Deformation Microstructure under a Nanoindent in Copper

W. Yang, B. C. Larson, G. E. Ice, J. Z. Tischler, J. D. Budai, K.-S. Chung
Oak Ridge National Laboratory, Oak Ridge, TN, U.S.A.

Introduction

The resistance to deformation by sharp point indentation is a standard method to measure the hardness of materials. However, the hardness of metals measured by nanoindentation is found to depend on the depth of the indent. The (apparent) hardness decreases by a factor of about 3 with increasing indent depths. Theoretical efforts to remove this ambiguity have led to a consideration of strain-gradient effects and the introduction of a material-dependent length scale for deformation [1, 2]. Unfortunately, measurements of the deformation microstructure under nanoindents have been very limited, and detailed nondestructive measurements have not been available. We have used polychromatic x-ray microbeams at the APS to make the first nondestructive, micrometer-resolution measurements of the 3-D deformation microstructure under nanoindents.

Methods and Materials

Micrometer-resolution measurements of the deformation microstructure in a nanoindented Cu single crystal were performed by using differential-aperture x-ray microscopy (DAXM) [3] in connection with Laue patterns obtained by using ~0.5-µm-diameter polychromatic microbeams. DAXM measurements provide direct, 3-D measurements of the plastic deformation induced lattice rotations as a function of depth along the penetration direction of the microbeam. Fig. 1(a) shows a schematic of microbeam probes entering a sample at an angle of 45° with the surface; also shown is an atomic force microscopy (AFM) image of a 100-mN Berkovich indentation in a <111> oriented single crystal of Cu. Figs. 1(b) and 1(c) show Laue diffraction charge-coupled device (CCD) images of the distorted lattice for microbeams entering the sample at positions B and C. The beam entering at position B penetrates beneath the center of a single flat face of the indenter, while the beam entering at position C probes near the intersection of two indenter faces and passes under the sharp edge of the Berkovich blade. Absorption limits the penetration depth of the x-ray probe to ~40 µm in Cu; however, the deformation distribution from the ~2-µm penetration depth of a 100-mN indent is contained within this volume.

Results

The CCD images in Fig. 1(b) contain Bragg diffraction streaks instead of sharp Bragg spots. Since white-beam Laue diffraction produces a one-to-one mapping of specular Bragg scattering onto the CCD x-ray detector, these streaks provide a direct measure of the local plastic strain (i.e., lattice rotations) along the path of the microbeam [4]. The relatively straight diffraction streak at position B indicates that the lattice rotations beneath the center of indenter faces are around an axis normal to the streak and along the top (horizontal) edge of the indent. The full pole figure in Fig. 1(b), obtained through detailed DAXM analysis of the position B diffraction pattern, indicates the positions of the four 111 poles. It also shows an expanded view of the pole figure containing micrometer-resolution depth-dependent measurements of the local lattice rotations starting at the surface position **s** and moving directly toward the undistorted lattice position **x**.

The triangular diffraction streak pattern for position C illustrates the increased complexity of the lattice distortions as the microbeam passes under the sharp blade at the intersection of two indenter faces. The schematic drawing in Fig. 1(a) depicts the expectation of both positive and negative rotations for position C, but the drawing does not predict the strong modulations in the diffraction streak intensities, nor does it anticipate the presence of three distinct rotation directions. The expanded pole figure for position C in Fig. 1(c) shows the results of the DAXM analysis of the triangular streaks, where **s** denotes the starting or surface rotation relative to the undistorted (111) Bragg position **x**. As the dashed arrows indicate, the rotations initially increase to the upper right and then jump rapidly (in <1 µm) to the lower right of the undistorted position **x**. The rotations then proceed rather quickly to a position near the **x** position.

Discussion

These results indicate that the deformation field below Berkovich indents in Cu is highly heterogeneous, with sharp angular rotation jumps. More complete nanoindent microstructure measurements are presently being analyzed in connection with the predictions of

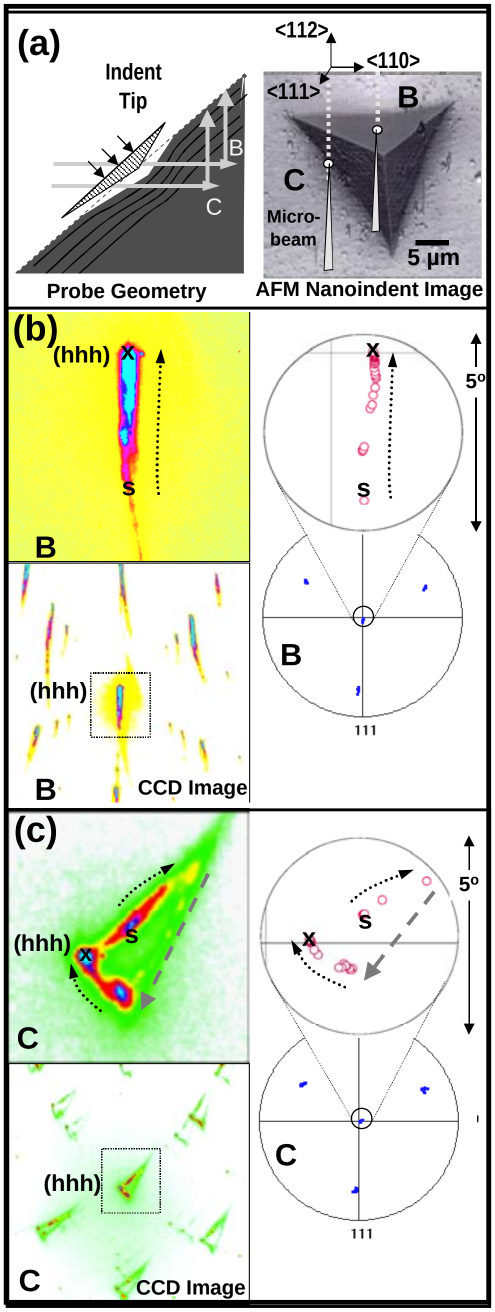

FIG. 1. Deformation microstructure in nanoindented Cu. (a) Illustration of nanoindent and microbeam geometry depicting microbeams probing the distorted lattice and an AFM showing the indent geometry and the microbeam probe for positions B and C. (b) White-beam Laue image and micrometer-resolution depth-resolved pole figure for position B. (c) White-beam Laue image and micrometer-resolution depth-resolved pole figure for position C.

strain-gradient and materials length scale theoretical models. The aim is to develop a fundamental understanding of deformation microstructure and its relationship to materials properties such as hardness.

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