3D ORIENTATION MICROSCOPY IN A FIB-SEM

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Electron backscatter diffraction (EBSD) is a well established technique for the study of the microstructure of crystalline materials. The ability to describe quantitative microstructure parameters such as grain size and grain size distribution, phase distribution, boundary character and to relate these parameters with the local texture makes orientation microscopy via EBSD a powerful technique in the field of materials science [1,2]. However there are a number of microstructure features such as true grain size over the sample volume or grain shape that cannot be described using a two dimensional characterization technique. For this reason it is evident that three dimensional analyses is essential for microstructure research.

During the last decade 3-dimensional characterization of crystalline microstructure has gained increasing attention due to the development of new tomographic techniques based on synchrotron and electron transmission techniques. Both techniques are based on 3D microstructure reconstruction after recorded projections of the crystals. Another approach to obtain the 3D-microstructure information is the serial sectioning and subsequent reconstruction of objects by stacking the information obtained after consecutive slices of the material.

A very powerful implementation of serial sectioning is now available by the combination of a high resolution field emission SEM-EBSD set up with a Focused-ion-beam (FIB) system in a dual beam microscope (Fig.1). The technique consists in a successive set of thin slices of material (minimum thickness ~ 50 nm) which is removed from the surface by sputtering with a high-energy ion beam (Ga^+ source). The so prepared smooth surface is in most of the case very well suited for the formation of electron backscatter diffraction (EBSD) patterns. After the milling process, the sample is tilted to the EBSD position, which means a tilting angle of 70° in order to observe the diffraction pattern. The microstructure of this surface can then be analysed by conventional 2D EBSD-based orientation microscopy. By successive sputtering and EBSD measurements the 3-dimensional microstructure can be explored. Each of the sections obtained will be used to reconstruct the original microstructure in three dimensions [3,4].

There are two strategies to prepare a smooth surface with the high-energy ion milling. The grazing incidence milling is the alternative if the feature that will be analysed is located on the edge of the sample (Fig. 2a). When this is not the case a low incidence milling is the option (Fig. 2b). In this circumstance a larger area has to be sputtered, so that possible shadowing effect for the EBSD measurement can be avoided.

The combination of EBSD-based orientation microscopy with serial sectioning via FIB has a number of interesting features. First, it allows a highly controlled removal of thin layers with a minimum slice thickness of 50 nm. Once that the spatial resolution of the 2D-EBSD technique is of approximately 50 nm or less depending on the material under investigation, the highest achievable resolution of the 3D-EBSD technique is in the order of 50 x 50 x 50 nm^3. Second, the serial sectioning can be performed over a relative large sample volume of approximately 50 x 50 x 50 μm^3. Third, the technique runs fully automated once the measurement is set-up. Additionally, all the advantages of the EBSD technique in the field of materials characterization can be extended now to the 3rd dimensions. This allows one to characterize not only feature size but also its shape and volume distribution by for example the characterization of porous materials or by understanding recrystallisation processes or deformation events. In a more complex case, by applying the 3D-EBSD technique, the nature of grain boundaries and triple junctions can be studied allowing a better understanding of grain growth and phase transformation processes. Unfortunately the technique is a destructive method. This means that in-situ observations are not possible.

The automated 3D-EBSD technique by serial sectioning using a dual beam FIB-SEM will be described and few application examples will be shown. Furthermore the 3D-study of the growth behaviour of nanostructured electrodeposited CoNi-samples will be discussed [2]. Moreover a description of the further application of a dual beam microscope and the explanation of some known difficulties of the technique will be presented.

References:
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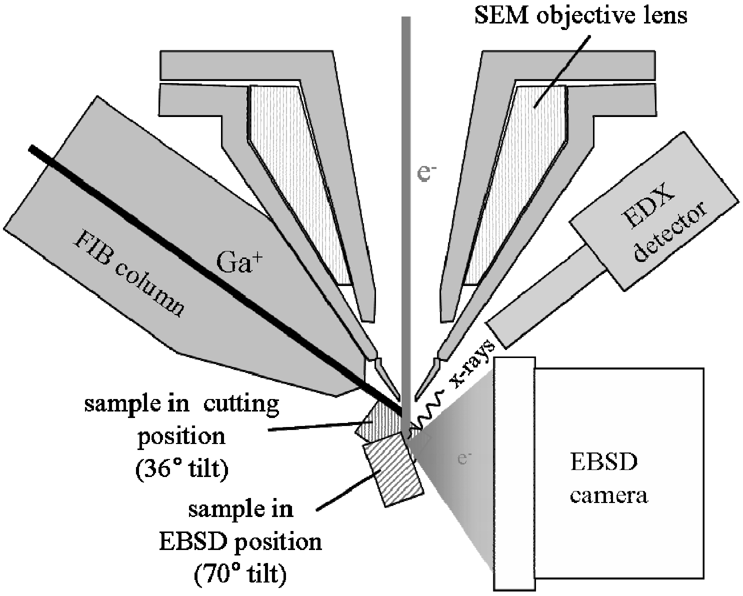

Figure 1 – Schematic representation of the experimental setup for the 3D EBSD –FIB method

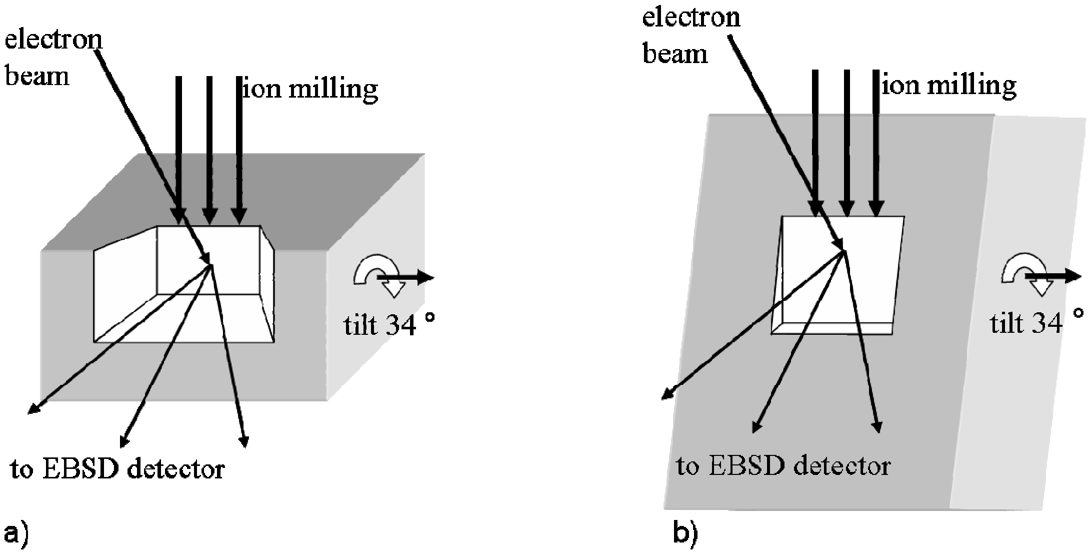

Figure 2 – Milling strategies. a) Grazing-incidence milling (edge milling), b) Low-incidence milling (surface milling)