

Orientation Imaging Microscopy Using ESEM

H. Garmestani, K. Harris, P. Kalu

2525 Pottsdamer Rd., FAMU/FSU College of Engineering, Department of ME
& Center for Materials Research and Technology (MARTECH),
Tallahassee, Florida, 32310

We have used an Environmental Scanning Electron Microscope equipped with the Orientation Imaging Microscopy attachment to obtain single orientation measurement in conducting (metals) and non-conducting materials. Electron Backscattered Pattern (EBSP) technique has been shown to be an effective tool for measuring lattice orientation of bulk polycrystalline materials in an SEM^{1,2}. Automation of this technique through Orientation Imaging Microscopy (OIM) has opened a new realm of materials characterization which was previously available only through Transmission Electron Microscope (TEM) of thin foil specimens³. Grain orientation and grain-to-grain misorientation are among a number of parameters which are crucial in studying the mechanisms of deformation in advanced materials. Such measurements have been successfully obtained for conducting specimens and under high vacuum conditions. The combination of ESEM and OIM provides the capability for measuring grain orientations in non-conducting crystalline materials with grain sizes of 1 micron or larger by eliminating the need for a conductive coating.

Our microcharacterization studies have initially focused on metals, specifically superplastic aluminum (Al-8090). TEM analysis of grain structure and subgrain formation could only provide information for individual grains or at best small regions of the microstructure, hence it does not provide a global picture. On the other hand, the data obtained by OIM technique and the regeneration of the microstructure based on different criteria to define grain boundaries by misorientation angle can provide a better understanding of superplasticity. Figures 1 and 2 show OIM images of a 25% biaxially deformed Al-8090 specimen (early stage of superplastic forming). To form these images, EBSPs were collected at points 1 micron apart (a six sided hexagon is used to represent each data collection point). Grain boundaries (sub-grain boundaries) are identified using different line widths for different small ($1-3^\circ$, $3-7^\circ$ in Figures-1a,b) and large (10° and above in Figure 2a) misorientation angles. The first micrographs in Figure-1 shows the presence of equiaxed structures. However, if the classical definition of 10° is chosen to represent the cut-off for large angle boundaries, a new microstructure is obtained which is shown in Figure 2-a. The corresponding pole figure is shown in Figure 2b. Detailed texture studies clearly identified the presence of a recrystallization texture at the early stage and texture randomization at the later stages of deformation.⁴

This study has recently been extended to investigate the effects of increased chamber pressure and the possibilities for using OIM to analyze non-conducting (semi-conducting) materials (ceramics and crystalline polymers). This technique shows promising results in metals under low vacuum conditions. The patterns showed very little increase in diffusivity under low water vapor pressure (less than 1 torr). EBSPs have been obtained from $\text{Bi}_2\text{Sr}_2\text{CuO}_x$ and efforts are currently underway to obtain OIM images.

References:

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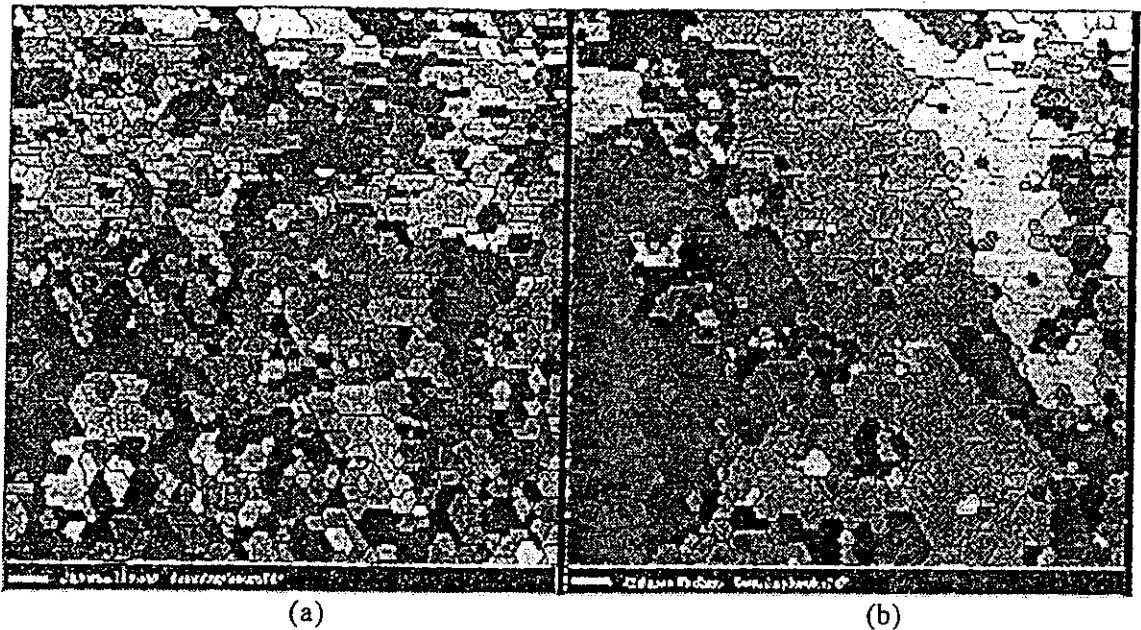


Figure 1- Misorientation boundary micrographs. Black lines depict boundaries with misorientations a- greater than 3° greater than 7°.

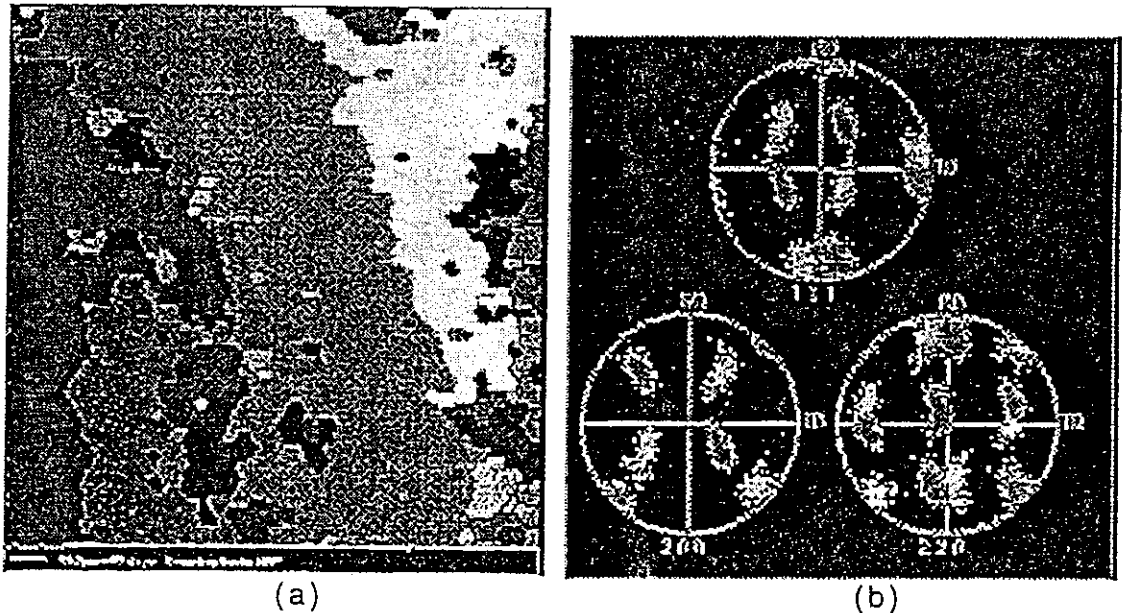


Figure 2- (a) 10° Misorientation boundary micrographs. (b) Pole figure representation of the previous figures. Different regions are represented by poles of the same color.