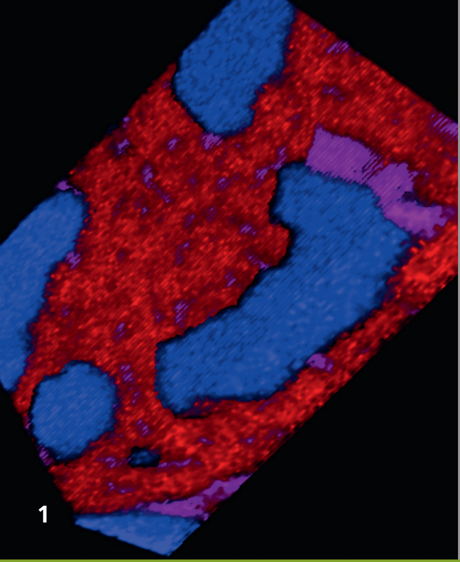

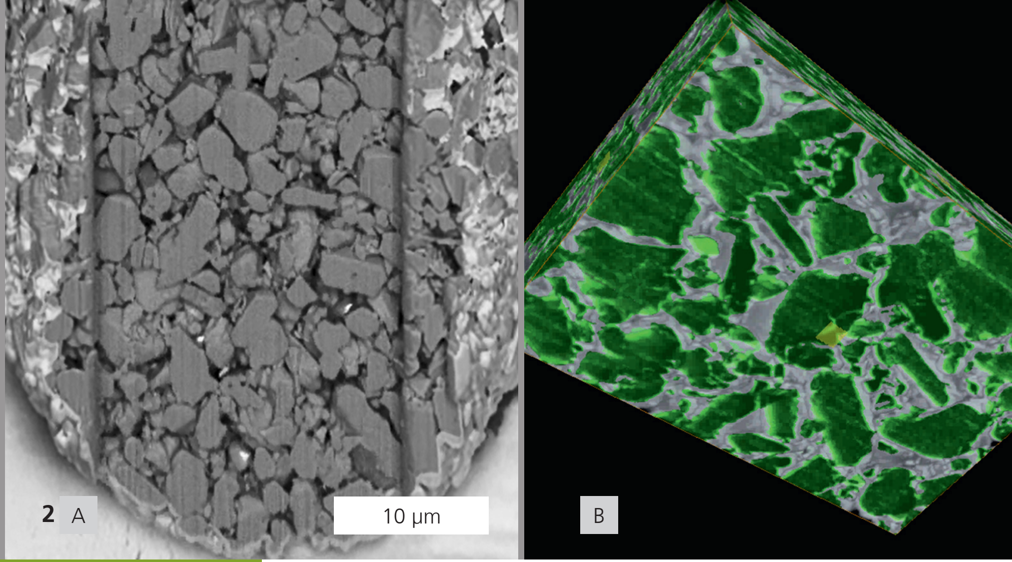

HIGH-RESOLUTION THREE-DIMENSIONAL CHARACTERIZATION OF CERAMIC MATERIALS

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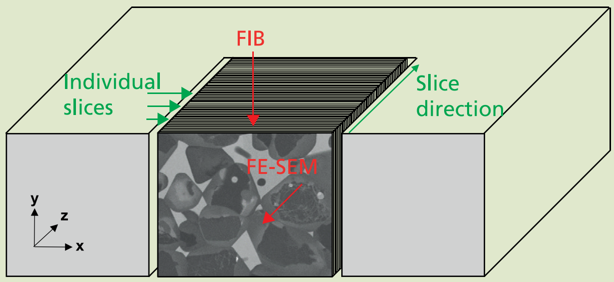
Development and optimization of high-performance materials hinge on the availability of high-resolution analysis methods. For the majority of samples, conventional two-dimensional images of cross-sections provide limited information about shape, stereoscopic layout, and character of individual components. The three-dimensional representation of structures and defects yields additional information about expected material properties. As an example demonstrating the scientific validity of this method, computed tomographic measurements were performed on ceramic foams. The microstructure was geometrically characterized and material data for the component construction were derived from the results. For high-performance ceramic materials, in which structural sizes are in the submicron and nanometer ranges, the lateral resolution of conventional computed tomography is generally not sufficient.

At Fraunhofer IKTS, two techniques, focused ion beam (FIB) tomography and X-ray nanotomography, are established for three-dimensional structural analysis down to the nanoscale.

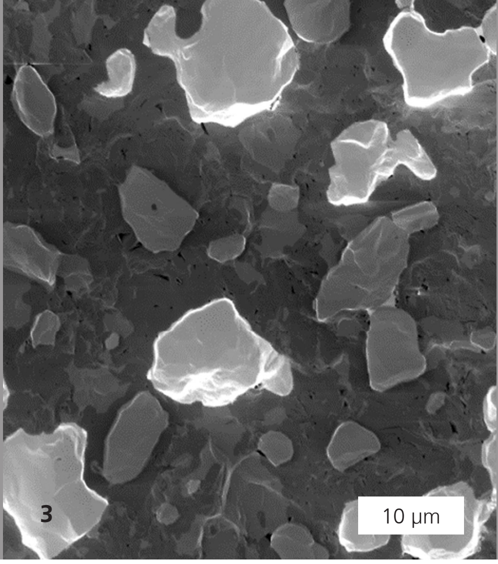
FIB tomography is based on the preparation of a series of slices using a focused ion beam (FIB) and a high-resolution, high-contrast image from a field-emission scanning electron microscope (FE-SEM). With this method, structures can be displayed up to a lateral resolution of about 10 nm (scheme on the right- hand side). Suitable 3D software can produce a volume data set by combining several individual cross-sectional images. In combination with energy-dispersive X-ray analysis (EDS), additional three-dimensional element distribution images can be created, as shown in Figure 1 for a $MoSi_2/Si_3N_4$ composite material. A titanium diboride/boron nitride (TiB_2/BN) composite material was used to demonstrate the capabilities of the FIB tomography technique.

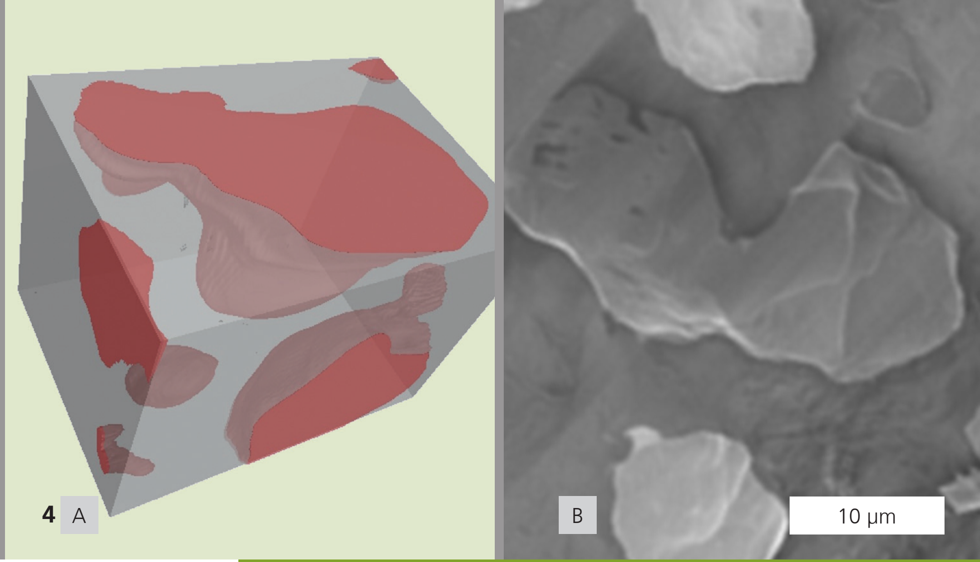
Principle of FIB tomography



This composite material is usually used for evaporation boats, e.g., for evaporation of aluminum. The boron nitride content in this material generates good thermomechanical properties. The electrical conductivity, which is needed for the direct heating, is provided by TiB_2 particles. In order to achieve reproducible conductivity, the TiB_2 needs to form a three-dimensional network. If the network is disturbed by local inhomogeneities or by aging, uniform heating of the material is not possible. In order to understand the performance of the material over time, it is important to know the distribution of the TiB_2 phase. A prerequisite for efficient material design is appropriate three-dimensional characterization. This was achieved using field-emission scanning electron microscopy.

By choosing the right imaging conditions, i.e., by using the in-lens detector, it was possible to differentiate between percolated and isolated TiB_2 grains (Figure 3). The electrically conducting percolated TiB_2 phase appeared bright in the FE-SEM

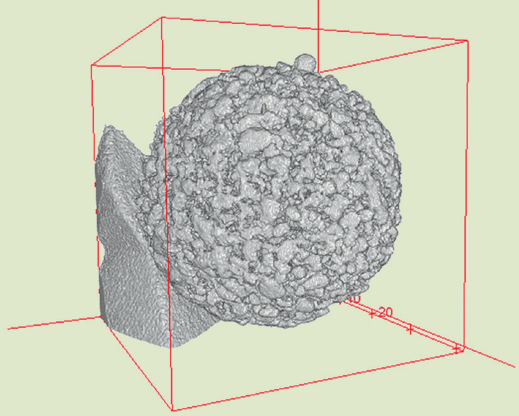




image, whereas the non-conducting isolated particles were dark. A three-dimensional representation of the TiB_2 network was produced using FIB tomography. The three-dimensional slicing technique (Figure 4A) confirmed the assumption that dark TiB_2 particles are not incorporated in the three-dimensional network. This technique verified the results that were obtained by two-dimensional FE-SEM imaging (Figure 4B).

Fraunhofer IKTS has established X-ray nanotomography as a modern non-destructive method for the analysis of structures and defects in ceramic materials. The method permits the non-destructive investigation of structural and functional materials at a microscopic level with a resolution down to 50 nm. If the X-ray absorption contrast between the components of a material is too low, contrast enhancement is achieved through Zernike phase contrast. This accentuates interfaces and surfaces as well as delamination and cracks. Through use of in-situ test stages, various experiments can be carried out under direct observation. This enables the recording of four-dimensional data sets in the X-ray microscope to supplement the three-dimensional information. With miniaturized thermal and mechanical equipment, which are positioned in the beam path of the X-ray microscope, experiments can be performed on a microscopic level and their effects can be observed. The 3D figure on the left-hand side shows the three-dimensional visualizations of an Al_2O_3 granule recorded by X-ray nanotomography. A FIB tomography generated structure belonging to the same granule batch is shown in Figure 2A and 2B.

3D visualization of the micro-structure of an Al_2O_3 granule by X-ray nanotomography



Services offered

- Generation of high-resolution 3D data sets for 3D micro-structural analysis composition (EDS) and failure analysis
- High-resolution 2D and 3D X-ray microscopy with a pixel resolution of 32 nm
- Investigation of kinetic processes, in-situ experiments: temperature chamber, chemical reaction chamber, mechanical tests
- High-contrast imaging with various detection methods
- Recording and reconstruction of 3D and 4D data sets (tomography, laminography, time-lapse imaging, and time-resolved tomography)
- Data evaluation, segmentation
- Characterization of devices and materials

1 *3D-EDS of a $MoSi_2/Si_3N_4$ sample (magenta: Yb, red: N, blue: Mo).*

2 *3D visualization of the micro-structure of an Al_2O_3 granule by FIB tomography (A) and reconstructed 3D volume (B).*

3 *FE-SEM image of BN/TiB_2 composite material recorded by in-lens detector.*

4 *Reconstructed 3D volume (A) of an isolated TiB_2 particle (B) in the BN matrix.*

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