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PERSISTENCE OF TWINS IN HIGHLY NANOTWINNED COPPER UNDER INTENSE SHEAR STRAIN

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ABSTRACT

A nanostructured material must maintain its microstructure under deformation if it is to preserve its high strength and other desirable properties. The goal of the present study is to determine the changes produced by high pressure torsion in high purity copper consisting of highly aligned columns of nanotwins separated by coherent sigma 3 interfaces. Previous studies have examined the effect of tension-tension cycling, which leads to the creation of softer, apparently detwinned regions at the surfaces that form surface grooves and serve as crack initiation sites. Unidirectional high pressure torsion (HPT) is used here to examine the effect of shear strain. Deformation by HPT is found to be highly concentrated near a sample’s surfaces, with the twin structure destroyed some distance into the sample interior. Between the sample surface and the remaining twins is an apparently detwinned region consisting of large grains. Closer examination by TEM shows that these “large grains” still remain nanotwinned even though subjected to very high shear strains. Furthermore, apparently detwinned columns in the sample interior consist largely of elements of nearby twins and matrix. Although deformation can cause notable changes to the microstructure, the nanotwinned structure is remarkably persistent.

1. INTRODUCTION

It has been well–established that nanotwinned materials possess a number of attractive properties (e.g., Lu, Lu and Suresh 2009). Like nanocrystalline metals with 3D grains, nanotwinned materials are strong, with the twin boundary spacing playing an analogous role to nanocrystalline grain size (Shen, Lu, Lu, Jin, Lu 2005). However nanotwinned metals tend to have higher ductility and improved microstructural stability under deformation (Anderoglu, Misra, Wang and Zhang 2008; Shute, Myers, Xie, Li, Barbee Jr., Hodge and Weertman 2011a; Qin, Lu, Tao, Tan, and Lu 2009). High purity nanocrystalline metals undergo rapid grain growth under stress/strain, even at cryogenic temperatures (Zhang, Weertman and Eastman 2005). Recent work has shown the relative stability of high purity nanotwinned Cu under
various forms of deformation, including compression, tension-tension cycling, and torsion (Hodge, Furnish, Shute, Liao, Huang, Hong, Zhu, Barbee Jr., and Weertman 2012). In the case of tension-tension cycling detwinned regions were seen to form at the sample surfaces (Shute et al. 2011a). These discontinuities in the structure are sites for crack initiation and failure. In the case of torsion, twins appear to be destroyed near the surface, where the shear strain is highest, turning into “large grains” (Shute, Myers, Liao, Li, Hodge, Barbee, Zhu and Weertman 2011b). However recent careful TEM observations show that at least some of the “large grains” are still twinned (Hodge et al. 2012). The nanotwinned structure can persist to higher strains than previously believed. It is the aim of the present research to determine how well the original nanotwins actually survive under high pressure torsion (HPT).

2. EXPERIMENTAL PROCEDURES

High-purity (99.999%+) Cu foils were deposited onto Si (100) wafers by interrupted DC magnetron sputtering, as described in previous publications (Hodge, Wang and Barbee 2006, Hodge, Wang and Barbee 2008). The films, which were ~ 170 μm thick, could be removed from their substrates and handled as free-standing foils. After polishing the surfaces the thickness of a typical sample was ~ 150 μm. The initial microstructure consisted of columns, aligned parallel to the growth direction, that contain twins separated by coherent Σ3 interfaces (normal to the growth direction). The columns viewed in a plane parallel to the twin boundary (TB) interfaces were generally equiaxed and varied widely in diameter, usually between 0.5 and 1.0 μm. Spacing between twin boundaries varied even more widely, with a median value of 35-40 nm. Most of the samples in the present research came from a single deposition.

For the study of the effect of shear strain on the nanotwinned Cu, a 10 mm diameter disk was cut from the as-deposited material by electrodischarge machining. Rough edges and scratches were polished with a series of diamond suspensions to a 1 μm finish. The flat surface of the disk was closely parallel to the plane of the TBs. Shear deformation was carried out by HPT between flat anvils under a compressive stress of 3 GPa. The disk was twisted by 180° and examined ~ 1mm from the center of rotation. The disk rotation rate was 1.6 rpm. Microstructural changes produced by the HPT were examined on cross-sectional cuts milled out with a focused ion beam (FIB) and by transmission electron microscopy (TEM). The cuts were oriented for maximum shear strain. Figure 1 shows the orientation of the cut surface with respect to the rotation direction.

![Fig. 1. Sketch of a Cu disk and orientation of FIB cuts made 1 mm from the center of rotation and oriented for maximum shear strain.](image)
3. RESULTS

3.1 Columns of twins in Region 1. The near-surface structure of a nanotwinned sample after twisting through a half-turn, as seen from one mm from the center of rotation, is shown in Fig. 2.

It can be seen that the structure varies considerably with depth from the surface. The first few hundred nm consist of very fine grains (Region 3), which gradually change into what appear to be large grains (Region 2), and finally about 5 \( \mu \)m from the surface the original columns of twins are evident (Region 1). The median twin boundary (TB) spacing and column widths in Region 1 do not seem to have changed notably but the columns are sheared in a direction parallel to the strain imposed by the HPT operation. (See Fig. 3a, which shows that the TBs remain parallel to the specimen surface.) In many cases adjacent columns of twins in Region 1 transition from one to another with no intermediate structure (Fig. 3a, left side) but in others there appear to be detwinned regions between adjacent columns (Fig. 3a, right side). Close examination of the pair of columns labelled 1 and 2 in Fig. 3a show that they have similar orientations. Henceforth the components of the pair are labelled M-1 and T-1, for Matrix and Twins of pair 1/2. Columns 3 and 4 also have similar orientations; their components are named M-2 and T-2. The dark field images of M-1 and T-1 (Fig. 3b) demonstrate the twin/matrix relation between them. While columns 1 and 2 join without intermediate structure, as do 3 and 4, large areas between the two pairs (of which Region C is a part) appear somewhat detwinned (Fig. 3a). Figure 3c is an enlarged bright field image of Region C obtained when the sample is tilted near to the \(<011>\) zone axis. Relatively coarse twins are found and dislocation cells are developed, e.g., cells labeled as A-F.
Fig. 3. (a) TEM image of an area in Region 1. Note the numbering of columns 1, 2, 3, 4. $N$, $R$ and $T$ indicate the normal to the surface, and the radial and torsion directions with respect to the center of rotation, respectively. Extension of the torsion direction shows that the TBs are still parallel to the sample surface.
Fig. 3. (b) Dark field images M-1 and T-1, showing a matrix-twin relationship. The red arrows indicate matching points. (c) Enlarged bright field TEM image of Region C in the dotted oval in 3(a).

Figure 4 is a sketch of the results of an analysis of the extended detwinned column surrounded on either side by the pairs M-1,T-1 and M-2,T-2. Two additional M-T pairs (3,4) are found in the column. Most all of the larger structures are similar to components from the 4 M-T pairs. Only the cells labelled X have large misorientation angles with respect to the other components.
3.2 Structure of the “large grains” in Region 2. The structure between the very fine grains at the surface and the remaining twin columns in the interior appears to consist of large grains (Fig. 2, see also Fig. 3b in Shute 2011b). However a closer examination reveals that at least some of the “large grains” still are nanotwinned. The TEM image in Figure 5 covers parts of all three regions.

The small square white box indicates the location of the large grain in Region 2 to be re-examined. Figures 6a,b show dark field TEM images in this large grain of twin lamellae and the corresponding matrix. Although the shape of the large grain is no longer similar to that of the parallel-sided structures that make up the twin columns, Selected Area Diffraction (SAD) indicates a near-perfect twin/matrix relationship over the entire grain. However the boundaries between the twin lamellae and the matrix are still approximately parallel to the sample surface. While the large grain is still nanotwinned the twins seem to be disappearing, leaving a single crystal grain. The white square (marked b) in Fig. 7a delineates the crooked region of a twin lamella that is examined by HRTEM in Fig. 7b. Intense activity of Shockley partials is seen at the ledge and twin boundaries. The tip of the twin (square marked c) highlighted in Fig. 7a is also seen to involve large numbers of Shockley partials (Fig.7c)
Fig. 5. TEM image of nanotwinned sample after torsion showing all three regions, separated by dotted lines. The white box encloses the large grain to be studied. $N$, $R$, and $T$ have the same meaning as in Fig. 3.

Fig. 6. (a) Dark field image of twin lamellae in the large grain indicated in Fig. 5. SAD pattern shows near perfect twin-matrix relationship. (b) Dark field image of the corresponding matrix lamellae. Entire grain is either twin or matrix. Note: assignment of terms “twin” and “matrix” is arbitrary.
Fig. 7. (a) Crooked region of a twin lamella (square marked b) and end of twin lamella (square marked c) to be examined by HRTM. (b) Observation by HRTEM of the region inside square b. Note intensity of Shockley partials at ledges and twin boundaries. (c) Observation by HRTEM of the region inside square c. Large number of Shockley partials are observed at twin boundaries and at the end of the twin lamella.

Although the extent of the twinning is diminished in Region 2, a nanotwinned structure persists in this strained region. Figure 8, a modification of Fig. 4 in Shute (2011b), shows that the twins are more persistent than originally thought, sustaining very high shear strains. (See Shute (2011b) for calculation of the shear strain.)
Fig. 8. Shear strain vs. depth into the sample. The twin columns in Fig. 2 first appear at arrow $B$, but the new observations show that nanotwining persists closer to the sample surface and thus at higher shear strains, as estimated by the red arrow.

4. SUMMARY

A copper sample containing columns of highly aligned nanotwins was subjected to HPT involving a half turn under a compressive stress of 3 GPa. TEM and HRTEM measurements showed that at least some of the “large grains” in the region between the highly concentrated deformation at the surface and the sheared twin columns in the interior, actually still are nanotwinned. Thus it is seen that a nanotwinned structure can persist to higher shear strains than originally thought. Furthermore, careful TEM examination in the sample interior of a detwinned column between two pairs of sheared twin/matrix columns showed that the detwinned column consisted almost entirely of nearby twin and matrix elements with only minor components having high angles of misorientation.

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