System for creating orientation maps using TEM

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Abstract

Electron back-scattered diffraction (EBSD) in scanning electron microscopy (SEM) is already extensively used for creating orientation-based microstructure images of polycrystalline materials. In an analogous way, Kikuchi patterns have been recently applied for creating polycrystalline orientation maps using a transmission electron microscope. Main components of the new system are similar to those of the SEM-based systems. The first steps are pattern acquisition and correction of the images. They are subsequently followed by automatic indexing of the patterns. Finally, from orientations obtained in a grid of points, a map is created. The system using transmission electron microscopy (TEM) has a good spatial resolution of about 10 μm. Its accuracy in orientation determination (≈0.1) is better than the accuracy of EBSD systems.

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1. Introduction

Microscopic images based on crystallite orientations have become an important element in research on polycrystalline materials. Generation of orientation maps on scanning electron microscopes by EBSD patterns is a well established technique [1]. Recently, a new automatic system for creating orientation maps has been constructed using TEM pure Kikuchi patterns (generated by inelastic scattering) and convergent beam patterns. The goal of this communication is to provide a concise description of the system's main components.

Kikuchi patterns have been applied for orientation determination for a long time; see e.g., [2]. They have been also employed for manual grain orientation mapping with location of grain boundaries obtained from contrast images [3]. Using a so-called circular scan mode, grain maps are automatically created by the "Automated Crystallography for the TEM" software distributed by EDAX/TSL. This system, however, has a poor accuracy in the orientation determination (≈5).

SEM and TEM orientation imaging systems are based on the same idea. In both cases, maps are obtained in a step-by-step beam scan with crystal orientation determination in each step. However, there are a number of fundamental dissimilarities. These are not only hardware differences but also software differs considerably because of specific features of TEM and SEM diffraction patterns. The image analysis and indexing of the TEM patterns require more refined approaches. Moreover, the TEM orientation maps differ from those obtained by SEM/EBSD. The former have a better spatial resolution and a better accuracy of (the relative) orientation determination. On the other hand, the map acquisition time on TEM is long and this limits applicability of the new system.

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Because of the good spatial resolution, the TEM mapping system is suitable for the investigation of fine grain materials. The high precision in determining misorientations opens new possibilities for research on orientation relationships and subgrain characterization.

2. Hardware

The TEM-based mapping system was implemented at LETAM, using a computer controlled Philips CM200 operating at 200kV and equipped with a LaB6 cathode, a goniometer stage (CompuStage), and a slow scan CCD camera (GATAN 791) with a YAG scintillator used as the primary screen. The camera is mounted on a 35 mm port above the viewing chamber; this location provides a large pattern acquisition angle which is essential for reliable pattern indexing. The camera and the TEM are controlled from a personal computer running the GATAN Digital Micrograph. The same computer is used for image correction. A separate computer is employed for processing the corrected patterns and for post-processing of orientation lists.

3. Software

After acquiring the pattern, the main steps in the process of creating an orientation map are image correction, line detection, indexing, and, finally, construction of the map from a set of stored orientations (Fig. 1).

The 1024 x 1024 pixel images provided by the camera are consolidated to 512 x 512 pixel images. The main reason for the binning is to speed up the data transfer and the further treatment of the image. The pattern acquisition is still the main factor determining the time needed for map creation. For the consolidated images, the rate is about 10 patterns per minute.

The intensity on the original pattern decreases with the distance from the center of the image. The system deals with this problem by taking two pictures with different exposition times. The one with short exposition time provides the central area; it is combined with the other one in which the borders of the pattern are enhanced. Moreover, the diffraction patterns are corrected using a procedure consisting of taking the logarithm of the intensity and applying a low-pass filter modifying the image background.

The main clearly visible feature of the corrected Kikuchi patterns are line pairs corresponding to crystallographic planes. In principle, the detection of lines in an image is relatively simple. However, the images provided by the system exhibit a variety of characteristics which depend on the sample and operating conditions of the microscope and may considerably differ from one measurement session to another. Therefore, the line detection procedure must be, to a certain extend, universal. In turn, this requirement lowers the efficiency of the procedure. We applied a kind of the Hough transform, which is the most frequently used algorithm for line detection, plus a number of additional routines. Various versions of the programs were judged based on their performance in detecting line pairs and their execution time. The best one which is used in the system is quite efficient. However, it is not completely universal; there are a number of parameters, and assigning proper values to them may improve the program's performance. The time needed for line detection depends on the required level of reliability. A reasonably good procedure takes about 2 seconds per pattern on a 1.7 GHz Pentium IV processor. It is expected that the performance of this part of the system will be improved in the future.

Although, the mechanisms of pattern formation are different, inelastic scattering Kikuchi patterns and convergent beam patterns have the same geometry. In both cases, the visible line pairs are sections of two wide-angle conics symmetrically located on two sides of a reflecting plane. Pattern indexing, i.e., assigning plane indices to the line pairs is done based on the geometry of diffraction patterns without considering intensities of reflections. This significantly simplifies the computer routines responsible for indexing.

The indexing principles applicable to the Kikuchi patterns are the same as in the case of the EBSD patterns. In practice, however, there some differences. The main reason lies in the sample-to-detector distance which in transmission microscopes is large in comparison to scanning microscopes. Because of that, the acquisition angle is relatively small, and the indexing procedure must allow for high index reflections. With high index reflections taken into account, the indexing requires more time and becomes less reliable. However, assuming correct indexing, the larger sample-to-detector distance leads to a better accuracy in the determination of the relative orientations. (For more details on the applied line detecting and indexing programs see [4, 5].)

With orientations determined at points of a grid and a color ascribed to each orientation, a topographic map of the microstructure is constructed. This is done in the same way as in the case of orientation data obtained by EBSD/SEM. Actually, in both cases the same post-processing software can be used, and one can take advantage of numerous additional options available in commercial EBSD/SEM systems.

3. Accuracy, resolution and applications

The new system has an important advantage of relatively high precision in determination of misorientations. The accuracy of absolute orientations is of secondary importance because it is limited by the precision of keeping orientations in preparation and positioning of the sample. Fortunately, TEM users are usually interested only in misorientations. It is easy to notice that the accuracy of relative orientations is linked to the sample-to-detector distance; roughly, the larger the effective camera length, the better the accuracy. However, this is true for only two (out of three) orientation parameters. The precision of the third parameter - the angle of rotation about the microscope axis - is not influenced by the camera length. Still, the accuracy is quite impressive. E.g., for a camera length of 159 mm at the CCD level, the precision in determining misorientations is estimated to be better than 0.1 . Finally, it must be noted that very large camera lengths cannot be used because patterns with small acquisition angle cannot be indexed.

The spatial resolution is an important aspect of the microscope images. In the age of nano-scale research it gets special attention. Also, the most interesting problems in the area of polycrystalline materials call for a high spatial resolution. Because of a large number of factors involved, the quantitative evaluation of the spatial resolving power is difficult. Humpreys [6] analyzed the resolution of the EBSD/SEM system. With a conventional filament it is about 100 nm, and one can reach about 40 nm in the case of a microscope equipped with a field emission gun. Our experience shows that the spatial resolution of a TEM-based mapping systems can be even better. For the experimental set-up we used, it is estimated to be about 10 nm.

The relatively high spatial resolution opens the possibility of numerous applications of the new mapping system because many problems in polycrystalline research lie at the lower end of the nano-scale. The investigation of severe plastic deformation, early stages of recrystallization and all other ultra-fine microstructures requires a high resolution. Moreover, the orientation data combined with the TEM ability to estimate the boundary inclination may be useful in grain boundary analysis. On the other hand, the new system cannot provide large scale maps. (An EBSD/SEM system with stage control is capable of covering multi-millimeter areas.) Therefore, the TEM-based system can be seen as complementary to the conventional EBSD orientation imaging.

The new orientation imaging method has been already applied to a number of metallic samples after severe plastic deformation. Interesting orientation maps have been obtained for aluminum samples deformed by equal channel angular extrusion and for shear bands in deformed copper. The results will be published elsewhere [7].

4. Conclusions

Our experience shows that it is relatively easy to build an orientation imaging system on a transmission microscope. Because of its high spatial resolution and good precision in determination of misorientations, this new instrument well complements the established EBSD systems and other conventional methods of investigation of polycrystalline materials. Although, it is less accessible than the orientation imaging on SEM, the new technique is expected to become a standard in investigation of fine grain materials. Further development of the system is expected. Most likely, other set-ups of this type will be created. As in the case of the EBSD orientation mapping, one can assume that the map acquisition will be considerably faster, and that some other tools will be added (e.g., phase identification). The orientation maps will be used not only as a separate source of quantitative information but also to enhance the conventional contrast images.

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Caption

Fig. 1. Schematic illustration of some elements of the system. (a) Corrected diffraction pattern. (b) Automatically detected line pairs. (c) Indexing; bars are omitted. (d) One of the first maps created by the system. Al sample deformed by equal channel angular extrusion at room temperature.