Microstructural Evolution and Characterization of Al-8090 Superplastic Materials

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Abstract

'he grain and subgrain structure of superplastically formed Al-8090 specimens has been tudied using Orientation Imaging Microscopy (OIM). The microstructure and microtexture for hree strains corresponding to ε=15%, ε =70% and ε =660% were characterized for uniaxial ensile specimens deformed at 520° C and $5x10^{-4}$ sec^{-1} strain rate. Using a misorientation ngle criteria resulted in structures which differed in appearance. The generated nicrostructures and the resulting microtexture data will be discussed in the context of the OIM echnique.

INTRODUCTION

The impetus to reduce the manufacturing costs of components has resulted in considerable attention being devoted in recent years to the phenomena of superplasticity and superplastic forming. In this process elongations as high as 4850% have been recorded [1]. The ideal microstructure for superplastic deformation consists of small (~10 micron) equiaxed grains. During superplastic deformation a gradual increase in grain size can occur and any initial grain alignment of the microstructure will disappear [2]. These findings are based on traditional microstructural characterization using conventional optical, scanning electron microscopy (SEM) and transmission electron microscopy (TEM) techniques in addition to standard X-ray texture techniques [3].

TEM analysis of grain structure and subgrain formation can only provide information for individual grains or at best a small region of the microstructure. Such analyses do not provide an adequate global picture, and can result in erroneous conclusions. The X-ray technique provides an average texture data which may not include the interaction of individual grains and subgrains. The SEM technique, used in the conventional method, is just an optical microscope with much higher resolution and magnification. However, when used in conjunction with the micro-texture attachment, it can be used effectively to provide a detailed microstructural characterization of the material.

Previous works have demonstrated that grain boundary sliding is the main mechanism of superplastic deformation [4] and that this is accommodated by slip or diffusion creep [5]. Thus, in addition to standard microstructural characterization investigation of grain orientation and grain to grain misorientation, it is crucial in studying the deformation mechanisms of such materials. Some recent studies on superplastic deformation revealed, in this respect, that nearest neighbor grain misorientation were high angle [6]. These misorientation measurements were accomplished by analyzing Kikuchi or channelling diffraction patterns in the TEM and SEM respectively. However, in order to obtain detailed information that will result in a global picture on the deformation of superplastic materials, large areas of the samples have to be studied in this way. TEM and SEM channelling methods for such large surveys are impractical. Optical microscopy reveals only part of the information, and x-ray diffraction provides only an overall average picture of orientations.

A detailed microstructure and micro-texture study of superplastically deformed Al 8090 material has been carried out as the basis for this paper. The microstructures of this material at three stages of deformation were recorded, using a new technique, Orientation Imaging

Microscopy. This enables the crystallographic features of the microstructure to be depicted over large areas of the sample. The need to investigate and apply this new technique is described first.

MICROSTRUCTURAL CHARACTERIZATION

Orientation Imaging Microscopy

In conventional optical microscopy, grains and grains boundaries are revealed by the chemical etching of the specimen surface. This technique may give a false impression of the microstructure since it is difficult to distinguish in the image small from large misorientation difference amongst the grains. For instance slight changes in the misorientation angle between adjacent grains can produce significant changes in shade. Also, grain boundary etchants which are sensitive to residual strain or energy gradients at the boundary and should distinguish low from high angle boundaries may in practice not do so, for solute segregation and precipitation will mask the etching differential expected. It is thus uncertain and unknown whether the true microstructure or what particular aspects of that microstructure may have been revealed.

In transmission electron microscopy, the diffraction contrast image shows all changes in orientation and hence distinguishes each grain and the grain boundaries that separate them. However, quantification of the microstructure in terms of types of grain boundary requires that electron diffraction patterns are obtained from each area. Even distinguishing low angle from high angle boundaries can not be done from examination of the conventional image alone. A simple tilt boundary of 2 degrees misorientation has edge dislocations in it spaced 7.2nm apart. The overlapping strain field from each dislocation prohibits resolving them individually and, consequently, any chance of recognizing the boundary for what it is. Furthermore, TEM analysis of grain structure and sub-grain formation can only provide information from a small region of the microstructure. Such analyses do not provide an adequate global picture and may result in erroneous conclusions.

X-ray diffraction technique, on the other hand, though providing average texture data from a large area neither provides the important information on the spatial distribution nor interrelation of individual grains and subgrains.

Scanning electron microscopy, used in both the conventional secondary electron imaging mode and in the channeling contrast mode, provides similar information to the optical microscope though at much higher resolution. Again the essential crystallographic information is missing.

Following upon the work of Venables [7], who developed the Electron Backscattered Diffraction (EBSD) technique in the SEM, Dingley [8] developed a computer assisted method for their on-line analysis. Adams et al [9] extended this to enable full automation of the technique. These diffraction patterns are formed in the same manner as Kikuchi patterns in the TEM, but result from backscattering of electrons out of the top surface of the sample. Hence they can be obtained from bulk samples. The specimen is inclined in the SEM at 70° to the incident electron beam. The diffraction patterns are imaged on a phosphor screen placed close to it, as illustrated in figure 1. The phosphor screen is viewed through an optical port using a high gain television camera which in turn is interfaced to a computer. By indexing successive diffraction patterns from hundreds of selected points on the sample surface, sufficient data can be collected to determine both macroscopic and local orientation texture and to provide a detailed survey of nearest neighbor orientation relationship.

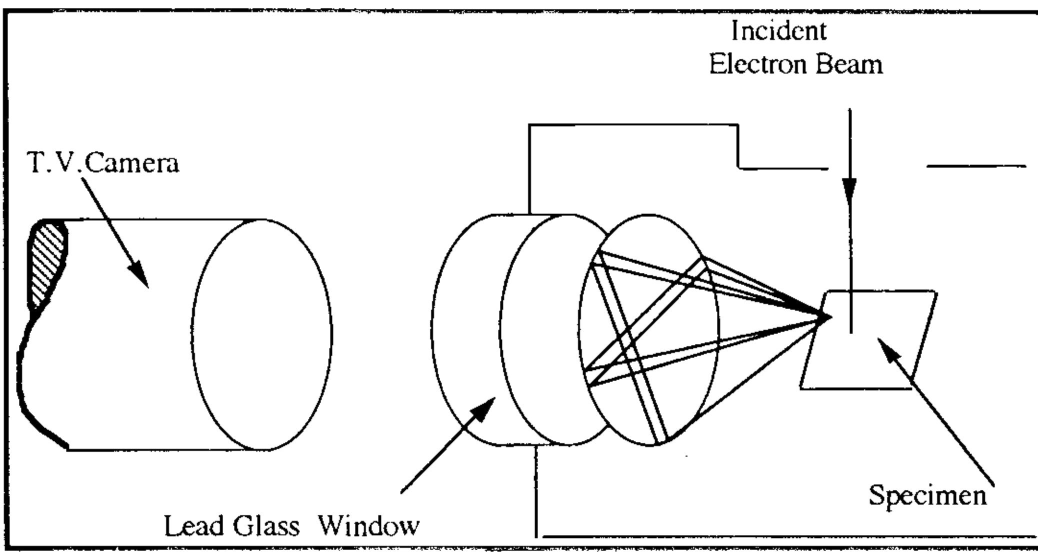


Figure 1. Schematics of BKD technique

Automation of this technique [10,11] through Orientation Imaging Microscopy (OIM) opened a new perspective in materials characterization which previously had only existed using TEM for thin foil specimens. In OIM the electron backscatter patterns are collected from points on the samples surface distributed over a regular grid. They are automatically indexed. From this data, a map, called an Orientation Image Micrograph (OIM), is constructed displaying changes in crystal orientation over the specimen surface. In the Orientation Image Micrograph the orientation of each point in the microstructure is known and hence also the location, line length and type of all boundaries. This information is used to construct a micrograph based on criteria input by the investor. For example, a contiguous grain may be defined as a crystallographic entity on the basis that all points within it must have an orientation within 15°. Its neighboring some other angle may be chosen. The resulting appearance of the microstructure may or may not vary significantly with the choice. What is important is choosing what the relevant criterion is for the material property being investigated.

Detailed microstructural and microtexture analyses of the specimens deformed to 15% and 660% were carried out using the OIM characterization procedures. Typical OIMs required several hundred to several thousand EBSP measurements to be taken on a hexagonal grid of points, with spacings of 0.2μm to several microns at each step. In addition to the orientation measured at each point, a QI parameter which represents the image quality of the electron backscatter patterns was determined. This parameter is associated with the presence and intensity of local plasticity and other crystal defects. Using the measured orientation for each pixel, grain boundary maps were constructed. At this point the criterion which defined grain contiguity and boundary misorientation was chosen. By assigning several different criteria in turn different images of the microstructure were constructed.

Material

The material used in this study was an 8090 Al-Li alloy of nominal composition Al-2.39 Li-1.21 Cu-0.64 Mg-0.12 (in wt%). Tensile Coupons were prepared with gage dimensions of 0.5x0.25x0.1 in. (in length, width, and thickness respectively) with the tensile axis parallel to the rolling direction. The specimens were superplastically deformed to different strains at 520° C and a nominal strain rate of $5x10^{-4}sec^{-1}$. Samples for both microstructural and microtexture analyses were electropolished, following an initial sequence of mechanical polishing.

RESULTS AND DISCUSSION

The as-received material was unrecrystallized and consisted of grains elongated in the direction of rolling, typical of similar studies on Al-8090 material [12]. An orientation image micrograph of the sample deformed 15%, showing image quality only is presented in figure 2. No grain boundary information is shown in the micrograph. In this figure, the darker regions represent locations in the microstructure where the quality factor is low, i.e. where poor quality EBSPs were obtained. Figure 3 is a second OIM constructed using the same data set as in figure 2 but now drawn to reveal grain boundaries across which the misorientation lies between 1 and 10 degrees, as thin lines and those boundaries across which the misorientation exceeds 10 degrees as thick lines. The thin lines may be considered as sub boundaries whilst the thick lines as

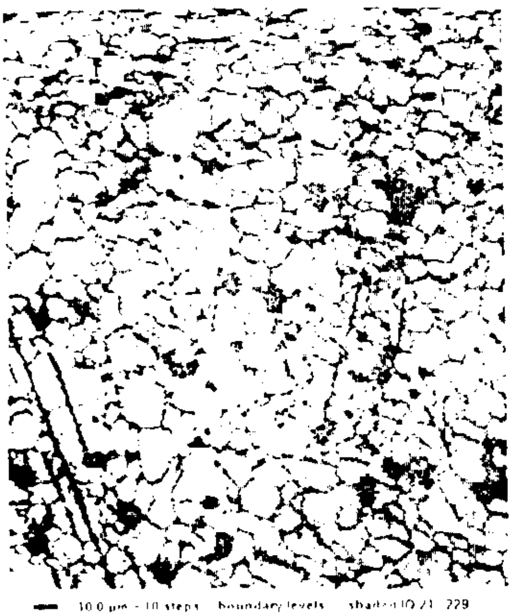


Figure 2 - An image quality micrograph for a 15% deformed SPF sample of Al-8900 material obtained from the Orientation Imaging Microscope.

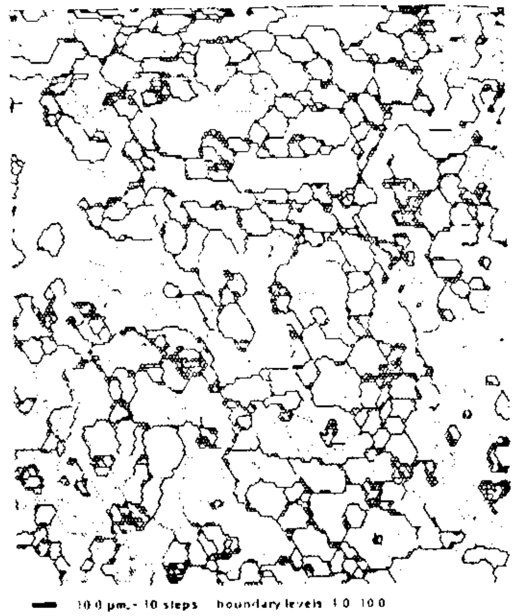


Figure 3 - An OIM Micrograph with thick lines representing high angle grain boundaries (>10°) and thinner black lines depict low angle grain boundaries.

Figure 4- Same as the previous figure with black lines depicting high angle boundaries. Three regions representing large grains are distinguished with three shadings the mid-region constitutes small grain structure.

true, or high angle boundaries. Both microstructures show the presence of an equiaxed microstructure of average grain size 10 μ diameter. Figure 4 is a third OIM from the same data set in which only grain boundaries across for which the misorientations greater than 10° are shown. It is now seen that the microstructure consists of fine grains sandwiched between a coarse grain structure. The corresponding pole figures obtained from the EBSP data are shown in figure 5. It is clear that the microstructure exhibits a [100] type texture with three strong variants.

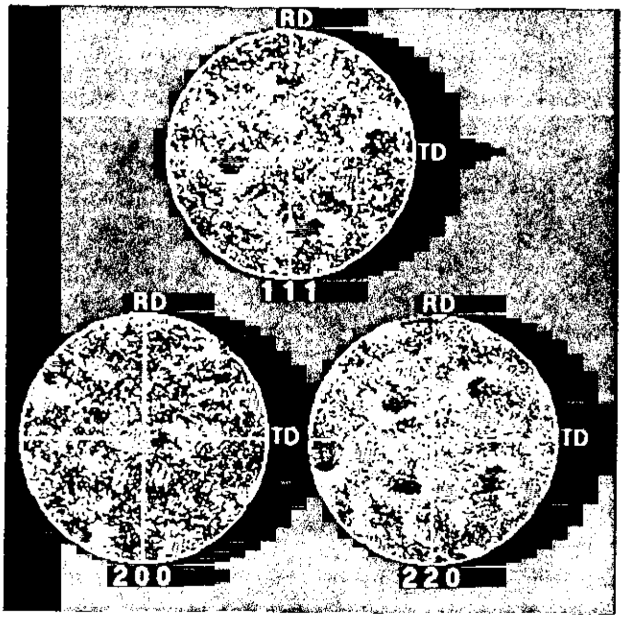
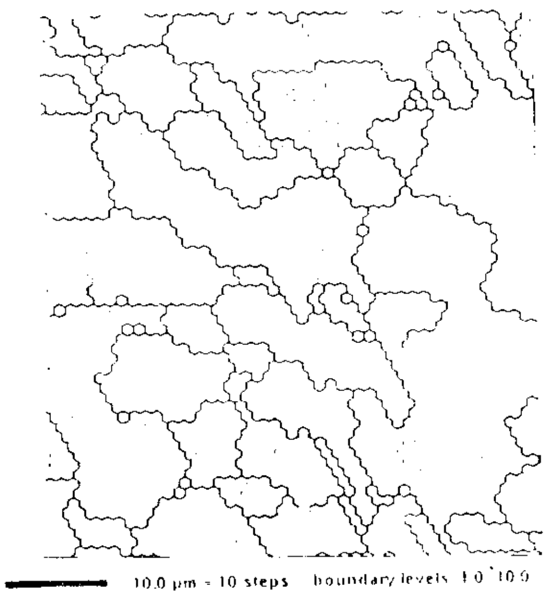


Figure 5- Pole figure representation of the previous figures. Black and gray and white high intensity poles represent the left and right regions of the microstructure.

The OIM micrographs for the specimen deformed to 70% elongation are shown in figures 6a,b. Figure 6a shows the microstructure constructed as in figure 3 with thin lines depicting grain boundaries across which the misorientation lies between 1° and 10°. The structure appears more equiaxed than that observed at the lower strain. the corresponding pole figures in figure 7 show a tendency towards randomization. In figure 6b is shown the corresponding OIM in which only the 10 ° boundaries are drawn. The visual impression is now quite different.



(a)

(b)

Figure 6- The OIM micrograph for the specimen deformed to 70% elongation. Figure 6a shows the microstructure constructed with thin lines depicting grain boundaries across which the misorientation lies between 1° and 10°. In figure 6b is shown the corresponding OIM in which only the 10° boundaries are drawn.

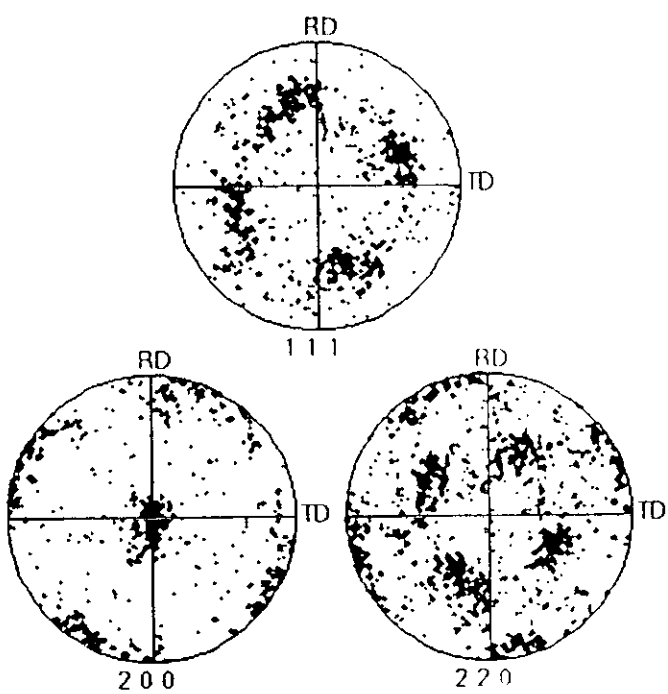
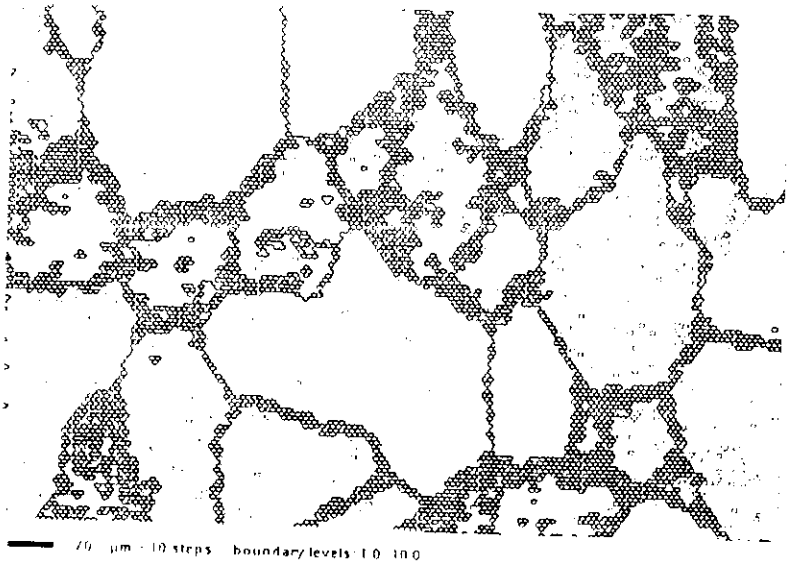


Figure7- The Pole figure representation of the microstructure in figure 6. This pole figure shows a tendency towards randomization.



(a)

(b)

Figure 8- OIM micrograph of superplastic specimens deformed to 660% deformation. Figure 8a shows an OIM in which the quality factor is depicted. In figure 8b, both low angle and high angle boundaries are drawn in as before as thin and thick lines respectively.

At the termination of superplastic forming, 660%, a significant amount of grain growth was observed (Figures 8a,b). Figure 8a shows an OIM in which the quality factor is depicted. In figure 8b, both low angle and high angle boundaries are drawn in as before as thin and thick lines respectively. However, it is seen that very few low angle boundaries exist and the structure consists of coarse grains bounded by high angle boundaries. The grain size is almost 200% larger than that observed in the corresponding OIM in figure 4 for the material deformed 15%. The thickness of the grain boundaries as depicted in figure 8a indicates a high level of damage at these interfaces. Pole figures for this level of strain indicates complete randomness of the microstructure.

CONCLUSION

This study has shown that Orientation Imaging Microscopy is an effective technique to investigate the evolution of the grain boundary microstructure in superplastic materials. Superplastic Al-8090 specimens were deformed in uniaxial tension to different strains at 520° C and $5x10^{-4}$ strain rate. The microstructure and microtexture for three strains corresponding to e=15%, e=77% and e=660% were characterized using Orientation Imaging Microscopy (OIM) technique. Using misorientation angle criteria of $\omega=5^{\circ}$, 10° or 15° to define grain boundaries, resulted in microstructures that differed in appearance. When a criteria of 5° (or less) was used to define the grain boundaries, three stages of deformation revealed equiaxed microstructures. When a definition of 10° (or more) was used, in the early stage of superplastic deformation (15%) the microstructure consisted of cluster of clusters of small equiaxed grains sandwiched by distinct large grains. At the later stage of deformation 660% strain, the microstructure was equiaxed and essentially the same as when the 5° criterion had been used.

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