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Automated Three-dimensional EBSD Analysis of Materials

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The micro-crystallographic structure of a bulk sample can be studied in detail by routine EBSD analysis. With this technique, sample preparation focuses on creating a flat surface at the area of interest, usually applying an electro-polish step as the last cleaning step. The area is suited for crystal orientation mapping (2D information). Another way to prepare the top surface of a sample for EBSD is to use Focused Ion Beam milling. A surface prepared by FIB milling is directly suited for EBSD analysis [1]. With this technique it is possible to remove material very locally and in a very controlled way - as a consequence the technique can be used to create local access to the 3rd dimension. By removing a thin slice at one of the edges of a bulk sample, a new internal surface area is revealed. Using a DualBeam system with both ion beam and electron beam capabilities, this newly revealed surface can directly be used for EBSD analysis using the electron beam. By removing sequential slices, successive internal areas become available and hence the full third dimension can be used for crystallographic measurement. In this way the crystallographic map of each slice can be created one by one and by combining the EBSD information of all slices a true three-dimensional crystallographic volume can be created. Another way to understand the 3D reality of a sample is by applying stereological methods and estimates for grain boundary planes from 2D maps [2] and these calculation methods may be verified by the experimental data collection as described in this paper.

In a DualBeam system with vertical electron column and inclined ion column, the geometry of both beams has to be taken into account when creating successive slices and EBSD maps. In fact two major positions are defined for each function:

- a milling position, where the ion beam is parallel to the surface and perpendicular to the third dimension: by milling along the side of the sample a thin slice is removed
- the EBSD position where the sample's surface of interest is tilted at 70° to allow EBSD analysis with the electron beam: the freshly revealed surface when set to this position can then be used for EBSD mapping

The two positions mentioned above do not coincide in space and need multi-axis stage-moves to go from one position to the other and back. As the process includes successive repositioning the accuracy of this must be very high i.e. to (sub) pixel level.

The process to collect all the data can be very time-consuming and, depending on the applied resolution of the EBSD map and the amount of material that needs to be milled away, can easily be in the range of 6 – 40 hours. This is especially true if the resolution in the third dimension (i.e. the number of slices) has to be of the same order as the lateral pixel resolution of the EBSD map. The total process, when executed manually step-by-step, is not only time consuming, it also requires accurate control and constant attention and therefore it is has been completely automated. The automation was realized on a Nova600 with Channel 6 EBSD system and uses a dedicated SW module including a wizard-type set-up and full control of all required functions. It also includes the fully embedded EBSD functions, such as camera control, automatic indexing and mapping. The

standard control software of the DualBeam system and the EBSD system are not used. The new software module also includes dedicated routines for automatic pattern recognitions and the creation of special markers that serve as positional anchor points for the definition of the slicing process as well as the EBSD mapping area. Also the milling strategy has been optimized to allow the EBSD signal to escape from the newly revealed surface and reach the detector without excessive shadowing, which may reduce the pattern indexing accuracy. During the actual run all data are stored and a continuous update, with the last result presented on the monitor. In the set-up phase of the process, feed-back and checks will ensure a proper setting of the relevant parameters (e.g. the position on the sample, the accuracy of indexing, the confirmation of pattern recognition). The set-up phase takes between 20 and 40 minutes and the actual run typically takes 14 hours, but can easily be expanded to 40 hours or more.

The total dataset stored during the unattended acquisition phase can be analyzed with a 3D crystallography module and provides both 3D visualization functions and arbitrary cross-sectional planar views, but also generates a fully quantified volume distribution. Per 3D-grain, the volume, shape, orientation and neighbor data are determined. As an example a Ti6246 sample has been automatically analyzed, using a volume of 20 x 15 x 15 micron, using 75 slices (200 nm step size) and a pixel map of 100 x 75, so the resolution in any dimension is the same. A total of 562500 "voxels" have been analyzed quantitatively and unattended in a time frame of 19 hours.

[1] T.L. Matteson et al., Journal of Electronic Materials, Vol. 31, No. 1, 2002
[2] G.S. Rohrer et al., Zeitschrift fuer Metallkunde, 95, pp. 197-214, 2004.

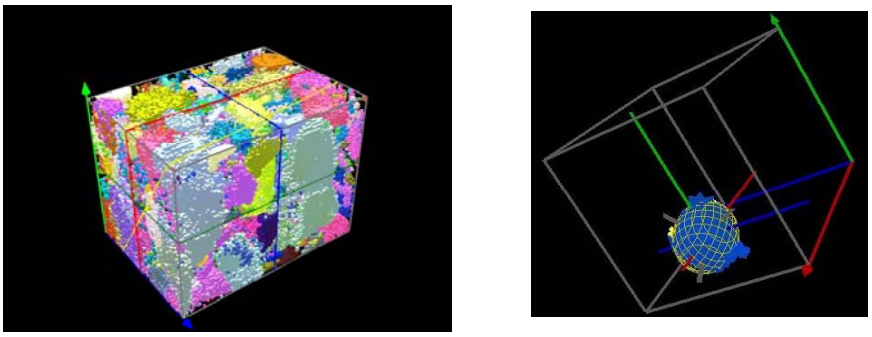

Figure 1 a, b): Analysed volume of Ti6246 showing the orientation of the hexagonal phase (left) and an ellipsoidal match of a single grain -number 339- within the volume (right).

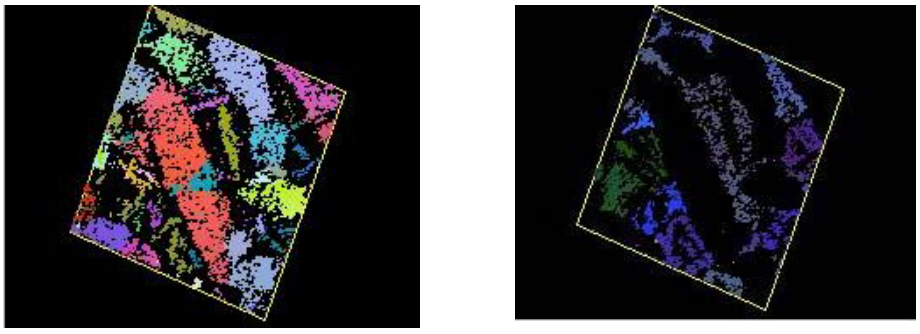

Figure 2 a, b): Cross-sectional view along the arbitrary plane (yellow line in figure 1a) showing the orientation distribution of hexagonal phase (left) and the complementary cubic phase (right).

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