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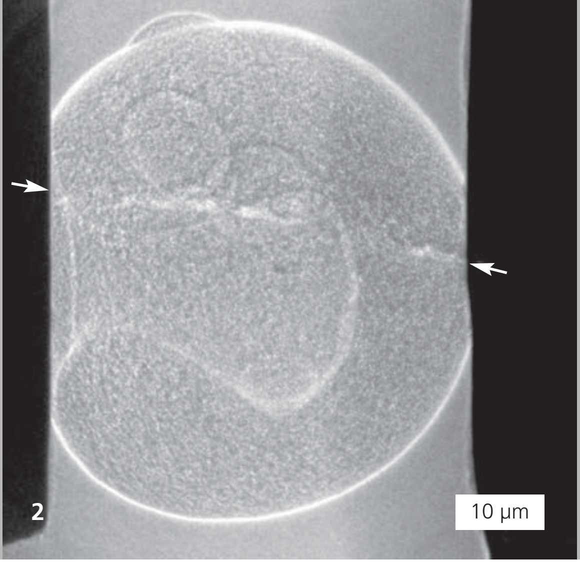
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X-RAY MICROSCOPY FOR MATERIALS RESEARCH, ENERGY MANAGEMENT, MICROELECTRONICS

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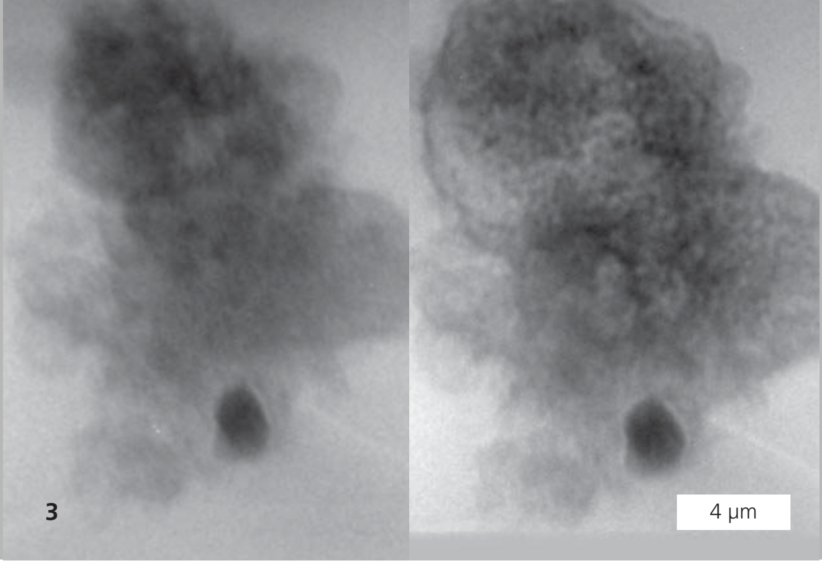
X-ray microscopy is a modern and non-destructive method, which is increasingly used for high-resolution analysis of structures and defects in materials as well as biological objects. The applications range from materials science, energy management, geological science and microelectronics to biology and medicine.

Today's laboratory-based x-ray microscopes (Figure 1) mostly use Fresnel zone plates as focusing optics, reaching a resolution of approximately 50 nm. Thereby, the x-ray microscopy fills the gap between visible light microscopy and electron microscopy. The x-ray computed tomography (CT) reconstructs a three-dimensional representation of an object from multiple projection images taken from different directions. Virtual cross sections through the reconstructed volume are applicable to show buried structures and material components (e.g. in composite materials), as well as to discover defects in materials and devices, like inclusions, pores and cracks. Furthermore, it is possible to analyze the sub-structure of biological tissue.

In materials science, x-ray microscopy is used for both the investigation of structural as well as functional materials. The three-dimensional microstructure, the morphology and topology of microstructure components, such as precipitations, pores and – in the case of composite materials – fibers and particles are analyzed by nano x-ray CT. If the x-ray absorption contrast or the differences between structural constituents is marginal, it is possible to capture high-contrast images by utilizing the so-called Zernike phase contrast. It emphasizes not only interfaces and surfaces, but also delamination and cracks. This contrast mechanism allows to image, for example, high-strength oxide ceramic fibers inside a matrix of the same material. With the help of nano x-ray CT, it is possible to visualize the arrangement of fibers inside the digital volume model of the sample matrix. These data are used as input for simulations to model mechanical properties of modern high-performance ceramics and optimize them through improved materials design. Likewise, the method enables the localization of cracks and delaminations after loading composite materials mechanically. By using in-situ test devices, experiments can be carried out inside the x-ray microscope under observation in order to extract 4D information in addition to 3D data sets. A miniaturized double cantilever beam (DCB) test, i.e. a piezo-driven mechanical test device, which is positioned inside the beam path, allows to adjust defined small values of mechanical strain and observe the effects. Figure 2 shows a spray-dried ceramic granule, made of aluminum oxide, between the two flat jaws of the DCB device under mechanical load, simulating the process of compacting a green body. If a certain load is reached, the granule fractures and gets compressed. Radiography with phase contrast does not only show the inner structure – e.g. in Figure 2 this granule is hollow – but also the cracks (arrows) and inclusions of impurities if present.

Among others, representative applications for x-ray microscopy are found in the research and development of energy storage materials and processes. To study kinetic processes, it is necessary to place and operate miniaturized reaction chambers inside the x-ray microscope. Such a microreaction chamber is able to run chemical processes at temperatures of up to 700 °C and under inert or reactive atmosphere at standard pressure, so that innovative processes for hydrogen storage can be analyzed. The steam iron process stores hydrogen, operates at low pressure and does not require rare noble metals. Therefore, it is a potential technology for decentralized energy storage management. The morphological change in the iron/iron oxide powder during the cyclic oxidation and reduction reaction influence the lifetime and the storage capacity of the storage material. If nanosized iron powders are used, the process temperature can be lowered due to the high surface area and therefore, the high reactivity. The oxidation reaction of very fine iron powders with particle sizes below 100 nm was investigated by in-situ experiments with temperatures of up to 500 °C in an inert atmosphere laden with water vapor. The formation



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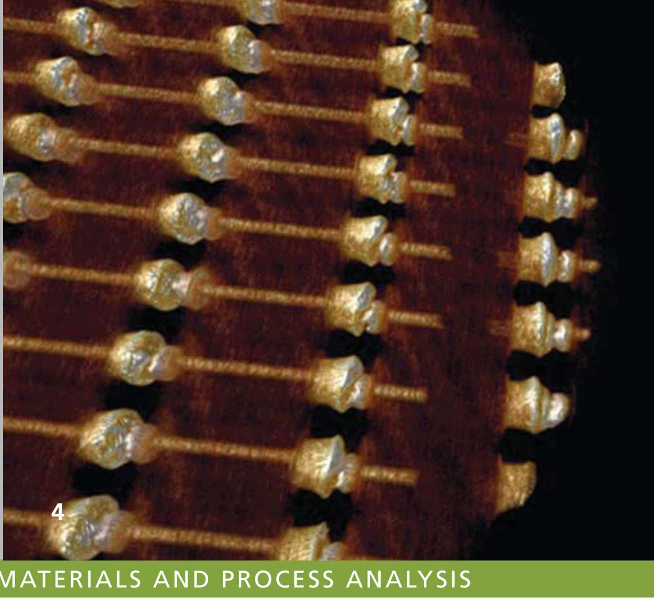

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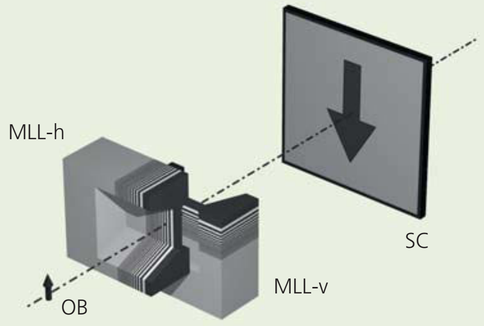
of a dense oxide layer hampers the gas exchange – i.e. release of hydrogen and entry of water vapor – and leads to the expansion of the powder agglomerates (Figure 3). The formation of such an oxide layer has to be prevented by material and process parameter selection. The in-situ x-ray microscopy is a suitable method for the characterization of processes on a microscopic scale within the development of new storage technologies.

The imaging of buried structures and defects becomes more important in the microelectronics industry, both for process control and quality assurance. For novel techniques in the packaging of integrated circuits, e.g. the 3D stacking of dies or the application of interposer structures, x-ray tomography is a convenient method to localize and measure defects regarding the electrochemical filling of metal vias in the die (through-silicon via – TSV). Conventional micro-CT provides informative overview data, e.g. on microelectronic products (Figure 4). The region of interest can be identified and subsequently investigated in detail by nano x-ray tomography. The nano x-ray CT is able to detect pores with less than 100 nm dimension in copper TSV structures (several micrometers in diameter and several 10 micrometers in height). Furthermore, irregularities in the formation of intermetallic phases and possible cracks in micro solder bonds can be identified (e.g. silver tin alloys that connect the vertically stacked dies).

A further improvement of the resolution in the x-ray microscopy will be achievable with novel x-ray optics, Multilayer Laue Lenses (MLL), that replace the Fresnel zone plates as focusing optics. A resolution of 10 nm and below seems possible. The successful integration of crossed MLLs into a laboratory-based x-ray microscope for full-field imaging and the proof of an undistorted projection are the basis for future applications of high-resolution x-ray microscopy, also regarding the analysis of modern microelectronic devices with dimensions of less than 100 nm on the wiring level. An investigation of defects in those pathways requires a resolution of 10 nm. Another field of application is the analysis of biological structures, e.g. the imaging of sub-structures in cells.

Schematic representation of crossed Multilayer Laue Lenses for full field imaging



Services offered

- High-resolution x-ray microscopy: 2D and 3D imaging in x-ray microscope
- Characterization of kinetic processes, in-situ experiments: thermal treatment chamber, chemical reaction chamber, mechanical test (micro-DCB)
- Imaging in absorption and Zernike phase contrast mode
- Highest resolution, smallest pixel width 32 nm
- Recording and reconstruction of 3D and 4D data sets (tomography, laminography, temporally resolved tomography, image series)
- Data analysis, segmentation

1 *Inner view of the x-ray microscope.*
2 *Ceramic spray-dried granule under mechanical load in the moment of fracture.*
3 *In-situ oxidation of iron powder during hydrogen release.*
4 *3D interconnect structure of a microelectronic chip with TSVs and solder bonds.*

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