Quantitative High-Resolution TEM/STEM and Diffraction

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HRTEM investigation of grain boundaries starting from orientation maps

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Many properties of polycrystalline materials are influenced by the geometry of the interfaces between the grains (e.g. mechanical or electrical properties, corrosion, segregation, etc.). This is why it is important to be able to characterize the geometry of grain boundaries (GB) by using both conventional TEM and high resolution TEM (HRTEM) effectively.

The optimum condition for HRTEM is that the sample must be oriented along a low index zone axis to visualize differently oriented crystallographic planes simultaneously, i.e. make use of the point resolution of the TEM. If the limited tilting range of the goniometer prevents us from orienting along a zone axis, we need to put up with the line resolution and a single set of planes are only imaged for a given grain. Investigation of GBs needs to fulfill similar conditions for both neighboring grains simultaneously.

In the case of grains with random orientation distribution in a polycrystalline material, the chance is low that there exist parallel pointing low index zones for two neighboring grains and it is even lower chance that we can reach them within the limited tilting range of the goniometer in a HRTEM (if they exist at all). There is larger chance that if we project one grain along a zone axis, its neighbor can only be imaged along a single plane only. The highest chance is that we can find an orientation where both grains can be imaged with lattice resolution (showing only one set of planes for either of them). However, it is very difficult to find and set any of such orientations manually. We have to be lucky to reach the proper orientation if we are not helped by a computer.

Development of a computer program is reported here that facilitates identifying and setting such orientation-pairs. Our software starts with recording an orientation map, using the commercial ASTAR software. Then our own software identifies the net of GBs found in the orientation map by assigning an average orientation to each grain and locating lines where this orientation changes above a threshold. The program highlights those parts of the GBs for which it is predicted that they can be investigated by HRTEM, because lattice resolution can be reached for both neighbors simultaneously within the tilting ranges of the given TEM. The program also gives advice, which tilt angles to set to reach that condition. Using this process increases the efficiency of the investigation of GBs. By scanning an area with several grains, we obtain information about the orientations of many boundaries in a short time. We have a good chance to find a few GBs among the many, which can be investigated by high resolution techniques under the given experimental conditions.

As an example, results are presented here on a polycrystalline TiN thin film. The investigations were carried out in a JEOL 3010 HRTEM with 1.7 Å point resolution. We used a GATAN double-tilt holder with the tilting ranges of ±20° and ±10° for the two orthogonal axes. An orientation map was recorded b the ASTAR precession system of NanoMegas, installed on the microscope.

The example on the TiN shows that using our method, it was easy to locate a low angle grain boundary in the orientation map, whose lattice imaging conditions were predicted within the possibilities of the TEM. The selected orientation is close to the <100> zone. After tilting the sample to the predicted orientation one set of {200} planes are seen in one of the grains, while the other set of {200} planes are seen in the other grain (due to the orientation difference between the two grains). Even though none of the grains were in perfect zone axis orientation, we were able to record an image from the row of dislocations that induced the orientation difference between the two neighboring grains (Figure 1.). The perfect dislocations in this fcc material have an <110> character, with the length of the Burgers-vector b=3 Á. The distance of the dislocations in the row (two of them are indicated by arrows in Fig. 1) is measured from the images to be 17 Á, resulting in 10° orientation difference, in agreement with the approximate value read from the orientation map. The orientation difference measured from the orientation map suggested that the orientations of the two grains differ by 12° (Fig.2). However,

simulated orientations are calculated at 1° steps for determining the orientation maps, so the accuracy of orientation is 1° for either grains. Consequently we can not anticipate prediction of the orientation difference between the two grains better than ±1-2° from the orientation map. Measurement form the HRTEM refined the value of the disorientation from 12° to 10°.

Additionally, the program can also give advice if pre-selected orientation of any selected grain is within reach. Obviously, the number of appropriate orientations increases with improving point resolution of the TEM and a wider pool of defects and boundaries offers themselves for examination if we can orient the sample into orientations from where they can be seen. Consequently, the program can be even more useful for new Cs-corrected TEMs, because it can predict how to set the sample if a special boundary is to be imaged. Very similarly settings for imaging special defects within a selected grain of a polycrystalline sample can also be predicted by the program. Altogether, atomic resolution imaging of defects and grain boundaries should benefit from the usage of that computer program.

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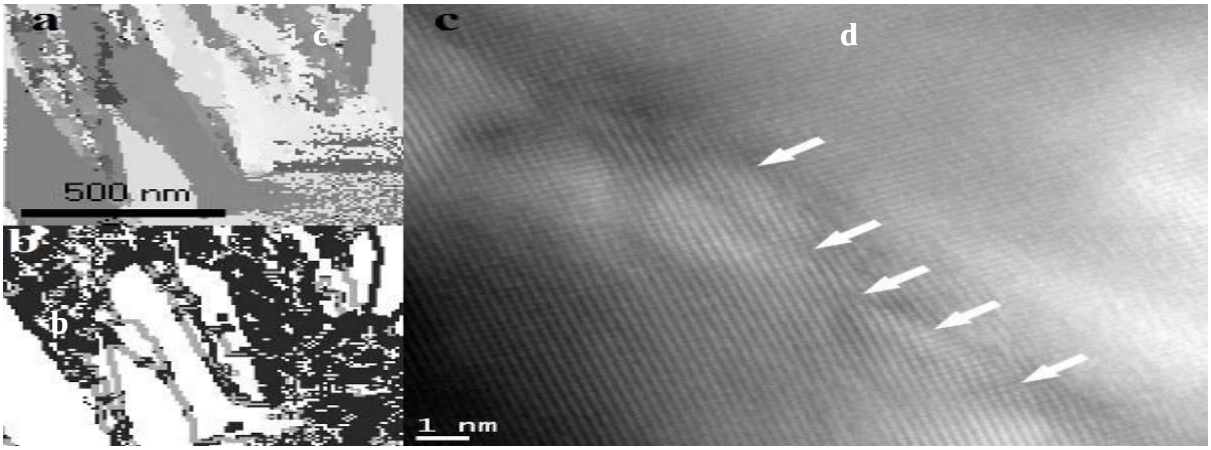

Figure 1. TiN sample: a) orientation map; b) part of GBs (red) to be investigated; c)high resolution image taken on the boundary denoted by arrow; (200) set of planes are shown in both neighboring grains; d) dislocations are present in the enlarged area at places denoted by arrows. The row of dislocations builds up the low angle boundary in the image.

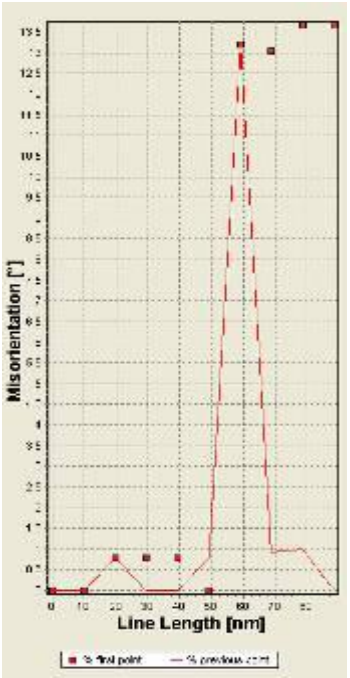

Figure 2. Orientation difference measured along a line crossing the selected grain boundary.