

## FRAUNHOFER-CENTER FOR HIGH TEMPERATURE MATERIALS AND DESIGN HTL

FRAUNHOFER-INSTITUTE FOR SILICATE RESEARCH ISC



## FRAUNHOFER-CENTER FOR HIGH TEMPERATURE MATERIALS AND DESIGN HTL



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# FRAUNHOFER-CENTER HTL IN PROFILE

HIGH TEMPERATURES – EFFICIENT SOLUTIONS



The Fraunhofer-Center for High Temperature Materials and Design HTL develops materials and components as well as measuring and simulation methods for the use at high temperatures. Important applications are in energy, drive and thermal technology.

The center is organized into four working groups: Composite Material Technology, Polymer Ceramics, High Temperature Design and Metal Ceramic Composites. It currently has around 70 employees at its two locations in the German towns of Bayreuth and Würzburg. More than 2000 m<sup>2</sup> of high-quality laboratory and pilot plant space with state-of-the-art equipment are available for development projects and R&D services. In addition, the HTL has an application center for Textile Fiber Ceramics in Münchberg that emerged from a cooperation between Fraunhofer and the Hof University of Applied Sciences.

The primary area of research is improving quality as well as material and energy efficiency of industrial heating processes. In Germany, more than 10 % of primary energy is currently used for industrial heat treatments. There is significant potential for improvement with regard to saving costs and energy as well as for improving quality.

The following working areas contribute to this research focus:

- Optimizing thermal processes in material production
- CMC components for gas turbines
- Developing ceramic fibers for high temperature insulation
- Designing kiln furniture and high temperature components
- Development of high temperature measuring methods

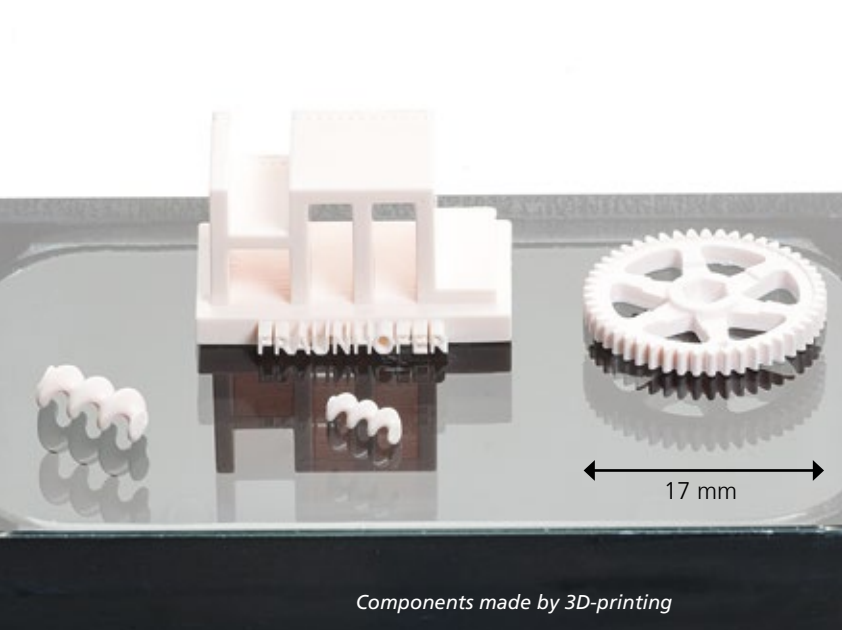
At Fraunhofer-Center HTL, ceramic and metal components as well as composites are developed in a closed process chain from component design and material design to pilot plant scale production. Technological focus is the production of lightweight components made of ceramic matrix composites CMC; but also 3D printing processes are available for the production of metal and ceramic components with complex geometries.

Ceramic fibers mark the beginning of the production chain of CMC. Fraunhofer-Center HTL develops oxide and non-oxide ceramic fibers which are produced in pilot plant scale starting with precursor synthesis, followed by the spinning process and ending with the pyrolysis and the coating of filaments. The fibers are processed by textile manufacturing techniques to preforms, then embedded in a matrix and converted to CMC components by a heat treatment.

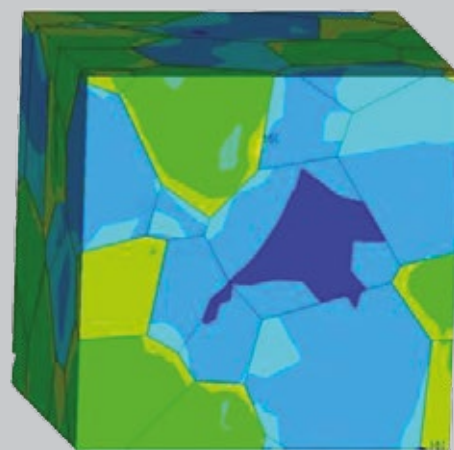
Thermooptical measuring (TOM) furnaces are developed at Fraunhofer-Center HTL for testing high temperature materials and optimizing manufacturing processes. They are used, for example for the optimization of debinding and sintering processes. Materials and components can also be characterized with different non-destructive or mechanical and thermal testing methods. Fraunhofer-Center HTL has numerous computer programs at its disposal for the design of materials, components and processes – from in-house-software via material databases up to commercially available finite element simulation tools.



Fraunhofer-Center HTL is  
certified acc. to ISO 9001:2008



Components made by 3D-printing



Computer simulation of the thermal stresses in a zirconia/alumina ceramics



Spools with continuous non-oxide ceramic fibers



Fiber pilot plant with fiber drawing tower

## HIGH TEMPERATURE MATERIALS

### OUR EXPERTISE

Fraunhofer-Center HTL has the entire production chain to develop technical ceramics. Starting with raw material conditioning via forming and heat treatment up to finishing, oxide, non-oxide and silicate ceramics can be produced. Likewise metal ceramic composites, carbon and metal components can be manufactured. A special feature is additive manufacturing. It can be realized both based on slurries and as powder bed process. Thus, ceramic, metallic and metal-ceramic components can be produced with complex geometries.

Material development starts with the selection of suitable mineral phases. Extensive material- and thermodynamic databases are available. Using special developed in-house software, material properties of multiphase – also porous – ceramics or composites can precisely be predicted. By that the microstructure of materials is optimized. Component design is done by means of the finite element method. In particular, thermomechanical loads during the application of the components are minimized.

For forming – in addition to 3D-printing – customary methods as cold isostatic pressing, extrusion, slip casting or wet pressing are available. Raw materials can be milled, dispersed, mixed and homogenized with different techniques. Particle size- and zeta potential analyzers as well as rheometers are used for process related analyses. The quality of green bodies is examined by special in-house procedures. The heat treatment of the green body is carried out in various electric or gas-fired furnaces. During the process, inert, oxide or reducing atmospheres as well as vacuum and overpressure can be realized. A well-aimed optimization of the parameters during heat treatment is carried out with special thermo-optical methods (see section „Thermal Processes“). For finishing, a computer controlled 5-axis machining center is

provided. Components with complex geometries can be constructed with high temperature joining techniques from simple parts. Material and component characteristics are evaluated with non-destructive methods, material testing and microstructure analysis (see section „Characterization“).

### OUR SERVICES

#### Material Selection and Microstructure-/ Component Design

- Identifying suitable ceramics for customer-specific needs
- Design of components for complex thermomechanical stresses
- Identifying optimal microstructures for customer specific requirements

#### Material and prototype development

- Green body production as well as green body evaluation
- Optimization of heat treatment processes
- Materials and component testing

## CERAMIC FIBERS AND MATRICES

### OUR EXPERTISE

Fraunhofer-Center HTL develops organic silicon precursors and pre-ceramic fibers together with suitable fiber coatings, and manufactures fibers on a pilot scale. Material tests are carried out on fibers and micro composites.

Ceramic fibers are developed according to customer-specific requirements. This includes chemical synthesis of precursors, fiber spinning and the subsequent processing steps (coatings, pyrolysis). The main fiber spinning techniques are melt spinning (solid sol-gel precursors and preceramic polymers) and dry spinning (preceramic polymer solutions and liquid sol-gel precursors).

We develop novel coating precursors and optimize existing systems for our customers. The purpose of coating ceramic fibers is to set a fiber/matrix interface that enables composite materials to behave in a damage-tolerant manner. A further objective is to protect ceramic fibers against corrosive attacks. Two different approaches are used for applying coatings: chemical vapor deposition (CVD) and wet chemical deposition of layers.

Customized inorganic-organic hybrid polymers in the Si-C and Si-B-N-C material systems can also be modified for other fields of application, e.g. for high temperature coatings. To this end, along with chemical laboratories we have pilot plant facilities at our disposal where polymers can be manufactured at a scale of up to 50 kg per batch.

### OUR SERVICES

#### Ceramic fibers

Ceramic fiber sampling from laboratory scale to small-scale series:

- Various spinning techniques such as melt spinning and dry spinning
- Oxide and non-oxide ceramic fibers
- As individual fibers or as roving with up to 1000 filaments

#### Ceramic coatings

- For metals, bulk ceramics and glass
- On ceramic filaments and fabrics
- As corrosion protection or oxidation protection and for setting the fiber/matrix interface when using CMC
- Coatings applied primarily by low-cost wet chemical means
- As varnishes or already applied; and if required: Given thermal refinement as ceramic layer

#### Matrix materials

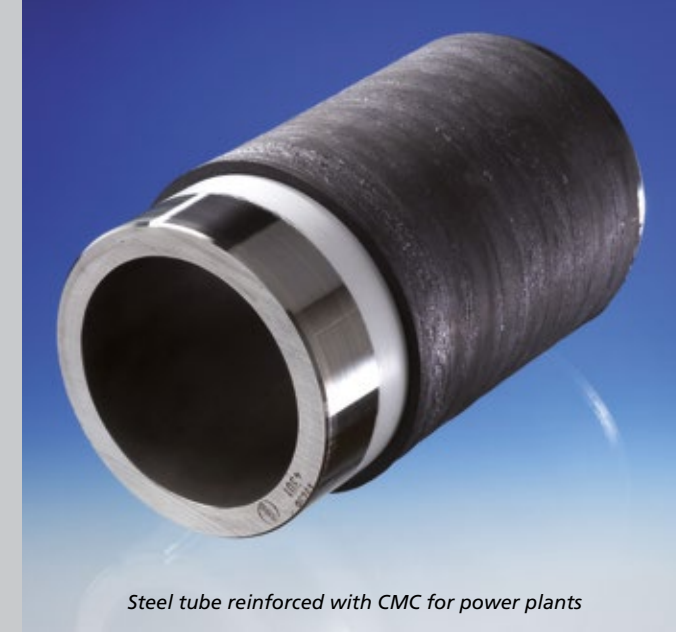
- For building up oxide and non-oxide CMC
- Available for non-oxide CMC based on preceramic precursors and as thermoplastic polymer or polymer solution
- For oxide CMC based on aqueous sol-gel precursors or on aqueous suspensions of ceramic powders



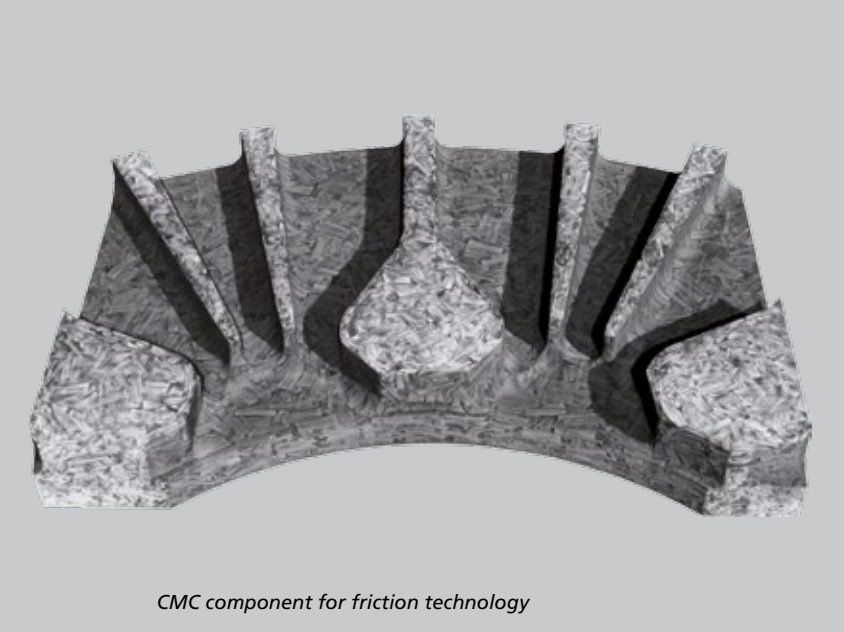
Multifilament yarn on a feeder



Nextel weft yarn processed on a Dornier rapier weaving machine



Steel tube reinforced with CMC for power plants



CMC component for friction technology

## TEXTILE FIBER PROCESSING

### OUR EXPERTISE

The application center for Textile Fiber Ceramics TFK in Münchenberg is a joint venture with Hof University of Applied Sciences. The application center TFK uses the existing competences and technical facilities in the field of textile fiber processing and testing and transfers textile processing techniques to ceramic fibers.

Together with the Fraunhofer-center HTL, projects and services are feasible for developing ceramic fiber composites covering all process steps from the fiber to the finished CMC-part. This continuous process chain is supposed to address national and international companies from all sectors, from material production to material application.

During the last decade the processing of textile fibers into 2D and 3D structures developed rapidly by the implementation of new technologies. Those innovative production methods are now transferred to inorganic fibers to exploit new applications. The biggest challenges for commercial success are the high costs of the fibers and of the processing steps. The application center TFK is working on the development of methods which are cost-effective and capable of series production to be used for processing inorganic fibers to load-conform 2D and 3D structures.

We offer a range of services according to market requirements and customer requests. Our aim is to successfully perform research activities in close collaboration with companies.

### OUR SERVICES

The application center for Textile Fiber Ceramics TFK is able to examine, test or process ceramic fibers such as SiC- and Al<sub>2</sub>O<sub>3</sub>- as well as carbon-, glass- and basalt fibers for a huge variety of purposes.

Focused dialogues identify the customers' requirements and objectives and define the common approach. Depending on the requirements, the customer receives an examination report and / or a presentation of the results including its interpretation. Further options include the development of customer-specific solutions and the initiation of R&D-projects.

In addition, we provide training courses and seminars about textile processing methods.

#### Textile Testing Methods

- Linear density according to DIN EN 1007-2
- Filament diameter according to DIN EN 1007-3
- Determination of tensile properties of filaments at ambient temperature according to DIN EN 1007-4
- Determination of the tensile properties of fibers within a multifilament tow at ambient temperature according to DIN EN 1007-5

#### Technical Facilities

In our technical facilities we provide the equipment of traditional textile production processes such as weaving, braiding, warp and weft knitting as well as nonwoven technology for development projects.

## CMC COMPONENTS

### OUR EXPERTISE

At Fraunhofer-Center HTL, components made from ceramic matrix composites (CMC) are developed in a closed process chain from component design and material design to pilot-scale production. CMC materials are distinguished from monolithic ceramics by virtue of their significantly higher fracture toughness, which results in damage-tolerant component behavior.

At the component design stage, we carry out the simulation and dimensioning of structures made from fiber-reinforced ceramics. We come up with a low-strain design by simulating mechanical and thermal loading conditions and give you a proposal for a prototype geometry. When using hybrid constructions ceramic components can be combined with metallic components. Using FE modeling, the different expansion behaviors of the materials at high temperatures are taken into account.

On customer's request, we develop application-specific ceramic matrix composites (CMC). This material group comprises carbon-fiber-reinforced carbon, carbon-fiber reinforced SiC (C/SiC), silicon-carbide-fiber-reinforced SiC (SiC/SiC) and oxide-fiber-reinforced oxide ceramics (O-CMC). The desired material properties are attained by selecting suitable starting materials – fibers, matrices and additives – and their spatial arrangement.

We possess the full range of technologies for the manufacture and ceramization of fiber-reinforced green bodies. Statistically reliable characteristic data are obtained from laboratory samples. For manufacturing prototypes, pilot plant facilities are available that permit upscaling to component dimensions up to approx. 700 mm. This makes it possible to elaborate concepts for the series production of components made of CMC. Manufacturing can be expanded to incorporate customer-specific quality

assurance measures, allowing the results and technology to be subsequently transferred to production scale.

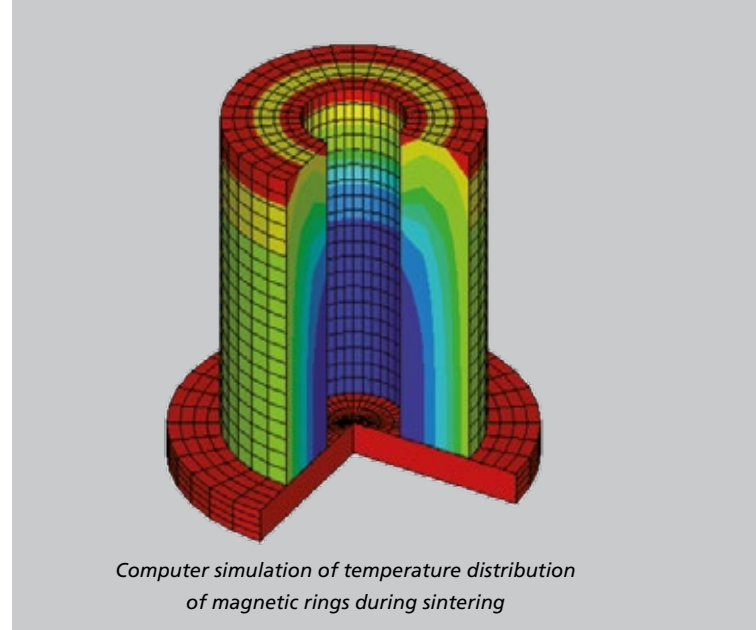
### OUR SERVICES

We provide you with samples of fiber-reinforced ceramic composite components and advise you on making the right choice for your applications. The initial orientation tests permit experimental investigation of whether the application properties can be obtained with available materials. Experience shows that materials have to be adapted to the specific application and optimized in the course of R&D projects.

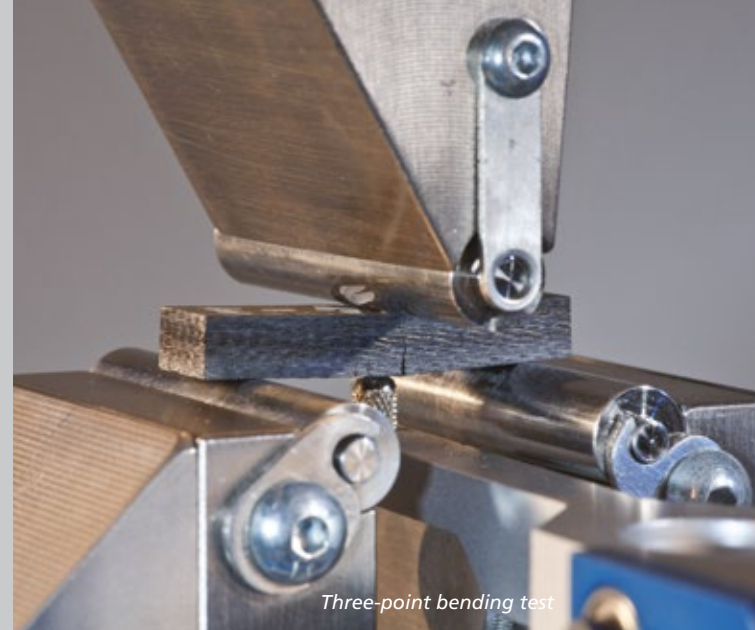
- Advice on selecting appropriate CMC materials
- Providing material samples made of carbon-fiber reinforced carbon (C/C) and oxide and non-oxide CMC for new applications
- Testing application properties
- Coating rovings and fabrics including thermal processing / ceramization
- Cost analysis for the manufacture and quality assurance of CMC components in different amounts and quantities



Thermo-optical measuring device TOM\_air



Computer simulation of temperature distribution of magnetic rings during sintering



Three-point bending test



Furnace system for heat treatments up to 2400 °C

## THERMAL PROCESSES

### OUR EXPERTISE

We optimize the heat treatment processes used in manufacturing ceramics, metals and metal-ceramic composites, such as drying, binder burnout / pyrolysis and sintering or melt infiltration. Thus, time-temperature cycles can be improved as well as the furnace atmosphere or the set-up of the charge in the industrial furnace. Our aim is to receive high and reproducible quality with good material-, energy- and cost efficiency of thermal processes.

Fraunhofer-Center HTL develops thermo-optical measuring furnaces (TOM), in which the industrial heat process is simulated in the laboratory. TOM-devices are able to provide all relevant industrial atmospheres: gas burner atmosphere, air, inert gases, forming gas, hydrogen, vacuum, overpressure, etc. They are equipped with detectors monitoring material changes with high accuracy during the heat treatment. For example dimensional changes can be measured during sintering with extremely good reproducibility or acoustic emission signals can be detected during debinding with sensitive microphones. Also high temperature properties of materials can be measured with TOM-devices.

The data measured are parameterized – in particular the kinetics of thermally activated reactions are described with robust models – and then used in FE simulations to optimize the heat treatment on the computer. In the FE models the interaction between the industrial furnace and the charge is taken into account so that the laboratory results can be transferred to the production scale.

In addition, we offer methods for investigating temperature distribution, furnace atmosphere and heat balance of production furnaces. These data can be displayed in the FE models and used for process optimization as well.

### OUR SERVICES

- In-situ characterization of the behavior of solids and melts during heat treatment
- Analyzing sintering, debinding, melting and infiltration processes
- Measuring dimensional changes (sintering, distortion, expansion)
- Measuring gas-phase reactions (changes in weight, gas emission)
- Thermophysical characterization: Thermal conductivity, creep resistance, emissivity, high temperature strength, high temperature elastic modulus, thermal shock properties
- Characterization of melting: wetting, viscosity
- Simulating heat flow and temperature field during heat treatment
- Developing time-temperature cycles with shorter overall duration (cold/cold)
- Developing heat treatment processes with fewer rejects and output that requires less finishing work
- Developing time-temperature cycles conditions that consume less energy
- Customer specific development of high temperature measuring methods
- Measuring of industrial furnaces: temperature distribution, furnace atmosphere, heat balance

## CHARACTERIZATION

### OUR EXPERTISE

We measure the composition, microstructure and properties of high-performance ceramics and composites. If required, we develop application-specific characterization methods and give customers advice on potential improvements to processes.

The focus of testing at Fraunhofer-Center HTL lies in non-destructive as well as mechanical and thermal testing methods. Samples and components are examined non-destructively by means of computerized tomography (CT) measuring up to 700 mm in diameter or achieving resolutions down to 3 µm. CT is completed by ultrasound and terahertz wave imaging and thermography. For mechanical testing all common test methods are available. The thermal tests are carried out by means of standard methods or with TOM-furnaces (see section „Thermal Processes“). Our cooperation with the Center for Applied Analytics at Fraunhofer ISC in the German town of Würzburg gives us access to numerous other measurement techniques in addition to the methods we have on site. This allows us to take a problem-centered approach, whereby we use the most suitable characterization methods for the job at hand.

### OUR SERVICES

- Non-destructive testing of materials and components
- Measuring thermal and mechanical material properties
- Qualitative and quantitative microstructural analysis
- Chemical and elementary analyses
- Density measurements
- Dimensional and damage analyses of components
- Consulting, carrying out studies

## TEST FIRING

### OUR EXPERTISE

Our pilot plant facilities contain various furnaces for the heat treatment of oxide and non-oxide ceramic materials and components. The following processes can be carried out in different atmospheres and optimized in close cooperation with the customer: debinding, pyrolysis, graphitization, melt infiltration (particularly with silicon) and sintering. Various furnaces are connected to a thermal afterburner system, making it possible to carry out processes that produce large volumes of condensates.

### OUR SERVICES

- Assessing and carrying out test and contract firing according to customer specifications from room temperature to 2400°C
- Defined atmospheres such as vacuum, inert gas, hydrogen and air
- Furnace utilization volumes from 1 liter up to 385 liters; suitable for manufacturing large individual components or small-scale series
- Possible to manufacture components up to a maximum size of 800 x 800 x 600 mm<sup>3</sup> via sintering and pyrolysis
- Optimizing manufacturing processes to obtain ideal material properties
- Test firings in a rotary kiln up to 1100°C
- Process documentation and final inspection of components according to the customer's request