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Nanoanalysis of Advanced Materials

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Advanced materials are mostly designed so that they possess specific properties for specific applications. Quite often it is also important that the overall dimensions of a component made out of advanced materials is small since for many applications in telecommunication systems and microelectronics only restricted space is available. In addition, often nanomaterials are also used as advanced materials. For nanomaterials at least one dimension of the component is in the nanometer scale. As an example, one-dimensional nanomaterials are thin films or thin film systems, two-dimensional nanomaterials are nanowires or nanotubes and three-dimensional nanomaterials are those where the grain size (in all dimensions) is in the nanometer range [1].

By advanced TEM techniques the structure and composition of specific areas of advanced materials can be analyzed to the atomic level. The most interesting areas are concentrated around crystal lattice defects, such as interfaces, dislocations, etc. It is well established that the properties of internal interfaces control the properties of the material. Hence for a science-based technological design and optimization of advanced materials it is of great importance to characterize and understand the structure, chemistry and bonding across interfaces. Those quantities can be determined by different TEM techniques. At this point it has to be emphasized that the results of those detailed investigations stem from a rather small area of the specimen, often only of a volume of 10^5 nm^3! For a generalization of the results it is critical that the selected area is "typical" of the whole material - often a difficult choice.

For specific material systems (e. g. thin films) TEM techniques allow the determination of the structure and composition of defects which are typical for the bulk material. The TEM results can be used for the correlation between microstructures and properties of the materials. Examples will be shown for grain boundaries and dislocations in $SrTiO_3$ and Al_2O_3, and heterophase boundaries. For quantitative investigations specific conditions have to be fulfilled, especially for high-resolution TEM (HRTEM) where the orientation has to be adjusted so that the direction of the incoming electron beam is exactly parallel to a specific zone axis of the crystal(s) as well as to the projection of the plane of the defect.

Experimentally, those model boundaries can be selected from local regions in a polycrystal (real microstructures). Or they can be produced as artificial bicrystals with the desired grain misorientation, containing one interface with a specific geometric translation state. Model grain boundaries, bicrystals in single-phase materials (homophase boundaries) are obtained, for example, by growth from misoriented grain seeds, by mechanical twinning or by diffusion bonding of pre-oriented surfaces at elevated temperatures and pressures [2]. The positions of columns of atoms can be determined with an accuracy of a fraction of 1 Å, see e.g. [3]. However, the results and the accuracy depend critically on assumptions which were made for the evaluation of the micrographs.

Heterophase boundaries between materials of different structure and/or composition can also be prepared by diffusion bonding or alternatively by microscopic film growth on atomically flat substrate surfaces with molecular beam epitaxy or sputtering. The different approaches provide a broad variety of interfaces between metals and ceramics and different metals and ceramic systems, respectively. Those interfaces can appear atomically sharp and coherent, semicoherent [4] with a network of interfacial dislocations or incoherent. The latter may contain thin interlayer films of strong structural disorder or new crystalline phases.

One very crucial step for the quantitative evaluation of TEM micrographs represents the preparation of electron beam transparent and resistant TEM specimens of regions adjacent to the defects. Recently, it turned out that specimen preparation by focussed ion beam (FIB) techniques results in high-quality electron transparent specimens as will be shown for TEM cross-sections taken from carbon nanotubes-containing ceramics [5].

HRTEM always provides geometric translation states and positions of columns of atoms in the materials. High-resolution EELS delivers information about the corresponding composition and local electronic structure. An important aspect is always to compare the experimental results with results from theoretical modelling. Theoretical studies lead also to information on the atomistics of interfaces, energetics and bonding [6]. If there is good agreement between experimental observations and theoretical studies, the theoretical studies can be used for the prediction of the properties of defects which cannot be easily investigated by TEM.

A real challenge will be the analysis of defects (mostly grain boundaries) in nanomaterials where the grain size (diameter) is often smaller than the thickness of the electron transparent specimen. The electrons pass not only along a specific grain boundary but also through material on top or below the defect to be investigated. The interpretation of HRTEM micrographs of the complicated microstructure does not lead to an accurate structure of the defect. It is therefore again critical to have specimen preparation techniques available allowing the preparation of extremely thin specimens. The chemical composition, however, and information on bonding can be obtained from thicker crystals. The defect specific signal can be deconvoluted from the measured signal. These investigations apply specifically to investigations of segregations at grain boundaries.

The advent of new instruments with monochromatic electron beams and lenses with C_s-corrections will lead to advanced microscopy with a sub-Angström spatial resolution and sub-electron volt energy resolution. However, these instruments are extremely sensitive to environmental disturbances, such as mechanical vibration and electromagnetic stray fields. Specific measures have to be taken to avoid those disturbances.

References

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