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FOCUSED ION BEAM (FIB) ANALYSIS AND MICROSTRUCTURING

THE TASK

To take full advantage of the mechanical strength potential is an essential goal in the development of advanced materials, layer systems and manufacturing techniques. Detailed knowledge of the microstructures is very important here; the heterogeneities intentionally induced in the material, such as grain boundaries and precipitations, must be studied, on the one hand. On the other hand, the consequences of material defects, such as discontinuities, microcracks or delaminations must also be known. In this classical field of materialography and materials testing, there is a need to obtain data in greater resolution for the observation of ever-smaller structural lengths and to analyze representative or intentionally selected volumes. The established cross section preparation method with its planar images must be extended into a 3D tomography imaging and analysis for the exploration of anisotropic structures in composite and gradient materials, as well as in layer systems.

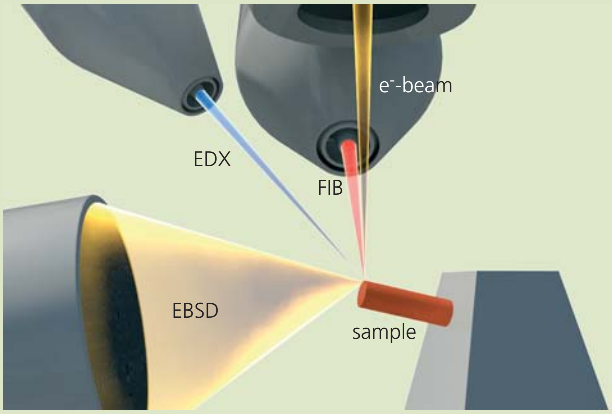
OUR SOLUTION

The application of the focused ion beam (FIB) has been established for high-precision, low-damage, pinpoint and efficient preparation of the material structure in a volume element that determines the properties. When combined with a scanning electron microscope (SEM) in a so-called dual-beam device, the samples can be fabricated and analyzed in linked process steps.

For the requirements of the Fraunhofer IWS Dresden and its cooperation partners, a special FIB/SEM dual-beam system is used. It is equipped with detectors for secondary, backscattered and transmitted electrons and with systems for the analysis of chemical composition (energy dispersive X-ray spectroscopy, EDX), as well as crystal orientation analysis (electron back scattered diffraction, EBSD), and with a micromanipulator (Fig. 1).

One advantage of the system setup is that the detectors and the EDX and EBSD analysis systems are aligned to the ion and electron beam in a way that works without moving the sample from the preparation to the measuring processes and vice versa (Fig. 2). Thus, complex analyses, such as tomography measurements, can be performed with simultaneously high geometric accuracy in an automated mode. Moreover, miniature components whose dimensions with sizes in the micrometer range and accuracies below 100 nm can also be fabricated.

Geometry of ion and electron beam, EDX and EBSD analysis systems and the sample



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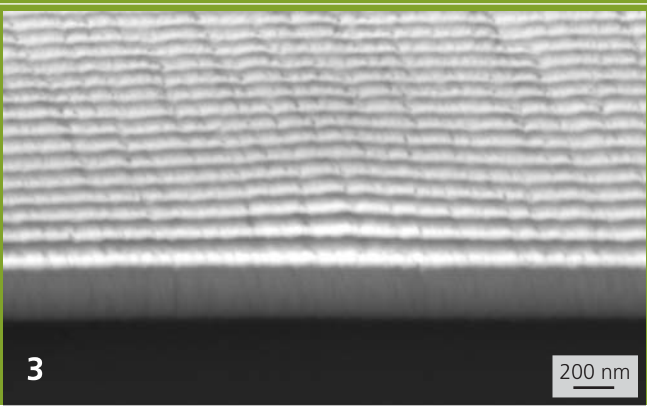


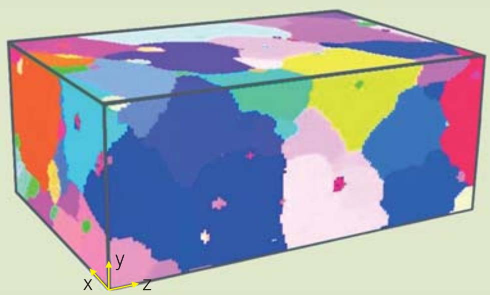
RESULTS

When analyzing layer systems, the advantages of the FIB preparation, mainly for highly accurate and low-damage sample preparation, take effect. A cross-section of a stack of silicon oxide/hafnium oxide coatings on an aluminum substrate with an adhesive coating (nickel) is depicted in Figure 3. The coatings with hafnium content appear brighter, whereas the coatings with silicon appear darker. The high resolution at thickness values of approximately 40 nm is apparent.

The microstructure of a Sn-Cu-Ag alloy is represented in Figure 4, with color coded distribution of the grain orientations. It was prepared and analyzed by combined ablation of material slices in z-direction and mapping of orientations in x-y planes by means of electron backscatter diffraction (EBSD). The 3D model of the examined volume is generated by stacking the planar mappings in z-direction.

A great deal of information can be derived from the 3D structure analysis with resolutions in the order of 10 nm, such as how the manufacturing processes influence the formation of the structure and the resulting characteristics. This analysis can also be used as a basis for the modelling of microstructures in a simulation model (for example, to forecast the deformation characteristics of a structure).

Color coded 3D representation of crystal orientations in a Sn-Cu-Ag alloy



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The FIB technology is particularly suitable to generate geometric structures in the micro- and nanoscale. To fabricate special X-ray optical components (multilayer Laue lenses), thin slabs with parallel surfaces and predefined thickness must be precisely cut out from very detailed small preforms. This precision together with the prevention of damage by amorphization is only feasible when using focused ion beams with optimized process steps in preparation. The example from Figure 5 depicts the smooth surface and homogeneous geometry achieved in the FIB cut. Slab thickness is 1 µm.

FIB technology offers many options for better understanding the structure property correlations of the heterogeneous structure of the materials and layer systems generated at the IWS and opens up new potentials for the fabrication of micro- and nanoscale components.

1 *FIB/SEM dual-beam device*

3 *Layer stack of SiO_2 and HfO_2*

5 *Micro-component for a multilayer Laue lens*

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