Characterization of Welds with a Polychromatic X-ray Microscope

G.E. Ice,[1] B.C. Larson,[1] R. Barabash,[2] F.J. Walker[2]
[1]Oak Ridge National Laboratory (ORNL), Oak Ridge, TN, U.S.A.
[2]University of Tennessee, Knoxville, TN, U.S.A.

Introduction

Polychromatic x-ray microscopy is an emerging tool for characterizing materials [1]. With this scanning technique, spatially resolved microdiffraction measurements of crystalline structure can be made in two [1] or three dimensions [2] (2-D or 3-D) with submicron resolution. Properties that can be measured include the local crystalline phase, local texture (orientation), and local elastic and plastic strain tensors. With polychromatic x-ray microbeams, the crystal structure of subgrain volumes can be measured without the complication of sample rotations. Welded materials are a class of materials for which the 3-D x-ray crystal microscope will provide unprecedented research opportunities. In this report, an iridium (Ir) weld sample is used to illustrate how the 3-D x-ray crystal microscope can be used to study deformation and local crystal structure in welds [3].

Methods and Materials

Ir alloys are high-temperature materials with excellent toughness that are used to protect radioactive power sources for space applications. Welded Ir joints are an area of particular concern, and considerable effort has been devoted to the microalloying and welding processes that are needed to join Ir without cracks. Here we study the mesoscale structure of an electron-beam weld in Ir. Because Ir is a high-Z material, x-rays penetrate only a few microns into the surface before absorption; the current 3-D x-ray crystal microscope uses x-rays in a bandpass from 10 to 24 keV that penetrate about 1 to 3 μm into Ir. For low-Z samples like aluminum (Al), regions up to 1 mm below the surface can be probed with our current instrumentation.

With a probe size of about 0.7 × 0.6 μm^2 and a penetration of about 2 μm, the total volume of material sampled with each Laue image is $<1\ \mu m^3$. A photomicrograph of the welded sample is shown in Fig. 1. Overlain on the photomicrograph is a false color map of the grain orientation as determined by the polychromatic x-ray microscope. Again, the grain structure is clearly resolved. Typically, x-ray microdiffraction measurements have an orientation uncertainty of about 0.01°, which is about 100 times more precise than electron backscattering diffraction (EBSD) measurements. Images of mesoscopic weld structures were constructed by analyzing an 80 × 80 grid of Laue images taken in 3-μm steps. The location of image 3031 (discussed later and shown in Fig. 2) is identified on the Fig. 1 image.

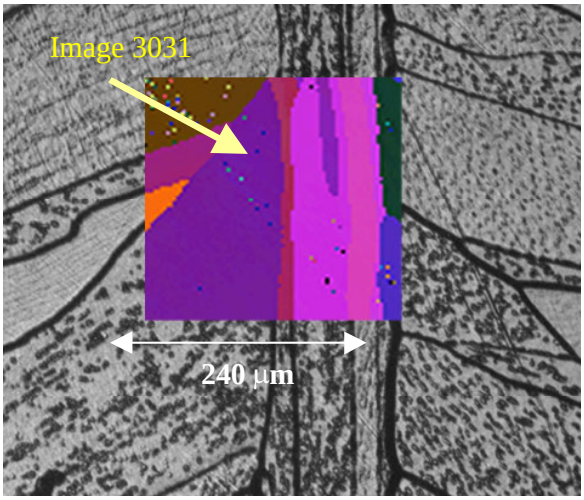

FIG. 1. False color x-ray representation of the surface grain orientation in an Ir weld overlain on an optical photomicrograph of the weld. Note that the grain orientations are measured to ~0.01°.

In addition to orientation information, the x-ray microdiffraction images are sensitive to the local elastic strain [1]. Angles between reflections in the Laue measurements are sensitive to deviatoric strains. Hydrostatic strains do not change the angles between planes, and the energy of at least one reflection must be measured to determine the hydrostatic strain. For materials in which plastic deformation is small, the strain tensor elements can typically be measured to $\sim 2 \times 10^{-4}$ to 2×10^{-5}. For highly deformed materials, elastic and plastic deformation must be treated simultaneously, and sophisticated analysis methods must be used to precisely determine the elastic strain tensor distribution. Welds studied so far have modest deformation.

Results

Although plastic deformation complicates the analysis of elastic strain, the measurement of local plastic deformation in welds is an area where polychromatic microdiffraction can have an immediate impact. As seen in Fig. 2, the Laue images from near a weld show significant streaking, even though the submicron x-ray beam samples only 1 μm^3 of material.

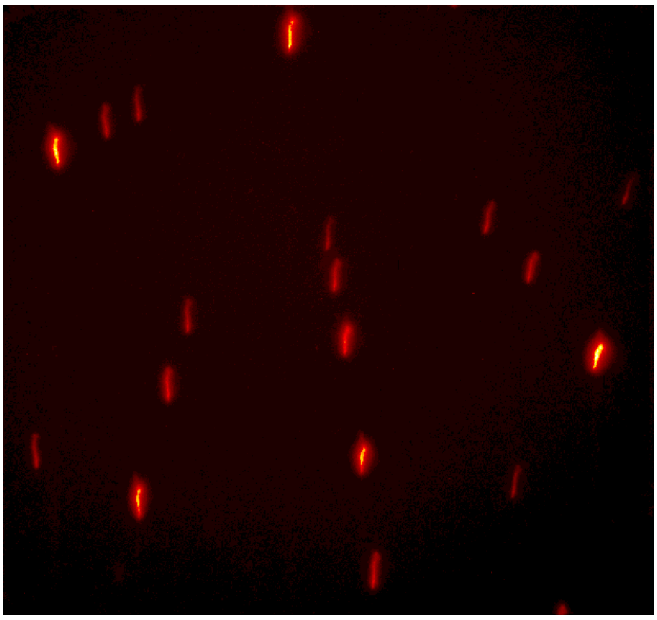

FIG. 2. Laue beam image 3031 from a single Ir grain in the weld. Within the boundary of this grain, the streaking direction did not change systematically as the x-ray beam was translated to different locations on the grain.

The presence of streaking means that the crystallographic planes are rotated within the 1 μm probed by the beam.

The length of the streaking is proportional to the number of unpaired dislocations, and the direction of the streaking is sensitive to the kind of unpaired dislocations and their distribution in the grain [4]. To ensure that the deformation is not a surface effect introduced by polishing, the samples were carefully polished and etched. Not only did the pattern remain the same, but measurements made on the base material showed orders of magnitude less streaking after the same surface preparation.

Streaking in image 3031 was modeled to identify the active slip system(s) at this grain location. As indicated in Fig. 1, image 3031 was recorded from a volume away from the grain boundaries. In fcc crystals, the most common edge dislocation system is a <112> type with <110> type Burger's vectors. There are 12 <112> type line directions and there is one <110> type perpendicular Burger's vector for each of the 12 line directions. Hence, there are 12 primary dislocation systems for each grain orientation. In addition, various other slip systems are observed. As a first approximation, we searched for 30 single slip systems or 18 multiple systems to find the best fit to the measured streaking.

Image 3031 is taken from a grain with an ~001 surface normal. The streaking of the Bragg peaks runs almost parallel to the weld axis. The best fit to the streaking direction is found for an edge dislocation slip system with a 211 line direction and with a $1\underline{1}0$ Burger's vector. Figure 3 shows the sample reference frame (xyz) and four unit cells that illustrate the dominant unpaired dislocation line direction and Burger's vector. The sample surface normal z is about 9° off parallel to the unit cell 001 axis. The weld axis runs parallel to y and is perpendicular to the 211 unit cell direction, as illustrated. For this particular case, the good fit to a single primary dislocation system suggests that the local deformation arises from this one system. In other grains, such simple fits are not always found.

In addition information on the types and approximate numbers of unpaired dislocations, some information about their organization within the probed volume can be inferred from the Laue images. As shown in Fig. 4, the overall pattern of the streaking fluctuates from point to point but remains surprisingly uniform over the surface of the grain; local fluctuations within a small (i.e., 27×27 μm^2) area are typically as large as they are over the entire grain surface. Indeed, as shown in Fig. 4, whereas the streaking in image 3031 is primarily along one direction, in neighboring images, there is evidence for multiply active dislocation systems; the streaks are curved. The overall nature of the images from a single grain, however, does not change systematically across the weld grains measured.

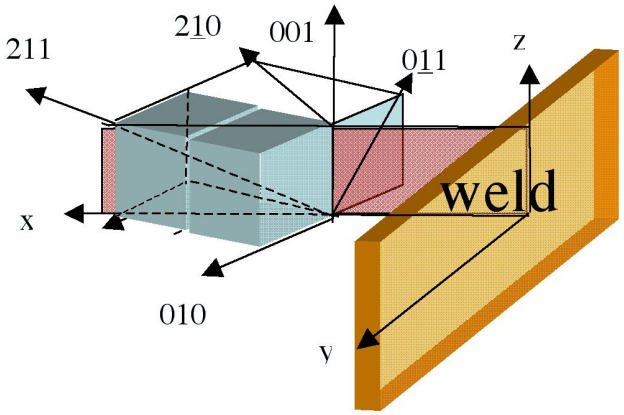

*FIG. 3. Weld axis and unit cell orientation at grid position 3031. The weld axis runs in the y direction with the surface normal of the sample in the z direction. The unit cells of the grain have their 001 axis nearly parallel to the sample z direction (about 8° of misalign*ment*). The 211 line direction of the dominant unpaired dislocation system is perpendicular to the y axis and about 17° from the x axis in the x-z plane. Lines observed at an angle of 45° to the weld axis in the false color map of streak length (Fig. 4) are in the 2$\underline{1}$0 direction.*

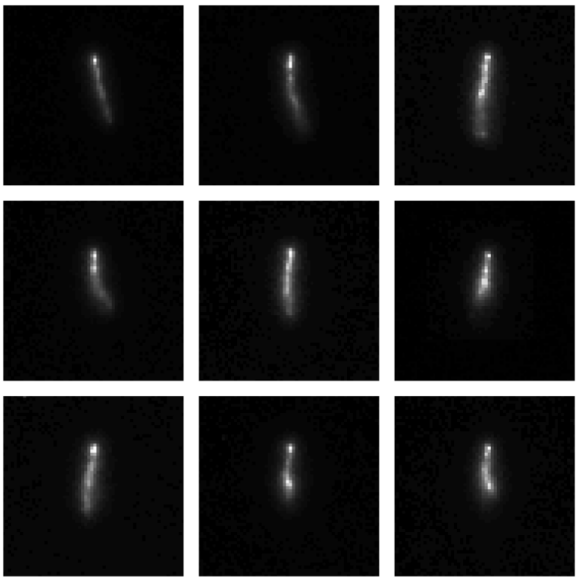

FIG. 4. -114 Laue reflection for the 3031 Laue image and its eight nearest neighbors. Note that although the overall direction and magnitude of the streaking are similar, there are subtle differences in the direction, magnitude, and structure of the streaking.

Discussion

As illustrated above, the study of welded materials with the 3-D x-ray crystal microscope is very much in its infancy. Efforts are underway to automate the analysis of the Laue images so that information about deformation and elastic strain is provided automatically. Ir weld samples are particularly easy to analyze with x-ray microdiffraction, because only one or two grains can be illuminated simultaneously. However, Ir samples are among the worst possible for 3-D studies; even 20-keV x-rays can penetrate only a few microns into Ir.

An area of particular interest for future development is an understanding of the grain boundary types in welds. With polychromatic x-ray microdiffraction, it is possible to survey the grain boundary morphology and crystallographic mismatch. Until now, this important information was very difficult to obtain because it required painstaking serial sectioning of specimens. However, serious grain boundary studies will require the use of lower-Z materials like Al or the development of techniques that extend the 3-D x-ray crystal microscope to higher x-ray energies.

Acknowledgments

This work was sponsored by the U.S. Department of Energy (DOE), Office of Science, Office of Basic Energy Sciences (BES), Division of Materials Sciences and Engineering. ORNL is operated by UT-Battelle, LLC, for DOE under Contract No. DE-AC05-00OR22725. Measurements were performed at the MHATT-CAT sector 7 beamline at the APS. MHATT-CAT sector 7 is supported in part by DOE Grant No. DE-FG02-99ER45743. Use of the APS was sponsored by DOE BES under Contract No. W-31-109-ENG-38.

References

[1] J.S. Chung and G.E. Ice, J. Appl. Phys. **86**, 5249-5256 (1999).

[2] B.C. Larson, W. Yang, G.E. Ice, J.D. Budai, and J.Z. Tischler, Nature **415**, 887-890 (2002).

[3] G.E. Ice, F.J. Walker, B.C. Larson, J.-S. Chung, S.A. David, and E.K. Ohriner, "Quantitative three-dimensional x-ray microprobe measurement of plastic and elastic deformation in welds," in *Proceedings of the 6th International Conference on Trends in Welding Research* (ASM International, Materials Park, OH, 2002), p. 873.

[4] R. Barabash, G.E. Ice, B.C. Larson, G.M. Pharr, K.-S. Chung, and W. Yang, *Appl. Phys. Lett.* **79**(6), 749-751 (6 August 2001).