3-D Measurement of Crystal Microstructure with Submicron Polychromatic X-ray Beams

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Introduction

An x-ray microprobe is capable of precisely measuring the strain, and orientation or crystal grains buried tens-to-thousands of microns deep in a sample. We are exploring methods of measuring the 3-D position of the grains so a real-space map of crystallographic strain and texture can be recovered for polycrystalline samples. Here we describe the use of depthtriangulation. This approach is very similar to the one taken by Stock and co-workers[1] but with vastly higher precision owing to the smaller incident spot size, rapid detector readout, more reliable positioning of the x-ray area detector, higher spatial resolution, and the close proximity of the x-ray area detector to the sample.

Methods and Materials

The basic approach used to measure the position of the diffracting grain is illustrated in Fig. 1. A high-resolution X-ray CCD area detector with ~1 Megapixels and with 22.5 μm pitch is used to observe the Laue pattern generated by a polycrystalline sample. The Laue pattern is observed over a series of distances from the sample. Characteristic features on the pattern are then fit and ray-traced back to the plane, parallel to the detector face, in which the incident beam passes through the sample. The intercept of the traced ray along the incident beam is the location of the diffracting grain. The plane-intercept of the traced ray perpendicular to the incident beam gives a measure of the uncertainty of the technique since the beam path is a straight line in the sample.

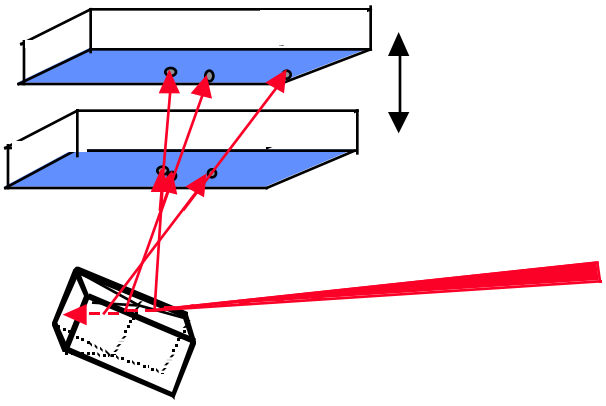

Fig. 1 Triangulation approach used to determine the grain location along the incident beam path.

measurements was grown at the Alcoa Technical Center and deformed in plane strain.[2] The tri-crystal had near columnar grains with [001] orientations near the surface normal. The (compression) deformation of the ~10x15x20 mm3 sample was carried out at 200° C in a channel die with the columnar and triple-junction directions along the zero strain direction of the channel.

The CCD image in Fig. 2 contains a diffraction pattern collected with the x-ray beam intercepting all three grains near the triple junction point. Distinct diffraction patterns from the different grains are present in the image. As can be seen the original triple grain has broken into a complicated subgrain pattern. For the measurements reported here, the sample normal was oriented at an angle of 45° toward the beam. Each diffraction pattern provides information on the deformation microstructure and sub-grain orientations in the deformed sample along a line with 0.7 x 0.7 μm^2 cross-section penetrating ~0.25-0.5 mm into the sample at a 45° angle.

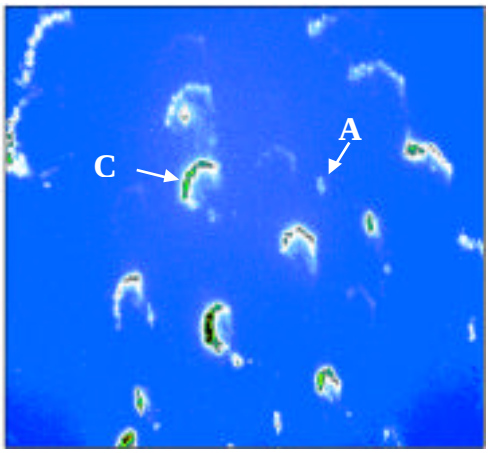

Fig. 2 Laue pattern showing characteristic crystallite grain patterns from two deformed grains at a grain boundary.

Results

The diffraction peak positions were determined by fitting two-dimensional Lorentzians to individual crystallite reflections within the arc A and cluster B of spots for 10 detector heights. These peak positions (in pixels) are plotted in Fig. 3. Figure 3 shows the locus of the peak positions as a function of detector distance projected onto the plane containing the x-ray microbeam (i.e. $Z_{Det} = 0$). The expanded view in the right hand panel indicates the position along the

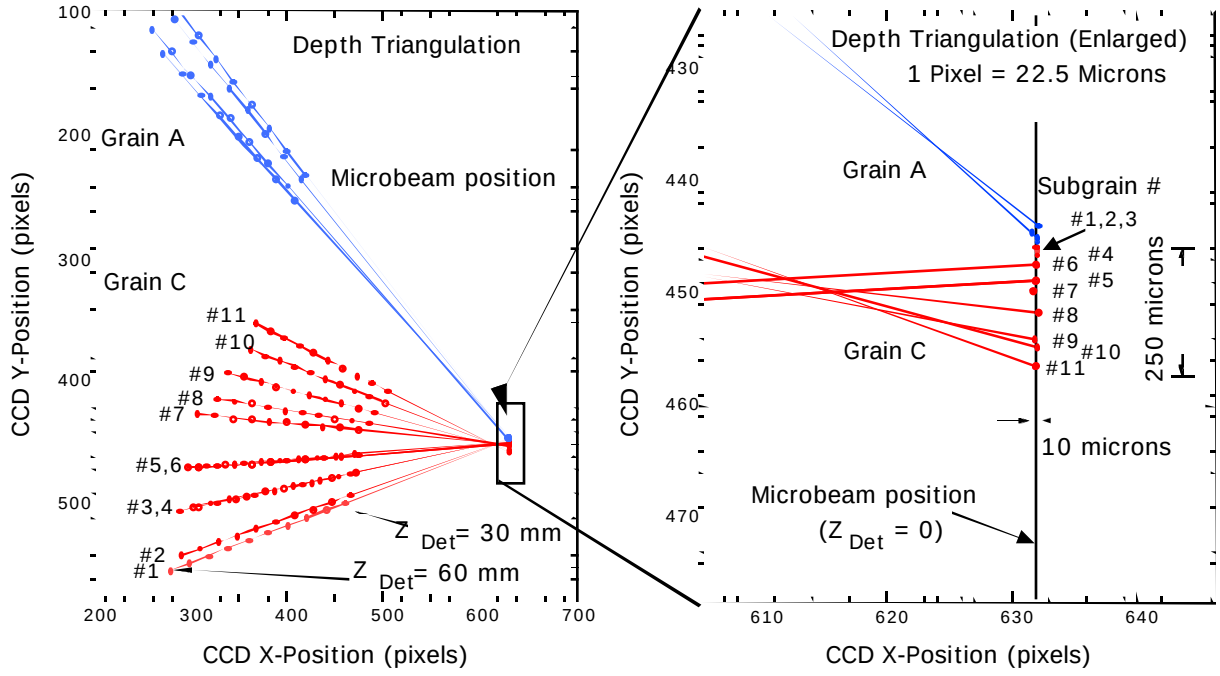

Fig. 3. Projection of diffraction peak positions for detector distances varying from 30-60 mm. The fit positions of various features in the diffraction pattern is indicated by the circles. The best fit to the fit positions is the straight line through the circles. As can be seen from the enlargement of the intersection region, the loci of the intercepts form a nearly straight line along the path of the incident beam.

beam that is identified as the source position for each of the diffraction peaks. Noting that the beam direction is implied to be from top-to-bottom in the figure, it can be inferred that the beam traverses ~50 µm through one grain and ~250 µm through another grain. We note that the x-position is determined by the microbeam path, so it is significant that the inferred x-positions from the triangulation form an essentially straight line. Since the x-position at $Z_{Det} = 0$ is determined in the same manner as the y-position, the deviations of the x-position from a straight beam line provide a measure of the uncertainties in the determination of the y-position (along the beam).

Using the deviations of the x-position and the fact that the two grains neither overlap nor leave a gap at the boundary, we estimate the uncertainty along the beam to be less than 5 microns, and in the 2-3 micron range in many cases. This precision is an order of magnitude smaller than the pixel pitch of the CCD detector and results from careful fitting of the peak intensities across several peaks and from an over-determination of the ray from the 10 positions of the detector. At present, triangulation on weak peaks or peaks that become multiple peaks for larger detector distances do not achieve this precision.

Discussion

Continued development of the x-ray microbeam techniques discussed here, including local stress/strain measurements[4], will provide new information regarding underlying issues involved in microstructural evolution. Microbeam x-ray techniques provide mesoscale information complementary to that presently available by TEM and electron backscattering OIM techniques. In particular, microbeam x-ray diffraction provides non-destructive bulk measurements and provide the possibility of measuring local stress distributions under loaded and/or dynamic conditions.

Acknowledgments

Research sponsored by the U.S. Department of Energy under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp. The x-ray measurements were performed on the MHATT-CAT beam line at the APS.

Use of the Advanced Photon Source was supported by the U.S. Department of Energy, Basic Energy Sciences, Office of Science, under Contract No. W-31-109-Eng-38.

References

1. S.R. Stock, A. Guvenilir, D.P. Piotrowsk and Z.U. Rek, Mat. Res. Soc. Proc. 375 275-280 (1995).
2. H. Weiland and R. Becker, in Proc. 20th Riso Intern. Symp. On Mat. Sci.: Deformation-induced Microstructures: Analysis and Relation to Properties, edited by T. Leffer and O.P. Pederson, Riso Intern. Laboratory, Roskilde, Denmark 213 (1999).
3. B. C. Larson, N. Tamura, J.-S. Chung, G.E. Ice, J.D. Budai, J.Z. Tischler, W. Yang, H. Weiland and W.P. Lowe, "3-D Measurement of Deformation Microstructure in Al(0.2%) Mg Using Submicron Resolution White X-ray Microbeams", MRS Fall Symposium (1999).
4. J.S. Chung and G.E. Ice, " Automated Indexing for Texture and Strain Measurements with Broad-Bandpass X-ray Microbeams", 86 J. Appl. Phys. 5249-5256 (1999).