

# Microstructure Analysis, Metallography and Mechanical Testing of Materials

Institute of Materials Research



# Microstructure Analysis and Metallography

**is one of the core teams of the Institute for Materials Research. Here, the fundamental characterisation of materials is carried out. For this purpose, an analytical scanning electron microscope and an analytical transmission electron microscope are installed. Furthermore, an X-ray powder and a texture diffractometer, a micro hardness tester and a variety of metallographic optical microscopes are available.**

The **metallographic** laboratory is the first stop for nearly all samples investigated at the institute. Here, the polished sections for the scanning electron and light microscopic investigations are prepared. If necessary, even new preparation methods are developed for new materials. The equipment consists of several sectioning, grinding and polishing machines, a micro hardness tester and different optical microscopes.

**Scanning electron microscopy** is applied in all central research areas of the institute. Typical investigations are, for example, quantitative phase analysis in ceramic and metallic systems, the analysis of volumes and interfaces in fibre matrix composites, analysis of fraction faces in ceramic composite materials and light metal, Ni-base and super alloys. Furthermore, microscopic texture investigations (EBSD), microscopic roughness profile determination and in situ tension and compression tests with up to 5000 N load are carried out.

The resolution of the field emission scanning electron microscope (Zeiss Ultra 55) is 1 nm at an acceleration voltage of 15 keV, and a resolution of even 4 nm at 100 eV is attained. Besides the usual Everhart-Thornely and BSE detectors, the microscope is also equipped with an in-lens secondary as well as an in-lens BSE detector for high resolution imaging.

An EDS system (Oxford INCA) permits quantitative elemental analysis, quantitative line scans and quantitative elemental mapping. Furthermore, a modern EBSD attachment (Oxford Nordlys II / Channel 5) for crystallographic phase and texture analysis is attached.

Modern SEM methods like EBSD require extremely well prepared distortion-free surfaces. To achieve this, several sputter, coating and ion beam etching machines are available.

Transmission electron microscopy (TEM) is the only method which allows for comprehensive characterisation of materials on the micro as well as on the nanoscale by combination of imaging with spatially resolved chemical analysis (EDS and EELS) and diffraction. It is applied to all fundamental applied research topics at the Institute of Materials Research.

By application of TEM, micro- and nanostructures are analysed and mechanisms relevant for synthesis, fabrication, properties, degradation and failure of materials are revealed. Typical examples are diffusion processes, segregation, phase transformations, the identification of grain boundary phases and the analysis of stacking faults and dislocations.

**The transmission electron microscope** (Phillips Tecnai F30 Super Twin) is

equipped with a STEM unit, bright field, dark field and HAADF detectors and an EDS system (EDAX). Furthermore, a Gatan imaging filter (GIF) for EEL spectroscopy and element selective imaging (ESI) and a CCD camera are installed. The sample holders available enable investigations from low (-194°C) to high temperatures (up to 1,300°C) as well as crystallographic and analytical investigations.

For TEM sample preparations, two ion beam thinning machines and a jet polisher are installed besides numerous mechanical devices.

#### X-ray diffraction

Powder X-ray diffraction is a fundamental characterisation technique for every

material. The technique allows a fast and reliable identification of the phases present in the sample by comparison with a database. Quantitative phase analysis is achieved by the Rietveld method, while the analysis of lattice parameters delivers further information on solid solution formation and composition.

Additionally, texture analysis by X-rays is used at the Institute. It allows the investigation of growth- or deformation-induced preferential orientations.

For X-ray investigations, a Bragg-Brentano powder diffractometer (Siemens D5000) and a texture diffractometer (Siemens D5000TX) are operated. The standard software packages EVA and Topas (Bruker) are applied for fast and reliable data analysis.

## Mechanical Testing of Materials

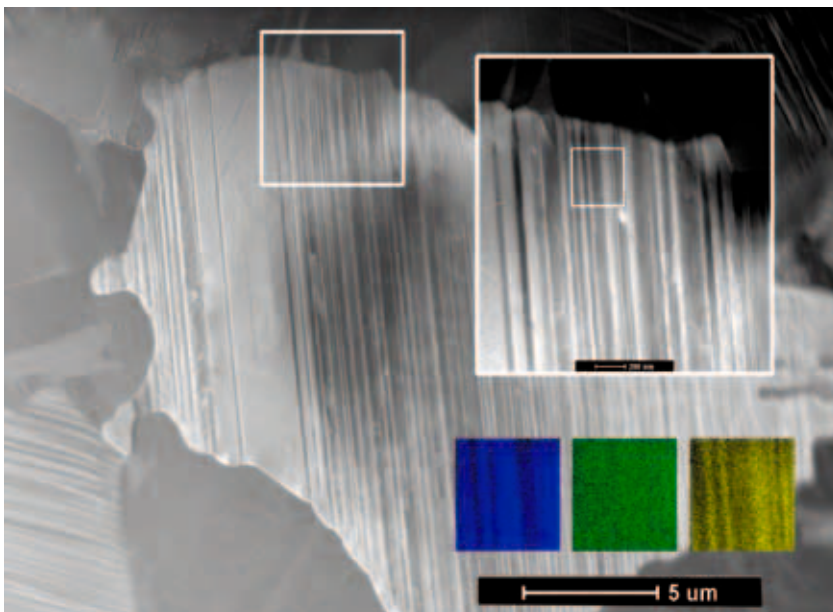
**offers mechanical material characterisation as scientific-technical service for all departments of the institute as well as for external customers. Mechanical material tests are performed under various loads, which can be uniaxial but also biaxial. Measurements are possible in air, vacuum, and corrosive environments at varying temperature ranges (-196° up to +1,400°C).**

A special expertise of the group is fatigue and fracture mechanics testing of all engineering materials including metals, ceramics, composites and multi-material hybrids. Investigations of crack growth behaviour with determination of the threshold  $\Delta K_{th}$ , monitoring of  $da/dN - \Delta K$ -curves, determination of crack resistance curves, and fracture toughness at different temperatures are part of the testing portfolio.

For answering current scientific and technical questions, experimental test plans are designed and special testing methods as well as test facilities are developed. The results from mechanical testing are evaluated involving microscopic characterisation of the material before and after testing in order to investigate changes such as deformation, damage, crack growth, and failure. These tasks are performed in close collaboration with the institute's departments and the microanalytic group.

Examples for in-house developments of test facilities are

- test facility for investigating thermal gradient mechanical fatigue of protec-



HAADF STEM image and EDS map of a lamellar TiAl alloy

tive coating systems for gas turbine blades by using hollow specimens, which are heated by using a radiation furnace and internally cooled with pressurised air. In doing so, testing conditions very similar to the service conditions of internally cooled gas turbine blades are achieved.

- test setup for fracture mechanics characterisation of delamination cracking of fibre reinforced polymers and hybrid laminates under clearly defined mixed mode crack opening conditions,
- test setups for investigating stress corrosion cracking under constant load in special corrosive media.

The mechanical testing laboratory owns several servo-hydraulic testing machines for uniaxial and biaxial loading, electro mechanical testing machines for different load ranges, mechanical resonance testing machines for cyclic loading at high frequencies, and constant load test rigs. For experiments on very small specimens a new testing laboratory has been established. Besides machines with smaller load capacity and high accuracy, modified hardness test machines are employed for exploring local mechanical properties. For testing at high or cryogenic temperatures, several furnaces, cooling and vacuum chambers are available. The laboratory equipment includes special measuring devices for monitoring forces, strains, temperatures, and crack propagation.

Mechanical material characterisation is performed also in co-operations with universities, other research institutions, and industry partners.

## Lab Equipment

- Universal testing machines for loads from 10 kN to 100 kN
- Single-arm type testing machine with load cells for 500 N and 1 kN, respectively
- Several servo-hydraulic testing machines from 10 kN up to 400 kN
- Biaxial test facility (4 cylinders up to 1,000kN)
- Electro-mechanical resonance testing machines up to 100 kN
- Constant load test rigs up to 60 kN
- Hardness and micro hardness devices
- Ultrasonic testing facility for sheets (400 mm x 1,000 mm)
- Several devices for monitoring crack growth based on the potential drop method
- Laser extensometer for contact-free strain with spatial resolution measurement and high temperature extensometers up to 1,200°C.



# Institute of Materials Research

The main research focus of the Institute of Materials Research is the development of new material solutions and their processing techniques for applications in aerospace, in energy technology, and in automotive engineering. In co-operation with DLR institutes as well as with national and international partners, the Institute of Materials Research is consistently working on fundamental and application-orientated topics.

The research portfolio spreads across the fields of metallic structures, hybrid material systems and intermetallics, ceramic

matrix composites, thermoelectric systems and high-temperature and functional coatings. The development of numerical methods to simulate the behaviour of materials and components completes these competencies aiming at supporting the transfer of materials into industrial applications.

In addition to the scientific research the institute is actively involved in the education and advanced training of young researchers at renowned universities in form of professorships and teaching assignments.

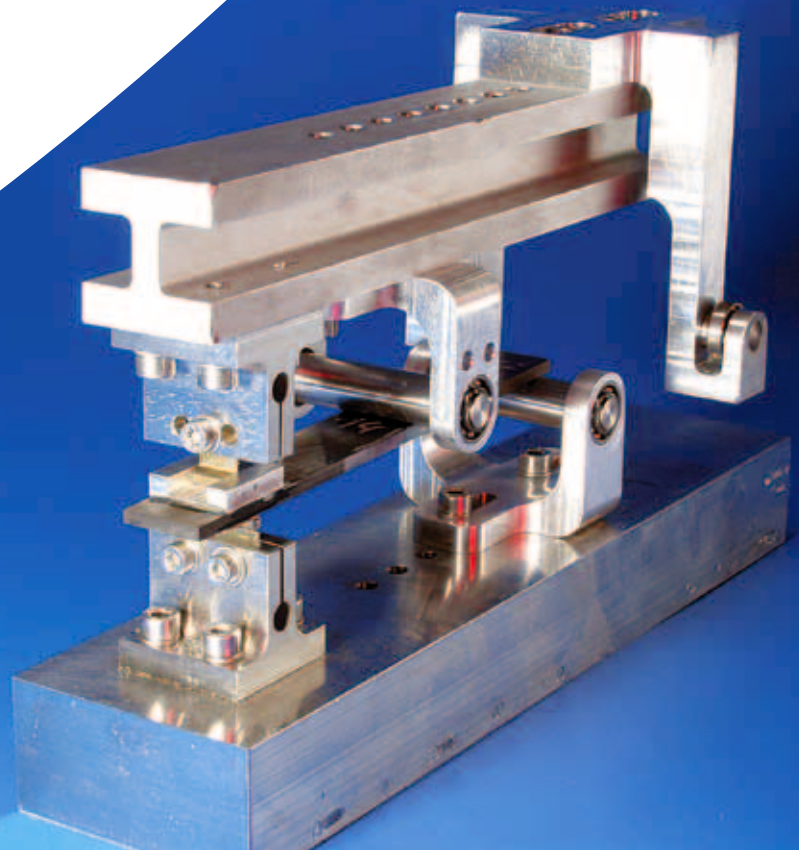
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## DLR at a glance

DLR is Germany's national research centre for aeronautics and space. Its extensive research and development work in Aeronautics, Space, Energy, Transport and Security is integrated into national and international cooperative ventures. As Germany's space agency, DLR has been given responsibility for the forward planning and the implementation of the German space programme by the German federal government as well as for the international representation of German interests. Furthermore, Germany's largest project-management agency is also part of DLR.

Approximately 6,500 people are employed at thirteen locations in Germany: Koeln (headquarters), Berlin, Bonn, Braunschweig, Bremen, Goettingen, Hamburg, Lampoldshausen, Neustrelitz, Oberpfaffenhofen, Stuttgart, Trauen and Weilheim. DLR also operates offices in Brussels, Paris, and Washington D.C.

DLR's mission comprises the exploration of the Earth and the Solar System, research for protecting the environment, for environmentally-compatible technologies, and for promoting mobility, communication, and security. DLR's research portfolio ranges from basic research to innovative applications and products of tomorrow. In that way DLR contributes the scientific and technical know-how that it has gained to enhancing Germany's industrial and technological reputation. DLR operates large-scale research facilities for DLR's own projects and as a service provider for its clients and partners. It also promotes the next generation of scientists, provides competent advisory services to government, and is a driving force in the local regions of its field centers.



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