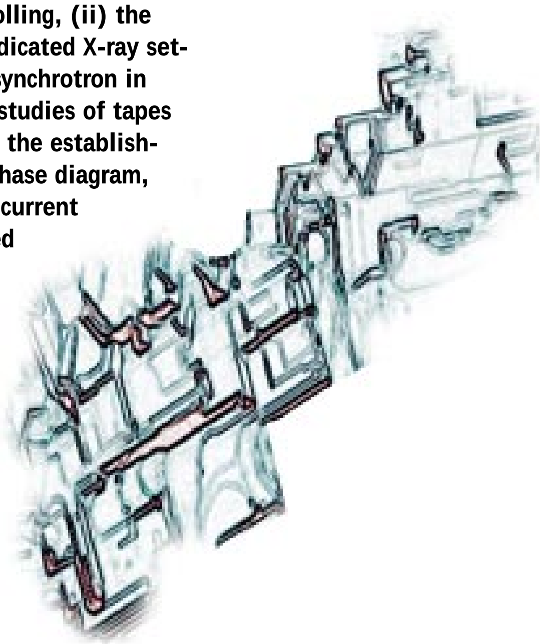
The Materials Chemistry research area consists of two programmes: Fuel cells and high-temperature superconductors. Both programmes deal with synthesis and processing of ceramic materials (oxides). The understanding of phase relations and defect chemistry is very important in both areas. Solid state electrochemistry is of particular importance for the fuel cell programme.

The fuel cell R & D concentrates on solid oxide fuel cells (SOFCs). Many results were obtained. As highlights one can mention (i) the development of Ni-based anodes, having a polarisation resistance of cathodes of only ~ 0.1 Ωcm^2 at 850 °C, (ii) the development a new cell design concept, (iii) the efforts of fundamental research on metal oxides with perovskite structure, and (iv) the work on high temperature oxidation of Fe-Cr-alloys, aimed at identifying the most suitable compositions for metallic SOFC-stack and -system components.

In the area of high temperature superconductors the following highlights can be mentioned: (i) a technique was developed for routine manufacturing of single filament tapes by means of rolling, (ii) the construction of a dedicated X-ray set-up at the HASYLAB synchrotron in Hamburg for *in-situ* studies of tapes was completed, (iii) the establishment of a (T, pO_2)-phase diagram, and (iv) the critical current density was increased by 35 % by cooling the tapes in an atmosphere with the oxygen partial pressure reduced to 7% (instead of air).

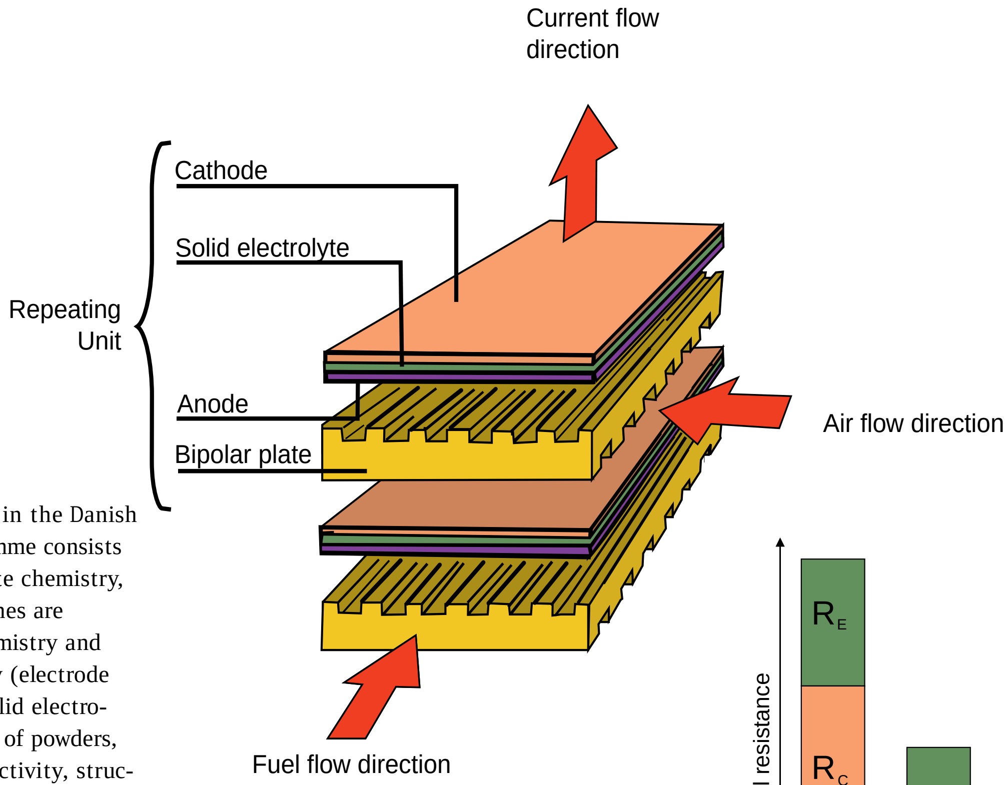
Project Funded Research: Materials Chemistry

Project type	Project name	Co-participants
The Energy Research Programme of the Danish Ministry of Environment and Energy (EFP)	DK-SOFC 1996-1998	• Haldor Topsøe A/S, Denmark • Innovision A/S, Denmark • Institute of Chemistry, OU, Denmark • Institute of Chemistry, DTU, Denmark
EFP	SOFC Module Design	• Haldor Topsøe A/S, Denmark
EFP	DK Superconductors	• NKT Cables A/S, Denmark • NKT Research Centre A/S, Denmark • Inst. of Product. Techology, DTU, Denmark • Electric Power Eng. Dept., DTU, Denmark • Research Assoc. of Danish Electric Utilities • Condensed Matter Physics and Chemistry Department, Risø
JOULE-THERMIE	Improving Durability of SOFC Stacks (IDUSOFC)	• Research Centre Jülich, Germany • ECN, Petten, The Netherlands • Imperial College, University of 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)	• Innovision 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

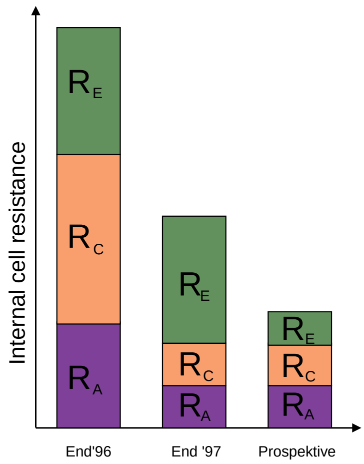


Fuel cells

The overall aim of this programme is to establish a basis for a Danish production of SOFC components and to explore the potential of SOFC in the Danish energy system. The programme consists of three parts: (i) Solid state chemistry, where the scientific diciplines are crystallography, defect chemistry and solid state electrochemistry (electrode kinetics, conductivity of solid electrolytes), (ii) characterization of powders, electrode properties, conductivity, structure of composites (electrodes), mechanical strength, cell and stack performance, and (iii) fabrication of electroceramic components to reduce cost and to obtain optimal properties.



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.



Achieved and prospective SOFC cell resistances in the Danish SOFC project compared on a relative scale at 850 °C. The contributions are electrolyte resistance, R_E, cathode resistance R_C and anode resistance R_A.

Improved performance of solid oxide fuel cell components leads to a new approach

Different designs of SOFC stacks are pursued world-wide. The bipolar flat plate design has, among other features, a minimum current path with minimum ohmic losses, and has been selected for the Danish SOFC programme.

Over the last few years the development of SOFCs for commercial use has reached a state where cost analysis is being used for identifying components and processes which must be substituted or modified to meet market costs of competitive technologies. The overall cost of electricity produced by SOFC stacks has been linked directly to the internal resistance of the stack, comprising reaction resistance in the electrodes and ohmic resistance in electrolyte, interconnect and contacts between components.

Recent developments made at the Department have reduced the electrode losses in the cell significantly. These improved electrodes now make it possible to achieve electrode resistances at temperatures of 800 - 850°C matching values, previously achieved at 1000 °C. Exploitation of this reduction in temperature in a complete cell leaves the resistance of the about 180 µm thick supporting YSZ electrolyte as the major contributor to the internal resistance. Significant reduction of this resistance may be achieved by a thickness reduction, which unfortunately diminishes the mechanical strength of the cell. As a consequence, a new approach is taken where the cell is built with a 1 - 2 mm thick porous anode, which provides the mechanical strength of the cell and acts as a support for a thin electrolyte. The electrolyte thickness is expected to be reduced to 30 - 40 µm, leading to a factor 5 reduction of the electrolyte resistance.

The cost of a mm-thick dense ceramic or oxidation-resistant metallic interconnect with a machined gas distribution structure on both faces is prohibitive for SOFC commercialization. A significant cost reduction can be obtained by splitting this component into several functional layers. Combined with the temperature reduction made available through better electrodes, the interconnector material can potentially be made from commercial FeCr alloys rather than from high cost ceramics and special alloys.

SOFC Component	Material
Electrolyte	Yttria stabilized zirconia (YSZ)
Cathode	Lanthanum strontium manganite (LSM)
Anode	Nickel-YSZ-cermet
Interconnect	Lanthanum strontium chromite (LSC) and/or chromium metal alloy

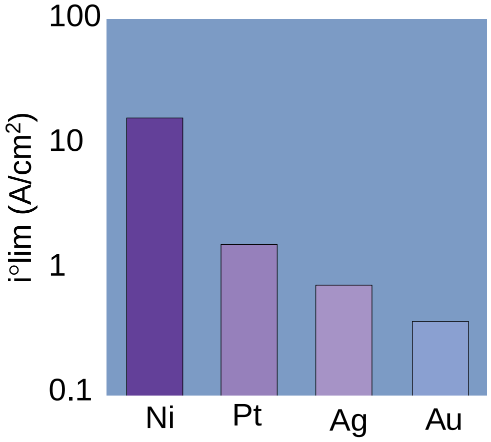
Oxidation of H_2 at the interface using a proton conducting oxide

In SOFCs based on oxide ion conducting solid electrolytes, the products of fuel oxidation are evolved at the anode. In an alternative cell design, based on high temperature proton conducting electrolytes, the fuel is hydrogen and the product, water vapour, is evolved at the cathode. The oxidation of hydrogen at the interface of a metallic electrode and the proton conducting perovskite $Sr_{0.995}Ce_{0.95}Y_{0.05}O_{2.975}$ was studied at 600 to 800 °C in hydrogen containing atmospheres, conditions relevant to SOFCs. Under anodic polarization different metals showed significant differences in the kinetics of hydrogen oxidation. Nickel, a preferred anode material for most SOFC systems, gave the highest current densities. Nickel, platinum and, to a lesser degree, silver displayed high activation energies for hydrogen oxidation. Gold, a relatively inactive material, displayed a low activation energy. Work is continuing to determine whether these differences can be related to the energy of adsorption and dissociation of hydrogen molecules on these metals.

Model studies of internal reforming in SOFC

In SOFCs methane cannot be oxidized directly on the actual Ni/YSZ cermet anodes, but has to be converted via steam reforming to hydrogen and carbon monoxide, which can then be electrochemically oxidized in the fuel cell. The high operating temperature of the solid oxide fuel cell allows the steam reforming process to take place inside the stack (internal reforming). Practical realization of internal reforming, however, poses some problems. Ni is a good steam reforming catalyst and on a Ni/YSZ anode the steam reforming rate is much higher than the reaction rates of the electrochemical processes. Therefore, the endothermic steam reforming process and the exothermic electrochemical oxidation processes may be very unevenly distributed over the stack volume resulting in a very inhomogeneous temperature distribution. This reduces stack efficiency and may even result in mechanical failure.

From a 3D model of the heat, mass and charge transfer processes, it was possible to identify what area specific reaction rate of the steam reforming rate is optimal. In addition, a more detailed (1D) model of the chemical and electrochemical process in the anode was formulated taking into account non-linear kinetics of the electrode reaction, gas-phase diffusion and effects of particle connectivity. This model allowed a study of how the area specific steam reforming reaction rate varies with the characteristics of the anode such as Ni-content, porosity, particle size and electrode thickness. The model studies showed that the most effective way to reduce the steam reforming rate to a suitable level is to reduce anode thickness to 5 - 10µm, and that this should not reduce the electrochemical performance. Model studies of this sort thus provide guidelines for focusing the work on developing electrodes suitable for realization of internal reforming.

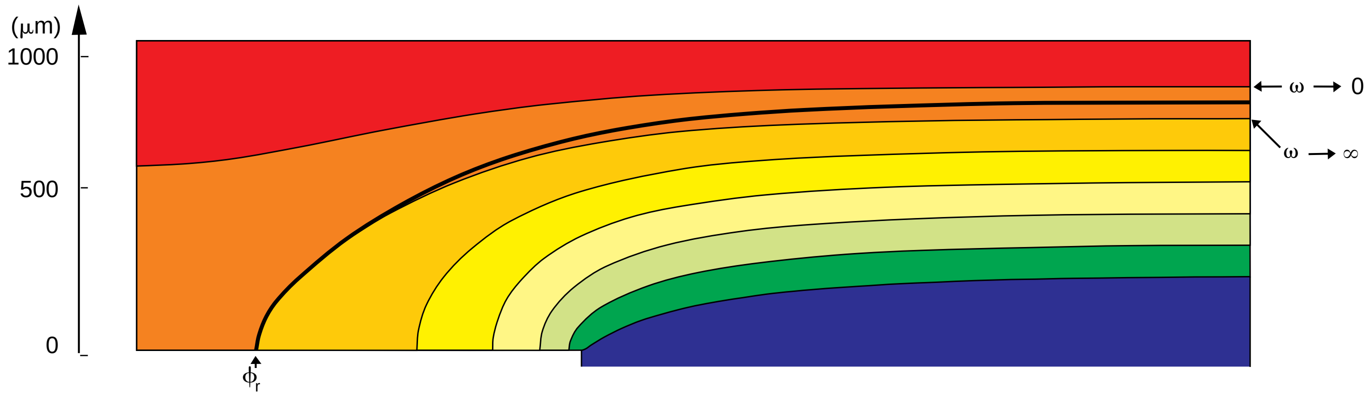


Limiting anodic current density for four metals, obtained at 800 °C in hydrogen, giving a measure of their performance as fuel cell anodes.

Modelling effects of electrode misalignment and gas conversion

Impedance spectroscopy is a popular experimental technique for studies of reaction mechanisms. A reference electrode is often introduced to measure the losses associated with the individual electrodes. A small oscillating voltage is applied between working and reference electrode. The resulting current is measured and the impedance of the electrode determined as a function of frequency. Interpreting the impedance spectrum and deriving information about the electrochemical process from such experiments is not trivial. When current flows through the cell, hydrogen is consumed and water produced at the anode (vice versa upon reversal of the current). This results in a variation in gas composition above the anode. Normally this variation is neglected in the analysis of the impedance spectra. However, a change in gas composition gives rise to a change in Nernst potential measured against a reference electrode in a stable atmosphere. Therefore, an impedance may be associated with the gas conversion. This effect is in fact the source of the dominant semicircle observed at low frequency in the impedance spectrum of SOFC anodes. A simple model of the impedance related to the gas conversion was formulated assuming perfect mixing of gasses in the volume above the electrode. The model was able to account well for the observed variation of the impedance with changes in gas composition and gas feed rate.

The effects of overall cell geometry of the three-electrode set-up were also analyzed. An important result of the analysis is that cells with thin electrolytes are unsuitable for fundamental studies of the electrode reactions. Even small misalignments of the order of half an electrolyte thickness of the two current carrying electrodes will lead to severe errors in the determination of the polarization resistance. The constraints that would have to be applied on the positioning of the electrodes to realize the usefulness of such thin electrolyte test cells are too stringent for normal SOFC electrode development work.



Example of a calculated potential distribution in the limit of very high frequency in the top part of a three electrode pellet designed for studies of the electrode kinetics of SOFC electrodes. The arrow marked Φ_r indicates the position of the reference electrode and the thick solid curve illustrates the position of the equipotential line $\Phi = \Phi_r$ in the limit of very low frequency (DC). A small change in the distribution is observed going from the high frequency limit to the low.

Materials for oxygen separation membranes

Ceramic membranes with mixed electronic and oxygen ionic conductivity can be utilized for (i) removal of oxygen from inert gases, (ii) massive oxygen production and (iii) conversion of methane to CO + H_2 without diluting the produced syngas by atmospheric nitrogen. Such devices are planned to operate in the temperature range of 500 to 900 °C. As they are pressure driven they need no external electrical driving force. The concept thus shows great industrial potential.

Experimental work was initiated on four compositions in the perovskite-like structure series $Sr_4Fe_{6-x}Co_xO_{13\pm\delta}$. A defect model explaining the equilibrium properties was formulated. The observed thermogravimetric data are in agreement with the defect model. Approximate values for the two equilibrium constants (including their temperature dependence) that describe the defect chemistry were determined.

A conductivity relaxation method was developed in order to investigate diffusion of oxygen through the ceramic. The model includes a 1'st order surface reaction. A powerful numerical least-squares recipe was developed, based on a universal numerical solution to the transcendental equations (including the 8'th term), entering the diffusion equations. Diffusion coefficients as high as 10^{-4} cm^2/sec were encountered. Permeation experiments with real membranes are planned.

High-temperature superconductors

The programme aims at forming a basis for utilization of high-temperature superconductors in the Danish electric power sector. At present the technological possibilities for utilization of superconductors in power transmission and power distribution are investigated with focus upon fabrication and characterization of superconducting high power cables. Establishment of a fundamental understanding of the mechanisms related to texturing and phase conversions, required for the establishment of superconductivity, is combined with the development of new fabrication technologies with a potential for industrial scaleability to improve critical current densities and decrease manufacturing costs of superconducting tapes.

Development of texture and progress of phase transformation during annealing in superconducting tapes

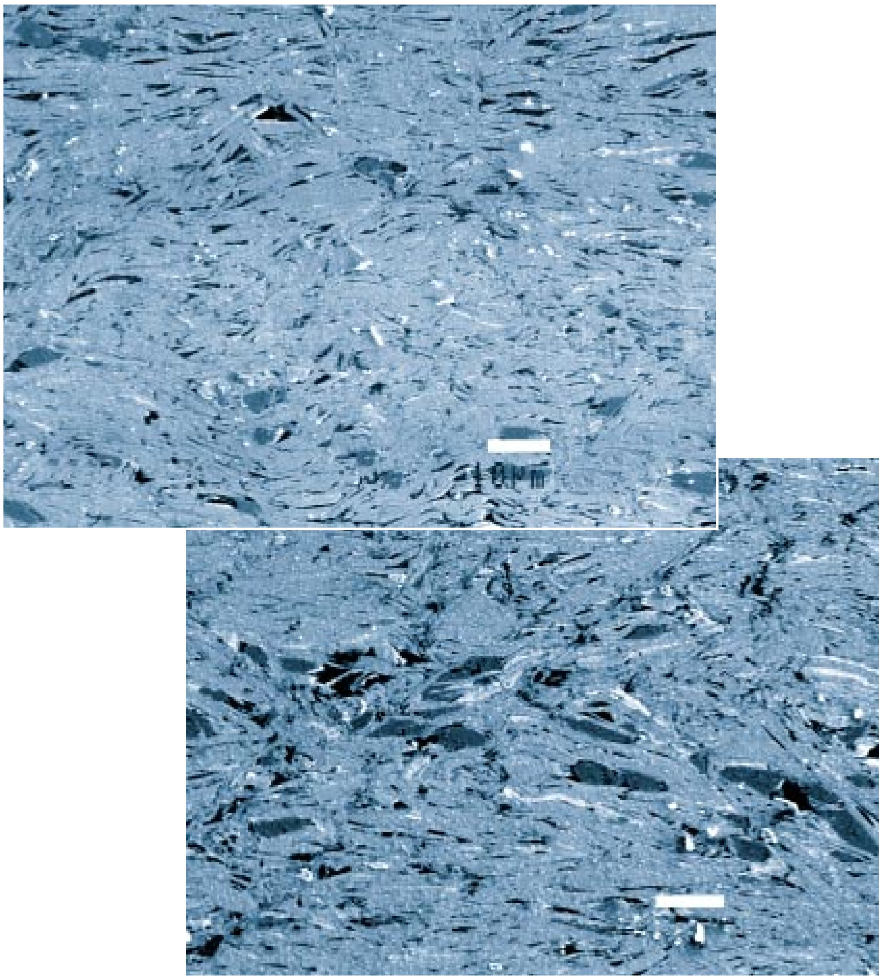
Despite much effort the structural effects of the two annealing cycles normally applied to the BiSCCO/Ag tapes are still poorly understood. In particular, the origin of the crucial texture development of the BiSCCO grains (c-axis alignment) and the solid state transformations are debated. The Department is collaborating closely with NKT Research Centre and the Department of Solid State Physics and Chemistry at Risø in an attempt to model these phenomena in order to optimize the annealing processes with respect to time, temperature, *T*, and oxygen partial pressure, pO_2. For structural characterization a series

Elements of high temperature superconducting tapes

	Explanation	Composition
BiSCCO	Generic name of ceramic family of compounds with HTSC properties	Bismuth-Strontium-Calcium-Copper-Oxide
Bi-2212	Compound of the BiSCCO family	$Bi_2Sr_2Ca_1Cu_2O_x$
Bi-2223	Compound of the BiSCCO family	$Bi_2Sr_2Ca_2Cu_3O_y$
Plumbates and Cuprates	Secondary phases. React Bi-2212 to Bi-2223 during fabrication of HTSC-tapes	ex. $(Ca,Sr)_2PbO_4$, CaCuO

of complementary techniques are used: synchrotron radiation, laboratory based X-ray diffraction, scanning electron microscopy, and measurements of the critical current densities. The synchrotron technique is based on a unique set-up at the synchrotron laboratory HASYLAB in Hamburg. Using high energy X-rays this set-up allows *in-situ* investigations of the superconducting powder inside the Ag cladding.

The major results obtained so far relates to the first annealing cycle: (i) The texturing mainly takes place during heating, and is related to grain growth of Bi-2212, which is drastically increased upon the dissolution of $(Ca,Sr)_2PbO_4$. The c-axis alignment of Bi-2223 is essentially constant during time and to a first approximation identical to the final alignment of Bi-2212. (ii) Upon dissolution of $(Ca,Sr)_2PbO_4$ a CaCuO phase is produced. The Bi-2223 phase is a result of a reaction of this phase with Bi-2212. In some parts of the (T, pO_2) phase diagram an intermediate phase comprising a stacking of Bi-2223 and Bi-2212 unit cells was observed. This provided clear evidence that the reaction takes place by means of an intercalation process. A sparse (T, pO_2) phase diagram was mapped. At higher temperatures a series of eutectica were identified.



SEM micrographs of (top) a quenched tape: the microstructure formed at the high temperature heat treatment has been 'frozen in', and (bottom) a slowly cooled tape. The size and total amount of non-superconducting secondary phases (black and white phases) are larger during slow cooling due to the decomposition of Bi-2223 phase.

Annealing of BiSCCO tapes at reduced oxygen partial pressure

The main superconducting phase of BiSCCO, the Bi-2223 compound, is formed by the reactions of many secondary phases during repeated heat treatments in a very narrow temperature range around 830 °C and it loses stability during cooling. It is fortunate that research results obtained the world over in the last few years have proven that heating under reduced oxygen partial pressures increases the stability range and the Bi-2223 phase can be cooled to low enough temperatures for the decomposition not to take place.

A proof that the Bi-2223 forms under the best conditions is given by the fact that the concentrations of the other phases have substantially decreased or become negligible and that a large electric current can be carried without resistance by the wire or tape. Laboratory experiments were carried out at the Department in collaboration with NKT/NST, the Danish manufacturer of superconducting tapes. The results show that: (i) The concentrations of the secondary phases are quite small during the heat treatments at high temperature and they increase during cooling in air (SEM and X-ray diffractograms have been used at the Department to identify and estimate the concentrations of the secondary phases present at high and low temperatures). (ii) Heat treatments in reduced oxygen partial pressures, (e.g. O_2 concentrations decreased from the 21 % in air to 7 %), increase the maximum superconducting current that can be carried by the standard tape from 20 to 27 A. The results are being implemented industrially to improve the characteristics of the superconducting tapes.

Techniques

A number of advanced techniques are used within the programme area. The methods for characterizing defect chemistry are thermogravimetry, differential thermal analysis and differential scanning calorimetry. Phase-changes are identified by dilatometry (volume change), diffraction of conventional X-rays or synchrotron radiation (changes in crystallographic lattice parameters). Since the electrochemical activity of the electrodes is very important, several methods are used for electrical measurements under controlled atmosphere and temperature. Electrical conductivity measurements are performed in the temperature interval -196 °C to 1050 °C.

Almost all characterization techniques available at the Materials Research Department are used within this programme area. Examples of techniques belonging to other programme areas are (i) electron microscopy methods (EDS, EBSP, etc.) for characterizing chemical composition, structure, texture and morphology, (ii) mechanical testing (stiffness, fracture toughness and strength), (iii) non-destructive evaluation (detection of flaws in SOFC components, quality control) and (iv) solid mechanics modelling of stress distribution and fracture mechanics analysis. The fact that so many methods and disciplines are used in these projects reflects a main feature of the strategy, namely to characterize the materials and the components as detailed as possible, in order to support the technological development. Another important consequence of the strategy is the availability of many shaping methods (and skills in using them) that makes it possible to make samples with pre-determined characteristics for the scientific studies.

Mathematical models of the processes in an operating SOFC, or in a stack of cells, may be valuable tools in the development and testing of SOFC stacks. Such models may be used for analysing the consequences for cell or stack performance of various changes in the cell or stack design. Another use of such models is in the specification of the properties of the individual components in the stack (anode, cathode, electrolyte and interconnect). This kind of information is very important in the search for new and better materials, i.e. it defines the targets for the researcher. The modelling methods that are used are mainly the finite volume method (modelling of heat, mass and charge transfer) and FEM (integral SOFC stack modelling and micromodelling of SOFC electrodes).

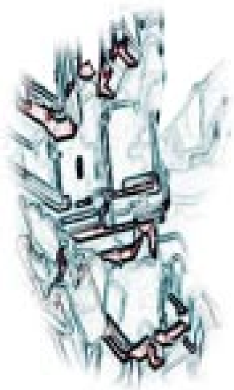
	Method	**Objective**
Ceramic Shaping	Tape casting/spraying/screen printing/dip coating/cold isostatic pressing	Fabrication of samples, components and devices
	High shear rolling/profile rolling/planar rolling	Deformation, texture generation
	Grinding by diamond tools	Machining of specimens and components
Characterization Techniques	Light scattering	Particle size distribution
	Thermogravimetry/differential thermal analysis/differential scanning calorimetry	Characterization of solid state processes
	Dilatometry	Volume changes as function of oxygen partial pressure and temperature, sintering characteristics
	X-ray diffraction/synchrotron radiation	Phase characterization, changes in lattice parameters
	Impedance spectroscopy/van der Pauw DC/classical 4 point DC	Electrical conductivity, electrochemical activity
	Direct current vs. voltage (chrono-amperometric and potentiodynamic)	SOFC performance, critical current density

MATERIALS ENGINEERING
– modelling and performance

The activities within Materials Engineering as described in the following sections have been focused on material properties for design application, materials engineering, related materials characterization and testing as well as component design.

The activities are presented in two sections, *Design and Mechanical Testing*, which concerns both component design and engineering materials, and *Properties of Advanced Composite Materials*, which covers characterization of materials reinforced with fibres or particulates.

The highlights within this area are (i) the studies of microstructure and mechanical properties of natural fibre composites, (ii) the investigation of the deformation characteristics of aluminium MMCs for forging, (iii) the development of a technique for measuring bridging laws during intralaminar cracking, and (iv) the development of a highly innovative radiographic system for real-time quantitative inspection of large thickness pipes.



Project Funded Research: Materials Engineering

Project type	Project name	Co-participants
Danish Research Councils	Natural Fibres for Polymeric Composites	• Royal Vet. and Agricult. University, Denmark • Inst. for Constr. and Materials, DTU, Denmark
EFP	Windturbine Blade Design	• LM Glasfiber A/S, Denmark • Vestas A/S, Denmark • ELSAM, Denmark • Wind Energy & Atmospheric Physics Dep. Risø • Dept of Fluid Dynamics, DTU, Denmark
EFP	Flywheel for Energy Storage for Add-on-Armour	• Per Udsen Co., Denmark • NESA A/S, Denmark • DEMEX A/S, Denmark
EUCLID	Advanced Techniques for Add-on-Armour	• DEMEX A/S, Denmark • DSM, The Netherlands • OTO Melara, Italy
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 Industri & Marine A/S, Denmark • Kuwait Petroleum Denmark A/S, Denmark • Photonic Science plc, UK • Thomsen Tubes Electroniques SA, France • Sauerwein Systemtechnick GmbH, Germany • Statoil a.s., Norway • Bundesanstalt für Materialprüfung, Germany • Instituto de Soldadura e Qualidade, Portugal • Institut de Soudure, France
BRITE-EURAM	Action for low weight automobile technologies (FLOAT)	• Rover Group Ltd., UK • GIE Renault, France • Centro Richerche FIAT SCpA, Italy • Ranshofen, Austria • Frauenhofer, Germany • Volvo AB, Sweden • SEPARIS, France • ISRIM, Italy • Daimler Benz AG, Germany

Design and mechanical testing

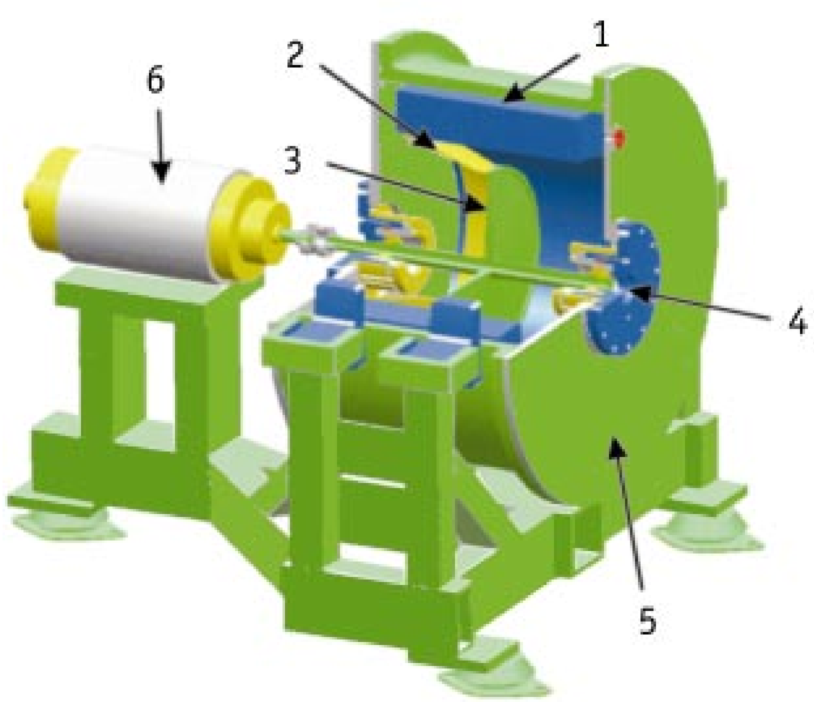
This research programme concerns the development of numerical and experimental methods for design of components with specific emphasis on new materials. The activities include development of advanced non-destructive and destructive experimental methods for mechanical testing and characterization of materials.

Design and manufacturing of 1 kWh flywheel for energy storage

The design work on a prototype flywheel system for energy storage has been finalized and the manufacturing of components started. The design is based on previous concept studies and patents on the critical connection between shaft and rim, which enables connection to either a mechanical transmission unit or to an electric motor/generator.

The flywheel was designed for an operational nominal speed of 36000 rpm and 20 - 50 kW power rating. The originally planned mechanical transmission had to be abandoned for budgetary reasons. A direct driven motor/generator and converter were chosen instead: a commercial spindle motor for tooling machines with a maximum speed of 50000 rpm and 6.5/14 kW power rating together with a versatile frequency converter. The motor/generator with converter as well as lubrication and cooling system were installed and initial acceptance tests performed.

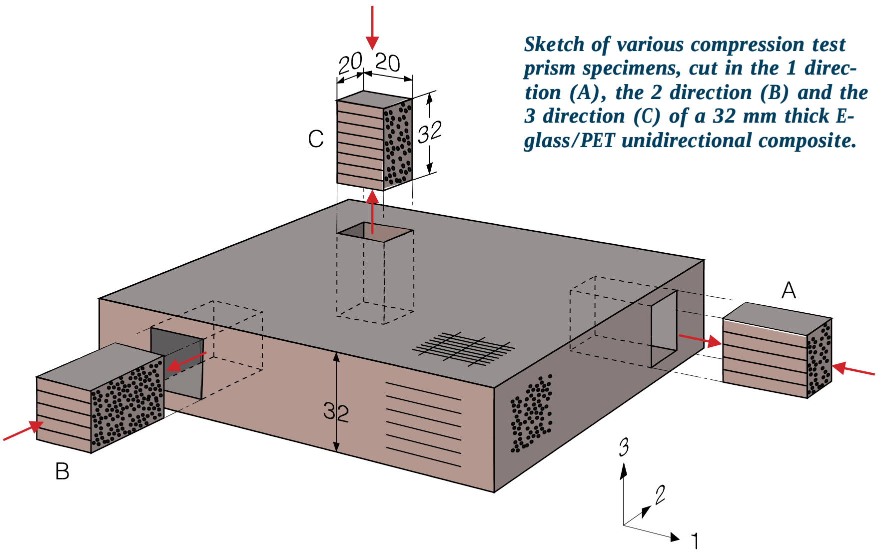
The flywheel rotor is supported on ball bearings and designed for operation beyond the lowest critical rotor speeds as a soft rotor. The bearings are supported in flexible bearing houses in the flywheel containment house. The flywheel rotor has to be operated in vacuum, whereas the chosen motor/generator shall be operated under atmospheric conditions. They are connected to each other through a flexible coupling and mounted in a common frame. The composite flywheel rim and connection element was manufactured by a filament winding technique, and additional materials testing was performed in order to verify the properties of the connection element. After initial test runs in order to verify the rotor-dynamic behaviour, the system is expected to be operational in the middle of 1998.



Test facility for prototype flywheel testing now being established in the Department. The 400 mm diameter, 20 kg carbon/epoxy rim (1) is connected to the thin composite shell element (2), attached to the integral steel disc and shaft (3). The shaft is supported in squeeze film damped ball bearings (4) in the end covers of the evacuated safety containment (5). The shaft is attached to a 6.5/14 kW motor/generator (6).

Out-of-plane elastic properties of polymer composites

Thick polymer composite laminates are used for an increasing number of applications such as wind turbine blades,



Sketch of various compression test prism specimens, cut in the 1 direction (A), the 2 direction (B) and the 3 direction (C) of a 32 mm thick E-glass/PET unidirectional composite.

flywheels and ballistic protection panels. Design and analysis of these applications requires a knowledge of the elastic constants and strength in the thickness direction (out-of-plane) of the laminate as well as in the directions of the laminate (in-plane).

Two methods were used for measuring the elastic constants of orthotropic laminates: A mechanical compression test and an ultrasonic technique. The compression test requires laminates of at least 25 mm thickness and uses prism shaped specimens with strain gauges attached to the four free faces. All three Young's moduli and the three Poisson's ratios can be determined in this manner. During the ultrasonic testing the specimen has to be immersed in water. However, all nine elastic constants of an orthotropic material can be measured with this technique.

Measurements on specimens from the same 32 mm thick unidirectional composite (E-glass fibres in PET matrix) show a good agreement between the methods. The ultrasonic technique is non-destructive in nature and gives the elastic properties for very low strain levels. In contrast, the compression test can be used to obtain material properties at strain levels limited only by the compression strain to failure. As an example consider the environmental degradation with time of a laminate. The compression test can be used to characterize the original material in the entire strain range. The ultrasonic technique can be used for the measurement of elastic constants at regular intervals during the environmental treatment. The changes in elastic constants with time can be used as a measure of the degradation of the laminate.

Fatigue testing and creep testing of thermoplastic polymer composites

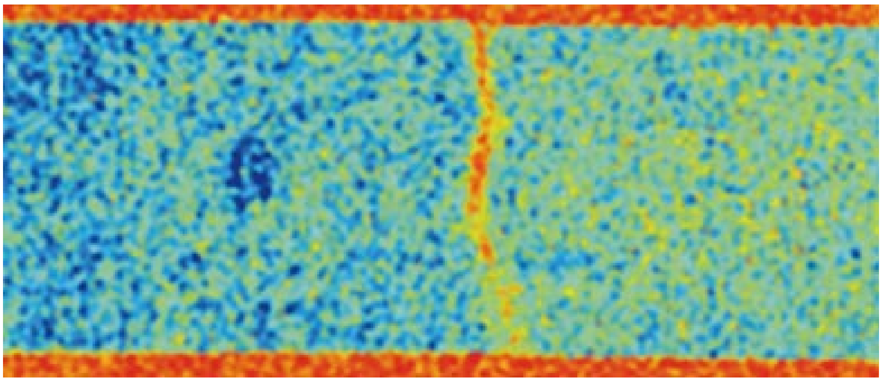
The fatigue behaviour of thermoplastic composites was investigated experimentally. Staircase tests, to determine the fatigue limit at given numbers of cycles (e.g. 10^5 to 2×10^6), were used to obtain materials data for design and to validate materials for production. Test specimens were cut from 1 - 3 mm thick panels. Cyclic tests (uniaxial loading) were carried out 0 º, 10 º, and 90 º relative to the fibre direction and were performed at frequencies up to 30 Hz. To minimise the testing costs the test frequencies were selected as high as possible without getting effects from internal heating due to hysteresis and damping. The tests are run at -30 ºC, at room temperature, and at temperatures up to 120 ºC. Creep tests on the thermoplastic composites were also performed. Creep curves were determined both at 25 ºC and at 120 ºC with loading directions 0 º and 90 º relative to the fibre direction. The creep tests were conducted for more than 1000 hours under constant load and temperature.

Non-destructive evaluation of advanced light alloys and correlation with destructive tests and microstructure

The Department is involved in two projects on advanced light alloys for automobile components, specifically, thixoforming of aluminium alloys and hyper-eutectic aluminium-silicon alloys. One of the main purposes is to establish a quantitative relationship between data from non-destructive evaluation (NDE), microstructure and mechanical properties. The perspective is to be able to predict component performance from a fast non-destructive inspection. High contrast and high resolution radiography and accurate through transmission and pulse-echo ultrasonic C-scanning have been applied. The detection limit was extended from conventional casting defects such as voids and cracks of mm-size to groups of micro-porosity. The latter type of defects, caused by shrinkage, is present at grain boundaries and of 0.1 - 0.2 mm in size. At the current stage, a qualitative relationship between NDE data and microstructure has been established. NDE has been applied to qualitatively predict the mechanical performance of samples, based on the apparent defect population. As an example, of a number of tensile specimens made from thixoformed AA6082 (aluminium-silicon-magnesium) components, two specimens which contain groups of micro-porosity were unveiled by NDE. The yield strength and tensile elongation of these two specimens were reduced by 30 % and 90 %, respectively.

Application of high resolution radiography on new plant composite materials

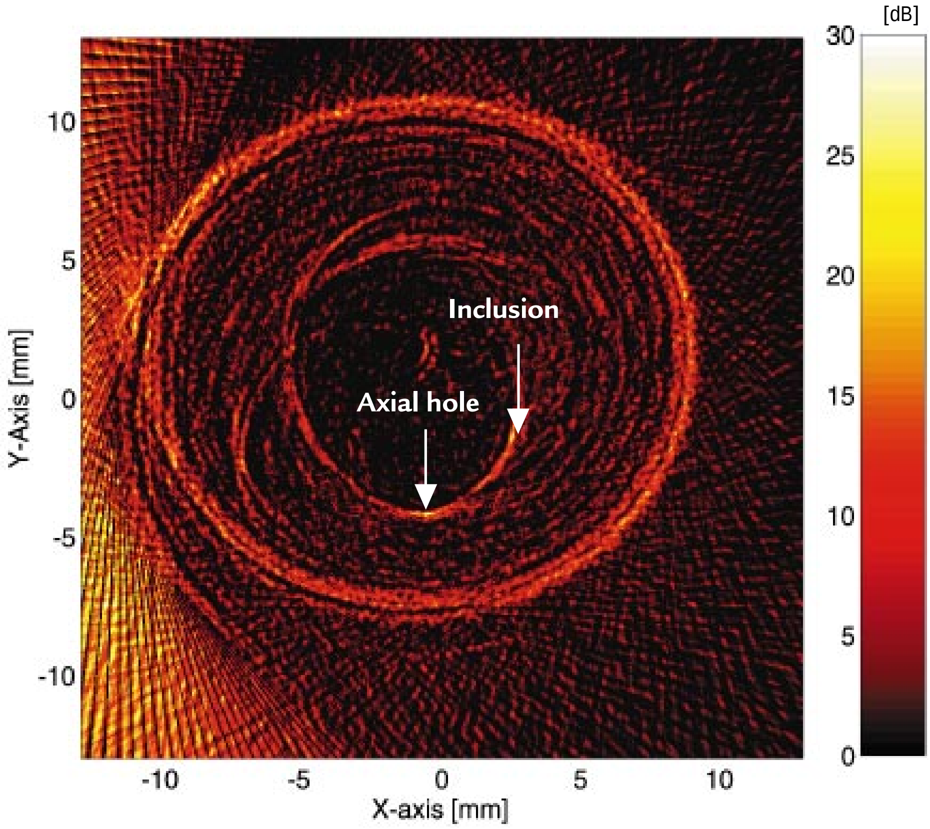
New advanced composites, made of plant and wood fibres, are becoming increasingly more attractive for environmental and political/economical reasons. These materials have proven suitable as re-



Digitized and false colored radiograph of a jute fibre-polypropylene test bar after strength testing. The predominant fibre orientation is N-S. Note low density spots and patches (blue area) and general sample homogeneity. Width of bar approx. 2 cm.

placements for conventional fibre composites as construction materials e.g. in the transport industry. Testing and characterization is important in order to ensure the material quality and performance. NDE, e.g. by radiography, offers a possibility to characterize flaws prior to destructive mechanical testing. The information obtained can be applied to assess the criticality of defects and to establish a performance to quality relationship. At the manufacturing control stage this knowledge can be applied to establish an accept - reject criterion.

Initial investigations have been conducted applying high resolution radiography on thin (3 mm thick) tensile tests bars made of jute fibres in a polypropylene matrix. Low energy radiation (peak energy 20 keV) was used in combination with a slow, fine grained film. The film was subsequently digitized in order to secure a high contrast between fibres and matrix. A spatial resolution of a few micrometres was obtained. The digitized image permits quantitative measures to be extracted, e.g. void sizes, dimensions of inhomogeneous patches and foreign inclusions, fibre alignment, etc. However, at the present stage, results have been applied only qualitatively to monitor the general homogeneity, distribution and direction of fibres. Future work aims at providing a correlation between destructive test results and quantitative data from radiography.



Reflection tomogram of a cylindrical AlSi-alloy showing: the cross-sectional dimensions (15-20 mm), an inclusion and a 2 mm axial hole. High reflectivity is indicated by yellow.

Non-destructive evaluation of solid oxide fuel cells

The plates functioning as electrolytes in SOFCs are only 0.2 mm thick and the thickness of the electrode layers is about 40 µm. It is important to ensure that the components used are free of large defects that can result in mechanical failure or lower the electrical performance of the stack. Therefore, ultrasonic through-transmission scanning was used to characterize cells for SOFC stacks.

In the through-transmission technique two separate transducers facing each other are used as transmitter and receiver of the ultrasonic signal. The amplitude and time-of-flight of the transmitted ultrasonic wave is measured during a raster scanning performed in water. High frequency (50 MHz) focused transducers were used in order to get a good resolution. The amplitude of the transmitted signal is damped by defects such as delaminations and cracks. In the present study plates with artificial defects (delaminations) were used as calibration samples. Destructive examinations have verified the capability of the technique to find the defects.

Ultrasonic reflection tomography

Ultrasound computed tomography (UCT) is a relatively new imaging technique in NDE of solid materials. It offers an improved characterization of discontinuities (inhomogeneties or defects) compared with the conventional A-, B- and C-scan technique.

In the UCT-technique, solid materials under investigation are insonified by ultrasound and the reflected (or transmitted) signals are used to reconstruct a two-dimensional cross-sectional tomogram of the material. A tomogram is simply a picture of a slice (Tomo comes from the Greek word tomos, meaning slice, and gram refers to a graphical representation). Ideally, this tomogram contains only an infinitesimal thin two-dimensional cross-sectional plane, without any interference from other planes. The tomogram may be displayed from any desired location and with any desired orientation relative to the specimen.

Discontinuities in cylindrical AlSi-alloys were studied by ultrasonic reflection tomography. Reflection data were acquired by high frequency (narrow beam) transducers in a synthetic circular aperture array. From these profiles reflection tomograms were constructed using a filtered back-projection technique. Straight line propagation was assumed. Three-dimensional information (i.e., discontinuity location, dimension and type) was obtained by stacking these reflection tomograms in multiple planes in the third dimension.

Properties of advanced composite materials

This research programme concerns the properties of advanced composite materials with either a metal, polymer or ceramic matrix. Focus is on the characterization of microstructure, mechanical and physical properties. Both inorganic and natural fibres are dealt with.

Improved methods for stiffness based fatigue diagrams for glass fibre/polymeric composites

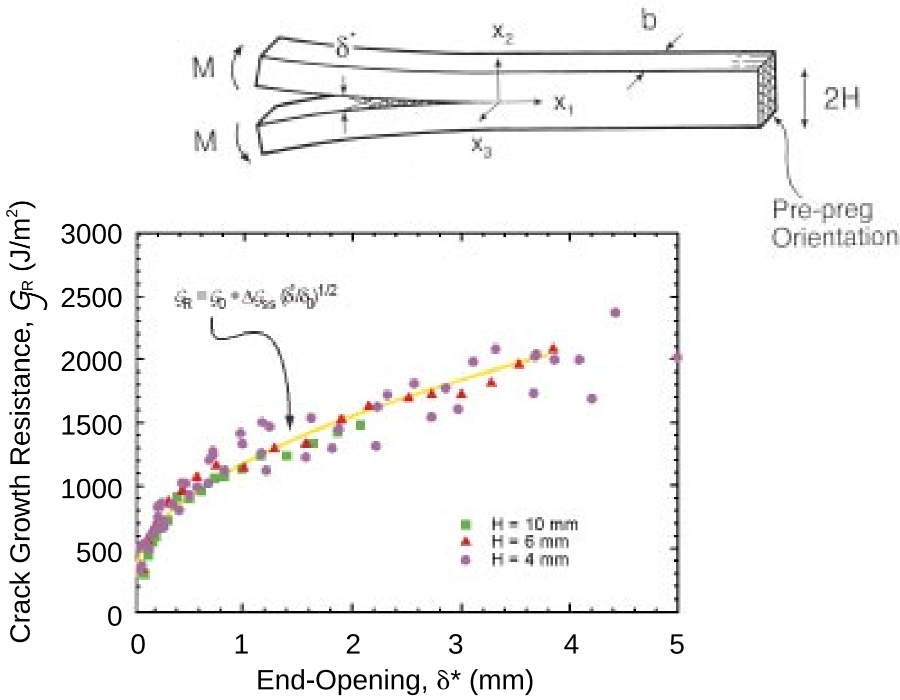
A model has been developed for the prediction of the damage evolution of fibre composite materials during cyclic loading. The model predicts the relative stiffness change per cyclic loading at given stress or strain levels; the stiffness decrease rate is described by a power dependence on the stress and the strain. This relationship allows a constant loading amplitude S-N curve to be derived based on experiments by recording the damage. It allows the fatigue lifetime to be predicted without running the fatigue test to failure. An interactive testing programme was developed where stiffness and damping behaviour can be followed experimentally cycle by cycle. Experiments were performed on carbon, glass, and carbon/glass-polyester composites in fatigue tests. Variable load amplitudes under block loading were also studied. The fatigue damage was recorded by measuring the stiffness changes in the material under this cyclic loading. Stiffness controlled fatigue curves were generated and presented in S-N diagrams based on normalized stress. It was found that the fatigue lifetime can be predicted based on constant load fatigue results, assuming independence of preceding load history.

Torsion test for determining the shear properties of composite materials

A fabrication procedure for the manufacture of high-quality tubes from prepreg material has been established. A problem in this connection is the thickness reduction during the consolidation of the material, which creates folds in the material if the compaction takes place from the outside. Split moulds were manufactured, and procedures for the location of the material in the split mould were established. These consist of unwinding a roll of the material in the mould, while simultaneously removing the peel-off layers. The method will probably produce the best quality tubes possible from pre-preg material.

Many tubes were produced. They were subjected to static and fatigue testing in torsion. This provided the much needed data on the static and dynamic shear properties of the material for wind turbine blades.

Static stiffness values were determined by strain-gauge measurements. These data will be very accurate, if the geometry of the tubes have been determined precisely. However, the fatigue properties should be regarded as lower limits for a hypothetical material with no end effects, since the location of the failure zones is, as expected, influenced by the unavoidable overlap regions.



Schematics showing a DCB specimen loaded with pure bending moments (end-opening of the bridging zone, δ*, is indicated). The measured relationship between the crack growth resistance G_R and δ* is independent of specimen geometry, indicating that the bridging law can be regarded as a material property.

Large scale bridging: Measurement of bridging law of fibre-cross-over bridging

Unidirectional laminae have a low resistance to cracking in planes parallel with the fibre direction. The crack growth resistance can, however, be enhanced significantly by fibre-cross-over bridging. Large scale bridging was studied experimentally in a unidirectional carbon fibre/epoxy matrix composite material. The experiments were conducted using double cantilever beam specimens loaded with pure bending moments. For this configuration, in contrast to most other standard test configurations, the global energy release rate is independent of the bridging traction from the fibres. For intralaminar cracks a significant increase in the crack growth resistance occurred with increasing crack extension. The increase was attributed to fibre cross-over bridging. The R-curve behaviour i.e. the relationship between crack growth resistance G_R and crack extension depended on specimen geometry. Therefore, the concept of R-curves cannot be used as a material property when large scale bridging occurs. The crack opening at the end of the bridging zone δ^* was measured by extensometers. Utilizing the path independent J integral the bridging law (the relationship between the local crack opening δ and the local bridging traction σ) was computed from

$$\sigma(\delta^*) = \frac{dG_R}{d\delta^*}$$

The bridging law was found to be independent of specimen geometry. A bridging law can therefore be used as a material property in composites with large scale bridging. The bridging stress was found to be related to the crack opening as $\sigma \propto \delta^{-1/2}$. This relationship was confirmed by an analytical model for fibre cross-over bridging.

Mechanical properties of jute fibre/polypropylene composites compare favourably with glass fibre/polymeric composites

Plant fibre composites were manufactured from jute mats and polypropylene (PP) foils, and from flax mats and PP-foils. The fabrication was made under pressure in an autoclave where the PP-foils melt and form the matrix. The materials (both composites and fibres) were characterized with respect to microstructure, physical and mechanical properties.

The composite density was found to be about 1.0 g/cm^3; the volume fraction of jute fibres was 25 - 30 %, and the porosity content was 3 - 9 %. This rather low porosity indicates a composite of good quality. This was confirmed by non-destructive evaluation based on X-ray radiographs. The mechanical properties were characterized under tensile loading; the stiffness and strength were measured. The jute/PP composites had stiffness values of 3.5 GPa in the 'weak' direction, and of 7.0 GPa in the 'strong' direction. This anisotropy is assumed to be caused by the fibre orientation in the jute-mats. The corresponding strengths of the composites were 27 MPa and 55 MPa, respectively. The stiffness and strength are comparable to those of other natural composites based on PP-matrix and plant fibres, such as flax, wheat straw, and kenaf, as well as with values for glass fibre/PP composites.

The effect of porosity was evaluated from tests at a fibre volume fraction of 31 %. The stiffness and strength values were found to decrease with porosity content in a steep, non-linear way, which is typical of synthetic composites. The fracture zone of the tensile loaded jute/PP composites showed some fibre pull-out, indicating moderate interface bonding between (non-treated) jute and PP.

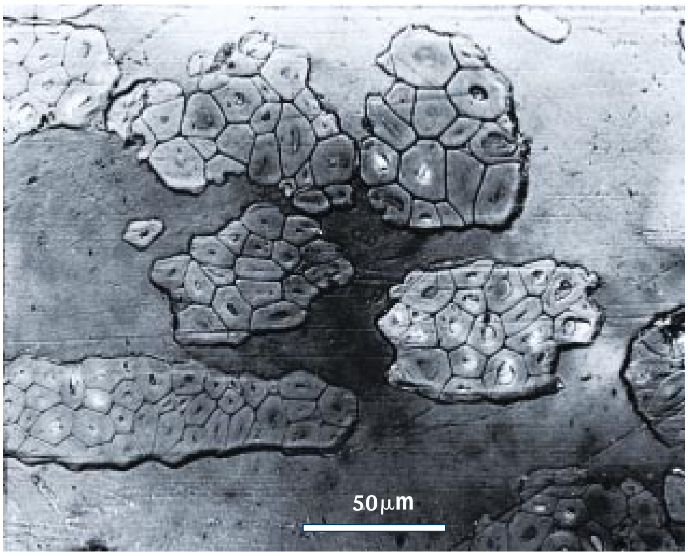
The long term aim is to fabricate and characterize plant fibre/polymer composites, with the goal of making stiff and strong composites of low weight and with a (potential) biodegradability.

Natural jute fibres characterized in terms of physical and mechanical properties

The properties of the jute fibres are of great importance for the properties and performance of the composites. Therefore, fibres were characterized by several direct and indirect methods. The fibre morphology was found to consist of approximately equiaxed single fibres of diameter of about 20 µm, assembled in bundles of 10 - 20 single fibres. The fibre density was 1.46 g/cm^3. The chemical composition was about 60 % cellulose, 12 % hemicellulose, 18 % lignin and 10 % other substances. The cellulose content is important for the (potential) crystallinity of the plant fibres, and thereby for the stiffness and strength of the fibres.

The characteristics of the jute/PP composites were used to calculate the properties of the jute fibres, based on composite theory. From the stiffness and strength in the 'strong' and in the 'weak' direction under tensile loading of the composites, the orientation-bias of the fibres in the mats was calculated. This gives a deviation from fully random planar orientation (orientation factor 0.33), such that the 'strong' direction has an orientation factor 0.44 , and the 'weak' direction has a factor of 0.22.

From these results it is possible, based on composite theory, to estimate the stiffness and strength of the jute



Jute fibre/polypropylene-matrix composite, sectioned transverse to the local fibre direction. The structure and morphology of the jute fibres can be seen. Note, that the lumen of the fibres seems to be filled by polypropylene.

fibres; the value for stiffness is 62 GPa and for strength 545 MPa; these values are comparable with other estimates. The stiffness is comparable with the value for glass fibres (70 GPa), and thus gives the potential for jute fibre composites to match glass fibre composites in (certain) mechanical properties.

Composites of NiTi fibres and polyethylene matrix show large shape recovery capability

The shape active effect in the alloy of NiTi was used to impose super visco-elastic response in a composite, containing NiTi fibres in a polyethylene matrix. The technical interest in such composites relates to their potential use as actuators responding to external changes in stress and/or temperature.

The composites were manufactured from NiTi-fibres of diameter 190 μm and a low density polyethylene matrix by consolidation in an autoclave under pressure at 150 °C. The content of fibres was 8.5 %. The NiTi fibres were heated to 450 °C for 30 minutes and quenched before the composites were made; this left the NiTi fibres in the austenitic condition.

Unidirectionally reinforced composites were studied experimentally during a loading/unloading cycle at 60 °C. The tensile direction was parallel with the fibres. The loading was continued until the austenite to martensite transformation was completed, which took place at an axial strain of about 6 %. During the subsequent unloading the martensitic to austenitic transformation brought the material back to nearly the original length. This demonstrates the large viscoelastic shape recovery effect of the composites.

The shape active behaviour of the composites was described by a model accounting for the stress and temperature dependent transformation of the NiTi fibres and on the viscoelastic behaviour of polyethylene. The model predicts a loading/unloading behaviour which is in good agreement with the experiment.

The experimental facilities and methods for characterizing the mechanical properties of materials are widely used within this programme area and in other programmes in the Department.

The facilities consist of a number of servo-hydraulic Instron testing frames (100 and 250 kN) with actuators ranging from 5 to 250 kN, load cells from 20 N - 250 kN, strain measuring equipment (strain gauges, extensometers) and temperature controlled chambers and furnaces for testing at low and high temperatures (up to 1000 °C). A number of load frames for creep testing of metals and polymers are used. Computerized control and data acquisition is used during testing. In some cases commercial data acquisition programs are used, while in other cases special programs are developed in-house.

For mechanical testing, both design of the test specimen, of the experimental set-up and of the load introduction are crucial. Some tests require specially designed tests specimens and fixtures. A number of different grips, specimen geometries and experimental set-ups have been developed and successfully used. Thermal imaging and acoustic emission systems are routinely used during testing, in order to study the evolution of damage.

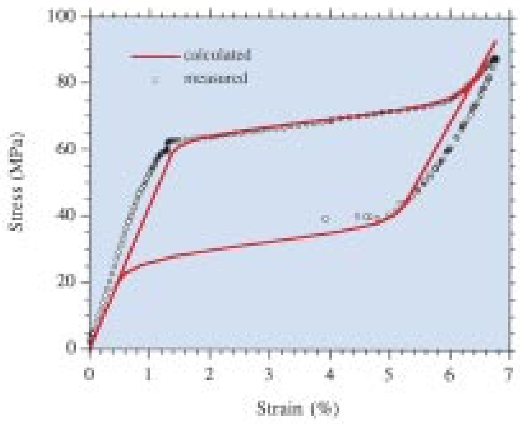
Non-destructive examination by ultrasonic or X-ray methods is widely used for inspection and characterization, in particular for quality control of manufactured parts or components, and in connection with mechanical testing for damage or failure analysis after testing.

Extensive modelling of stress and strain fields of specimens and experimental set-ups are performed by the finite element method for the interpretation of test results. Likewise, digital imaging techniques are used for the interpretation and visualization of results from both radiographic and ultrasonic inspections.

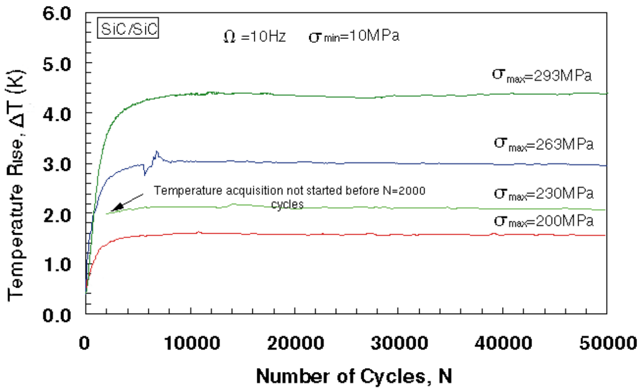
Techniques from other programme areas of the Department are sometimes used in projects belonging to Materials Engineering. For instance, test specimens are often processed by other programme areas. Also, characterization of microstructural damage or studies of fracture surfaces are done by the use of SEM. Several mechanical tests may also be conducted in the ESEM, providing a direct relationship between the evolution of damage (observed *in-situ* during the tests) and mechanical behaviour.

Methods for controlling test lapse

Computer programs for controlling tensile test machines were developed for two purposes: (i) to control the fatigue testing of materials, and (ii) to save data as required during the testing. The programs are flexible with respect to testing cycles (load versus time), and they can calculate the materials response, stiffness, energy dissipation etc. with high accuracy during the test. Furthermore, the programs can adjust



The loading/unloading response of the shape active composite of NiTi fibres in a polyethylene matrix, performed at 60 °C. The large strain shape recovery covers a range of about 6 %. The theoretical model prediction is shown by the fully drawn curve.



During cyclic loading, ceramic matrix composites (CMCs) may experience temperature rise due to internal heating (sliding along fibre/matrix interfaces). The plot shows the temperature rise of a CMC as a function of the number of cycles for various applied stress ranges. When the damage state is stable the temperature rises to a steady-state level (the energy dissipation equals the heat loss).

the control parameters continuously, for example fatigue testing with constant stress, or constant strain ranges. There are no particular requirements to the test machine, so the programs can be used in combination with most testing machines of the Department.

Isothermal chamber for mechanical testing

During the last three years the Department has worked on establishing a mechanical testing facility capable of quantitatively measuring the amount of internal heat generated within a test specimen due to cyclic loads. Several gripping arrangements and testing geometries have been implemented and the technique is now a part of the standard mechanical testing facilities in the Department. Emphasis has been on testing unidirectional and woven fibre-reinforced ceramic matrix composites. These systems develop internal heating due to frictional sliding between fibres and matrix. The amount of heat generated per second can also be measured as the area of the stress-strain hysteresis loop times the cyclic frequency. The strength of the testing technique lies in its capability of detecting hysteresis in materials with low thermal diffusivity like ceramic and polymeric composites. In these cases the technique of detecting hysteresis from temperature rise measurements gives a resolution higher than the detection limit of conventional strain measurements.

The technique is also well suited for monitoring long-term fatigue tests as the time-temperature relationship requires much less storage capacity than the stress-strain data. Then, significant changes in temperature can be used for triggering stress-strain acquisition, as changes in the shape of the cyclic stress-strain curve are usually more informative with respect to evaluating details of the material degradation. Another feature of the technique is the capability of continuously detecting temperature distributions and thus damage development with an infrared camera. This gives a possibility of monitoring more complex fatigue behaviour and it is aimed at testing structural components with e.g. holes.

	Method	Objective
Characterization Techniques	Static tensile/compression/ torsion tests	Elastic constants, stress-strain behaviour, strength, creep
	Dynamic tensile/compression/ torsion tests	Fatigue behaviour, damage development, damping properties
	Fracture mechanics testing	Fracture toughness, fatigue crack propagation
	Acoustic emission	Crack initiation, crack growth, damage development
	Thermography	*In-situ* detection of damage evolution by temperature rise
	Ultrasonic scanning	Detection of flaws, elastic constants
	Radiography	Location of flaws, porosities, water content, etc.
Data Analysis and Presentation	Digital imaging	Radiography, ultrasonic scanning

High energy real-time radioscopy detector

Within the framework of an EU-funded industrial R&D a new and versatile quantitative γ-radioscopic inspection system was developed. The system consists of (i) two different high-energy and high performance radiation imaging detectors, (ii) a versatile and rigid robot unit aimed at inspection of major pipes and (iii) an expert system driven computer control with integrated procedures for flaw sizing and image processing. Risø, which is coordinating this research project, is also responsible for the main part of the procedures for quantitative image analysis, allowing improved perceptibility and size determination of flaws. The procedures are basically developed on two approaches: (i) the measurement of the remaining wall thickness of pipes, based on profiles across the pipe tangential section, and (ii) an innovative approach which makes use of a priori information of the pipe in order to correlate radiation attenuation with wall thickness variations. The developed procedures have proven highly efficient and thickness of the pipe walls was determined with an accuracy of 4 % for the tangential measurement technique (type (i)) and approximately 10 % for type (ii). The procedures are required to promote the use of electronic imaging systems in radiographic inspection, since new detectors do not, as radiographic film, permit manual ruler-based measurements of image details. The central issue in this context is to achieve an improved inspection efficiency and an improved reliability of quantitative radiographic condition monitoring.

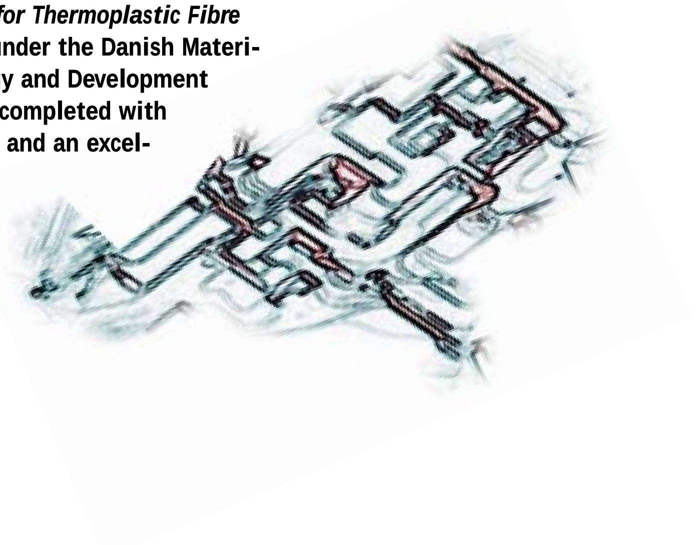
MATERIALS TECHNOLOGY
– synthesis, processing and product

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

The area also covers more fundamental activities with the character of long term basic research. These projects are often done in collaboration with universities in Denmark, frequently as PhD projects.

The activities in this area are described under the subheadings *Powder Technological Materials* and *Manufacturing Technologies for Advanced Fibre Composites*.

The highlights in the area of Materials Technology are (i) the development of a numerical model for simulation of the thermal history of atomized particles in the spray-forming process, (ii) the development of an integrated oxygen sensor based on Mg-$SiTiO_3$ semiconductor, (iii) the development of a new type of hybrid yarn and associated process technologies for production of composites with thermoplastic matrix, and (iv) that the framework programme *'Hybrid Yarn for Thermoplastic Fibre Composites'* under the Danish Materials Technology and Development Program was completed with great success and an excellent international evaluation.



Project Funded Research: Materials Technology

Project type	Project name	Co-participants
Danish Agency for Trade and Industry, Centre Contract	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 • Danish Technological Institute (DTI), Denmark
MUP	Electro-Ceramic Functional Graded Materials	• Ferroperm Components, Division of AMP, Denmark • Haldor Topsøe A/S, Denmark • PBI-Development A/S, Denmark • Chemical Institute, KU, Denmark
MUP	New Alloy Systems for Corrosion Resistant Powder Materials	• Grundfos A/S, Denmark • Danfoss A/S, Denmark • Ferritslev Jernvarefabrik A/S, Denmark • Dansk Sintermetal, Denmark • APV Pasilac A/S, Denmark • Dept Corrosion and Surface Technology, DTU, Denmark
MUP	Hybrid Yarn for Thermoplastic Fibre-Composites: Materials and Process Technology	• A/S Kaj Neckelmann, Denmark • Komposit Procesteknik ApS, Denmark • LM Glasfiber A/S, Denmark
Nordtest	Fractography of Cyclically Fatigued Zirconia Ceramics	• Swedish National Testing and Research Institute, Sweden • University of Tampere, Finland
JOULE-THERMIE	New generation Wind Turbine Blade	• Bonus Energy A/S, Denmark • Kemijoki Yo, Finland • Technical Research Centre of Finland • Garrad Hassan & Partners, Ltd., UK
EUREKA	New Compaction Methods for Gas-Atomized Powders	• Norsænk-Aalykke A/S, Denmark • Impactor Technology AB, Sweden • Shockwave Metalworking Technology, The Netherlands • DTI, Denmark • Swedish Institute for Prod. Eng. Research • TNO Research Centre, The Netherlands

Powder technology materials

This programme is concerned with the development and optimization of new powder-based materials and process technologies aiming at improved application properties, as well as testing and characterization of the materials produced.

Centre for powder-based metal matrix composites

The Materials Research Department is a partner in a Danish centre for powder-based metal matrix composites (MMCs). The centre involves a number of Danish industrial enterprises and the centre activities focus on steel-based materials with additions of rather cheap types of ceramics like alumina. The applications for the MMCs are in friction materials, grinding and cutting materials, and for wear purposes.

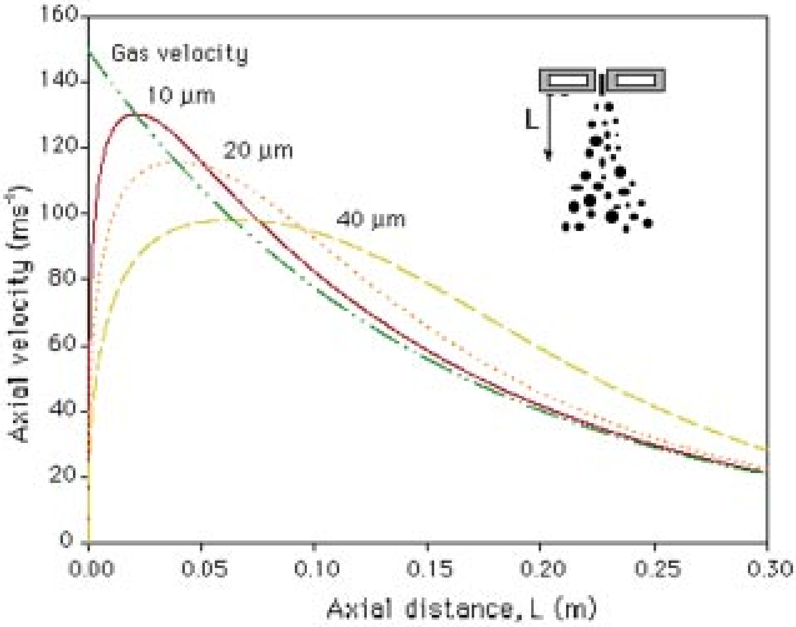
The Department is particularly involved in the centre sub-project on spray-forming of MMC materials. The former gas atomizer for production of powder has been reconstructed to allow also the production of spray-formed, purely metallic bodies, and currently the equipment for admixing ceramic particles is in preparation. Special efforts were put into the development of new metal outlet tubes and gas nozzles. For the outlet tube porous (approx. 30 % porosity) alumina and zirconia constructions were tested. The results indicate that the re-use of outlet tubes is possible 5 to 10 times.

During 1997 a series of 13 % Cr steels was produced. They showed very fine microstructures in the sprayed state, in particular regarding porosity and grain size. The latter was found to be in the range from 10 to 20 µm. The as-sprayed measured hardnesses were up to 800 Hv. The experimental activities on spray-forming are closely coordinated with the work on numerical simulation of the process.

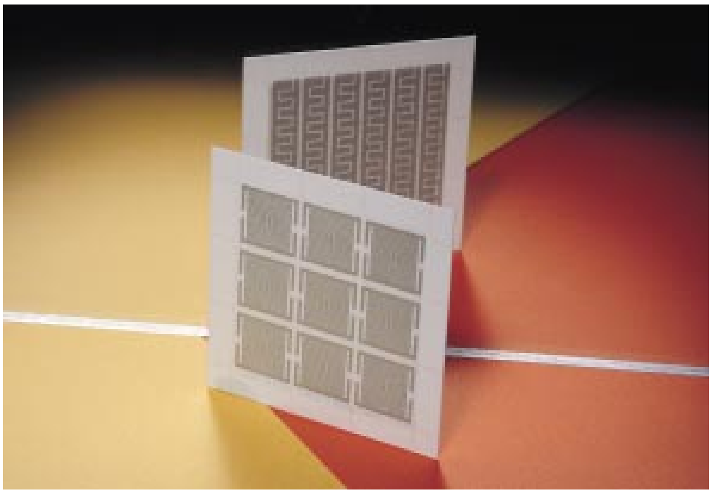
Numerical modelling of rapid solidification processes

Spray forming of metals is anticipated to achieve commercial importance for Danish industry as a new and advanced production method during the next decade. A technical and scientific understanding of the process and of the materials properties is therefore of interest.

It is the aim of this study to formulate numerical computer models for a quantitative simulation of dynamic and thermal behaviour of individual droplets during spray forming. The simulation incorporates the entire history of the droplets from the formation to deposition and cooling in the solid state. The model predicts droplet velocity and temperature as a function of flight distance, the extent of droplet solidification on arrival at the substrate, and temperature distribution in the deposited material. The effects of process parameters on droplet histories are then



Gas and droplet axial velocities are shown as functions of the axial distance travelled from the atomizer, calculated by the numerical model. Results are shown for droplets of 10, 20 and 40 µm in diameter. The initial gas velocity is 150 m/s.



Alumina support plate (50 mm x 50 mm) with screen printed Mg - doped $SrTiO_3$ semiconductor and Pt-electrodes on one side (background) and Pt-heaters on the other side (front) for production of nine integrated oxygen sensors.

used for optimization of the microstructure in droplet consolidation. The materials simulated are steel and copper based alloys.

Development of ceramic oxygen sensors

In an attempt to improve the properties of Mg-doped $SrTiO_3$ sensors, new techniques were exploited for the preparation of starting material and sensor fabrication. Fine and highly crystalline Mg-doped $SrTiO_3$ powder was obtained at a low temperature (80 °C) by the use of a sol-precipitation process. Thus, high temperature calcination, generally needed in the preparation of ceramic powders, could be avoided. A screen-printing technique was used to fabricate the sensors. Thanks to these new techniques, the sensors showed good properties, such as short response times to the oxygen partial pressure changes and good reproducibility. Furthermore, a new type of integrated oxygen sensor was also developed. This sensor has the sensor material and the electrodes on one side of an Al_2O_3 support plate and a Pt-heater on the other side. These integrated sensors, which were made by screen-printing, showed all good adhesion. Testing of the heater indicated that even the high operating temperature of 700 °C could be reached by a low power consumption of the heater. Thus, screen printing technology proved very attractive for sensor fabrication as it is possible to produce sensors with reproducible properties and a compact structure of several integrated functions.

Fabrication process for barium titanate-ferrite functionally graded ceramics

The purpose of this project is to develop a process for fabrication of electronic noise filters from barium titanate-ferrite multilayer ceramics. In order to be able to fabricate crack free filters, two main problems have to be overcome: (i) introduction of cracks during the forming process (uniaxial pressing), and (ii) delamination caused by residual stresses originating from the rather large difference in thermal expansion of the two materials. In the case of non-symmetric configurations a technique was developed for pressing crack free tubes and rectangular and disc shaped specimens. For these samples thermally-induced delamination could be avoided by using two interlayers of intermediate compositions.

To understand the problems of thermally induced delamination of graded multilayers, a fracture mechanics model was developed. The analysis showed that the steady state energy release rate G_{ss} is proportional to $\Delta\alpha^2\ \Delta T^2\ E\ H\ /(1-\upsilon)$,

Sintered barium titanate-ferrite multilayer components for fabrication of integrated electronic noise filters.

10 mm

where $\Delta\alpha$ is the difference in the thermal expansion coefficients of the base materials, ΔT is the temperature difference from stress-free state, H is the multilayer thickness, E and υ are the Young's modulus and Poisson's ratio, respectively. For non-symmetric specimens G_{ss} was found to decrease very fast as the number of layers with intermediate properties increased. Thus, the use of interlayers may be very efficient for preventing delamination of non-symmetric multilayers. In contrast, G_{ss} was higher and decreased with a much slower rate for symmetric specimens. Thus, for this configuration multilaye-ring is not efficient in preventing delamination. Instead, for symmetric multilayers the experimental work focuses on modifying the thermal expansion coefficients of the base materials in order to reduce G_{ss} by minimizing $\Delta\alpha$.

Manufacturing of low enriched uranium fuel elements

The fabrication of low enriched uranium (LEU) fuel elements was continued in 1997. For more than 20 years all fuel elements for the research reactor DR3 at Risø have been produced in the Materials Research Department. The first LEU elements were tested with success in the reactor more than ten years ago. Since then more than 300 elements of this type have been used in the reactor.

The LEU fuel elements consist of 4 concentrically arranged tubes. Each tube is made of 3 fuel plates welded together. Each fuel plate (1.5 mm in thickness) is made as a rolled sandwich of three layers: a core - containing the uranium - covered with Al sheets on both sides. The core is made powder metallurgically from a mixture of U_3Si_2 and Al powder.

Inspection and evaluation of bowlshaped test specimens (240 mm i diameter) manufactured by a press consolidation technique from hybrid yarn. In front of the picture are the male and the female press tools and next to them a stack of fabrics is ready for processing.

Manufacturing technologies for advanced composite materials

This programme covers the development and optimization of new thermoplastic fibre composites and associated process technologies, aiming at improved occupational health and application properties, as well as characterization of the produced materials.

Press consolidation of thermoplastic composites into 3D-shapes in minutes

Press consolidation of thermoplastic composites is a very fast process. It is suitable for production of smaller components in larger series. In principle, the process consists of four steps: (i) Heating the material to the process temperature, (ii) transfer of the heated material to the press, (iii) shaping, consolidating and cooling, and (iv) removal of the finished part from the press.

A new research press facility has been designed and built. The equipment consists of a press, a material transport arrangement, a PC/PLC control unit, and a process data sampling unit. The press is a single active hydraulic press with a maximum press force of 200 kN and a maximum press area of 1000 mm x 800 mm. The material is heated under vacuum to prevent/minimize the degradation of the thermoplastic matrix material prior to press forming. The process control and data sampling system regulates all process parameters: Process temperature, vacuum, speed of material transportation, speed of pressing, pressure or position of the pressing tool, and the pressing time.

Optimization of the press consolidation technique was performed on balanced woven fabric of glass/PET hybrid yarn. It was found to be a very robust process where high material quality (fibre content 45 %, less than 2 % porosity and no unwetted fibres) is obtained for a large process window. The press time was only 15 seconds.

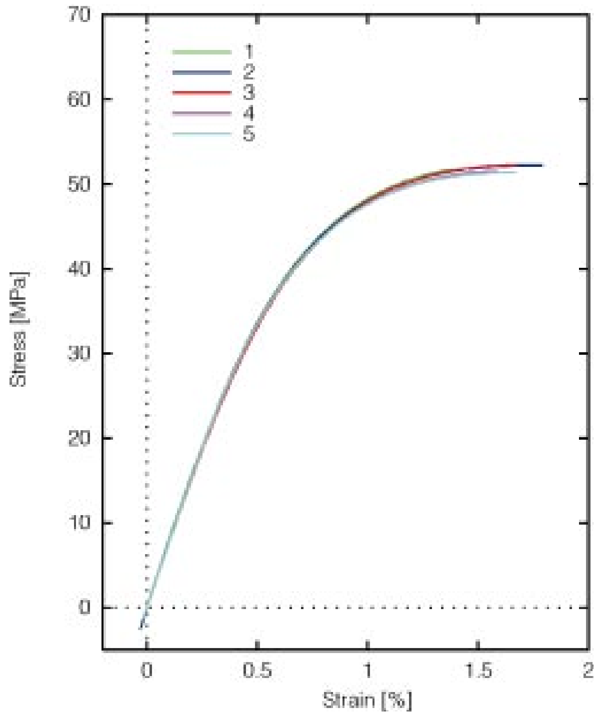
Press consolidation of a car door-post in thermoplastic composites

A fibre reinforced thermoplastic car door-post was produced from hybrid yarn fabrics by a press consolidation technique. The door-post was made of a balanced woven fabric produced from a hybrid yarn mixed of glass fibres and PET fibres. The fabrics were stacked into a laminate with the desired thickness, heated to the process temperature (290 - 300 °C for the PET), transferred to the press mounted with a cold pressing tool, and finally pressed and consolidated into shape.

Manufacturing of the door-post has with success been used to test if components with rather complex geometry could be produced on an industrial press line (Komposit Procesteknik). The final car door-posts were manufactured using a pressing force of 900 kN and a pressing time of only 12 seconds. The quality of the material of the fabricated part was examined. The material was well consolidated with a fibre content of 45 % and a porosity of about 2 %.

Light weight parabolic antenna in thermoplastic composites

The Department's autoclave and know-how has been used to manufacture a light weight prototype parabolic antenna shell from an Aramide/PEI hybrid yarn. The task was commissioned by Saab Ericsson Space AB, Sweden, and involved autoclave process optimization on flat specimens, and manufacturing of parabolic antenna shells by the optimized autoclave consolidation technique. The driving force for using PEI as matrix material is the electrical properties and the lower moisture absorption compared with Aramide/Epoxy composites.



Stress-strain curve in the transverse direction of press consolidated unidirectional glass fibre/PET laminates. (Five samples on the same plot).

Materials and mechanical properties of hybrid yarn thermoplastic composites

Two combinations of glass fibres and polymer fibres have been developed for the hybrid yarn. A 300 Tex glass fibre roving with a special developed sizing was used in both combinations, but two different types of polymer fibres, also specially developed, were used for the hybrid yarns. One type of polymer fibres was PET, and the other type was a modified PET with a lower melting and processing temperature. The processing temperature for the hybrid yarn based on PET was 280 - 300 °C. For the modified PET based hybrid yarn, the processing temperature was 220 - 240 °C.

Composite laminates consolidated form the hybrid yarn were found to have excellent room temperature tensile properties. Both filament wound unidirectional laminates and laminates consolidated from balanced woven fabrics (Twill 3/3) were evaluated and characterized. All the laminates have a porosity lower than 1 % and a fibre content of 41 % and 44 %, respectively, for the modified PET laminates and the PET laminates.

Since the cooling rate in press consolidation is high, the crystallinity in the press consolidated PET laminates is low, which again leads to high ductility of the PET matrix material. This was observed on static tensile test in the transverse direction of unidirectional laminates, where the strain to failure was as high as 1.7 %.

The high bonding strength between the fibres and the matrix material is reflected by the high shear strength of the laminates, which is 50 MPa and 60 MPa, respectively, for the modified PET and the PET matrices, measured by 12 ° off-axis tensile tests on unidirectional laminates.

	Unidirectional				Woven fabric	
	0 ° - direction		90 ° - direction		Balanced Twill 3/3	
	Mod. PET	PET	Mod. PET	PET	Mod. PET	PET
E-modulus [GPa]	32	35	8	10	20	22
Tensile strength [MPa]	580	520	45	55	280	260

Tensile properties of composite laminates consolidated from hybrid yarn

Techniques

Process development and optimization of autoclave consolidation for manufacturing of parabolic antenna shells. The task was commissioned by Saab Ericson Space AB, Sweden.

In the Materials Technology Programme a number of production facilities for advanced materials are available in addition to a series of characterization techniques. The equipment is also used by other programmes of the Department as well as in collaboration projects with external partners.

In the area of Powder Technological Materials an inert gas atomizer was established a few years ago and has been reconstructed for production of spray-formed materials. In the powder mode the equipment can handle gas pressures up to 60 bar and a number of nozzle systems has been fabricated for precise control of gas/metal ratios. Furthermore, the melting chamber can be pressurized relative to the spraying chamber. The final powder can be further processed in an inert gas glove box, without exposure to the atmosphere.

A test system is available for the evaluation of electrical properties of gas sensors. In this system the electrical response can be measured as a function of oxygen pressure, temperature and time. The sensors can also be thermally cycled.

The facilities for fabriction of PMCs include a high temperature autoclave (530 °C, 20 bar, vacuum), two filament winding machines (two axes) and a new computer controlled press. The autoclave and filament winding machines are used for manufacturing of test coupons and components with both thermosetting and thermoplastic matrix materials. The pressing facility is combined with a pre-heating, vacuum and material transport system designed for fast processing of thermoplastic composites.

	Method	Objective
Materials Shaping	Inert gas atomizing	Rapidly solidified metal powders
	Spray forming	Direct formation of compacted structures from liquid particles
	Inert gas glove box	Handling of reactive materials
	Uniaxial powder pressing	Green bodies
	Rolling	Deformation
	Heat treatments	Sintering (ceramic and metal parts)
	Filament winding	Continuous fibre composites (test specimens and components)
	Autoclave technique	Consolidation or cure of PMCs
	Press consolidation	Consolidation of PMC composites
Characterization Techniques	BET	Surface area of powders
	Thermal analysis	Thermal response (expansion/weight/heat evolution)
	Sensor testing	Electrical properties, long term stability
	Microscopy	Fibre distribution and orientation, evaluation of materials
	Matrix digestion	Fibre and porosity content in composites

FINANCES 1997

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.

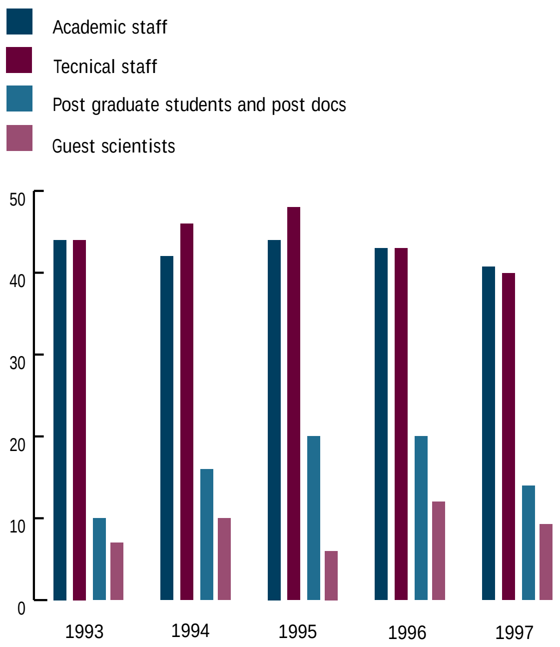
The numbers given in the tables are in units of 1000 Danish Kroner (DKK). The equivalent amount in US Dollars is also shown (DKK 1000 equal US$ 144, alternatively, 1 US$ equals 6.92 DKK).

The decrease in direct funding from 1996 to 1997 is due to the fact that payment of electricity and heating was removed from the budget of the Department.

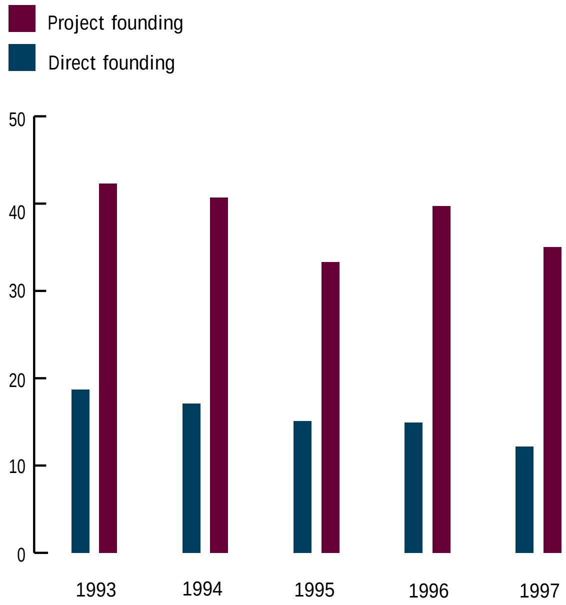
The difference between Income and Expenditure for 1997 turned out to be 951,000 DKK (138,000 US$) i.e. less than in 1996, but still satisfactory.

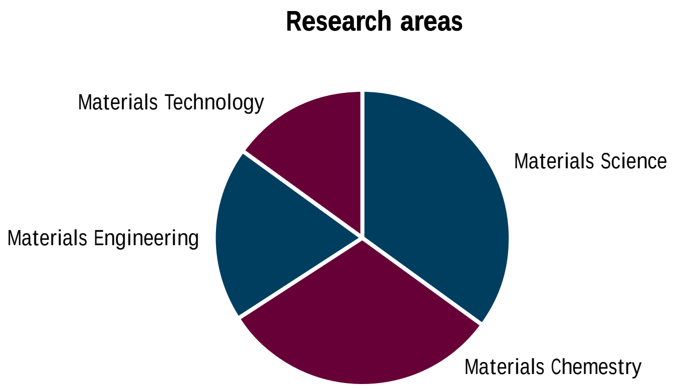
Income 1997	**DKK 1000**	**US$ 1000**
Direct funding (Ministry of Research and Technology)	12 893	1 862
Project funding	35 681	5 153
	48 574	7 015
Expenditure 1997		
Salaries	33 547	4 845
Operating expenses	11 964	1 728
Equipment	2 112	305
Total	47 623	6 877

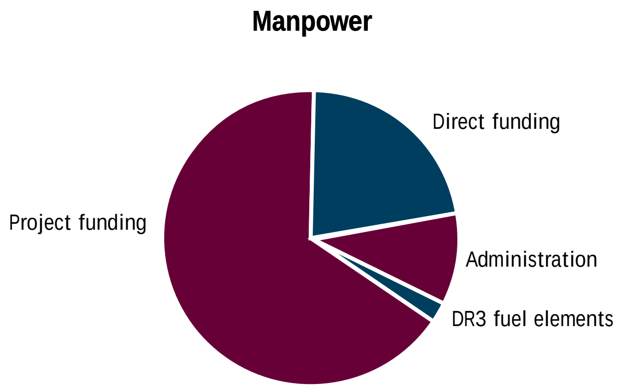
Personelle

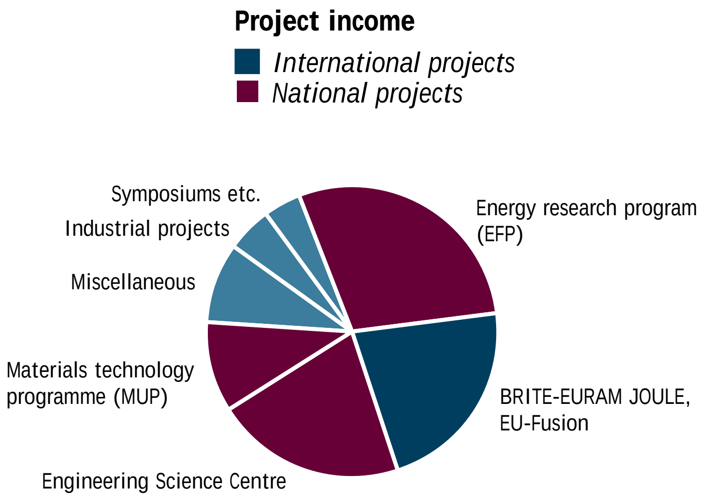


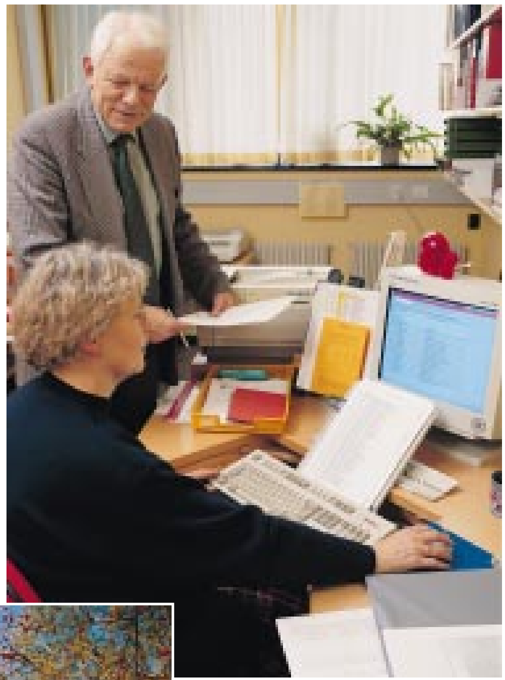
Income 1993 - 97 (DKK millions, 1997 kroner)




Research areas
Materials Technology
Materials Science
Materials Engineering
Materials Chemestry


Manpower
Direct funding
Project funding
Administration
DR3 fuel elements


Project income
International projects
National projects
Symposiums etc.
Industrial projects
Miscellaneous
Materials technology programme (MUP)
Engineering Science Centre
Energy research program (EFP)
BRITE-EURAM JOULE, EU-Fusion



PERSONNEL 1997

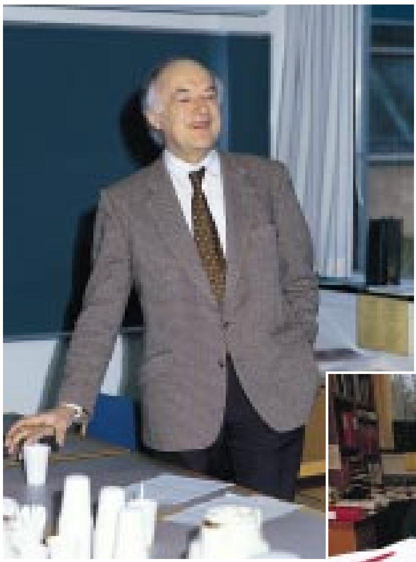
In 1997 18 members of the Department left and 15 new members joined (*) the Department

Head of Department

Niels Hansen

Scientific staff

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



Postgraduate students

Carstensen, Jesper Vejlø
Clausen, Bjørn *until 31 May*
Hansen, Jesper Rømer*
Larsen, Dorte
Mullit, Paw M.
Nielsen, Steen Arnfred
Nielsen, Søren Fæster*
Petersen, Kenneth*
Pryds, Nini H. *until 1 Mar.*
Rasmussen, Torben
Sörby, Lennart
Thorsen, Peter A. *until 15 Aug.*

Post docs

Ahlgren, Erik *until 31 Jul.*
Armstrong, William D. *until 24 Jun.*
Christoffersen, Henrik *until 31 May*
Dam, Niels Ebbe*
Garbe, Stephan *until 1 Oct.*
Holtappels, Peter W.*
Huang, Xiaoxu
Jacobsen, Torben, K.*
Krieger Lassen, Niels C.
Lyttle, Mark*
Kindermann, Lutz *until 30 Nov.*
Laffarague, Denis*
Lienert, Ulrich
Marina, Olga A.
Mishin, Oleg V.*
Pantleon, Wolfgang*
Pryds, Nini H.*
Sarroute, Sabine *until 31 Dec.*
Schiøtz, Jacob*
Thomsen, Niels, B.@ *until 31 Oct.*
Thorsen, Peter A.*

@ Industrial Post Doc, Danfoss/Risø

Consultant

Waagepetersen, Gaston

Technical staff

Adrian, Frank
Borchsenius, Jens F.S.
Dreves Nielsen, Poul
Frederiksen, Henning
Gravesen, Niels Nørregaard
Hersbøll, Bent
Jensen, Finn E. *until 31 Jul.*
Jensen, Knud
Jensen, Palle V.
Jespersen, John
Kjær, Anne-Mette Heie
Kjøller, John
Klitholm, Cliver
Larsen, Bent
Larsen, Birgit N.
Larsen, Jan
Larsen, Kjeld J. C.
Lillegaard, Keld
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
Porsdal, Helle *until 31 Jan.*
Pedersen, Niels Jørgen
Robl, Steen *until 31 Aug.*
Sandsted, Kjeld
Schmidt, Jesper*
Strauss, Torben R.
Sørensen, Erling
Aagesen, Sven

Office staff

Dreves Nielsen, Elsa
Hoffmann Nielsen, Lis
Kristiansen, Lisbeth Aa *until 10 Jun.*
Lauritsen, Grethe Wengel
Mortensen, Jytte
Sørensen, Eva M.
Thomsen, Ann
Voss, Anita

Apprentices

Christensen, Lars F.
Fabritius, Nicolai
Hammershøj Olsen, Casper
Klein, Roland
Nilsson, Jesper

Postgraduate (PhD) projects

Many PhD projects are conducted at the Materials Research Department in collaboration with universities in Denmark and other countries.

PhD Projects finished during 1997

Bjørn Clausen
'Characterization of polycrystal deformation by numerical predictions and neutron diffraction experiments'.
The Technical University of Denmark, Lyngby.
Supervisor: *Torben Lorentzen*

Torben K. Jakobsen
'Influence of holes and notches on the fatigue behaviour of 2-D woven ceramic matrix composites'.
The Technical University of Denmark, Lyngby.
Supervisor: *Povl Brøndsted*

Nini H. Pryds
'Rapid solidification of stainless steel'.
The Technical University of Denmark, Lyngby.
Supervisor: *Allan Schrøder Pedersen*

Torben Rasmussen
'Structure and dynamics of dislocations'.
The Technical University of Denmark, Lyngby.
Supervisors: *Torben Leffers, Ole Bøcker Pedersen*

Chris Pickup
'A study of the annealing and mechanical behaviour of electrodeposited Cu-Ni multilayers'.
University of Cambridge, UK.
Supervisor: *Andy Horsewell*

Peter A. Thorsen
'The influence of the grain boundary structure on diffusional creep'.
The University of Copenhagen, Denmark.
Supervisor: *Jørgen B. Bilde-Sørensen*

Ongoing PhD Projects

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.
Supervisor: *Mogens Mogensen*

Jesper Vejlø Carstensen
'Structure development and mechanisms in fatigue of polycrystalline brass'.
The influence of stacking fault energy on fatigue is studied with polycrystalline brass as a model system. Specimens of Cu with 15 % Zn and Cu with 30 % Zn with well-characterized grain size distribution and texture are subject to cyclic deformation at constant plastic strain amplitude. Dislocation microstructures and surface damage evolution are characterized by TEM, SEM and optical microscopy.
The Technical University of Denmark, Lyngby.
Supervisor: *Ole Bøcker Pedersen*

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.
Supervisor: *Mogens Mogensen, Finn W. Poulsen, Peter V. Hendriksen*

Ninette Kjerulf-Jensen
'Raman and IR-spectroscopic investigation of SOFC materials'.
Solid oxide fuel cell materials will be characterized individually and in fuel cells under operating conditions by vibrational spectroscopy and other suitable spectroscopic methods. Correlations with structure and defect chemistry will be made.
The Technical University of Denmark, Lyngby.
Supervisor: *Finn Willy Poulsen*

Darja Kek
'Electrochemical properties of interfaces between metals and solid state ion conductors'.
The interfaces between ceramic ionic conductors ($SrCeO_3$-based proton conductor and YSZ oxide ion conductor) and metals (Ni, Ag, Au, Pt) are studied at various temperatures using impedance spectroscopy. Also the electrode kinetics in hydrogen at low partial pressure are investigated.
National Institute of Chemistry/University of Ljubljana, Slovenia.
Supervisor: *Mogens Mogensen*

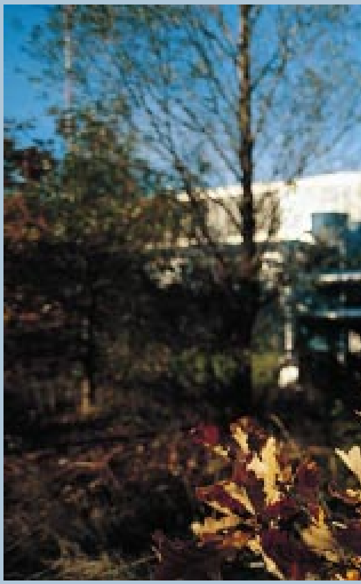
Dorthe Larsen
'Structural and electrical properties of perovskites'.
Oxides with perovskite structures such as $LaAlO_3$, $LaGaO_3$ and $LaScO_3$ doped with SrO and MgO are studied. The main purpose of the study is to find relationships between the detailed crystal structures and the conductivities of the materials.
The Technical University of Denmark, Lyngby.
Supervisors: *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.
The Technical University of Denmark, Lyngby.
Supervisor: *Andy Horsewell*

Steen Arnfred Nielsen
'Ultrasonic characterization of materials using tomographical methods'.
This project focuses on ultrasonic characterization of polymer materials using computer tomography. The main goal is the investigation of image reconstruction techniques based on fan-beam projections. Examination with ultrasonic through-transmission scanning equipment is used to verify the applicability of fan-beams for non-destructive applications, in particular the study of defects.
The Technical University of Denmark, Lyngby.
Supervisors: *Hans Erik Gundtoft, Svend Ib Andersen, Jørgen Rheinländer*

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 sample is compressed.
University of Copenhagen, Denmark.
Supervisor: *Torben Leffers*

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.
Supervisor: *Allan Schrøder Pedersen*



Lennart Sörby
'Structural study of SOFC materials'.
Diffraction techniques based on X-ray, neutron and synchrotron radiation are used to study solid oxide fuel cell materials, both under equilibrium and dynamic conditions. Structure refinement is done by the Rietveld method.
University of Uppsala, Sweden.
Supervisor: *Finn Willy Poulsen*

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: *Mogens Mogensen, Finn Willy Poulsen*

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: *Mogens Mogensen*

Undergraduate projects

Jesper Rømer Hansen
'Conducting perovskites: Investigation of $La_{0.8}Sr_{0.2}Cr_{0.97}V_{0.03}O_{3-\delta}$ structure, conductivity and oxygen diffusion'. The Technical University of Denmark, Lyngby.
Supervisor: *Mogens Mogensen, Finn W. Poulsen, Peter V. Hendriksen*

Michael Nielsen
'Classification of a filmless radiographic system'.
The Engineering College of Copenhagen, Ballerup, Denmark.
Supervisor: *Jørgen Rheinländer*

Jacob Olsson
'Measurement of vein thickness in digitized radiographs of rabbits (drug testing)'.
The Engineering College of Copenhagen, Ballerup, Denmark.
Supervisor: *Jørgen Rheinländer*

Andrew J. Perry
'Investigation into semiconducting oxygen sensors based on Mg doped strontium titanate'.
University of Bath, UK.
Supervisor: *Ole Toft Sørensen*

David Coimbra
'Nernst-type oxygen sensors: synthesis and characterization of samaria/praseodymia doped ceria and testing of yttria-stabilized zirconia sensors'.
Ecole Nationale Supérieure de Céramique Industrielle, Limoges, France.
Supervisor: *Ole Toft Sørensen*

External lecturers and examiners

Many of the staff members of the Materials Research Department are actively involved in education and training in materials science as university external lecturers and examiners.

External lecture courses

Charlotte C. Appel
Jørgen B. Bilde-Sørensen
Andy Horsewell
'Electron microscopy and microanalysis'. Course no. 10455. The Technical University of Denmark, Lyngby.

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

Mogens Mogensen
'Oxygen ion and mixed conductors and their technological applications'. 33rd Course. NATO Advanced Study Institute: International School of Materials Science and Technology. Erice, Sicily, Italy.

Svend Ib Andersen
Jørgen B. Bilde-Sørensen
Torben Lorentzen
Allan Schrøder Pedersen
Bent F. Sørensen
Ole Toft Sørensen
'Introduction to new materials'. The University of Aarhus, Denmark. *(Distance learning)*

External Examiners

Svend Ib Andersen
Jørgen B. Bilde-Sørensen
Christian P. Debel
Morten Eldrup
Andy Horsewell
Hans Lilholt
Finn Willy Poulsen
Ole Toft Sørensen
Members of the officially appointed corps of Danish university examiners (MSc and BSc).

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

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

Christian C. Debel
BSc examiner. Engineering College of Copenhagen, Ballerup, Denmark.

Peter V. Hendriksen
PhD examiner. Norwegian University of Technology and Science, Trondheim, Norway.

Hans Lilholt
PhD examiner. University of Aalborg, Denmark.

Allan Schrøder Pedersen
PhD examiner. University of Copenhagen, Denmark

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

Staff members on assignment abroad

Charlotte C. Appel
Statoil R&D Centre, Trondheim, Norway. 5 - 12 January.

Nikolaos Bonanos
National Technical University of Athens, Greece. 7 - 25 April.

Niels-Ebbe Dam
Carnegie Mellon University, Pittsburgh, PA, USA, 20 October - 1 December.

Mette Juhl Jørgensen
Birchall Centre for Inorganic Chemistry and Materials Science, Keele University, Staffordshire, UK. 13 - 22 February.

Torben Leffers
National University of Rosario, Argentina. 3 - 15 November.

Jørgen Lindbo
Sandia National Laboratories, Livermore, CA, USA. 1 - 22 March.

Palle Nielsen
School of Mines, St. Etienne, France. 9 - 19 September.

Bachu N. Singh
AEA Technology. Harwell, UK. 21 - 25 April.

Ole Toft Sørensen
University of Science and Technology of China, Hefei, China. 29 September - 12 October.
Xi´dian University, Xian, China. 13 - 20 October.

Roadshow

The Engineering Science Centre has applied the concept of 'Roadshow' to materials research: a number of staff members from the Department turn up at an industrial company and present lectures about the activities within the centre. The lectures are selected by the company from a long list (at present comprising 32 titles). Several shows have been organized in 1997. The two largest were at Danfoss A/S (70 industrial participants, 8 from the Department) and at Grundfos A/S (about 30 industrial participants, 4 from the Department). Roadshows have also been held at Dronningborg Industries A/S, Elektro-Isola A/S, Adtranz Danmark A/S, and TIC Roskilde Amt/ Ingemann Maskinfabrik by one lecturer from the Department and about 10 industrial participants each time.

Visiting scientists at the Materials Research Department

Many guests visited or worked in the Department during 1997.

Dr. Nina Aas
Statoil R&D Centre, Trondheim, Norway. 3 - 6 November.

Prof. Brent Adams
Carnegie Mellon University, Pittsburgh, PA, USA. 16 June - 10 July.

Dr. V. S. Ananthan
17 May - 17 October.

Dr. Claire Y. Barlow
University of Cambridge, UK. 22 August - 5 September.

Dr. Alberto Borrego
CENIM, Madrid, Spain. 7 August - 12 October.

Michael S. Brown
University of Waikato, Hamilton, New Zealand. 9 June - 9 December.

Gabriele Brückner
RWTH Aachen, Germany. 11 - 24 August.

David Coimbra
Ecole Nationale Supérieure de Céramique Industrielle, Limoges, France. 3 March - 31 December.

Prof. Julian Driver
School of Mines, St. Etienne, France. 7 - 20 September.

Dr. Dan J. Edwards
Pacific Northwest National Laboratory, Richland, WA, USA. 15 November - 14 December.

Chen Fanglin
University of Science and Technology of China, Hefei, China. 15 January - 15 April.

Dr. Stanislav I. Golubov
Institute of Physics and Power Engineering, Obninsk, Russia. 1-16 April, 1 August - 30 September.

Dr. Tetsuya Hirade
Chalmers University of Technology, Gothenburg, Sweden. 10-12 April, 15-26 September, 9 - 22 November.

Michael Hanke
Technical University, Braunschweig, Germany. 7 - 14 September.

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

Malte Hollmann
Technical University, Dresden, Germany. 22 - 27 November.

Prof. John W. Holmes
University of Michigan, Ann Arbor, MI, USA. 2 - 6 April.

Dr. Darcy Hughes
Sandia National Laboratory, Livermore, CA, USA. 28 July - 24 August.

Byoung-Sam Kang
Korea Electric Power Research Institute, Taejan, Korea. 3 - 28 November.

Darja Kek
National Institute of Chemistry, Ljubljana, Slovenia. 1 February - 31 July, 15 September - 19 December.

Jens Keuerleber
Freiberg University of Mining and Technology, Germany. 2 - 10 December.

Dr. Volker Klemm
Freiberg University of Mining and Technology, Germany. 2 - 6 December.

Anne Köntges
RWTH Aachen, Germany. 11 - 24 August.

Prof. Amar N. Kumar
Indian Institute of Technology, New Delhi, India. 1 - 31 January.

Carrie B. Manon
Alfred University, New York, NY, USA. 1 - 31 January.

Rozlyn Phillips
University of Waikato, Hamilton, New Zealand. 1 May - 28 August.

Robin Preston
Cambridge University, UK. 19 - 27 May.

Prof. Brian Ralph
Brunel University, London, UK. 26 - 31 July.

Jaydeep Sarkar
Cambridge University, UK. 19 - 27 May.

Prof. Ramesh Talreja
Georgia Institute of Technology, Atlanta, GA, USA. 4 - 15 August.

Prof. John Wert
University of Virginia, Charlottesville, VA, USA. 5 June - 16 July.

Prof. Kjell Wiik
The Norwegian University of Science and Technology, Trondheim, Norway. 4 August - 17 December.

Dr. Erik Woldt
Technical University, Braunschweig, Germany. 1 - 26 September.

Hong Zheng
University of Science and Technology of China, Hefei, China. 1 January - 31 December.

Prof. Francis W. Zok
University of California, Berkeley, USA. 30 April - 4 May.

Rolf Jarle Åberg
Norwegian University of Technology and Science, Trondheim, Norway. 8 - 20 September.

Colloquia

Dr. Hiroshi Takagi
Murata Company, Kyoto, Japan.
'Status of the SOFC programme at Murata Co.'
23 January.

Prof. John W. Holmes
The University of Michigan, Ann Arbor, MI, USA.
'Fatigue behaviour of ceramic matrix composites'.
4 April.

Prof. Erik Johnson
University of Copenhagen, Denmark.
'TEM studies of nanosized inclusions in aluminium'.
6 May.

Prof. Brent L. Adams
Carnegie Mellon University, Pittsburgh, PA, USA.
'A mesoscale study of the interaction of grain boundaries with the deformation field'.
8 July.

Prof. Meng Guangyao
University of Science and Technology of China, Hefei, Anhui, China.
'Applications and preparation of ceramic membranes'.
11 July.

Prof. Ramesh Talreja
Georgia Institute of Technology, Atlanta, GA, USA.
'Recent studies in damage mechanics of composite materials'.
14 August.

Dr. Laurence Mott
Bio Composites Centre, Bangor, UK.
'Testing of plant fibres and microtomography of fibres and composites'.
21 October.

Malte Hollmann
Technical University, Dresden, Germany.
'Cyclic plasticity of nickel single crystals characterized at different temperatures and different length scales'.
25 November.

Prof. Amiya K. Mukherjee
University of California, Berkeley, CA, USA.
'New results on nanometals, nanointermetallics and nanoceramics: Processing and some mechanical properties'.
8 December.

Distance learning using video conferencing

As described in other sections, the staff of the Department has become increasingly involved in educational activities, for example by teaching undergraduate and PhD-students both through lecture courses and project work. To further increase the possibilities for interaction with the university world and its students and to promote the communication with industrial partners, a video conferencing system was installed at the end of 1996 in a dedicated room in the Department. The facility was established in collaboration with all the major universities in Denmark who also acquired similar facilities. The aim is that lecture courses given at one institution can be followed by students at other universities by two- or multi-way video links, i.e. using the so-called distance learning principle. Similarly, meetings can be arranged between participants at different locations, so-called video conferences. In all cases travel money and time are saved. After the official opening in May by the Minister of Education of the eight collaborating Video Conferencing Centres a distance learning course, 'Introduction to New Materials', from Risø to Aarhus University has been carried out, as well as a number of meetings with Danish and foreign participants to discuss research collaboration and planning of education.

Participation in committees

Danish committees

Eivind Adolph
Technical Assessor, DANAK. Copenhagen.

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.

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

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

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

Technical Assessor, DANAK. Copenhagen.

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

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

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

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

Board of the Danish Research Councils. (*Chairman)*.

Peter Halvor Larsen
Danish Ceramic Society. *(Chairman from 19 November)*.

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

The Chemistry and Materials Commission of the Danish Technical Research Council. (*Until June 30)*.

The Coordination Committee for the Danish Materials Technology Development Programme. Copenhagen. *(Chairman)*.

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

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

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.

Contributor to 'The Great Danish Encyclopedia' (in Danish 'Den Store Danske Encyklopædi'). Copenhagen.

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

Ole Bøcker Pedersen
Contributor to 'The Great Danish Encyclopedia' (in Danish 'Den Store Danske Encyklopædi'). Copenhagen.

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

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

Ole Toft Sørensen
Danish Ceramic Society. *(Chairman, until 19 November)*.

International committees

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

Editorial Board of 'Journal of Strain Analysis'.

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

Member of the board of the Scandinavian Society for Electron Microscopy.

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

Povl Brøndsted
Editorial Board of 'Advanced Composites Letters'.

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

International Programme Committee for the 11th International Conference on Positron Annihilation. May 1997. Kansas City, MO, USA.

Advisory Board of 'Materials Science Forum'.

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

The COST Technical Committee on Materials. Brussels, Belgium.

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

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

Editorial Board of 'Revue de Metallurgie'.

Editorial Board of 'Monographs in Materials Science'.

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

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

International Committee for the International Conference on The Quantitative Description of Materials Microstruture (Q-MAT). April 1997. Warsaw, Poland.

Peter Halvor Larsen
Nordic Society for Thermal Analysis and Calorimetry.

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

Torben Leffers
Editorial board of 'Textures and Microstructures'.

International Advisory Committee for the International Conference on Texture and Anisotropy of Polycrystals. September 1997. Clausthal, Germany.

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

Symposia and workshops

18th Risø Symposium

The 18th Risø International Symposium on Materials Science for 1997 was entitled: 'Polymeric Composites - Expanding the Limits', and took place in the great lecture hall at Risø on 1 - 5 September 1997. The Symposium had 83 participants from industry, research institutes and universities, representing 15 countries, including a strong representation from Danish industry; special funding support from the Nordic Academy for Advanced Study (NorFa) enabled 7 researchers from the Baltic states to participate.

Composite materials are becoming an increasingly important part of the spectrum of materials available for the design and manufacturing of products and components. This situation is of special importance in the energy, transport and environmental industries. In this field of materials engineering the polymeric composites are the most developed and most successfully used materials within the composites group.

There is continuing scope for expanding the limits, both in terms of materials constituents and properties and in terms of component design and manufacturing. Materials science forms the basis for designing of components, ranging from small functional devices, to large load bearing structures.

The theme of the symposium was covered by the 15 invited review papers and 43 contributed papers addressing materials, properties and applications within polymeric composites. The approach was that of considering the topics in context, with focus on new aspects which were likely to expand the limit for the engineering applications of polymeric composites. The subjects concerned further developments in established fields, such as cracks and fatigue as well as design and non-destructive evaluation. The subjects also addressed new developments, such as polymeric fibres and plant fibre composites, as well as elastically isotropic composites and shape active composites. Furthermore, manufacturing and interfacial aspects were presented, and emphasized the need to produce high quality composites and related components so that the industry can use them with confidence.

The symposium was organized by the Materials Research Department, in collaboration with the Engineering Science Centre for Structural Characterization and Modelling of Materials and with the Danish Materials Technology Development Programme (MUP-2-framework programme). Composite materials have been the theme of previous Risø Symposia in 1982, 1988 and 1991, where polymeric, metallic and ceramic composites were covered.

Organizers: ***Svend Ib Andersen, Povl Brøndsted, Niels Hansen, Ole Jørgensen, Grethe W. Lauritsen, Hans Lilholt, Aage Lystrup, Bent F. Sørensen, Helmuth Toftegaard***

Nordic Ceramics' 97

The annual Nordic Ceramics meeting was arranged together with the ceramic societies in Denmark, Norway and Sweden at the Risø National Laboratory during 26 - 27 May 1997. This symposium attracted 90 participants especially from the Nordic Countries. 10 invited lectures and 26 contributions were presented at this symposium.

Organizers: ***Torben R. Strauss, Bent F. Sørensen, Ole Toft Sørensen, Anita Voss***

Teaching of high school students - an investment in the future

In recent years Danish students have shown less interest in education within the scientific fields. This is a disturbing development in our modern society which depends on a work force possessing a high standard of scientific and technical skills. Thus, there is a great need to stimulate the interest of young people in scientific subjects. As a part of Risø's efforts in this respect, the Department is running a one day course for high school students. The course covers theory and practice within the field of materials science, specifically polymer chemistry and polymer based fibre composites. This provides the students with some knowledge about the production and the properties of materials along with the experience that science can be exciting. The course has been developed as a joint venture between the Materials Research Department, the Condensed Matter Physics and Chemistry Department and teachers at Himmelev Gymnasium. As background material, the book 'On polymeric materials' (in Danish) by J.S. Petersen and L. Schulz, Himmelev Gymnasium, has been written and published. In 1997, three classes (47 students) have participated in the course and the feedback from students and teachers has been very positive.

The Department also supervises individual projects for a limited number of high school students. One such project was carried out in 1997:

Jakob Hessel Andersen
'Environmental scanning electron microscopy'.
Roskilde High School (in Danish 'Amtsgymnasiet'), Roskilde, Denmark.
Supervisor: *Jørgen B. Bilde-Sørensen*

Scientific Programme Committee of the 10th International Conference on Mechanics of Composite Materials (MCM). April 1998. Riga, Latvia.

International Advisory Committee for the International Meeting on Composite Materials: 'Advancing with Composites '97'. May 1997. Milan, Italy.

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 4th International Conference on Deformation and Fracture of Composites. March 1997. Manchester, UK.

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.

International Committee for Composite Materials. Philadelphia, USA.

European Association for Composite Materials, Standing Committee.

Editorial Board of 'Advanced Composite Materials'.

Editorial Board of 'Composite Science and Technology'.

Editorial Board of 'Polymers and Polymer Composites'.

Editorial Board of 'Applied Composite Materials'.

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

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

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

Editorial board of 'Journal of Neutron Research'.

Mogens Mogensen
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).

The Organizing Committee of the ‘Third International Symposium on Ionic and Mixed Conducting Ceramics’, part of ‘Joint International Meeting - ECS and ISE’. September 1997. Paris, France.

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

The Nomination Commitee for the Outstanding Achievement Award of the High Temperature Materials Division of the Electrochemical Society. Pennington, NJ, USA.

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

Ole Bøcker Pedersen
Scientific Committee for the International Conference on Fatigue of Composites. June 1997. Paris, France.

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

The Fuel Cell Committee under the Nordic Energy Research Programme. Ås, Norway.

Organizing Committee for the Third European Solid Oxide Fuel Cell Forum. June 1998. Nantes, France.

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

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

International Organizing Committee for the 8th International Conference on Fusion Reactor Materials. October 1997. Sendai, Japan.

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

Organizing Committee for the International Workshop on Basic Aspects of Differences in Irradiation Effects Between FCC, BCC and HCP Metals and Alloys. October 1998. Santillana del Mar, Spain.

Bent F. Sørensen
Editorial Board of ‘Key Engineering Materials’.

Ole Toft Sørensen
Nordic Society for Thermal Analysis and Calorimetry.

ICTAC Standardization Committee.

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

Editorial Board of ‘Journal of Thermal Analysis’.

Committee for the election of Professor in inorganic chemistry at Norwegian Technical-Natural Science University, Trondheim, Norway.

Social activities

The Materials Research Department’s social committee, MAK, keeps track of special birthdays, anniversaries, weddings etc. MAK is also responsible for the traditional Christmas party and a summer picnic. In 1997 the summer picnic consisted of a fondue party in Copenhagen and included a visit to the Tycho Brahe Planetarium. The Christmas party was held at Risø.

Members of the Department are very active in organization of athletic activities throughout Risø. The badminton club, for example, plays two hours every Monday evening in a nearby hall and holds two annual tournaments in a relaxed atmosphere. The winners are given small prizes accompanied by teasing praise at a celbratory dinner. Jogging is another favourite sport of the Department. A team of relay runners from the Department was the second best team from Risø in a major fitness arrangement in Copenhagen and members from the Department also did well in the prestigious annual ‘Risø competition’.

The Department is also strongly represented in the less physical activities, e.g. the Risø art society which sponsors regular exhibitions in the foyer of the Risø canteen and lends pictures to members’ offices and to departments. Department staff are also active in ‘Culture at work’, a Risø organization which arranges visits to the theatre, museums and other cultural events.

These departmental social activities promote interactions between the regular staff and the increasing numbers of new graduate students, post docs and visiting scientists, many of them from abroad.

PUBLISHED WORK

International publications

1. *Ahlgren, E.O.*, Thermoelectric power of $SrCe_{0.95}Y_{0.5}O_{3-\delta}$. Solid State Ionics (1997) v. 97 p. 489-495

2. *Ahlgren, E.O.*, Thermoelectric power of Gd-doped barium cerate. J. Phys. Chem. Solids (1997) v. 58 p. 1475-1480

3. Almazouzi, A.; Diaz de la Rubia, T.; Ishino, S.; Lam, N.Q.; *Singh, B.N.*; Trinkaus, H.; Victoria, M.; Zinkle, S., Defect production, accumulation and materials performance in an irradiation environment. Summary. J. Nucl. Mater. (1997) v. 251 p. 291-294

4. Almazouzi, A.; Victoria, M.; *Singh, B.N.*; Diaz de la Rubia, T. (eds.), Defect production, accumulation and materials performance in an irradiation environment. Proceedings. International workshop on defect production, accumulation and materials performance in an irradiation environment, Davos (CH), 2-8 Oct 1996. (Elsevier, Amsterdam, 1997) (J. Nucl. Mater., vol. 251) 300 p.

5. *Armstrong, W.D.*, Magnetization and magnetostriction processes in $Tb_{0.27-0.30}Dy_{0.73-0.70}Fe_{1.9-2.0}$. J. Appl. Phys. (1997) v. 81 p. 2321-2326

6. *Armstrong, W.D.*, Burst magnetostriction in $Tb_{0.3}Dy_{0.7}Fe_{1.9}$. J. Appl. Phys. (1997) v. 81 p. 3548-3554

7. *Armstrong, W.D.*, The magnetization and magnetostriction of $Tb_{0.3}Dy_{0.7}Fe_{1.9}$ fiber actuated epoxy matrix composites. Mater. Sci. Eng. B (1997) v. 47 p. 47-53

8. *Armstrong, W.D.*; *Lorentzen, T.*, Fiber phase transformation and matrix plastic flow in a room temperature tensile strained NiTi shape memory alloy fibre reinforced 6082 aluminium matrix composite. Scr. Mater. (1997) v. 36 p. 1037-1043

9. Bak, J.; *Kindl, B.*, Quantitative analysis of mineral powders by DRIFTS: Determination of $SrCO_3$ in superconductor precursor powders. Appl. Spectrosc. (1997) v. 51 p. 1730-1735

10. Berbon, M.Z.; *Toft Sørensen, O.*; Langdon, T.G., Creep behavior of superplastic 2.5% yttria-stabilized zirconia. In: Creep and fracture of engineering materials and structures. Earthman, J.C.; Mohamed, F.A. (eds.), (Minerals, Metals and Materials Society, Warrendale, PA, 1997) p. 193-202

11. Berg, R.W.; Nielsen, K.; *Poulsen, F.W.*, Redetermination of the crystal structure of Al_2Br_6. A comparison of three methods. Acta Chem. Scand. (1997) v. 51 p. 442-448

12. *Bergenstof Nielsen, C.*; *Horsewell, A.*; Østergård, M.J.L., On texture formation of nickel electrodeposits. J. Appl. Electrochem. (1997) v. 27 p. 839-845

13. *Bonanos, N.*; *Mogensen, M.*, H_2 oxidation at the interface $Ni/Sr_{0.995}Ce_{0.95}Y_{0.05}O_{2.975}$. Solid State Ionics (1997) v. 97 p. 483-488

14. Cai, S.; Millar, C.E.; Pedersen, L.; *Toft Sørensen, O.*; Xu, Y., Extrusion and properties of lead zirconate titanate piezoelectric ceramics. Ferroelectrics (1997) v. 196 p. 389-392

15. *Carstensen, J.V.*; *Pedersen, O.B.*, Texture and grain-size effects on cyclic plasticity in copper and copper-zinc. Mater. Sci. Eng. A (1997) v. 234/236 p. 497-500

16. *Chen F.L.*; *Toft Sørensen, O.*; Meng, G.Y.; Peng, D.K., Chemical stability study of $BaCe_{0.9}Nd_{0.1}O_{3-\alpha}$ high-temperature proton-conducting ceramic. J. Mater. Chem. (1997) v. 7 p. 481-485

17. *Chen, F.L.*; Wang, Ping; *Toft Sørensen, O.*; Meng, G.Y.; Peng, D.K., Preparation of Nd-doped $BaCeO_3$ proton-conducting ceramics by homogeneous oxalate coprecipitation. J. Mater. Chem. (1997) v. 7 p. 1533-1539

18. *Chen, F.L.*; *Toft Sørensen, O.*; Meng, G.Y.; Peng, D.K., Synthesis of Nd-doped barium cerate proton conductor from oxalate coprecipitate precursor. J. Thermal Anal. (1997) v. 49 p. 1255-1261

19. *Chen, F.L.*; *Toft Sørensen, O.*; Meng, G.Y.; Peng, D.K., Preparation of Nd-doped barium cerate through different routes. Solid State Ionics (1997) v. 100 p. 63-72

20. *Christensen, J.*; *Gotthjælp, K.*; Kjeldsteen, P., The effect of surface treatments on the brazing of iron-based powder metal compacts. Weld. J. (1997) v. 76 p. S239-S244

21. *Christoffersen, H.*; *Leffers, T.*, Microstructure and composite deformation pattern for rolled brass. Scr. Mater. (1997) v. 37 p. 1429-1434

22. *Christoffersen, H.*; *Leffers, T.*, The orientation of dislocation walls in rolled copper relative to the crystallographic co-ordinate system. Scr. Mater. (1997) v. 37 p. 2041-2046

23. *Clausen, B.*; *Lorentzen, T.*, Experimental evaluation of a polycrystal deformation modeling scheme using neutron diffraction measurements. Metall. Mater. Trans. A (1997) v. 28 p. 2537-2541

24. Daveau, S.; Brandon, N.P.; *Bonanos, N.*, Electrical conduction phenomena in coked industrial reforming catalysts. Mater. Res. Bull. (1997) v. 32 p. 205-212

25. Daymond, M.R.; Bourke, M.A.M.; von Dreele, R.; *Clausen, B.*; *Lorentzen, T.*, Use of Rietveld refinement for elastic macrostrain determination and for the evaluation of plastic strain history from diffraction spectra. J. Appl. Phys. (1997) v. 82 p. 1554-1562

26. Driver, J.H.; *Winther, G.*, Microtextures et hétérogénéités de déformation. Rev. Metall. (Paris) (1997) p. 1021-1028

27. Edwards, D.J.; *Singh, B.N.*; *Toft, P.*; *Eldrup, M.*, Recent results on the neutron irradiation of ITER candidate copper alloys irradiated in DR-3 at 250 °C to 0.3 dpa. In: Fusion materials semi-annual progress report for the period ending December 31, 1996. (1997) p. 183-193

28. *Eldrup, M.*; Sanders, P.G.; Weertman, J.R., Positron annihilation study of the influence of grain size and purity on the annealing behaviour of nano-crystalline copper. Mater. Sci. Forum (1997) v. 255/257 p. 436-438

29. *Eldrup, M.*; *Singh, B.N.*, Studies of defects and defect agglomerates by positron annihilation spectroscopy. J. Nucl. Mater. (1997) v. 251 p. 132-138

30. Frello, T.; Andersen, N.H.; Madsen, J.; Käll, M.; Zimmermann, M. von; Schmidt, O.; *Poulsen, H.F.*; Schneider, J.R.; Wolf, T., Dynamics of oxygen ordering in $YBa_2Cu_3O_{6+x}$ studied by neutron and high-energy synchrotron X-ray diffraction. Physica C (1997) v. 282/287 p. 1089-1090

31. Gillespie, J.W.; *Hansen, U.*, Transverse cracking of composite laminates with interleaves: A variational approach. J. Reinf. Plast. Compos. (1997) v. 16 p. 1066-1092

32. *Hansen, N.*; *Huang, X.*, Dislocation structures and flow stress. Mater. Sci. Eng. A (1997) v. 234/236 p. 602-605

33. Hattel, J.H.; *Jacobsen, T.K.*; Hansen, P.N., Real-time simulation of thermal stresses and creep in plates subjected to transient heat input. IMA J. Math. Appl. Bus. Ind. (1997) v. 8 p. 123-141

34. Heinisch, H.L.; *Singh, B.N.*, Stochastic annealing simulation of intracascade defect interactions. J. Nucl. Mater. (1997) v. 251 p. 77-85

35. Huang, X.; *Hansen, N.*, Grain orientation dependence of microstructure in aluminium deformed in tension. Scr. Mater. (1997) v. 37 p. 1-7

36. Hughes, D.A.; *Hansen, N.*, High angle boundaries formed by grain subdivision mechanisms. Acta Mater. (1997) v. 45 p. 3871-3886

37. Hughes, D.A.; *Liu, Q.*; Chrzan, D.C.; *Hansen, N.*, Scaling of microstructural parameters: Misorientations of deformation induced boundaries. Acta Mater. (1997) v. 45 p. 105-112

38. Jacobsen, T.K.; *Brøndsted, P.*, Strain redistribution around holes and notches in fiber-reinforced cross-woven brittle matrix composites. Key Eng. Mater. (1997) v. 127/131 p. 1083-1092

39. Jacobsen, T.K.; *Sørensen, B.F.*; *Brøndsted, P.*, Fatigue and frictional heating in ceramic matrix composites. Key Eng. Mater. (1997) v. 127/131 p. 745-752

40. Jean, Y.C.; *Eldrup, M.*; Schrader, D.M.; West, R.N. (eds.), Positron annihilation. IPCA-11. 11. international conference on positron annihilation, Kansas City, MO (US), May 1997. (Trans Tech Publications, Zürich, 1997) (Mater. Sci. Forum, 255/257) 852 p.

41. Jiang, J.Z.; Lu, S.W.; Zhou, Y.X.; Mørup, S.; Nielsen, K.; *Poulsen, F.W.*; Berry, F.J.; McManus, J., Correlation of gas sensitive properties with Fe_2O_3-SnO_2 ceramic microstructure prepared by high energy ball milling. Mater. Sci. Forum (1997) v. 235/238 p. 941-946

42. Jiang, J.-Z.; Lin, R., Mørup, S.; Nielsen, K.; *Poulsen, F.W.*; Berry, F.J.; Clasen, R., Mechanical alloying of an immiscible α-Fe_2O_3-SnO_2 ceramic. Phys. Rev. B (1997) v. 55 p. 11-14

43. Juul Jensen, D., Simulation of recrystallization microstructures and textures: Effects of preferential growth. Metall. Mater. Trans. A (1997) v. 28 p. 15-25

44. Juul Jensen, D., Effects of orientation correlations on misorientation distributions in cold-deformed aluminium. Mater. Sci. Eng. A (1997) v. 234/236 p. 762-765

45. Juul Jensen, D., Applications of orientation mapping by scanning and transmission electron microscopy. Ultramicroscopy (1997) v. 67 p. 24-34

46. Jørgensen, O.; *Horsewell, A.*, On the identification failure of carbon-epoxy cross-ply laminates, and its suppression by elasto-plastic interleaves. Acta Mater. (1997) v. 45 p. 3431-3444

47. Kettunen, P.; Lepistö, T.; Tiainen, T.; *Hansen N.; Brøndsted, P.; Liu, Y.L.*; Lohne, O.; Bauger, Ö.; Pedersen, K.; Tweed, J.H.; Young, R.M.K., Aluminium alloys with a sharp fatigue limit. FI Patent 923488 (10 July 1997)

48. Kindermann, L.; Das, D.; Nickel, H.; Hilpert, K.; *Appel, C.C.*; *Poulsen, F.W.*, Chemical compatibility of $(La_{0.6}Ca_{0.4})_xFe_{0.8}Mn_{0.2}O_3$ with yttriastabilized zirconia. J. Electrochem. Soc. (1997) v. 144 p. 717-720

49. Kuzjukevics, A.; *Linderoth, S.*; Grabis, J., Characterization of yttria-doped zirconia powders produced by plasma-chemical method. Solid State Ionics (1996) v. 92 p. 253-260

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ACRONYMS, ABBREVIATIONS, ETC.

AAU Aalborg University

AU Aarhus University

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

BiSCCO $Bi_2Sr_2Ca_2Cu_3O_{10}$. A material with superconducting properties

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

CMC Ceramic matrix composites

COM Crystal orientation microscopy

CVD Chemical vapour deposition

dpa Displacements per atom

DR3 Danish Reactor 3. A nuclear research reactor at Risø

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

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 Joint Opportunities for Unconventional or Long-term Energy Supply. An EU programme on non-nuclear energy and rational exploitation of energy

KU University of Copenhagen

LEU Low enriched uranium. Fuel for nuclear test reactors

LIP Large Installation Programme

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

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

MMC Metal matrix composites

MUP The Danish Materials Technology Programme

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

ODF Orientation distribution function

OU University of Odense

PAS Positron annihilation spectroscopy

PEI Polyetherimide

PET Polyethyleneterephtalate

PLC Programable logical controller

PMC Polymer matrix composites

SEM Scanning electron microscope

SFT Stacking fault tetrahedron

SIA Self-interstitial atom

SOFC Solid oxide fuel cell. High tech ceramic fuel cells

TEM Transmission electron microscopy

UCT Ultrasound computed tomography

YSZ Yttria stabilized zirconia

Risø National Laboratory is a broad-based research organization with primary research activities in energy, materials and the environment. There is a total of 891 employees.

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

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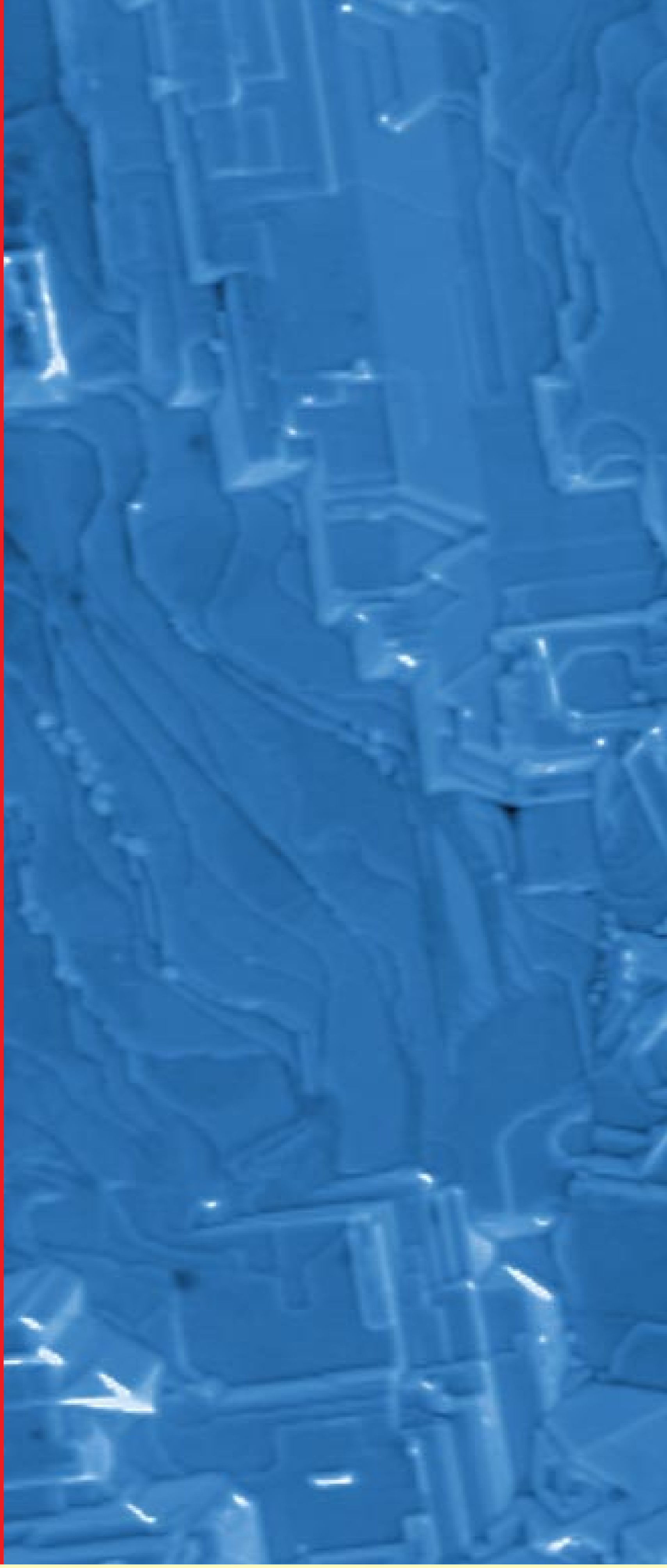

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