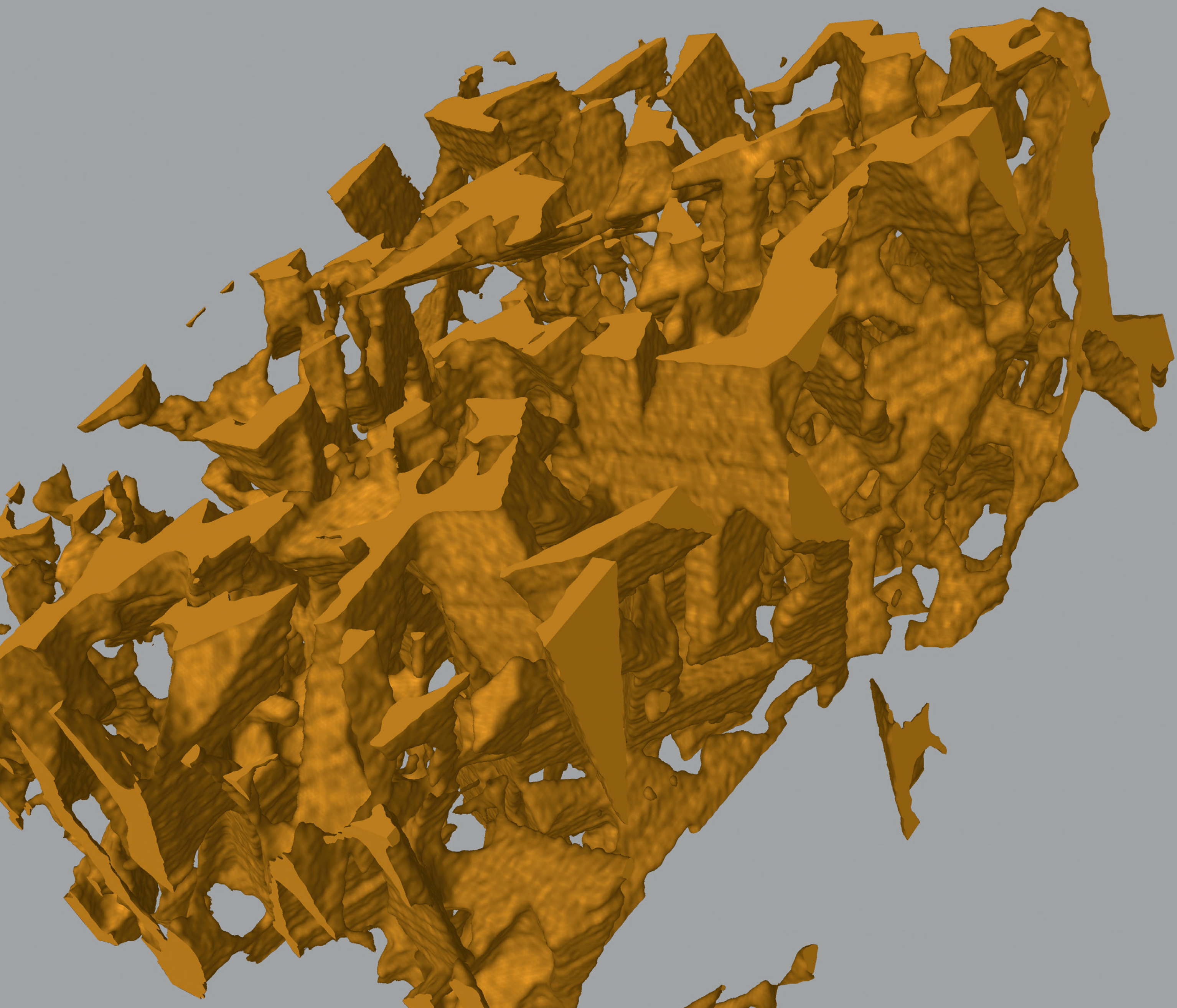
Fraunhofer IKTS

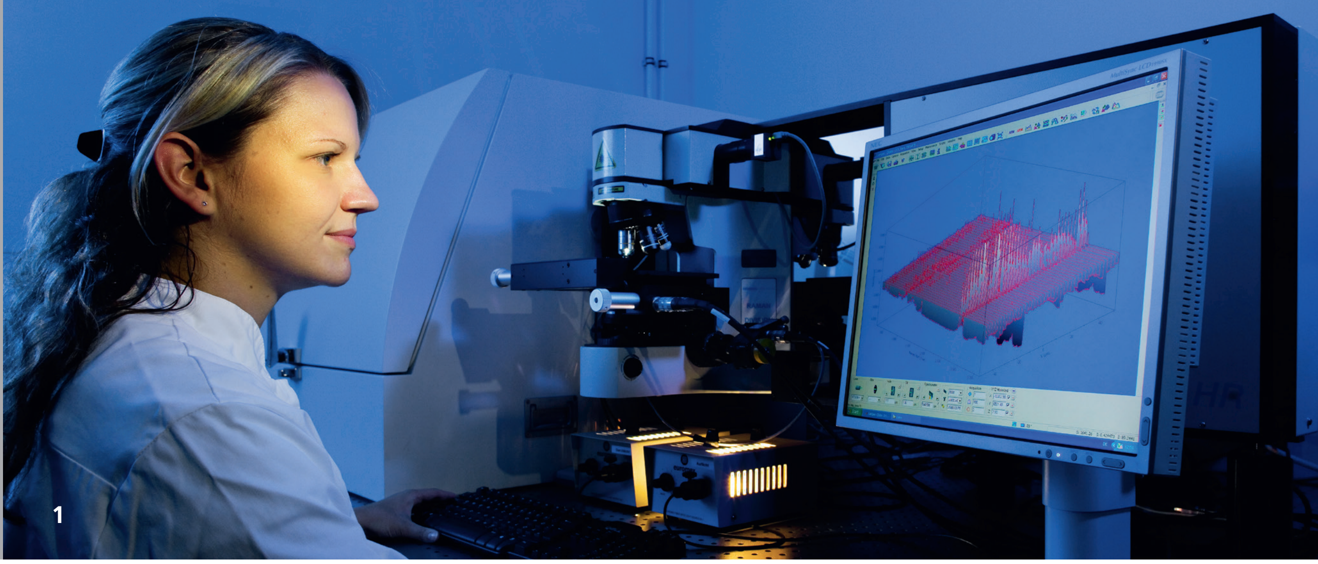
FRAUNHOFER INSTITUTE FOR CERAMIC TECHNOLOGIES AND SYSTEMS IKTS

INDUSTRIAL SOLUTIONS

CHARACTERIZATION I ANALYSIS I MEASUREMENT I TESTING



COVER IMAGE *Three-dimensional distribution of cobalt in a hardmetal.*
1 *Surface investigation with a laser scanning microscope.*



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ONE STOP SHOP FOR ALL ANALYSIS REQUIREMENTS

Fraunhofer IKTS serves as an initial contact for all problems related to analysis, evaluation, and optimization of materials and components as well as the associated manufacturing processes. Focus is primarily on ceramic and powder metallurgy (PM) materials and components as well as materials and components used in microelectronics.

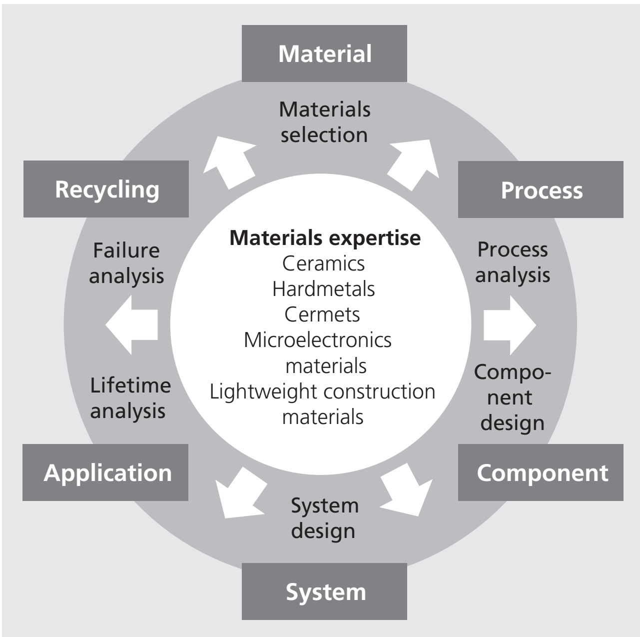
Fraunhofer IKTS possesses state-of-the-art equipment and an extensive range of modern analysis methods for

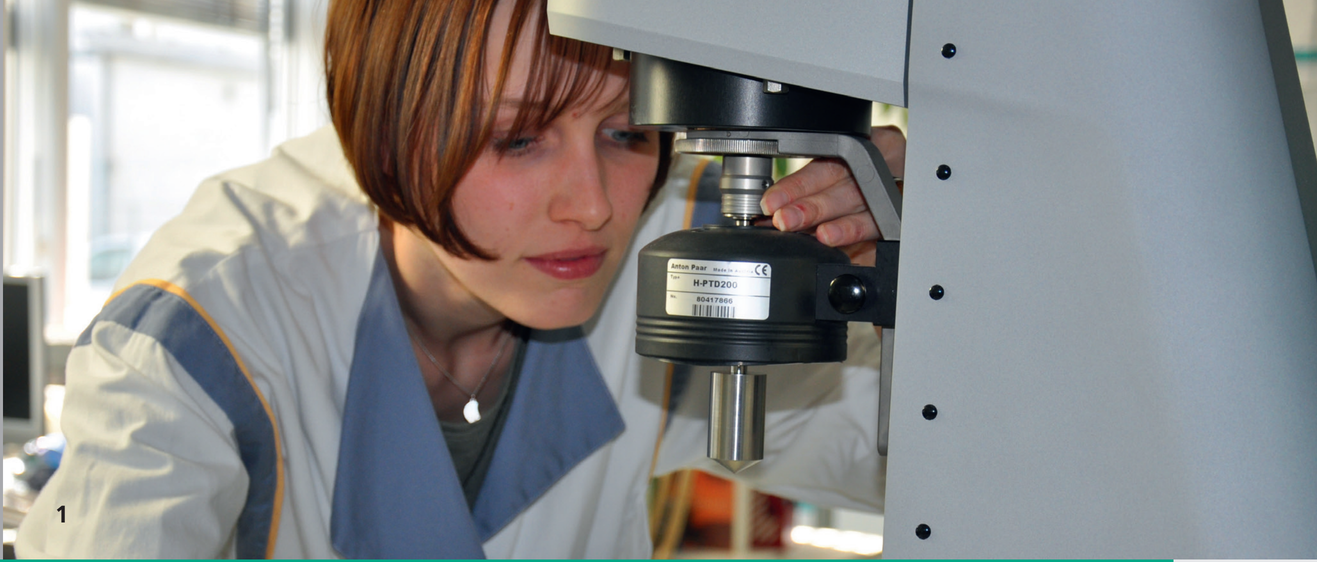
- Applied research and development
- Characterization of feedstocks and materials
- Monitoring and optimization of ceramic and PM production processes
- Quality assurance of components and parts
- Failure analysis and evaluation of behavior of materials and components in specific applications

Both standard and in-house, in part unique, analysis capabilities are available. With extensive materials, process, and analysis expertise, Fraunhofer IKTS provides support and advice in development of new materials and products, determination of characteristics, clarification of complex failure mechanisms, and compliance with regulatory and qualitative standards. Accreditation has been granted for specific measurement methods for determining powder and suspension characteristics as well as thermophysical and electrical/dielectric properties and for selected NDT methods.

This gives Fraunhofer IKTS the capacity to handle diverse tasks related to quality assurance and certification of products and processes all the way to prototype testing for customers.

Fraunhofer IKTS guarantees the confidentiality and security of all data and information handled within the scope of project work.





POWDER AND SUSPENSION CHARACTERIZATION

With the emergence of new application areas and higher demands being imposed on products, the quality of the starting materials is becoming a critical parameter in the production of advanced ceramics. Especially for ceramic and PM manufacturing processes, characterization of powders and suspensions allows potential quality defects and their root causes to be identified at an early stage.

Powder characterization

Measurement-based evaluation of powder raw materials and dispersions yields important insight into the applicability of materials as well as their processing properties in a given application. Powder analysis is hence indispensable for effective process description, control, and optimization as well as quality assurance. In addition, key information that can be used towards the registration, safety evaluation, and authorization of chemicals in accordance with REACH can be obtained through physicochemical analysis.

The DIN EN ISO/EC 17025:2005-08-accredited Laboratory for Powder and Suspension Characterization provides evaluation services for materials with various chemical compositions. The portfolio thus far has included oxide-, carbide-, and nitride-based ceramics, metals, hardmetals, and carbons as well as glasses, organic materials, and polymer particles. The multiscalar analysis extends from the nanometer to the millimeter range and encompasses both compact and porous materials.

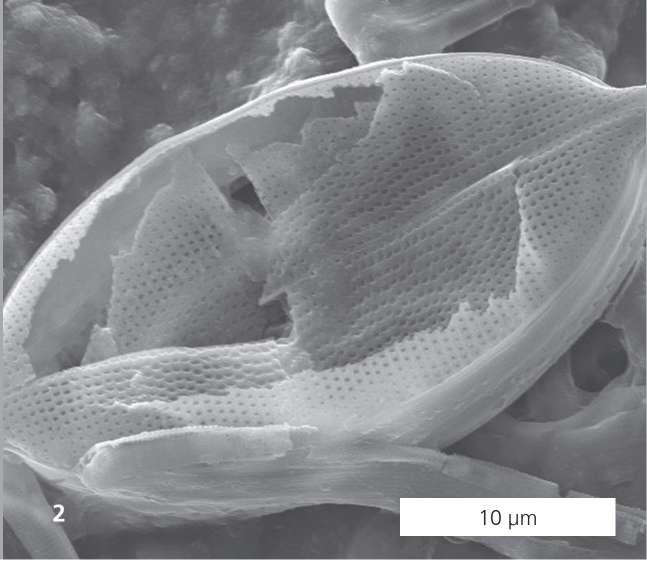
The best and fastest approach for yielding the necessary information is derived in a preliminary joint analysis of requirements. Interpretation of results and evaluation of improvement potential can be provided on request.

Suspension characterization

Development and optimization of stable, economical, and sustainable manufacturing processes and hence high-quality products are achieved through further powder handling. Introduction of raw materials into a liquid or a paste induces reactions between particle surfaces, dissolved components, and fluids. Application-relevant properties, such as suspension flowability or rheology and temporal and sedimentation stabilities, are influenced by these interactions.

A key task related to this is the defined modification and improvement of suspension properties based on measurement results. Processing and product properties can be optimized through adjustment of the energy input during dispersion or with additives. Depending on the project partner's or client's problem, suspensions with low or high solids concentrations (from a few mg/l to more than 30 % by volume) in water or in organic solvents and with adapted colloid chemical stabilities can be developed. With qualified measuring methods, the presence of inorganic and organic particles in suspensions can be quantified and changes, such as those occurring due to the presence of foreign substances, can be detected in situ.

1 *Suspension analysis with rheometer.*



Services offered

The Laboratory for Powder and Suspension Characterization is accredited to determine the dispersion and electrokinetic properties of liquids and solids (accredited equipment marked with * below).

Particle size distribution
- Laser diffraction (Mastersizer 2000)*
- Dynamic light scattering (Zetasizer Nano ZS)*
- Centrifugal separation analysis (LUMiSizer/LUMiReader X-Ray)
- Nanoparticle tracking (ZetaView)
- Dynamic image analysis (QICPIC)

Particle shape analysis
- Statistical image analysis (optical and electron microscopy)
- Dynamic image analysis (QICPIC)

Specific surface area determination
- Gas adsorption (ASAP 2020, test gases: N, Kr)

Bulk and true densities
- Helium pycnometry (Penta pycnometer)*

Porosity
- Mercury porosimetry (AutoPore V)
- Optical methods (optical and electron microscopy)

Surface charge (zeta potential)
- Microelectrophoresis (Zetasizer Nano ZS, ZetaView)*
- Electroacoustic measurement (ZetaProbe, field ESA measuring system)*

Rheology and viscosity
- Rotational and oscillation modes (MCR 301)
- Vibro viscometer (SV-10)

Sedimentation stability
- Centrifugal separation analysis (LUMiSizer)
- Gravity separation analysis (TurbiScan)

Special application areas

Raw materials selection and sampling
- Comparison of materials from different manufacturers
- Testing of different batches
- Dispersion of raw materials

Nanomaterials
- Evaluation according to defined standards/EU definitions
- Dispersibility of nanomaterials
- Presence in physiologically and ecotoxicologically relevant media
- Interaction with macromolecules (proteins/humic acids)
- Colloid chemical stabilization for different applications (e.g., coating deposition)

Suspension development
- Preparation of binary and ternary suspension mixtures, e.g., for thermal spraying
- Abrasive suspensions for finishing applications (adaptation of rheology, e.g., as a function of the applied magnetic field)

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2 *SEM image of the surface of an aged plastic particle exhibiting biological adhesion.*

THERMAL ANALYSIS AND THERMAL PHYSICS

Thermal processes have a major impact on the properties of materials, semi-finished products, and components. Lengthy know-how and a comprehensive equipment basis help to provide insight into these complex interactions as a function of temperature, time, and gas atmosphere.

Fraunhofer IKTS is a partner with accredited methods for investigation, modeling, simulation, and optimization of thermal processes. A wide range of thermoanalytical and thermophysical methods and equipment can be used individually, in combination, or directly coupled to each other. Thermodynamic phase equilibrium calculations are employed for predicting phase formation and transformations as well as interactions between materials/components and the furnace atmosphere. With this information, the energy and cost efficiency of processes and the materials properties can be analyzed and optimized. The know-how applied towards these tasks extends far beyond ceramic and PM materials and processes. Problems concerning ceramics, hard materials (e.g., borides), hardmetals, cermets, and, to a lesser extent, polymers, glasses, metals, alloys, biomaterials, foams, and composites are treated.

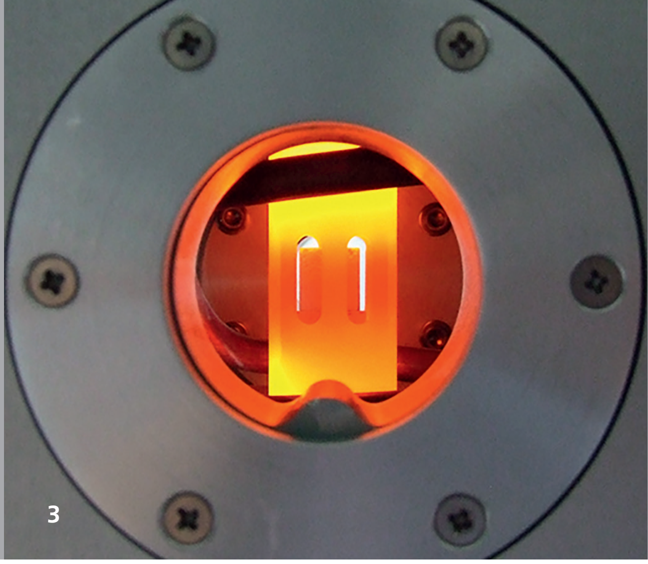
Methods

- Thermogravimetry (TG; mass changes)
- Emission gas thermal analysis (EGA) with mass spectrometry MS and FTIR spectrometry (gas analysis)
- Thermomechanical analysis (TMA), thermodilatometry (TD), hot stage microscopy (HSM), optical thermodilatometry (length or shape changes, wetting behavior, thermal expansion coefficient)
- Differential scanning calorimetry (DSC), differential thermal analysis (DTA; caloric effects, heat capacity)
- Laser flash analysis (LFA; thermal diffusivity)
- Calculation of thermal conductivity from density, heat capacity, and thermal diffusivity
- Comparative methods for determination of thermal conductivity
- High-temperature X-ray diffractometry (HT-XRD; phase transformations)
- Thermoconductometry (electrical resistance measurements)
- Kinetic analysis, modeling, and simulation (master curve, kinetic field, Netzsch Thermokinetics)
- Thermodynamic calculations with FactSage

Services offered

- Complex characterization of thermal behavior of materials (from -140 °C to 2400 °C) in oxidizing, inert, or reducing gas atmospheres (toxic, corrosive, or explosive gases and gas mixtures also possible)
- Optimization of laboratory mock-ups of technical processes (determination of temperature ranges critical for product quality, avoidance of defects, etc.)
- Kinetic analysis, modeling, and simulation of property changes (also in situ on real component geometries), e.g., for elucidation of reactions and effects of process and material parameters and for derivation or calculation of optimum process control
- Derivation of strategies for optimum outgassing and sintering as well as for adjustment of local property gradients

1 *Overview of thermal analysis infrastructure at Fraunhofer IKTS.*



- Determination of thermodynamic and thermophysical state parameters/properties (melting, solidification, and crystallization parameters, stabilities, enthalpies, heats of reaction, thermal capacities, thermal expansion coefficients, densities, thermal diffusivities, thermal and electrical conductivities and resistivities, wetting parameters, etc.)
- Supply of thermophysical data sets for finite element modeling and simulation calculations
- Determination of thermophysical properties for data sheets
- Development of data sets for thermodynamic material descriptions
- Thermodynamic phase equilibrium calculations and derivation of results pertaining to the manufacturing technology and materials optimization
- Thermodynamic analysis of the effects of interfacial reactions and corrosion under various loading conditions

Technical equipment

Accredited measuring stations

- Simultaneous thermal analyzers (STA 449F1, 429, 429C, 409), partially coupled (capillaries, apertures, or SKIMMER) with
  - Mass spectrometers (QMS403C, QMG420, 421, 422)
  - FTIR spectroscope (Tensor27)
- Macro thermobalance (MTG419/NGB)
- Thermomechanical analyzers (TMA402)
- Thermodilatometers (DIL402 and DILE7)
- Dynamic differential calorimeters (differential scanning calorimeter DSC 404, 404C, and DSC7)
- Laser flash analyzer (LFA427)
- Light flash analyzer (Nano-Flash) (LFA447)
- Thermal conductivity tester (TCT416)
- Automated laboratory gas supply system for mixing and doping of high-purity atmospheres on all units, pressure range from high vacuum to normal pressure and gas analysis with portable mass spectrometer

Non-accredited measuring stations

- Hot stage microscope and optical dilatometer (special feature: high-purity atmospheres)
- In situ resistance analyzer RIS
- In situ high-temperature X-ray diffractometer

Specific features

- Complex thermoanalysis/complex thermophysical analysis
- High-purity, corrosive and potentially explosive atmospheres
- Extreme temperatures ranging from -180 to 2400 °C
- Materials analyses (few mg/few mm³) up to component analyses (up to 500 g/250 cm³)
- Coupling of thermoanalytic experiments and thermodynamic modeling
- Kinetic analysis, modeling and simulation

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2 Selection of STA crucibles.
3 Hot stage microscope/optical dilatometer: 3D analysis of sintering.

MICROSTRUCTURAL AND MATERIALS ANALYSIS

Fraunhofer IKTS has numerous characterization methods and a comprehensive equipment basis at its disposal for developing diverse high-performance materials. Drawing on its exhaustive experience with various materials and composites, the institute offers customers and project partners a wide range of services. Established, standard, and new methods are continuously being optimized to expand the knowledge base.

The topical and methodological focus of activities at Fraunhofer IKTS is on ceramics, hardmetals, and cermets. However, customers and project partners from the areas of metals and composites have also long been seeking out the institute's expertise. Process-accompanying materials characterization, qualitative and quantitative microstructural analysis, and analysis of defects and weak points in components are the main investigations performed. Preparation and analysis methods are adapted to specific materials as well as continuously improved and optimized.

Work is done to the highest quality standards and supply of required information to customers is fast and efficient. Intermeshing of sample preparation and analysis equipment with different methods enables a variety of analyses to be performed all along the technology chain. Parameters are varied, e.g., in the FESEM, to yield the maximum amount of information from the sample.

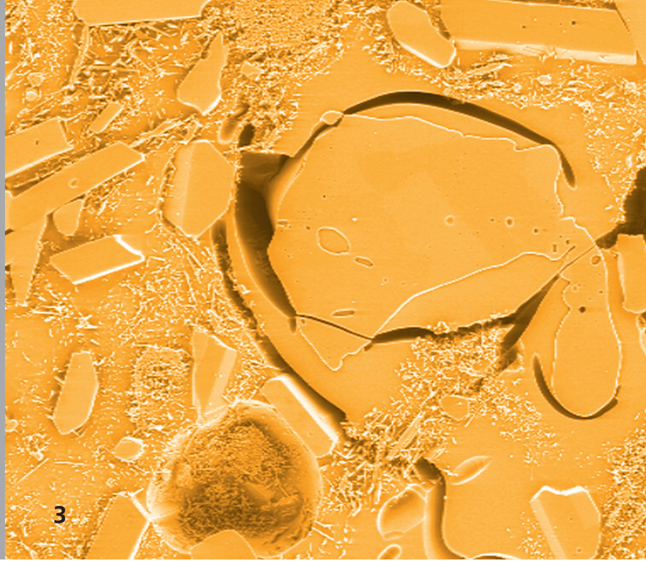
Depending on the given application and requirements, an optimized characterization method can be developed for a specific material or product. Evaluation and interpretation offer customers effective additional information for the optimal use of the analysis results.

Fraunhofer IKTS's range of offerings is continuously expanding in response to increasing demands on high-performance materials, innovative product fields, and new technical possibilities. Promising new methods, such as high-resolution three-dimensional imaging of small-volume samples via FIB tomography, have become established over the last few years. Three-dimensional element mapping can additionally deliver information on local compositions and inhomogeneities.

Ion beam-based preparation methods enable delicate materials, such as granules, green bodies, or semi-sintered samples, to be prepared in a way allowing for high-resolution artifact-free imaging of the structures and microstructures.

Local electrical conductivities can also be shown on micron and submicron scales.

1 *Materials analysis using scanning electron microscope (SEM).*



Services offered

Materialographic sample preparation

- Mechanical sample preparation
- Chemical and physical etching processes for enhancing microstructural contrast
- Ion beam preparation methods, e.g., focused ion beam (FIB) preparation, ion beam polishing, ion beam slope cutting
- Preparation of electron-transparent samples for STEM and TEM imaging
- Target preparation
- Preparation methods for granules, green bodies, semi-sintered, and delicate samples
- Development of individual preparation methods

Microscopy

- Optical microscopy
- Confocal 3D laser scanning microscopy
- High-resolution field-emission scanning electron microscopy
- Transmission electron microscopy
- Atomic force microscopy (e.g., LFM, MFM, EFM, STM)
- Acoustic microscopy

High-resolution analysis methods

- Energy-dispersive X-ray analysis (EDX)
- Wavelength-dispersive X-ray analysis (WDX)
- EBSD phase and orientation analysis
- TEM (incl. EELS)
- High-resolution 3D imaging via FIB tomography and 3D EDX
- EDX coating thickness analysis

Chemical and phase analysis

- X-ray diffractometry (XRD; qualitative and quantitative analysis, measurement at room temperature and at high temperatures, thin-film analysis, reflectometry)
- X-ray fluorescence analysis (XFA) on raw materials, components, and liquids
- Micro-Raman and IR spectroscopy
- Determination of oxygen and carbon contents

Additional services offered

- Quantitative microstructural analysis (e.g., grain size analysis, porosity analysis, phase analysis, coating thickness analysis, determination of shape parameters)
- Interfacial analysis
- Analysis of corrosive reactions
- Failure analysis (estimation of mechanical, thermal, tribological, and corrosive loads, fractography)
- Determination of surface roughness

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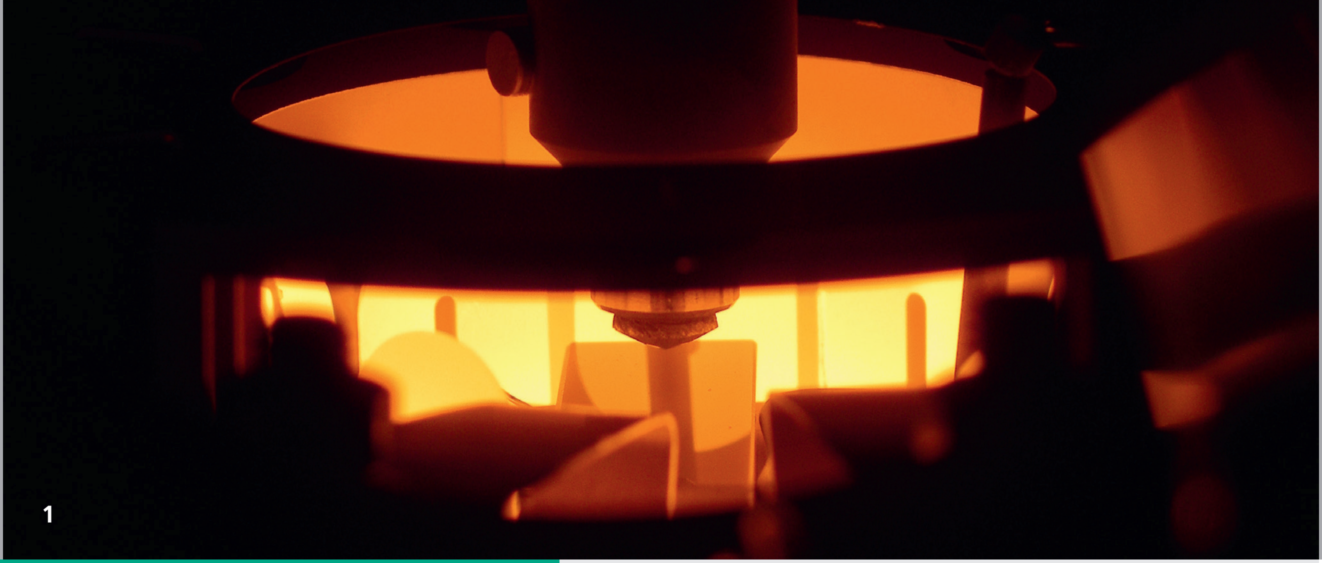
2 *Fusion device for RFA sample preparation.*
3 *SEM image of an electroporcelain specimen.*



MECHANICAL TESTING

Materials characteristics, such as strength, hardness, and fracture strength, are critical for component design and improvement of advanced materials. Fraunhofer IKTS draws on a number of standard and non-standard test methods to determine the mechanical properties of materials at all stages of the development cycle.

The Dresden and Hermsdorf siites are extensively equipped with in part unique facilities for performing numerous mechanical tests on materials, components, and products. Applications range from material and component tests within the scope of R&D, tests for process and manufacturing monitoring as well as QA, and customer- and application-based tests. All standard processes as well as application-specific processes for determination of materials characteristics, e.g., at elevated temperature or after aging in various media, can be performed. Measurement capabilities at IKTS range from high-temperature strength and creep resistance measurement to hardness measurement at temperatures ranging from room temperature to 1500 °C. Through this, materials properties can be determined at the temperature ranges relevant to the respective applications. As a result, better insight into the behavior of the materials can be gained and component design can be more specific.

Measurement quality and reproducibility are ensured through continuous improvement of test and analysis methods as well as regular calibration of test equipment.

Services offered

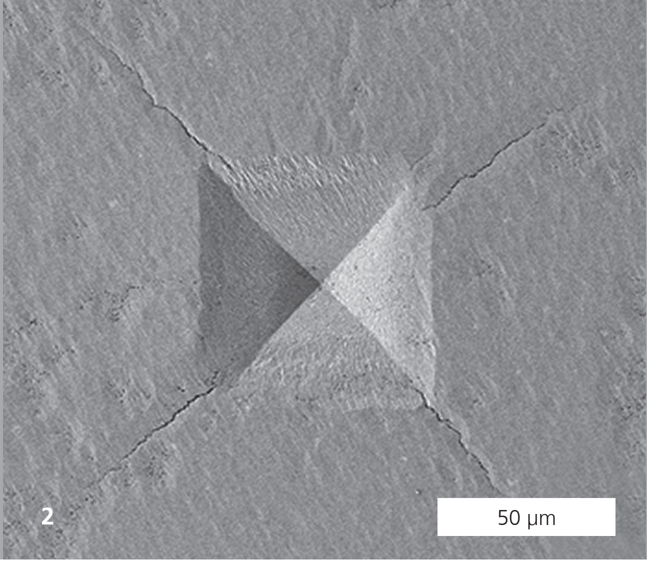
Determination of strength and elastic properties

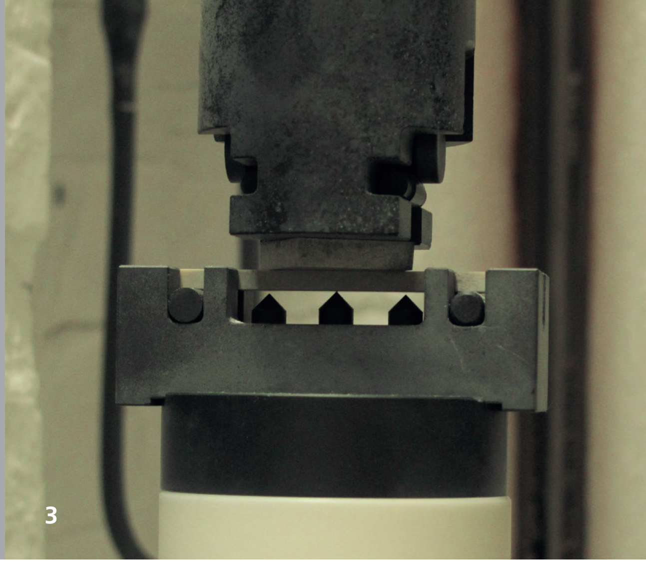
- Flexural strength (three- and four-point bending) up to 100 kN in accordance with DIN EN 843-1
- Compressive strength up to 100 kN (without measurement of change in length: up to 2 MN) in accordance with DIN 51104
- Combined tension and compression testing (100 kN) with torsional loading up to 100 Nm
- Torsion testing
- Tensile strength testing with strain measurement up to 100 kN
- Testing under multiaxial loads
- Tensile and flexural bond strength testing
- Determination of Young's modulus under static bending loads in accordance with DIN EN 843-2
- Statistical analysis of strength measurements (Weibull modulus determination in accordance with DIN EN 843-5)
- Fractography/fracture surface analysis (according to DIN EN 846-6)
- Determination of Young's modulus, shear modulus, and Poisson's ratio using pulse and resonance methods

Specific mechanical methods

- Burst strength testing
- Shear testing
- Testing of loads arising from vibration or mechanical shock

1 *High-temperature hardness testing.*





- Biaxial stress measurement
- Acoustic emission testing under mechanical and thermal loads
- Measurement of residual strength following thermal shock loading

Hardness

- Vickers and Knoop micro- and macrohardness (HV0.025 to HV50: 0.25 to 490.3 N) in accordance with DIN EN 843-4, Brinell and Rockwell hardness
- Characterization of thin films: instrument-based penetration testing (0.01–30 N), scratch testing in accordance with DIN EN 14577
- Production of hardness test blocks made of Si_3N_4 (1400 to 1600 HV1), SiC (2000 to 2200 HK1), and Al_2O_3 (2000 to 2200 HV1) with calibration certificates

Fracture toughness K_{IC}

- From bending tests on notched specimens (SEVNB method) in accordance with DIN EN ISO 23146, from crack lengths from Vickers hardness indentations (IF method)

Dynamic loading

- Impact testing in accordance with DIN EN 60068-2-75
- Pendulum impact testing

Testing at elevated temperatures

Strength in air < 1600 °C, alternatively in vacuum to 1400 °C, 1 kN

- Flexural strength (three- and four-point bending) in accordance with DIN EN 820-1
- Young's modulus (four-point bending in air)
- Compressive strength

Creep and fatigue testing in air < 1600 °C, 1 kN

- Creep under constant load (four-point bending) in accordance with EN 820-4
- Material fatigue with dynamic fatigue test (four-point bending)
- Determination of crack growth by means of fractographic investigations

High-temperature hardness up to 1500 °C in a vacuum

- HV0.2 to HV30 (2.0 to 294.3 N)

Micromechanical testing (see p. 16)

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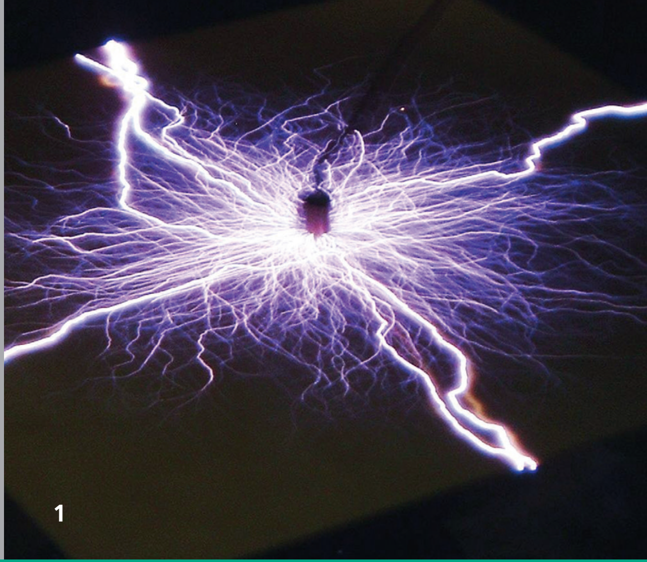
2 *Hardness indentation.*
3 *Apparatus for creep testing at elevated temperatures.*



QUALITY ASSURANCE LABORATORY

Testing can be performed alongside development in nearly all areas of quality assurance according to national and international standards or customer-specific programs. Detailed environmental, material, and safety tests are performed in a fully equipped testing laboratory by well-versed staff with extensive experience in test planning, execution, and evaluation. Other activities include jig/fixture construction and regular calibration of the measuring and test equipment.

Fraunhofer IKTS can perform a wide range of standard and non-standard, customer-specific tests in a laboratory that has been audited and accredited by VDE, TÜV Nord, and DAkkS (Deutsche Akkreditierungsstelle GmbH).

The technical infrastructure at Fraunhofer IKTS encompasses a laboratory equipped for conducting electrical, environmental, material, and safety tests, a laboratory for mechanical testing, and a calibration laboratory. Safety and reliability tests are performed on products and materials using standardized test systems and processes. The related test marks (e.g., TÜV "Type Approved" mark) can be issued based on the results of these tests. The testing laboratory has been certified as a CBTL (certified testing laboratory) by the IEC in cooperation with the national certification body VDE Prüf- und Zertifizierungsinstitut GmbH.

The Quality Assurance Laboratory has received flexible-scope accreditation from the DAkkS for certain electrical test procedures. The latest quality standards (ISO 9001, ISO 10012, etc.) prescribe regular and preventive monitoring and calibration of the test equipment used. The calibration laboratory offers DIN EN IEC 17025-compliant factory calibration by competent staff. To supplement DAkkS accreditation of the testing laboratory, accreditation of the calibration laboratory is being sought.

Services offered

Accredited electrical testing of materials

- Volume resistivity in accordance with DIN IEC 60093
- Surface resistivity in accordance with DIN IEC 60093
- Dielectric strength up to 100 kV AC/130 kV DC in accordance with DIN EN 60243-1
- IEC 250 dielectric properties (permittivity)

Non-accredited testing of materials

- Resistance measurement with temperature loading in accordance with DIN IEC 60345
- Arc resistance testing
  - Low-voltage high-current testing in accordance with DIN VDE 0303-5
  - High-voltage low-current testing in accordance with DIN EN 61621
- Determination of tracking and erosion resistance in accordance with DIN EN 60587 and DIN EN 61302
- Testing in accordance with DIN EN 60112 PTI/CTI
- Assessment of thermal hazards
  - Ball pressure testing in accordance with DIN EN 60695-10-2
  - Glow wire testing in accordance with DIN EN 60695-2-10
  - Burner testing in accordance with DIN EN 60695-11-4

1 *Dielectric breakdown in a ceramic material.*

2 *High-voltage AC/DC unit.*

Safety and environmental testing

- Climatic testing
  - Dual-chamber shock testing (-80/220 °C)
  - Single-chamber shock testing (-30/150 °C with vibration load of up to 50 g)
  - Temperature- and humidity-controlled aging (-70 °C to 190 °C and 0 % to 98 % relative humidity, chamber sizes up to 1.5 m^3)
  - Stress testing (-70 °C to 180 °C at 15 K/min)
  - Thermal aging (up to 1600 °C)
  - Condensation testing (100 % relative humidity)
- Vibration testing (5 to 2000 Hz, max. 600 kg, max. 100 g acceleration)
- Impact testing (max. 200 g, half-sine, trapezoid, saw-tooth)
- Salt spray testing in accordance with DIN EN ISO 9227

Safety and life testing

- Testing of components and products according to the latest standards as a basis for CE marking, TÜV "Type Approved" marking, or VDE marking pursuant to:
  - DIN EN 60335
  - DIN EN 60730
  - DIN VDE 0620
  - DIN EN 60669
  - DIN EN 60668
  - DIN EN 61210
  - DIN EN 61558
  - DIN EN 60598
  - DIN EN 131 others on request
- Component and product life testing
- Testing within the scope of development
- Reliability and serviceability testing

Calibration

- Electrical parameters
  - Voltage (0 to 40 kV)
  - Current (0 to 550 A)
  - Frequency (0 to 2 GHz)
- Electrical components
  - Resistors (0 to 100 TΩ)
  - Capacitors (10 pF to 1 mF)
  - Inductors (1 µH to1 H)
- Temperature calibration
  - Thermocouples and gauges (-75 °C to 1150 °C)
- Scale calibration (1 g to 100 kg)
- Force (0 to 100 kN)
- Torque (0 to 400 Nm)
- Humidity (20 % to 95 % relative humidity)
- Pressure (1 to 700 bar, oil or nitrogen as medium)
- Length calibration
  - Calipers, dial gauges, final dimensions
  - Micrometer screw gauges, geometry measurement
- Other types of calibration on request

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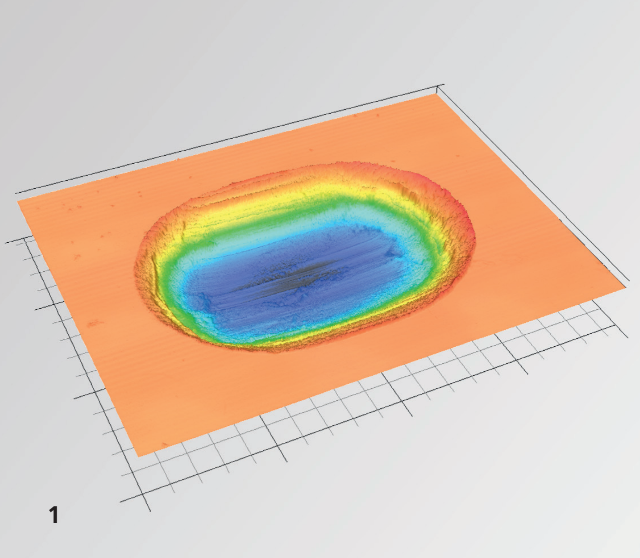
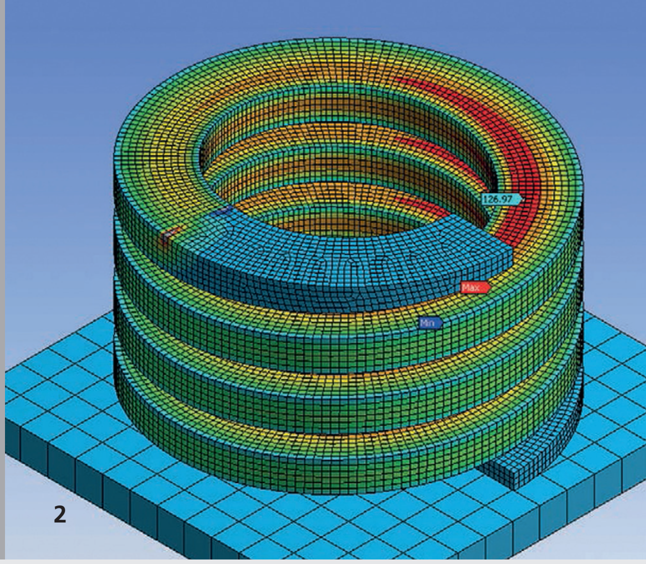
3 Shaker table for vibration testing.
4 Climate cabinet, 1.5 m^3, -70 °C to 190 °C, 5 K/min.





FAILURE ANALYSIS

Application-specific selection of materials for ceramic components and behavioral assessment (including failure analysis) of ceramic materials and components in use are complex processes requiring solid knowledge of materials, skill in materials preparation, and mastery of the relevant analysis methods.

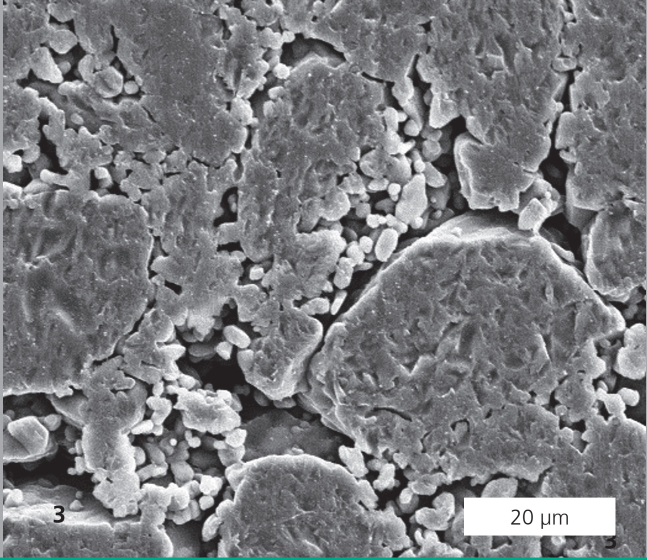
In many sectors of the economy, there is an urgent need for low-cost production components with extended service lives in both abrasive tribological and corrosive conditions. The result of this is greater loading of existing materials and components in various applications. Apart from development of high-performance materials, materials selection based on specific loading conditions is required for avoidance of premature component failure. However, this requires precise knowledge about the behavior of the materials under mechanical, thermal, corrosive, and tribological loads. This knowledge can be generated through sophisticated measurement and analysis methods that can reproduce the extreme loading conditions. Alternatively, reliable failure analysis and assessment can be performed to facilitate determination of the root cause of failure and materials qualification.

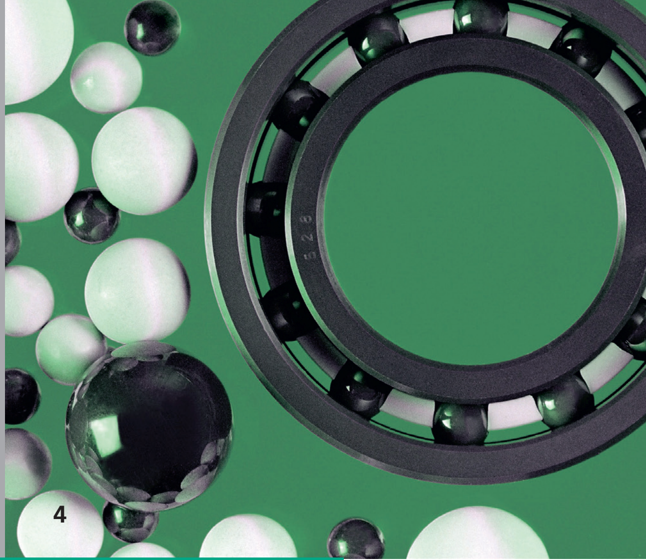
Failure analysis entails thorough fractographic investigation of the fracture surfaces as well as localized or more extensive changes in the microstructure and the properties under application conditions. Service life-limiting processes can be modeled through simulation of mechanical, thermal, corrosive, or abrasive tribological loads by means of adapted test methods or numerical simulations. This then enables the selection of materials to be qualified or the materials to be optimized for the particular application in collaboration with partners from industry.

To be able to model and understand service life-limiting processes, it is often necessary to have precise knowledge of the corrosion stability and the wear behavior. Fraunhofer IKTS has far-reaching experience and methods for investigating corrosion and wear behavior (see Tables 1 and 2). Both standard test methods and modified, load-adapted test benches are available. Optimal strategies for quality assurance of products can be developed with this know-how in combination with state-of-the-art non-destructive analysis possibilities.

The institute's know-how and "know-why" cover ceramic materials, hardmetals, and cermets. Solid results interpretation is possible thanks to the close meshing with the areas of materials and component development. Fraunhofer IKTS possesses a unique body of knowledge on ceramic materials, components, and systems stemming from a long history of development of ceramic materials, processes, and products. Customers and project partners benefit from this experience in materials selection and component design as well as in failure analysis.

1 *Wear track in a Si_3N_4 material.*
2 *Stress distribution in a ceramic spring.*





CORROSION AND TRIBOLOGICAL TESTING

Standard corrosion methods

Method	Temperature	Media
Corrosion by liquids under normal pressure	To boiling point	Acids, bases, salt solutions
Electrochemical corrosion in various electrolytes	Close to RT	Acids, bases, salt solutions
Hydrothermal corrosion	< 250 °C	Pressure < 200 bar, water, salt solutions, diluted acids, water vapor
Gas corrosion	< 2000 °C	Gas with various compositions, flowing
Hot gas/burner test bench	< 1600 °C	Flow rate v = 100 m/s, pressure 1 atm, up to 30 % water vapor
Salt spray test	35 °C	As per DIN EN ISO 9227 (NSS)
Humid heat	0 °C to 100 °C; 10 % to 100 % relative humidity	Constant climate, cyclic tests also possible
Tracking and erosion resistance	Normal climatic conditions	Normal and harsher conditions according to DIN EN 60112 and DIN IEC 60587
Arc resistance	Normal climatic conditions	Low voltage and high current, high voltage and low current

Tribological measurement methods

Method	Conditions	Result
Oscillation and rotational measurement with various pairs (SRV linear-oscillation test machine)	Controlled climatic conditions T < 800 °C force < 200 N	Frictional force, twisting moment, coefficient of friction, wear rate
Rolling-sliding friction (rolling friction with slip)	Dry running, various media (force to 2000 N)	Frictional force, wear rate, coefficient of friction
Abrasive wear according to ASTM G65 (TE 65 multiplex sand/wheel abrasion tester)	Frictional force < 200 N	Specific abrasive wear (mass change, volume change)

Services offered

- Consulting on application-based materials selection and component design
- Failure and failure mechanism analysis
- Determination of corrosion behavior and corrosion mechanisms occurring in ceramic materials
- Determination of wear mechanisms

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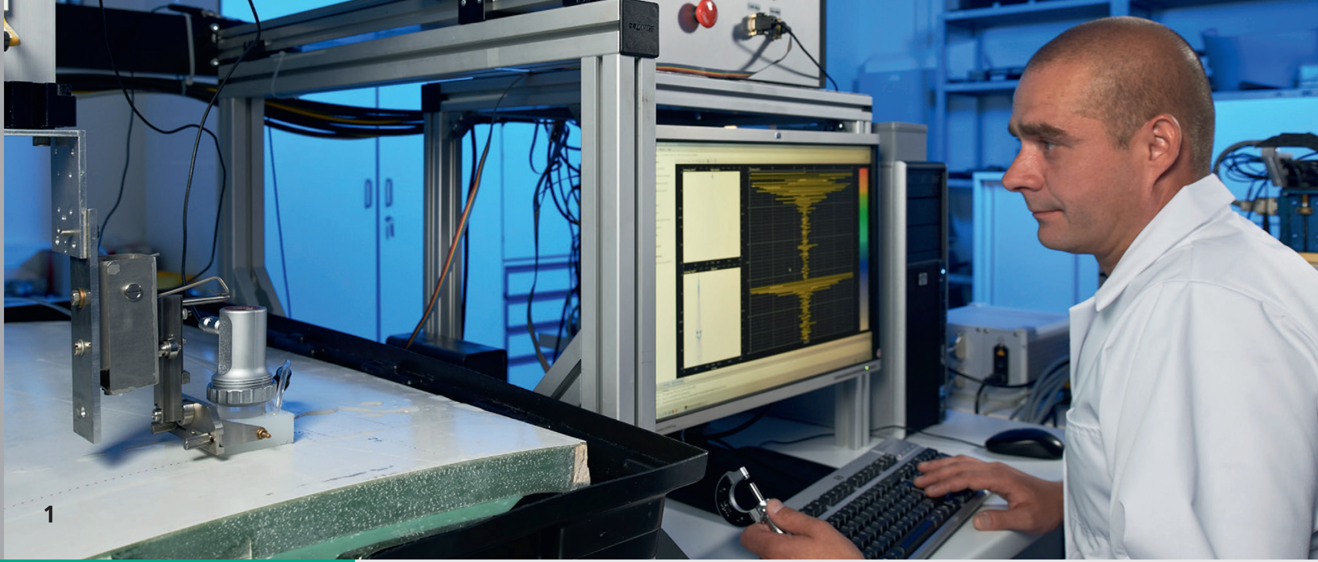
3 *Surface of an SiSiC material corroded in NaOH.*

4 *Si_3N_4 ball bearing.*

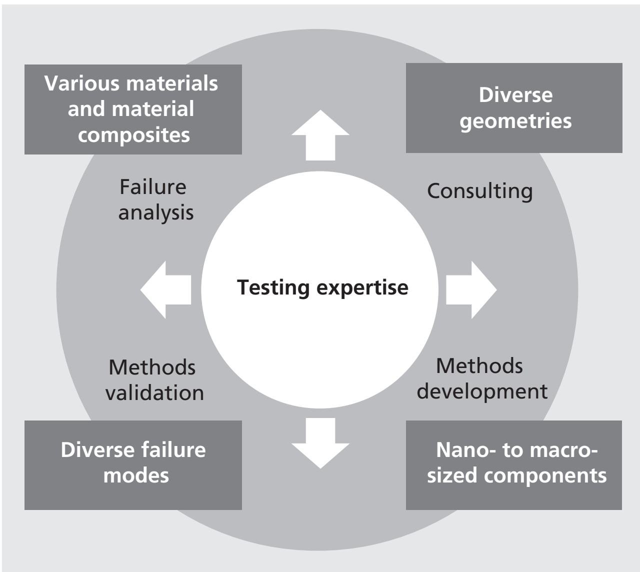


NDT TEST LAB

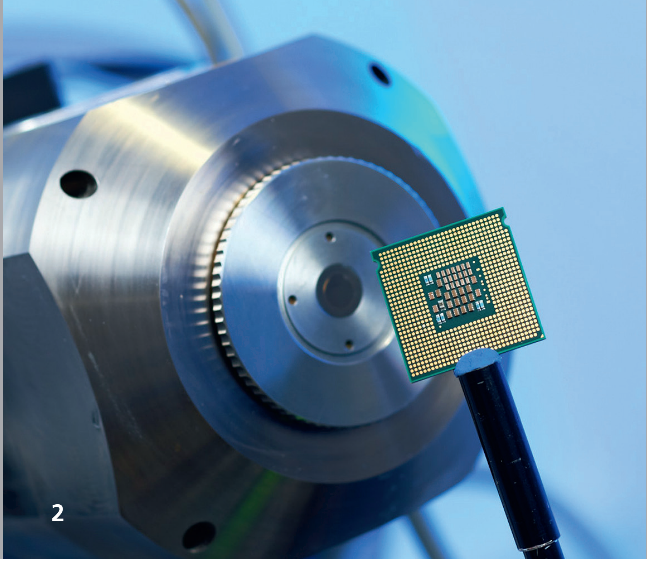
The accredited NDT test lab at Fraunhofer IKTS offers a complete range of non-destructive testing services for classic routine testing using standard or modified methods. Unconventional tasks, feasibility studies, development of test concepts, and validation/qualification of new processes and test concepts are all part of the accredited test lab's full-service NDT offering and can be performed for clients in all industries either at the client site or in the testing laboratory according to the specific task.

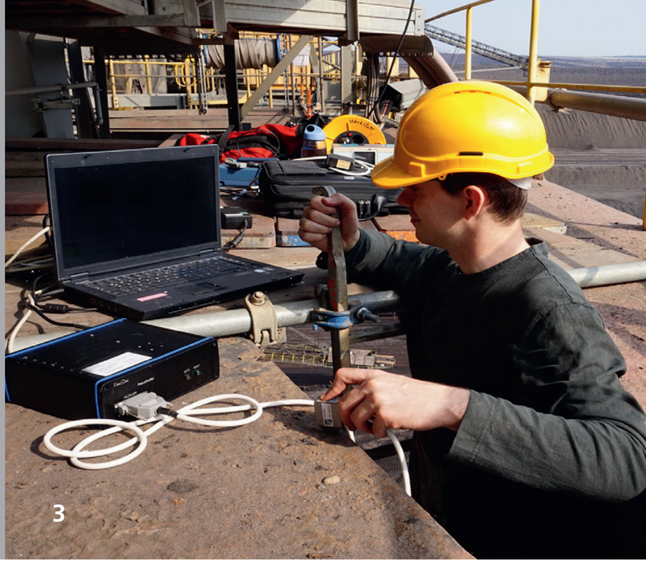
The full-service NDT offering encompasses all steps from consulting to the individualized test result. Activities can begin with feasibility studies for customized methods, modification, supplementation, or adaptation of existing test methods or development of new methods and continue with validation, conceptual design, and creation of test solutions and customer-specific services. Feasibility studies are conducted for use of existing NDT methods on new materials and applications and for modifications based on existing methods. For each defined problem, the feasibility under the given conditions is checked for a number of different approaches. The accredited NDT test lab draws on commercially available testing technologies as well as all new technologies developed at Fraunhofer IKTS. Individual problems can be solved using state-of-the-art sensors, equipment, and algorithms.

If no standards exist for a specific application case for NDT methods in the field of ultrasonics and eddy current, the NDT test lab optimizes, evaluates, and validates the method under the given customer-specific conditions. With the validation process the applicability and the conditions of applicability of a non-standard test method for a specific case are demonstrated through a more detailed investigation.



1 *Ultrasonic inspection of a GFRC component.*





Services offered

Ultrasonic methods

- Detection of volume defects with a material- and method-dependent resolution of Ø ≥ 100 µm
- Ultrasonic scan on planar, curved, and rotationally symmetric samples made of various materials (object parameters: weight < 100 kg, dimensions < 500 x 300 x 300 mm)

Probe characterization

- In accordance with DIN EN 12668-2, DIN EN 16392, and other methods

X-ray methods

- X-ray microtomography
- Laminography for partial areas of planar assemblies, such as printed circuit boards
- X-ray diffraction for analysis of phases and surface tension

Eddy current methods

- Manual testing of components with various geometries
- Eddy current scans, spatial resolution of up to 200 µm
- Inspection for surface defects, conductivity measurement following calibration, and determination of coating and wall thickness
- Investigations using various sensors (absolute and differential sensors), measurement frequencies up to 100 MHz

Micromagnetic materials characterization

- Use of the Barkhausen effect
- Testing with 3MA and FracDim® in the laboratory and on site
- Realistic estimation of material damage
- 2D measurement of internal stress with/without experimental calibration
- Determination of hardness and other microstructurally dependent parameters (e.g., hardening depth, defects)

Active thermography

- Lock-in thermography possible
- Various excitation modes: thermal (e.g., hot air), electrical (conduction, induction), or optical (laser, IR emitter, halogen emitter)

Other surface methods

- Penetrant, magnetic particle, or visual testing
- Inspection for near-surface defects and surface properties
- Can be used on nearly all types of materials

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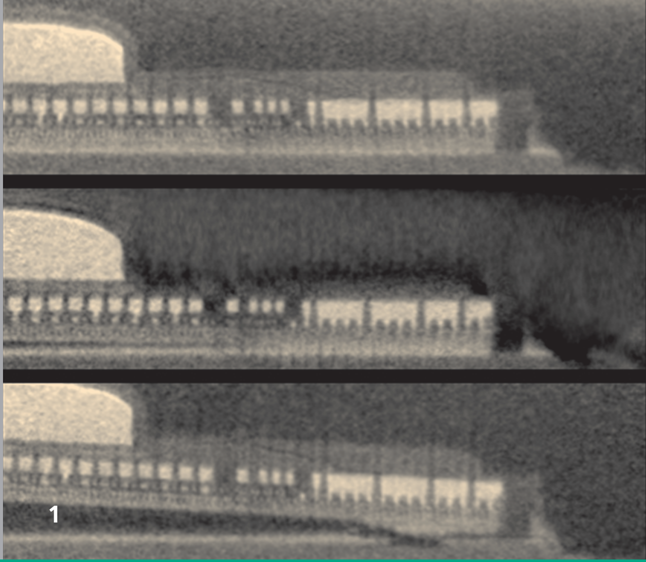
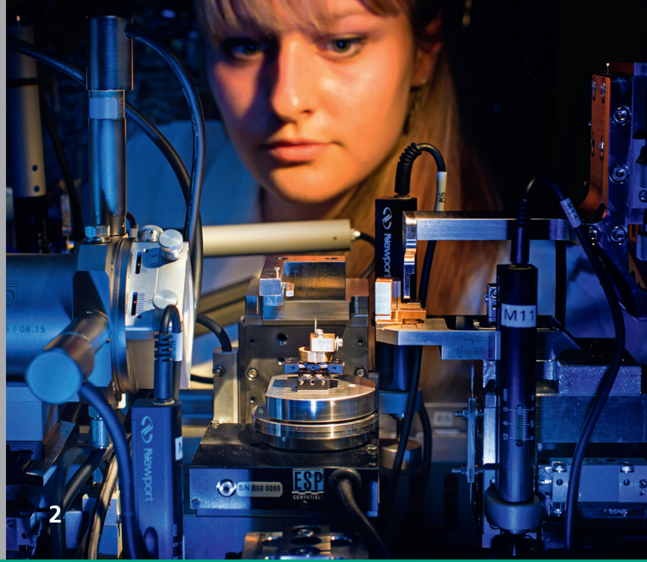
2 *X-ray inspection of an electronic assembly.*
3 *Micromagnetic inspection of a steel girder.*





MICROELECTRONICS AND NANOELECTRONICS TESTING

A special area of expertise at Fraunhofer IKTS is the use of microscopy principles in the transmission-based characterization of components and composites for micro- and nanoelectronics. For this, the application scope of X-ray-based methods is extended to approx. 50 nm to enable three-dimensional visualization of complex geometries.

X-ray microscopy and tomography

At Fraunhofer IKTS, structures and defects in materials are analyzed by means of X-ray microscopy (XRM) and a method based on it, X-ray nanotomography, performed in an X-ray microscope. These methods enable non-destructive examination of structural and functional materials on a microscopic scale with a resolution of up to approx. 50 nm. Imaging of pores, inclusions, and cracks in composite materials (e.g., CMCs), nanoporous materials (filter membranes), and microelectronics (through-silicon vias) represents the main application. Objects larger than the field of view (max. 65 x 65 µm) can be visualized through sectioning in the X-ray microscope and pasting together of the micrographs to form mosaics.

Especially for low-density materials, the contrast can be enhanced using the Zernike phase contrast technique. This allows for better visualization of interfaces and surfaces as well as delaminated regions and cracks. Thorough materials characterization can be performed at Fraunhofer IKTS through a combination of X-ray microscopy and X-ray nanotomography in conjunction with numerous imaging and analysis methods, such as optical microscopy, micro-CT, electron microscopy (REM, TEM) including spectroscopy (EDX, EELS), and micro- and nanoindentation.

In situ experiments

With miniaturized thermal and mechanical testing devices situated in the beam path of the X-ray microscope, both three-dimensional and four-dimensional data can be acquired in experiments. Currently, the following miniaturized testing capabilities are available for use in the X-ray microscope:

- Microindenter with variable test specimens (flat punch, sphere, Berkovich, cube corner) up to 1 N
- Micro double cantilever beam for studying crack propagation
- Microreaction chamber with temperatures of up to 500 °C (reactive or inert atmosphere)
- Electromigration and stress migration test methods

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1 *Tomographic sections through a microchip during a micro-DCB test.*

2 *Materials analysis in the X-ray microscope.*

OTHER APPLICATION-SPECIFIC TEST METHODS

The methods presented here for characterization of processes, materials, and components are suitable for a wide range of applications. Fraunhofer IKTS is also building up know-how in special niche applications.

Characterization of granules and compactibility of raw materials

With the state-of-the-art facilities at the Center of Excellence in Powder Technology, all process steps from selection of ceramic, metal, and composite materials to treatment and tailoring are supported by qualified characterization methods.

Workability of thermoplastic mixtures

Shaping of thermoplastic mixtures is a core competency at Fraunhofer IKTS and is accomplished with the help of extensive materials and process characterization experience.

Porous materials and membranes

One key area of activity at Fraunhofer IKTS is membrane development and technology. Unique methods for application-specific membrane testing (e.g., of permeability and separation behavior) using developed pilot processes and systems are available for this.

Burners and systems for exhaust gas after-treatment

Unique characterization methods are employed in the development of high-temperature ceramic materials and components. Characterization of porous materials especially for exhaust gas after-treatment has been driven forward over the last several years through close cooperation with leading manufacturers.

Characterization of catalytic activity

As a basis for energy and environmental systems, characterization of catalytic activity of substances and components generates one-of-a-kind solutions for energy and substance conversion.

Properties of battery components and batteries

All of the relevant characterization methods, especially for lithium-ion, sodium-nickel chloride (ZEBRA), redox flow, and metal-air batteries, are used.

Fuel cell technology

Fraunhofer IKTS is one of the world's leading institutes in the development of high-temperature fuel cells. With particular focus on SOFC and MCFC technologies, complex characterization methods covering materials through to qualification of prototype systems in the field are available.

Characterization of electrical and dielectric properties at room and elevated temperatures

Controlled adjustment of electrical and dielectric properties allows ceramic materials to be used in a multitude of applications. At Fraunhofer IKTS, knowledge of suitable multiscale characterization methods is supplemented by excellent technical facilities.

Characterization of optical properties

Transparent ceramics and ceramic phosphors can be made in efficient manufacturing processes. Fraunhofer IKTS has the characterization capabilities required for this.

3 *Test rigs for SOFC fuel cell systems.*

FRAUNHOFER IKTS IN PROFILE

The Fraunhofer Institute for Ceramic Technologies and Systems IKTS conducts applied research on high-performance ceramics. The institute's three sites in Dresden and Hermsdorf (Thuringia) represent Europe's largest R&D institution dedicated to ceramics.

As a research and technology service provider, Fraunhofer IKTS develops modern ceramic high-performance materials, customized industrial manufacturing processes and creates prototype components and systems in complete production lines from laboratory to pilot-plant scale. Furthermore, the institute has expertise in diagnostics and testing of materials and processes. Test procedures in the fields of acoustics, electromagnetics, optics, microscopy and laser technology contribute substantially to the quality assurance of products and plants.

The institute operates in eight market-oriented business divisions to demonstrate and qualify ceramic technologies and components as well as non-destructive test methods for new industries, product concepts and markets beyond the established fields of application. Industries addressed include ceramic materials and processes, mechanical and automotive engineering, electronics and microsystems, energy, environmental and process engineering, bio- and medical technology, optics as well as materials and process analysis.

www.ikts.fraunhofer.de

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Industrial solutions
Characterization – Analysis – Measurement – Testing

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