X-ray microbeam diffraction: Rapid multi-length-scale characterization spanning micrometers to millimeters. S.R. Stock. School of Materials Science and Engineering Georgia Institute of Technology

Keywords: instrumentation, materials, synchrotron.

With the advent of the newest generation of synchrotron x-radiation sources, capabilities for and interest in all x-ray microbeam techniques, including microbeam diffraction, has leapt forward. Diffraction with beam diameters one micrometer or smaller is a reality, allowing multi-length-scale, crystallographically-related characterization with micrometer resolution spanning fields (and depths) of view of millimeters and strains varying over at least four orders of magnitude. Furthermore, high brightness, high energy x-ray photons from the new storage rings allow diffraction from the interior of samples previously only accessible with neutrons. It is not yet clear what combinations of these attributes can be achieved simultaneously, and the focus here will be on different approaches to high resolution microbeam diffraction, with emphasis on the third-dimension, i.e., perpendicular to the sample’s surface.

First, formation of microbeams for x-ray diffraction will be reviewed briefly. Various approaches to determining the depth from which diffracted beams originate will be covered next. Different groups’ x-ray microbeam diffraction work will be used to illustrate areas of current activity, and these extend from grain subdivision processes to microtexture mapping to mesotexture quantification. Of particular importance is how these results can be incorporated into physically-based models of complex phenomena such as complex, high strain processing and fatigue crack closure. Challenges associated with the new capabilities will also be mentioned.

Application of electron backscatter diffraction to grain boundary surfaces. V. Randle, H. Davies, R. Wilson, I. Pearce, Department of Materials Engineering, University of Wales Swansea, Swansea SA2 8PP, UK.

Keywords: grain boundary, EBSD, serial sectioning.

Over the last few years the experimental technique of electron backscatter diffraction (EBSD) in a scanning electron microscope (SEM) has become a widely used materials characterization technique. One aspect of its use is the ability to record both the crystallographic orientation and spatial coordinates of a pixel and output these data pictorially as an orientation map of the microstructure.

Use of EBSD and mapping to investigate the distribution of grain boundary and facet crystallography is a key area of the Swansea research group. Much of the work is directed towards measuring not only grain orientations and grain boundary misorientations, which are extracted directly from EBSD data, but coupling the measurements to calibrated serial sectioning and trace analysis in order to extract the crystallographic indices of important surfaces such as fracture planes and internal interfaces. Whereas this process has as yet mainly concentrated on total section depths of less than the grain size, extension of the total section depth to greater than the grain size affords an opportunity to probe the connectivity of the grain boundary network. An additional requirement becomes, therefore, the ability to join orientation maps from sequential sections together to appraise the grain and boundary connectivity and subsequently to extract the crystallographic information.

A major advance has been made recently by using three-dimensional (3D) graphic utilities to reconstruct sectioned orientation maps. Although the original motivation for the research arose out of the need to find a less labour intensive (i.e. more automated) method for measuring the crystallography of surfaces, and for displaying the data in a map, the devised procedure can equally well be applied to a diversity of hitherto virtually inaccessible material measurements and problems arising from the link between topological parameters (grain volume, surface area, connectivity) and crystallography.

The material selected for pilot studies of 3D reconstruction was α-brass (63%Cu,37%Zn), since it is currently being studied in a large-scale investigation on the behaviour of grain boundaries in this alloy. The material was annealed for 24h at 900EC which resulted in a grain size of approximately 500µm. The procedures involved are:

X EBSD measurements
X calibrated sectioning
X 3D reconstruction
X extraction of data

This paper reports the crystallography of grain boundary surfaces in brass, the methodology involved in acquiring a 3D reconstruction of grain boundary surfaces, and some preliminary results.