ZEISS Microscopy Solutions for Industrial Ceramics Research
2D, 3D and 4D Solutions to Engineer New Advanced Ceramics for High Performance Industrial Applications
Designing High Performance Advanced Ceramics from Nanoparticles to Finished Goods

Analytical solutions to push the boundaries of engineered ceramic material properties

Ceramics have become the material of choice for a wide range of high performance applications thanks to the ability to design their macroscopic properties and combine these with modern processing techniques. In today’s fast moving environment, increasing the efficiency of products, meeting challenging customer demands, and reducing production costs while keeping ahead of the competition can be a challenge without an analytical approach. To drive innovation, a material’s microstructure and its interaction with the environment must be characterized in situ, in multi dimensions and using different modalities, taking advantage of modern microscopy solutions.

ZEISS’ powerful portfolio of 2D analytical characterization technologies combined with 3D non-destructive imaging and in situ capabilities, equips researchers with an unparalleled toolkit to pioneer solutions and further advance ceramics research and development. Combining innovative software packages for modeling, simulation and artificial intelligence, ZEISS solutions for microscopy unlock the power to engineer new ceramic material properties for the 21st century and beyond.
Today’s modern building materials are designed to produce significantly greener, more efficient building structures which have at least some ability to self clean or heal. The design of new cements and glasses for the building industry is focused on research into expanding the capabilities of these raw materials to address future needs of industry whilst increasing strength, durability and optimizing thermal insulating properties.

As structures weather over time, their strength can also be affected by chemical reactions which are governed by 3D structure as well as availability of certain elements in the building matrix. In spite of the newest development and improvements in materials, failures still occur, causing financial losses. Understanding the nature of these failures may be very difficult using traditional approaches.

The combination of non destructive 3D X-Ray microscopy with analytical and environmental microscopy in the SEM provides a unique combination of investigative tools for the civil engineering researcher. In parallel, automated mineral identification, in both the optical and electron microscope, allows for the spatial mapping of mineralogy in samples and can be used in the determination of sulfate attack processes.
Nanoparticles

Ceramic nanoparticles have a wide range of industrial applications, from additive manufacturing, catalyst research, drug-delivery/medical, highly functional devices and batteries.

The synthesis of ceramic nanoparticles is controlled by a number of interacting parameters which must be carefully optimized to ensure uniformity of chemistry, size, agglomeration and morphology of the desired resultant nanoparticle population. In order to determine if this has been successful, nanometer resolution surface imaging and chemical mapping is required.

Since chemical variations can occur over the nanoscale, only the combination of low kV EDS analysis and magnetic field free, high resolution SEM imaging provides this capability. This technique allows researchers to analyze versatile samples without any sputter coating or further sample preparation, to increase confidence and save research time.

Images were taken on unprepared/uncoated samples and no sputter coating was required.
Solid oxide fuel cells (SOFC) and solid oxide electrolysis cells (SOEC) represent more environmentally friendly solutions for energy production than combustion engines. Because of their increased efficiency and low CO₂ emissions, there is a global effort by companies (residential heating, automotive, power plants, ceramics, thermal engineering) and governments to actively focus on the implementation, development and commercialization of these technologies.

However, the commercialization is still challenging due to the increased cost of production. In order to overcome this challenge, lower cost, more reliable or less materials with increased performance need to be developed.

ZEISS’ complete portfolio offers solutions to develop SOFC and SOEC from ceramic particles to components, in both 2D and 3D. By analysing microstructure, interfaces and interlayers, voids, porosity, tortuosity, diffusion and degradation mechanisms, the researcher is equipped to better understand operational performance and design requirements.

3D Chemical analysis to study aging of SOEC.
High temperature operation, extreme wear and corrosion resistance as well as high strength and machinability make technical ceramics ideal materials for modern applications which operate at the edge of the performance envelope required by modern technologies.

By engineering microstructural properties of these materials, a blend of performance characteristics can be achieved and applied to substrates or as whole components to enable optimum operation of materials.

ZEISS’ portfolio is an analytical powerhouse designed to characterize new ceramic materials to understand their chemistry, grain size and porosity. In combination with in situ mechanical testing rigs, thermal, electrical and mechanical properties can also be analyzed at the nanoscale and correlated to microstructural design criteria.

By characterizing new technical ceramics at the nanoscale with analytical microscopy, industry gives itself an advantage when developing new materials.
Ceramic matrix composites (SiC/SiC, oxides/oxides or carbon/carbon) are materials in high demand in automotive, aerospace and defence, biomedical and nuclear industries, due to their lightweight, high-performance (wear resistance, thermal shock resistance, etc.) and non-conductive properties.

By improving the design of these materials and by understanding their failure mechanisms and in operando behavior, the operational envelope of composites can be increased.

ZEISS offers a complete portfolio of microscopy solutions to evaluate the design of composites, to study mechanical properties in situ (nano-indentation, tension and compression testing), microstructure, fracture propagation and behavior, and to non-destructively evaluate internal structures (3D porosity or fiber orientation).

By using advanced 3D image segmentation tools, correlative microscopy, and multi-modal analysis (chemical identification or mechanical property predictions), an up scaled analysis can be performed to get more insights into the performance of composites. Detailed microstructural analysis enables macro scaled properties to be designed from first principles.

Fluorescence image of a cross section of a carbon fiber in a hockey stick composite sample.

Non-destructive 3D analysis of fiber composite sample, showing both glass and carbon fibers, as well as defects and inclusions.

Von Mises strain analysis in a hockey stick composite sample, performed using correlative microscopy. Physical properties measured using GeoDict from Math2Market simulation engine.

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Youngs Modulus E (GPa)</td>
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<tr>
<td>Poisson Ratio v</td>
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<tr>
<td>Shear Modulus G (GPa)</td>
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</tr>
<tr>
<td>Lamé Modulus (GPa)</td>
<td>6.5</td>
</tr>
<tr>
<td>Bulk Modulus K (GPa)</td>
<td>11.0</td>
</tr>
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Technical Glass

Highly engineered technical glass has seen a renaissance in development recently arising from both the development of smart windows (self cleaning or reduced heat transfer), their use in modern tactile electronics such as smart phones, optics and telecommunications and space exploration. Requirements on new devices are for increased strength and scratch resistance, surface finish, chemical inertness and integration with touch sensitive and flexible electronic devices.

By applying transparent ceramic coatings, glass is being routinely used as the interface between regions of widely varying temperature and chemical environment. Recyclable glass, thinner but stronger glass and the integration of multiple functionalities in glass can only be achieved using research and development.

ZEISS correlative solutions enable a seamless integration of data from optical, electron and X-ray microscopy. The cause of defects can be identified by analyzing the same region of interest using correlative microscopy. Porosity and voids can be quantified by nondestructive interior tomography and substrates, coatings and interfaces can be analyzed using focused-ion beam cross sectioning.

Void segmentation in glass using non-destructive 3D tomography. Sample: courtesy of SCHOTT AG.

Fracture details on non-conductive foam glass used as an insulator. Image taken on unprepared / uncoated samples and no sputter coating was required. Sample: courtesy of Martin Bonderup Østergaard, Dr. Rasmus R. Petersen and Prof. Yuanzheng Yue from Aalborg University, and Dr. Jakob König from the Jozef Stefan Institute.

Non-destructive 3D reconstruction of foam glass used as an insulator, in low (green), medium (red) and high (blue) resolution. Sample: courtesy of Martin Bonderup Østergaard, Dr. Rasmus R. Petersen and Prof. Yuanzheng Yue from Aalborg University, and Dr. Jakob König from the Jozef Stefan Institute.
Increased biocompatibility, excellent toughness and strength, chemical inertness and advances in the manufacturing of ceramic materials makes them ideal candidates for biomedical applications such as teeth, hip and joint implants, glass beads for drug delivery and medical equipment.

3D non destructive imaging and 4D mechanical experiments enable the testing of devices such as dental implants and provides researchers with confidence in the lifetime of their products.

ZEISS Xradia Ultra’s 4D nano-indentation module allows researchers to observe and analyze impacts associated with use of the implant during its life in the human body at a scale of 50 nm.

Combine 2D analysis using scanning electron microscopy to study surface finish and wear, 3D confocal laser scanning electron microscopy for roughness measurement with 4D experimentation to obtain the complete analysis of your ceramic material.
Solution Spotlight
Correlative microscopy and machine learning

ZEISS Shuttle & Find is a correlative microscopy interface for light and electron microscopy, designed to provide new insights and enhance productivity in industrial ceramics research.

Identify cause of defects and analyse aging in energy materials by fast relocation of region of interest and automated calibration and work routines. Achieve reproducible results faster and save research time.

Once the correlative data has been acquired, it can be overlaid and simultaneously segmented using the Atlas 5 correlative workspace and ZEN Intellesis machine learning segmentation software.

Light and electron microscopy are two technologies that ideally complement each other. You can now combine them to gain new insights and enhance productivity. ZEISS as the world’s only manufacturer of both light and electron microscopes in all performance classes now provides a bridge between both worlds. Benefit from easy sample transfer, fast relocation of the region of interest and precise image correlation. Gain short time-to-result and maximum information from your samples.

Correlative and multi-modal analysis of layer structure (cathode, separator and anode) of an aged Li-ion battery: (A) brightfield, (B) polarized light microscopy, (C) backscattered SEM signal and (D) chemical analysis using EDS. Courtesy: Aalen University, Materials Research Institute, Aalen, Germany.
Analyze grain size automatically and precisely with the ZEISS ZEN Grains module, on both EM and LM images.

Better understand processing of advanced ceramics, such as sintering, and improve material performance by achieving a homogeneous microstructure. Identify large grains that are of critical importance and observe grain growth.

Three measurement modes are available: comparative diagrams, a purely interactive method, the semi-automatic intercept method and the fully automatic method, which reconstructs grain boundaries and calculates individual grain sizes.

Avoid human bias, obtain accurate and repeatable results, and automatically generate reports, following ASTM E112 or other international standards.

Original image (left) and analyzed image (right) of FE-SEM microstructure of additively manufactured ceramic sample. Image was taken of unprepared/uncoated sample and no sputter coating was required. Sample courtesy of Lithoz GmbH.

Grain size distribution

Grain size [µm²]
Improve surface quality of finished ceramic goods, optimize polishing processes and prepare surfaces for coatings by characterizing 3D micro structures and surfaces using the LSM 800 confocal laser scanning microscope.

Sensitive surfaces can be analysed using non contact roughness and topography measurement. Upgrade our leading ZEISS Axio Imager 2 microscope with LSM 800 to combine all essential light microscopy contrasting methods for ceramics research with high precision topography in a single instrument.

The powerful ZEISS Confomap software generates clear images, height maps and produces reports compliant to the relevant standards.

**Solution Spotlight**

**Roughness analysis**

Non contact topography reconstruction of a ceramic part.
Solution Spotlight
Pores, voids and phase analysis

Design desired microstructure and flawless ceramic parts by analysing pores, voids and phases in both 2D and 3D using the ZEISS ZEN Intellesis powerful machine learning segmentation tool.

Process and analyse images from different microscope modalities, beyond the traditional greyscale algorithms. Identify different textures or segment coloured images in four simple steps.

ZEISS ZEN Intellesis provides an additional tool for industrial ceramics researchers interested in getting more insights from their data: a data-agnostic machine learning system that can be used alone or in conjunction with other software platforms. ZEN Intellesis has the capability to segment large sets of single or multi-channel data generated using any microscopy method such as light, electron, X-ray microscopy, EDS in both 2D and 3D by applying the pre-trained model.
Solution Spotlight
Coatings analysis

Analyse multi-layers or coatings and determine correlation between thickness, porosity and performance to optimize process parameters and reduce material cost.

ZEISS ZEN Layer Thickness module provides a convenient assisted workflow, either in automatic or interactive mode. Measurements of layer thickness can then be reported.

ZEISS Xradia Versa X-ray microscopes provide nondestructive 3D characterization and quantitative analysis of thermal barrier coatings to extract volumetric and quantitative information of key features of interest, such as layer interface, volume percentage, pore morphology, voids and microcracks.

<table>
<thead>
<tr>
<th>Description</th>
<th>Distance (µm)</th>
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<tbody>
<tr>
<td>Minimum</td>
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</tr>
<tr>
<td>Maximum</td>
<td>17.11</td>
</tr>
<tr>
<td>Mean</td>
<td>15.14</td>
</tr>
<tr>
<td>Standard Dev.</td>
<td>1.26</td>
</tr>
<tr>
<td>Range</td>
<td>3.87</td>
</tr>
</tbody>
</table>

3D surface color-rendered image of a thermal barrier coating system showing internal voids and cracks.

Thermal barrier coating analysis with measuring chord.

Thermal barrier coating analysis with polarized light contrast.

Thermal barrier coating porosity measurement with brightfield contrast.

500 µm
ZEISS Products for Ceramics Research

- ZEISS Axio Imager 2
  - An industrial research workhorse, offering fast, reproducible results from tailored and automated workflows. Class-leading optics and contrast modes, full motorization, the flexibility of a modular design and an emphasis on usability.

- ZEISS EVO scanning electron microscope
  - Your interactive SEM for high productivity, intuitive and easy to use in a multiuser environment, the new EVO is the versatile analytical platform for both wet imaging and research applications.

- ZEISS SIGMA field emission scanning electron microscope
  - Sigma is the advanced analytical microscopy workhorse with usability and flexibility in market-leading detection technology, high resolution imaging even on non-conductive samples, and the proven Gemini column, giving fast crystallographic, chemical, textural and other analytical data streams.

- ZEISS Crossbeam FIB-SEM
  - High throughput 3D analysis and TEM sample preparation. Integrated imaging and analytical solution for near surface regions, failure mechanisms and 3D crystallographic investigations of ceramic samples.

- ZEISS Gemini SEM
  - Our flagship field emission system for the highest demands in ceramics characterization with sub nanometer resolution, high speed and surface sensitivity. With ease of use and stability by design, making fast analytical microscopy possible even at low kV, to analyze non-conductive samples without sputter coating.

- ZEISS Xradia Versa 3D X-ray microscope
  - High resolution, non-destructive imaging with in situ capabilities, designed to add the 4th dimension in ceramics research.

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ZEISS LSM 800 confocal laser scanning microscope
Our most advanced confocal laser scanning microscope combines all essential light microscopy techniques with high precision surface topography and texture analysis, using the advanced Confomap image analysis software.

ZEISS Axio Observer
Your open and flexible inverted microscope platform, offering all the capability, optics and imaging modes of the Axio Imager, with the capability to handle even large samples with easier sample preparation.

ZEISS Xradia Ultra Nanoscale X-ray Microscope
Your Synchrotron-quality nanoscale microscope performs non-destructive 3D imaging down to 50 nm resolution. Xradia Ultra Load Stage enables in situ nanomechanical, compression, tension and indentation experiments.

ZEISS Atlas 5 correlative microscopy workspace
Correlative microscopy for multi-scale, multi-modal, multi-dimensional imaging and analysis. Blended learning workspace enables remote control of instruments and collaboration across geographies and imaging modalities.

ZEISS Mineralogic Mining
Automated mineralogy software for building materials. Chemical and mineralogical analysis to provide insights into mineral distribution and thaumasite attack.
ZEISS Products for Ceramics Research

- **Stereo LM**: 1 µm
- **Sub-micron XRM**: 700 nm
- **Widefield LM**: 250 nm
- **Polarized LM**: 200 nm
- **Confocal LM**: 200 nm
- **Nanoscale XRM**: <50 nm
- **C-SEM**: <2 nm
- **FE-SEM**: <1 nm
- **FIB-SEM**: <1 nm
- **Helium Ion Microscope**: <0.5 nm

The most advanced technology for the highest quality data.