ORIENTATION IMAGING OF HEAVILY DEFORMED METALS

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Abstract

Orientation imaging has become a standard technique in materials laboratories worldwide. The application of this characterization technique to analysis of heavily deformed materials has become more feasible with the introduction of field emission source SEMs that result in improved spatial resolution. Electron back-scatter diffraction patterns can be obtained from regions as small as 10 nm in diameter in some instances. Applications of this characterization technique to heavily deformed Al and Cu alloys are described in the present work.

Keywords: Orientation imaging, heavily deformed materials, Electron back-scatter diffraction patterns

Resumen

Las imágenes orientadas se han convertido en una técnica estandarizada en los laboratorios de materiales de todo el mundo. La aplicación de esta técnica de caracterización para analizar materiales muy deformados se ha hecho más factible con la introducción de Microscopios Electrónicos de Barrido con filamentos de emisión de campo que permiten una mejor resolución espacial. Los patrones de difracción de electrones retrodispersados pueden ser obtenidos de regiones tan pequeñas como 10 nm de diámetro en algunos casos. En el presente trabajo se describen las aplicaciones de esta técnica de caracterización para aleaciones de Al y Cu.

Palabras clave: Imágenes orientadas, materiales muy deformados, patrones de difracción de electrones retrodispersados

1. Introduction

Over the past decade or so, microstructural characterization of polycrystalline materials using automated electron back-scatter diffraction (EBSD) techniques in the scanning electron microscope (SEM) has become routine. The high resolution obtainable in the field emission SEM enables EBSD analysis of materials with sub-micron grain sizes.

Conventional orientation imaging [1,3], whereby images of the microstructure are obtained by automated EBSD analyses over a regular array of points, requires that several measurements be made within each grain or dislocation cell in order to accurately reconstruct an image of the microstructure. Recent orientation imaging of a platinum film having an average grain diameter of 75 nm was successfully demonstrated [4]. In that study, it was evident that grains of less than 30 nm in diameter were successfully imaged. During interactive operation, clear EBSD patterns were obtained from grains of approximately 10 nm in diameter. Cell forming materials can generally be characterized accurately by EBSD assuming that cell diameters are of similar size to the interaction volume of the beam in the region from where the elastically back-scattered electrons originate. Alloys that are not cell-forming in nature can be characterized by EBSD to the point that dislocation density within the interaction volume is sufficiently low to allow coherent scattering over at least half the region of specimen-beam interaction, with the crystallite lattice mutually oriented to within one degree.

The following sections contain examples of some of the authors' recent research into heavily deformed materials. Examples from cold-rolled aluminum, equal channel extruded copper, and friction stir welded aluminum are presented.
Pure Al (99.99%) was cold rolled to 80% deformation. Electron transparent specimens were prepared from the cross section of the plate. Cell orientations were measured in the TEM using an enhanced variation of the technique described by Baggethun [5]. The foils were subsequently analyzed in the SEM by orientation imaging.

Figure 1 shows a bright field TEM image of the region analyzed indicating a well-defined cell structure. The nominal orientation of the grain from where the image was taken is (211) with respect to the sample surface normal orientation. The bright field image was manually divided into individual cells and the orientation of various cells was measured using Kikuchi patterns.

Figure 2 contains an orientation image map of the cells for which an orientation was measured by Kikuchi line analysis. The inset unit triangle in Figure 2 contains an orientation color key for the colored map. The orientations shown are those with respect to the specimen normal direction. The data were analyzed for average cell size and for the misorientation angle distribution between neighboring dislocation cells. The cell size distribution and the misorientation angle histogram are given in Figures 3 and 4 respectively. The average cell diameter using the assumption of equiaxed cells was 0.96 μm. The average dimension in the elongated direction was about 1.3 μm and 0.67 μm in the transverse direction as measured from the bright field image.

The same region was analyzed in the scanning electron microscope by orientation imaging in the SEM. An orientation image was measured from the (211) grain in the same region as that analyzed in the TEM. The field of view imaged in the TEM is a fraction of that seen in the SEM, and the images are rotated with respect to each other, so the regions analyzed are not readily comparable. An image quality map of this structure is shown as Figure 5. The dislocation cell morphology is apparent even though the misorientation angles between neighboring cells is typically less than five degrees. The average cell size (assuming equiaxed cells) as measured from the OIM scan in the SEM was 1.03 μm. This compares favorably with the value of 0.96 measured in the TEM. The cell size distribution and misorientation angle histogram for this region as determined from the orientation imaging technique are included in Figures 3 and 4.

The cell size distribution is skewed toward smaller cells as measured in the TEM and leans toward larger cells as measured by the SEM. These differences are small, however, and may be explained by the fact that the SEM measurements covered a significantly larger region than those from the TEM so the statistical distributions will differ somewhat. The differences observed in the misorientation angle distribution are also minor, but show a propensity for the SEM measurements to include more very low angle grain boundaries. This is attributable to the fact that the resolution in determining relative orientations by EBSD is on the order of 0.5 degrees and some artificial boundaries on this scale may be introduced.

Previous work has shown that in some instances, dislocation cell structure is a strong function of crystallite lattice orientation [6]. If measurements of dislocation cell morphology are to be used in relating structural variables to material behavior, the dependence of cell morphology upon crystallite lattice orientation must be determined. This requires a large number of measurements for statistically reliable information to be produced. To complicate matters further, grain interactions are known to play an important role in determining cell structure evolution, so the two-point orientation correlation function...
should also be simultaneously measured. Although it is feasible to implement fully automated orientation measurements in the TEM [7], it is unrealistic to assume that such a quantity of information could be obtained by TEM techniques.

Proper quantification of dislocation cell morphology must include a description of the anisotropy of dislocation cell shape, the clustering or banding of cells, and its dependence upon crystallite lattice orientation. One measure that can satisfy each of these objectives is that of a dislocation cell wall surface area per unit volume that includes all of the appropriate dependencies. One could envision a measure described as

\[ S_V = S_V(n,g,r,g') \]  

where \( S_V \) is the stereological measure of cell wall surface per unit volume with functional dependence upon cell wall orientation, \( n \), lattice orientation, \( g \), and neighboring lattice orientation \( g' \) which lies at a position \( r \) from \( g \).

An alternate approach is to develop a cell shape and orientation distribution function which could also have functional dependence upon the 2-point orientation correlation [8]. This function may be written in 2-dimensions as

\[ \xi = \xi(a,b,\Phi,g,r,g') \]  

where \( a \) and \( b \) are the major and minor axis dimensions of the cell, and \( \Phi \) is the orientation of the major axis normal with respect to a reference axis. The extension of the function to 3-dimensional space is straightforward and is accomplished by adding a third axis and an additional angle describing the orientation in vector space of the major axis. Correlation of cell shapes may be described by a two-point function, \( h_2(\xi,t,\xi') \), where \( t \) is a vector between cells of various shapes. The practical measurement of such a correlation function is still beyond reach as the function lies in a 14-dimensional space, which is prohibitive in relation to experimental and computational techniques. Functions with reduced dimensionality or fixed parameters are currently tractable, however, and may be reasonably employed in the investigation of material microstructure.

3. Equal-Channel Extruded and cold-rolled Cu

Equal channel angular extrusion (ECAE) is a process whereby large strains can be imposed upon a material without changing the initial shape of the billet. This is done by forcing the material around a corner, with the dimensions of the input and output channels being equivalent. The technique is discussed in detail by Segal [9] and Valiev [10].

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**Fig. 3.** Grain size histograms as determined by SEM and TEM techniques.

**Fig. 4.** Misorientation angle histograms as determined by SEM and TEM.

**Fig. 5.** Dislocation cell structure as observed by orientation imaging.

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Information derived from the OIM scan depends upon the requirements of the investigation and the preferences of the researcher. Figure 6 shows a series of images obtained from a Cu specimen deformed by ECAE. Defining a boundary as 15 degrees of misorientation or higher yields a view of highly strained grains having a nominal grain diameter of 35 microns in a structure of elongated grains in the direction of shear. If the definition of a grain, or cell, boundary is changed to 2 degrees (5 micron grain size) or 0.75 degrees (grain size indeterminate), the structure begins to appear very different as seen in the images presented in Figure 6. Boundary interpretation is 0.75, 2, and 15 degrees in the images from left to right as presented. Imaging the dislocation structure using a step size of 50 nm yields an interpretation of the average diameter of the cells to be about 0.3 \( \mu \text{m} \) using a cell definition of 0.75 degrees.

![Fig. 6. Orientation images of ECAE processed Cu billet showing grain structure as a function of GB angle definition.](image)

As with any imaging technique, microstructural interrogation of such heavily deformed material requires different imaging strategies based upon the type of information desired. For example, the determination of crystallographic texture alone requires a large step size where each measurement will preferably lie in a different grain or cell. Analysis of grain boundary structure requires a smaller step size with several measurements falling within each grain. Analysis of structure on the scale of dislocation cells requires an even more refined step size and eventually limits the total area that can be imaged in a reasonable amount of time. Figure 7 shows an image of a 60 percent cold-rolled Cu plate with increasingly finer step sizes. The relevant information that can be derived from each of the images is dependent upon the step size used during data collection.

![Fig. 7. Progression of orientation images taken at different step sizes.](image)

While information from various length scales can be accurately measured in a statistically reliable manner, there remains the challenge of linking the information obtained. It is not difficult, for example, to measure dislocation cell size using orientation imaging, but to obtain a statistical description of cell morphology as a function of orientation is not readily achieved because of the large number of measurements required. Any thought to measure correlation functions of such distributions and to obtain reliable data on the effect of neighboring grains and their orientations continues to be out of reach experimentally.

4. Friction stir welding in Aluminum

Friction stir welding is a relatively new joining process that holds considerable promise in that it has many advantages over conventional fusion welding. Some of these advantages include the elimination of cracking in both the fusion zone and heat affected zone, and elimination of porosity, filler metals, shielding gases and costly weld preparation. A description of the technique is given in the patent application from the Welding Institute (TWI) [11].

For this analysis contained in the present report, commercial purity Al plate 6.35 mm thick was sectioned into 10 cm wide by 61 cm long test plates and the edges were machined to be flat. Welding was performed using a tool that consisted of a 19 mm diameter shoulder with a 6.35 mm diameter right hand threaded point that was approximately 6 mm in length. The plates were aligned such that the weld travelled along the RD of the plate material and penetrated through the ND of the processed plate. All welds were performed such that full penetration...
plate. All welds were performed such that full penetration friction stir welds were achieved using a rotational speed of 700 rpm and a travel rate of 18 cm/min. Orientation imaging analysis was performed on entire weld regions on cross sections of the welds. Scans were made in 40 micron steps over areas of approximately 20 mm x 6.35 mm. The resultant grain size is, of course, much smaller than this, so only global trends in crystallographic texture can be observed, and no information is obtained on grain boundary structure. A representative orientation image is shown as Figure 8. Strong gradients in the texture can be observed through the stir zone of the weld nugget. Various regions throughout the weld were isolated and analysed independently in an attempt to identify the local deformation state responsible for producing the given texture.

The texture of the stir zone or tool region is presented as a set of re-calculated pole figures shown as Figure 9. Considering the convoluted deformation history through which stir-welded material must pass, these pole figures exhibit a surprisingly typical shear texture with the shearing direction aligned with the tangent to the edge of the tool and RD of the plate, and the shear plane normal aligned with TD. Strong texture gradients exist throughout the weld region in addition to that in the transition region from the weld to the thermo-mechanically affected zone and from the TMAZ to the base metal.

Fig. 8. Representative orientation image from a friction stir weld in Al.

Analysis of the weld region directly under the shoulder of the tool exhibited a strong, and well-defined texture gradient across the width of the weld. Pole figures for this region are shown in Figure 10. The texture of the entire region contains a (200) fiber component, characteristic of compressive deformation in fcc metals, and a (111) component indicative of shearing deformation in the plane of the plate (with no dominant shearing direction). Both of these texture components might be expected considering the deformation imposed by the compressive and rotating action of the weld tool. A more clear understanding is obtained by separating this region further into discrete segments representing the structure under the shoulder at a given location across the width of the weld. The results obtained from analyzing the texture in each region clearly show the texture gradient present in this region. Figures 11 a-g contain (111) pole figures for each region from left to right across the weld and directly underneath the tool shoulder. The dominant texture component seen in each figure is not a (111) lying in the plane of the sheet as indicated by the overall texture, but is a (111) component approximately 60 degrees from the specimen normal. The direction from the specimen normal that this dominant texture component lines appears to be aligned with the tangent of the tool edge as it travels from point to point along the weld.

Fig. 9. Pole figures from tool region showing a typical shear texture.

Fig. 10. Pole figures showing texture under shoulder region of weld tool.

The friction stir welding process produces a complicated microstructure consisting of widely varying
crystallographic textures from position to position through the weld. A quantitative assessment of these textures could be made by comparing these texture measurements to deformation models and iterating to determine the deformation gradient tensor necessary to obtain the textures observed at each point. Additional studies of these welds to determine corrosion resistance as a function of the grain boundary character distribution and the inhomogeneity in the dislocation sub-structure are currently in progress.

Fig. 11. (111) pole figures showing texture gradient under the shoulder of the weld tool.

5. Discussion

It is apparent from the above examples that heavily deformed materials can be successfully analyzed using automated EBSD techniques. The success that one will have in obtaining high quality diffraction patterns from deformed materials is primarily a function of the SEM capability and the Z number of the material.

In light of the data presented above, the question of when to use the SEM in analysis of deformed structures and when to continue with the more conventional TEM analysis can be reasonably asked. The work on cold rolled pure Al plate material presented herein has clearly established the feasibility of measuring dislocation cell morphology in the SEM. Work on the highly strained Cu (ECAE discussion above) and research not discussed in the present effort demonstrate the feasibility of obtaining information on crystallite lattice orientations and dislocation cell morphology in a number of deformed materials. The data of cell structure obtained by automated EBSD techniques differ from that derived from TEM techniques in a number of ways. First, the sharp Kikuchi lines in the TEM lend to a superior angular resolution in measuring lattice orientation. In addition, the orientation and thickness of the cell walls can be extracted directly from TEM images while the SEM technique will give no information on cell wall thickness, and yields orientation information only through serial sectioning. Finally, while the spatial resolution of the EBSD technique is becoming increasingly finer with each generation of SEM, the TEM has an undisputed advantage in spatial resolution.

Information from materials with nano-scale dislocation cell sizes can be obtained only from high resolution TEM imaging.

There are several advantages to performing the analysis by SEM techniques where possible. Specimen preparation is reduced to standard metallographic techniques that save considerable effort in comparison with the preparation of TEM foils. The large regions that can be imaged in the SEM allow for statistically reliable functions to be obtained, including the grain boundary character distribution, and the crystallite orientation correlation function.

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7. References