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Influence of Large-Strain Deformation on the Microstructure, Texture, and Mechanical Response of Tantalum Bar

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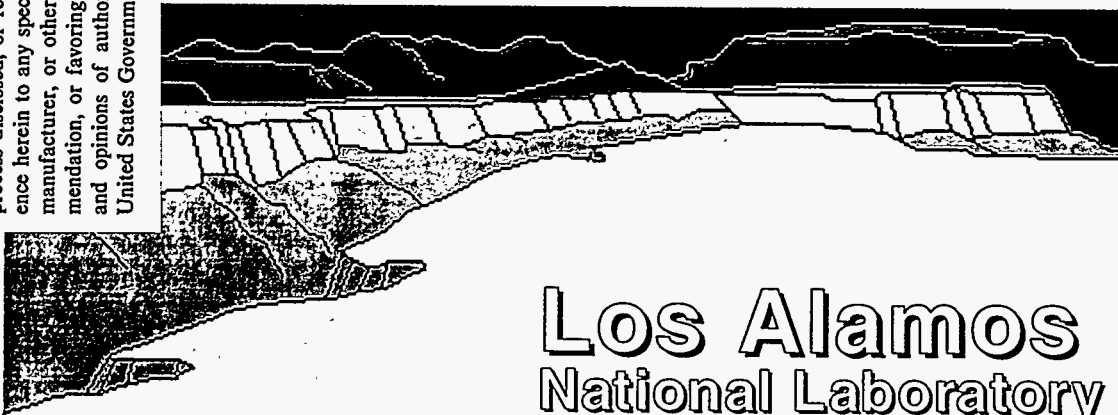
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INFLUENCE OF LARGE-STRAIN DEFORMATION ON THE MICROSTRUCTURE, TEXTURE, AND MECHANICAL RESPONSE OF TANTALUM BAR

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Abstract

Numerous studies have established the influence of impurities, crystallographic texture, temperature, and strain rate separately or collectively on the constitutive response of annealed tantalum, in particular plate Ta-stock. However, fewer detailed studies have examined the evolution of crystallographic texture and the mechanical response of tantalum bar or rod material following prestraining to large strains $\epsilon > 1$. In this paper the influence of large plastic prestraining on the microstructure evolution, texture evolution, and mechanical response of high-purity tantalum bar material is presented. Tantalum cylinders annealed at 1200 °C were quasi-statically upset forged, with intermediate lubrication, to true strains of 0.4, 0.95, and 1.85. Microstructural and textural banding within the starting Ta-bar was characterized in detail. It was found that different oriented bands evolved differently during large-strain forging leading to significant scatter in the mechanical response. Aspects of defect storage, work-hardening response, and texture evolution in Ta-bar as a function of forging strain are discussed.

Introduction

Since the 1950's, when widespread research on Ta, W, Nb, and Mo was escalated by widespread funding for the U.S. nuclear rocket-engine program, numerous studies have probed the microstructure-chemistry/property response of refractory metals[1-5]. During this period tremendous strides were made in understanding the melting and fabrication technologies, single- and polycrystalline deformation behavior as a function of interstitial and alloy content, creep properties, etc., driven by the focus of utilizing these metals in demanding applications where their high-temperature strengths and oxidation resistance were crucial. In more recent years the properties of a subset of these materials, specifically Ta, Mo, and W have again attracted intense scientific and engineering interest but this time focused on ballistic applications because these metals offer advantages due to their high density, excellent formability, and good fracture toughness (even at low temperatures). With this new application focus has come a shift in emphasis to: 1) understanding and

controlling production technologies affecting material behavior isotropy [crystallographic texture][4-7], 2) high-strain-rate mechanical behavior[3,8-10], 3) shock-loading behavior[11], and 4) alloying effects on mechanical response[12,13]. Tantalum, like all bcc metals exhibits deformation behavior which is markedly influenced by impurities, alloying additions, crystallographic texture, temperature, and strain rate[2-5].

Recent efforts have been focused on the optimization of tailored and reproducible microstructure / property relationships for a given application of tantalum. This emphasis has led to changes in the chemistry and processing controls used to produce current tantalum mill products, including control of crystallographic texture to affect in-plane homogeneity of Ta-plate materials. Concentrated studies of the influence of texture on the deformation response of tantalum alloys have been fostered by recent code simulations and experiments revealing an influence of anisotropy on material response in ballistic applications[14].

Understanding the mechanical response of metals at large strains is crucial to some defense applications where the materials are generally subjected to dynamic deformation to large strains (e.g., explosive-formed penetrators, shaped-charges, and Taylor impact tests). The other important issue is to investigate the effects of processing parameters on the properties of the metal in the deformed state since the final hardware is usually fabricated from heavily processed metal with possibly thermo-mechanical treatments during processing. The purpose of this paper is to report recent findings on the influence of upset forging to large strains on the mechanical properties and texture evolution, as a function of loading direction, strain rate, and starting texture of tantalum representing current tantalum bar-stock material.

Experimental

Material

High-purity tantalum bar material, hereafter referred to as Ta-bar, was purchased from Cabot Corporation; this bar

product is their marketed "ballistics grade" tantalum. The chemical composition of this Ta-bar is given in Table I. The bar was purchased in the as-rolled condition in order to perform annealing studies to determine the optimal recrystallization processing to produce the most uniform microstructure attainable. The Ta-bar material feed stock is triple electron beam melted under vacuum. It is then forged into a principally round cylinder. In some instances (depending on the end use), it receives an intermediate heat treatment prior to being rolled into bar. Throughout this processing, the original ingot axis is maintained in the cylindrical workpiece. The end result is that the centerline of the bar has received little deformation through all of the reduction steps described. The consequences of this on the microstructural and textural evolution will be discussed in the results section.

Table I Chemical Composition (ppm wt. %) of Ta-Bar

Nb	W	Mo	Ni	O	C	H	N
80	<25	5	10	65	10	<5	20

Following recrystallization at 1050 °C in vacuum, sections of Ta-bar were upset forged to true strains of 0.5, 0.95, and 1.85. A maximum true strain of 1.85 (84.3% height reduction) was achieved. The upset forging consisted of deforming the bar at a low strain rate along the axial bar direction between two flat steel platens. Two sets of experiments were performed. The first upset forging runs allowed for minimal stops in the deformation process, including minimal relubrication. The second set of forgings allowed for interruptions during deformation at approximately every 25% strain to relubricate the part and platens. Compression samples were machined from the annealed Ta-bar and the discs produced by upset forging. The large-strain compressive reload stress-strain behavior of the upset-forged bar, as a function of forging strain, was probed by reloading samples EDM-machined from the upset discs and reloading them at 298K at 0.001 s⁻¹.

The crystallographic textures of: the as-received, the Ta-bar as a function of annealing temperature, and the Ta-bar as a function of upset forging strain were measured using both x-rays and neutrons on sections normal to the bar axis. Bulk X-ray textures were measured on a Scintag X-ray unit using the Schulz reflection technique[15] with Fe K α radiation. Reduction and analysis of texture data were accomplished with the popLA software package[16]. Local texture gradients were characterized using orientation imaging microscopy (OIM) to discern local microtextural features as related to the microstructure. Neutron texture measurements were conducted at the Los Alamos Manuel Lujan, Jr. Neutron Scattering Center [LANSCCE].

The OIM interrogation technique provides a spatial distribution of texture through point-by-point measurements of crystal orientations utilizing an automatic technique based on rapid computer indexing of electron backscatter diffraction patterns in the scanning electron microscope[6,17,18]. Approximately 36,000 orientation measurements were made on a 2 mm x 14 mm hexagonal

grid with a step size of 30 μ m between measurements. The discrete measurements were shaded according to their orientation with respect to the sample axial direction (to facilitate comparison with the pole figures). For example, locations with <111> axes nearly parallel with the axial direction were shaded black, <100> axes are middle-gray, and <110> are white. The intensity of the shade and cross-hatching decreases with increasing misalignment of the particular <uvw> family of axes with respect to the ideal orientation represented by a given gray scale and shading.

Results

Annealing Study - Hardness & Microstructure

The microstructure of the as-received Ta-bar was characterized using optical metallography and then annealed at temperatures ranging from 900 to 1200 °C in vacuum to define the optimal heat treatment for recrystallization. Microhardness measurements suggested that the Ta-bar was recrystallized even at the lower temperatures as shown in Figure 1. However, optical metallography showed that the Ta-bar was not fully recrystallized until at T > 1000 °C.

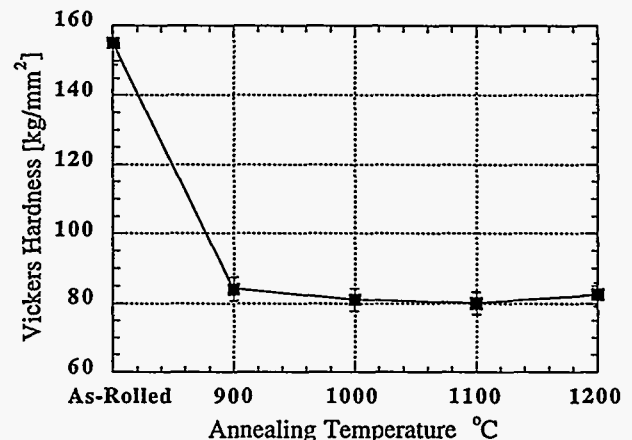


Figure 1: Effect of Annealing Temperature on Hardness of Ta-Round-Rolled Bar Material

Figure 2 shows the microstructures of the as-received Ta-bar, following annealing at 900 °C and after annealing at 1100 °C. Note that the as-received structure is extremely non-uniform with bands exhibiting heavy cold worked material adjacent to bands of material with minimal cold work. The as-recrystallized bar however, demonstrates significant grain size banding, having a minimum, maximum and average grain size of 19.9 μ m, 73.0 μ m, and 32.6 μ m, respectively.

The banding seems to be more evident from the centerline, see the micrograph on the far right, out to about one-half of the radius. The outer portions of the bar have recrystallized in a more uniform and consistent manner, although there is still some evidence of banding. During heat treatment, these different bands recrystallized to reflect this non-uniform deformation. The result is a microstructure with varying grain sizes and shapes, due to partial recrystallization. This type of microstructural

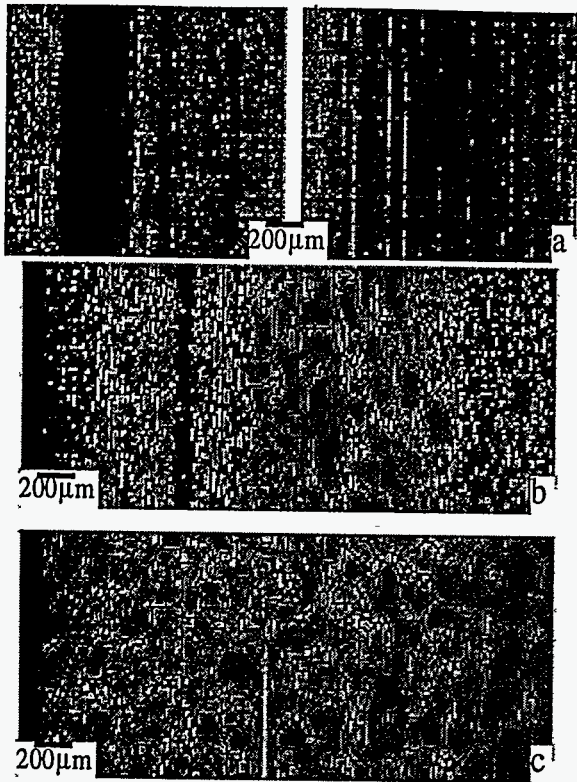


Figure 2: Metallography illustrating banding in Ta-bar in: a) as-rolled, b) annealed at 900°C, and c) annealed at 1100°C.

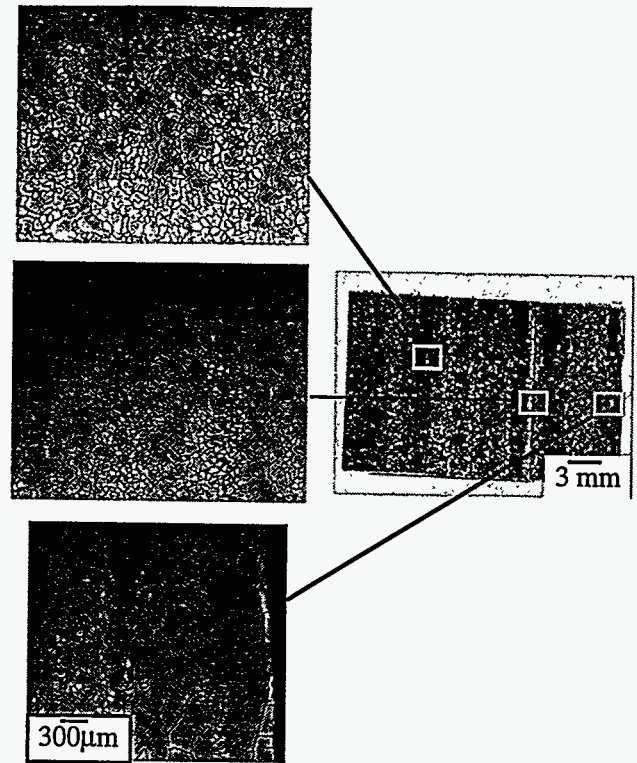


Figure 3: Metallography of Ta-bar recrystallized at 1050 °C illustrating microstructural banding

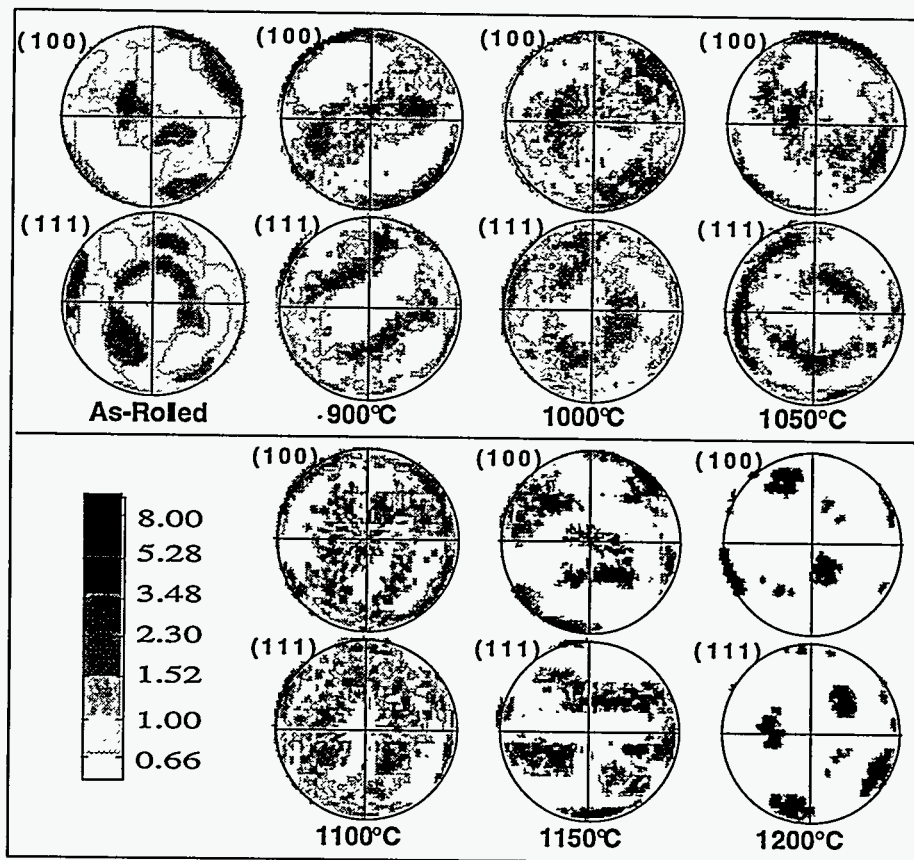


Figure 4: Texture of Ta-bar as a function of annealing temperature.

banding was found to strongly affect the subsequent mechanical properties and deformation behavior.

A recrystallization temperature of 1050 °C was chosen as the annealing temperature leading to the most uniform equiaxed microstructure although significant banding was found to persist as shown in Figure 3. This is demonstrated in the upset forging experiments that were performed on the bar recrystallized at 1050 °C, and discussed in the results section.

Annealing Study - Texture

The texture of the as-received and the Ta-bar material as a function of annealing temperature was measured using both x-rays and neutrons. Figure 4 displays the {001} x-ray pole figures as a function of annealing temperature. The most striking feature is the lack of a well defined symmetry about the axial direction to the pole figures. In addition, the trend of texture evolution with annealing temperature is ambiguous, except for the general increase in sharpness with annealing temperature. Since x-rays provide only near-surface information in Ta, neutron diffraction (ND) texture measurements were performed on selected samples to probe the through-thickness bulk texture.

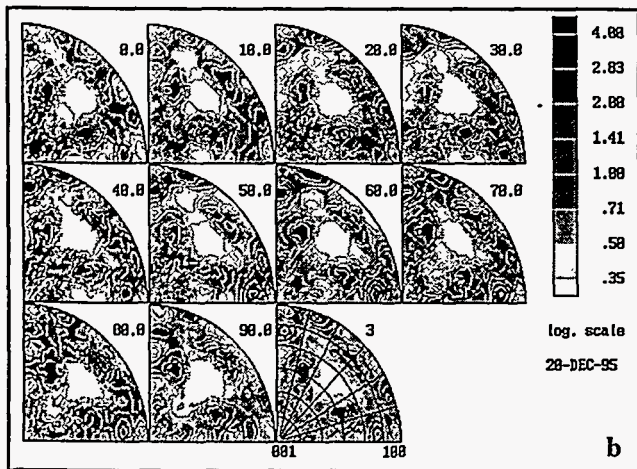
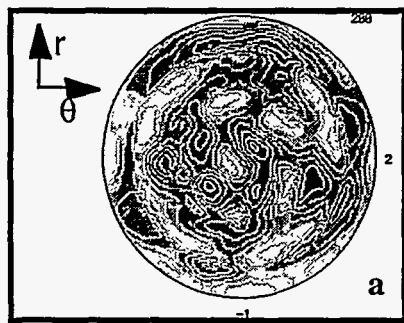


Figure 5: Neutron diffraction from as-annealed (1050 °C) Ta-bar: a) {001} pole figure, and b) axial-direction sample orientation distribution.

Figure 5 shows the results derived from ND measurements on the Ta-bar annealed at 1050 °C in the form of a {001} pole figure, and sections of the axial-direction sample orientation distribution function (SOD) plotted in polar coordinates. The projection of all SOD sections constitutes the inverse pole figure, which clearly shows a dearth of <111> and <100> axes aligned along the axial direction during forming operations. The asymmetric nature of the texture in the r-θ plane is still evident, indicating that the irregular texture is not limited to surface regions but exists through-out the bulk of the Ta-bar.

The texture results, in addition to the metallographic observations, support the supposition that the deformation processes employed to fabricate the Ta-bar lacked axial symmetry. A consideration of the typical method for fabrication of rolled bar as presented in Figure 6 lends credence to this hypothesis.

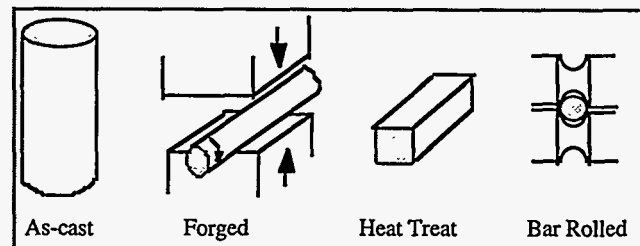
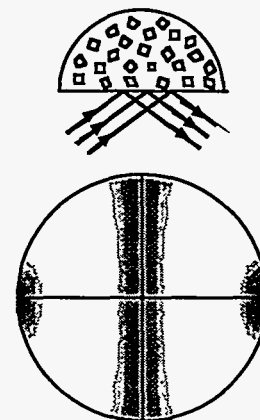


Figure 6: Schematic illustrating the processing used to produce round-rolled Ta-bar.

Unlike extrusion or wire drawing, both of which employ converging flow through conical dies and hence axisymmetric deformation, the forging and rolling stages of bar rolling are inherently asymmetric about the axis. This occurs due to tangentially non-uniform compressive stresses applied at discrete locations on the circumference of the bar followed by successive axial rotations. The process can be expected to impart a degree of preferred radial texture.

True Fiber Texture



Cyclic Texture

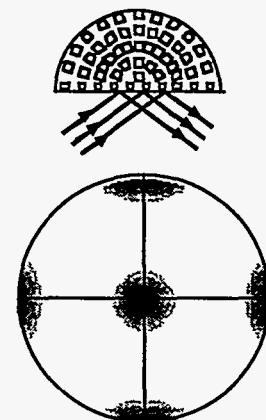


Figure 7: Schematic comparing orientation differences between fiber and cyclic textures.

Figure 7 shows a schematic contrasting a true fiber texture, which would be expected from uniform axisymmetric deformation processes, with an idealized cyclic texture. The corresponding pole figures display the difference in sample symmetry between the two textures. If pole figures were measured on the axial-normal face instead of the radial-normal face as shown, they would appear identical. Actual pole figures in this investigation were in fact measured on the axial-normal surface, but samples were sectioned small enough to isolate any in-plane anisotropy present. In addition to possible heterogeneities associated with the tangential direction, non-uniform through-radius deformations may also be encountered unless sufficiently large strains per pass are imposed. This could give rise to variations in both the deformed and recrystallized textures as a function of radial position.

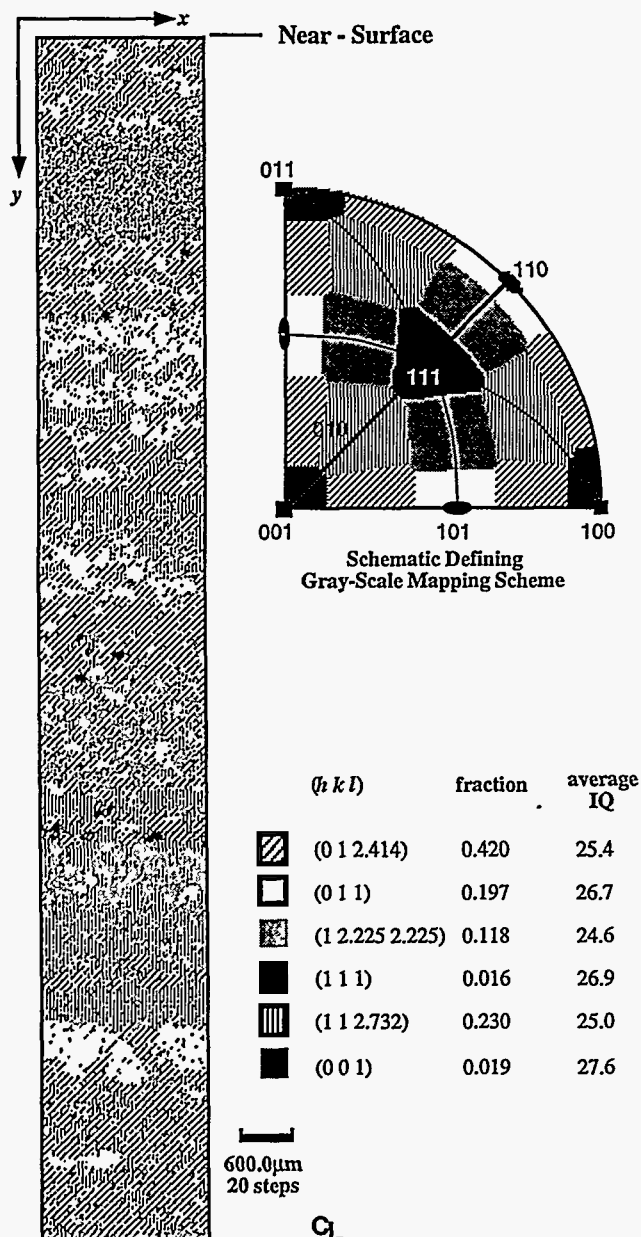


Figure 8: OIM gray-scale map showing (hkl) aligned with the rolling (x) direction for the round-rolled Ta-bar.

The 1150°C annealed bar was characterized by OIM to discern microtextural features to: 1) gain insight into the confusing bulk textures, 2) investigate the source of significant mechanical property variability as a function of position in the bar, and 3) determine if the microstructural banding previously observed (Figure 3) had a textural counterpart. Figure 8 shows the results of the OIM scan measured over the r-z plane, radially from the center-line to near-surface. The figure includes the intensity shading scheme projected on a quadrant of the (001) projection for a cubic crystal, along with corresponding volume fractions of (hkl) orientation components (planes parallel to the r-z plane), and the average IQ. The IQ values are a measure of the quality of backscattered patterns, and their insensitivity to orientation shows that the sampled microstructure was fully recrystallized.

The principal discovery from the OIM data is that a non-random spatial distribution of texture exists in the radial direction of the rolled bar, and this distribution is associated with grain size banding. The source of the position-sensitive texture gradient is thought to be due to insufficient or variable radial reductions per pass imparted during forge break-down and/or round rolling of the bar as detailed earlier. This can lead to radial strain gradients, associated inhomogeneity in deformation texture, and resultant non-uniform recrystallization textures.

Upset Forging Experiments - Microstructure / Hardness

The first set of forgings, recrystallization at 1050 °C in vacuum and upset forged to true strains of 0.5, 0.95, and 1.85, were characterized for microstructure and hardness as a function of total strain. Texture and mechanical property evaluation was done and as discussed in the following sections. Figure 9 details the results of the microhardness for the three upset forged Ta-bars.

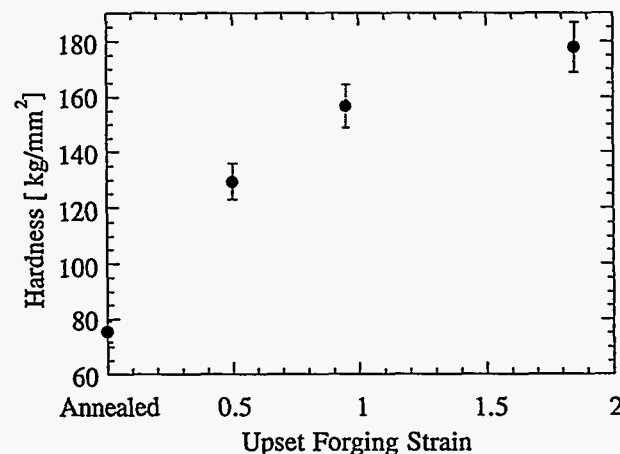


Figure 9: Hardness of Ta-bar as a function of upset forging strain.

The hardness of the forged discs is seen to increase as a function of forging strain with the absolute hardness increase per upset strain falling off at higher strains. This is consistent with the concept of a saturation stress, i.e.,

the attainment of a balance between work hardening and dynamic recovery processes leading to the asymptotic approach of a maximum flow stress level [dependent on temperature and strain rate] during forging. The microstructural banding present in the recrystallized Ta-bar, Figure 3, is seen to result in non-uniform deformation storage in the upset forged samples as a function of upset forging. At a forging strain of $\epsilon = 0.95$, as seen in Figure 10, differential slip-line density between differently oriented bands is observed consistent with different slip activation as a function of band orientation. The areas which were more uniformly fine and equiaxed grained deformed more uniformly, while the areas which had large, or unrecrystallized, grains demonstrated much less deformation substructure following forging.

Also as seen macroscopically in Figure 10, there is significant macrobanding present due to this non-uniform deformation. Finally, the effects of the banded structure are still evident in the sample upset to the true strain of 1.85, see Figure 11. Not only are there areas of minimal deformation and areas of very high local deformation, the macroscopic flow pattern indicates that there has been some friction effects present during this forging, note the sinusoidal flow patterns in the center of the lower micrograph; this sinusoidal flow pattern is on the outer portions of the forged discs where the classic "Friction-Hill" is known to be important. The outer portions of the forged disc have experienced lower constraint than have the central portions of the disc where the amount of surrounding material during forging is much greater. However, at the larger strain areas, the flow seems to be more uniform as expected with the majority of the material flow at the centerline of the forged billet.

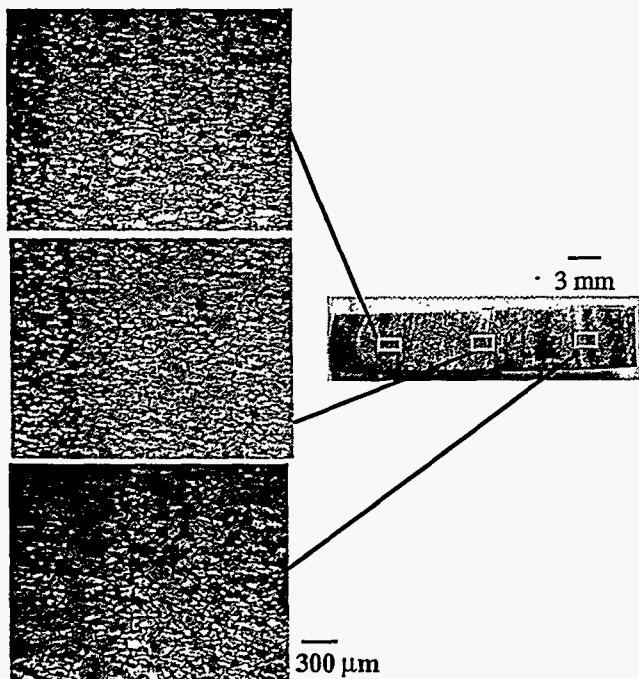


Figure 10: Optical photo of Ta-bar upset forged to $\epsilon=0.95$.

The macroscopic flow patterns also support that the measured textural banding would be expected to propagate through the deformation process.

The second set of forgings, with increased lubrication frequency, resulted in hardnesses and microstructures very similar to the first. The same macrostructural flow patterns were observed albeit the slight friction problem was reduced with increased lubrication. This finding supports that the majority of the observed forging behavior is primarily a function of the starting textural and microstructural banding rather than a manifestation of large frictional contributions. It was however noted that during the second forging run to $\epsilon = 1.85$, the forged disc became harder between upsets, as measured by the forging forces needed by the press on the disc to maintain plastic upsetting, after more frequent stopping for relubrication.

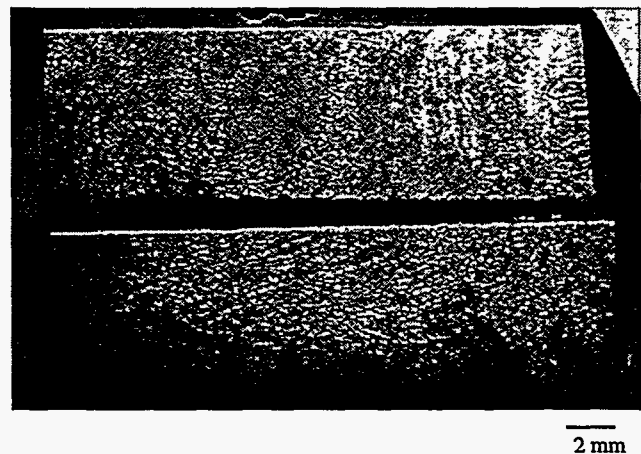


Figure 11: Metallography of macroscopic flow patterns in Ta-bar upset forged to $\epsilon=1.95$.

This observation is consistent with a contribution of strain hardening, and will be investigated further.

Upset Forging Experiments - Texture

An investigation of the effect of upset forge variables on deformation texture development was undertaken to determine those factors that contribute to the homogenization of the textural inhomogeneity inherent in the as-received and annealed bar. Texture was measured as a function of total true compressive, relative friction at the tool/workpiece interface (relubrication with 2 vs. 5 passes), radial position in the forged blank (center vs. edge), and axial position (surface vs. mid-plane). Slices were sectioned from the 1150°C annealed rolled bar such that the upset compression axis was parallel to the axial direction. In this orientation the texture banding would be expected to extend in rings from the center to the periphery on the faces of the blanks. The goal of upset processing is to break down the microstructural inhomogeneity in the starting rolled bar, and develop $r-\theta$ in-plane isotropy.

The measured response for each variable is presented in the form of the (200) experimental pole figure and (111) pole figure recalculated from the ODF. The ODF was reduced

from the (200), (211), and (220) experimental pole figures by means of the WIMV vector method, as incorporated in the popLA software package[16]. All pole figures are axial-direction normal and are plotted with consistent times-random density scales to facilitate comparisons. The {001} and {111} components dominate the compression deformation textures in most bcc metals, including Ta[6,19]. Although display of the ODF would present more information, in this case the aforementioned pole figures provide a straightforward method of correlating the influence of deformation processing on texture evolution. The "off-axis" variables were held constant for each comparison, and are noted in the individual figures.

The effect of total strain on reducing the r - θ in-plane anisotropy showed that strains of -0.95 were clearly insufficient to break down the asymmetry of the starting material, while near in-plane isotropy is attained at a strain of -1.85. This corroborates with the macrostructures of Figure 11, in which much of the banded structure remained undisturbed at smaller strains.

The effect of relative friction changes showed that increased friction resulted in an in-plane smear of both texture components, usually biased in the radial direction. This had the effect of reducing in-plane isotropy. Increased friction also results in a larger dead-zone of limited strain under the tooling and less uniform straining in both the radial and axial directions.

The effect of relative position on texture is dominated by the frictional conditions at the tool/workpiece interface. Under ideal lubrication conditions, all regions of a compressed disk would undergo equivalent strains. However, in practice a friction hill exists diametrically across the surface, with its maximum at the center. The effect of this gradient is to increase the hydrostatic stress and reduce the deviatoric stress component toward the center of the disk. Therefore, the effective strain of a given element is dependent on both its radial and axial position. In addition, surface shears may also occur, in which highly localized strains occur near the surface undergoing sticking friction conditions.

The texture as a function of radial position in the upset forging discs showed that symmetry is enhanced as the position is varied from center to edge. This is expected due to the effective strains being larger as one moves away from the center. The effect was not entirely systematic; at larger strains the magnitude of the effect is diminished. This may be attributed to the eventual reduction of the size of the dead-zone, and to the extreme positional sensitivity of texture displayed in the starting material. As expected, the surface regions showed more in-plane anisotropy, with smearing of the texture in the radial direction a generic feature. It should be noted that interactive effects between radial position, axial position, and relative friction may be present. For example, the relative effect of axial position may be dependent on the frictional conditions at the interface and/or the radial position.

A comparison of X-ray diffraction and ND interrogation techniques is shown in Figure 13 by way of (200) pole figures measured from the equivalent sample. The through-thickness sampling from ND reveals three distinct sets of {111}<uvw> orientations compared to only one at the sample surface measured by X-rays. The ND results provide bulk texture information averaged over an entire sample. These data are beneficial for anisotropy input to deformation models, where X-ray data provide information relevant only to the analyzed surface.

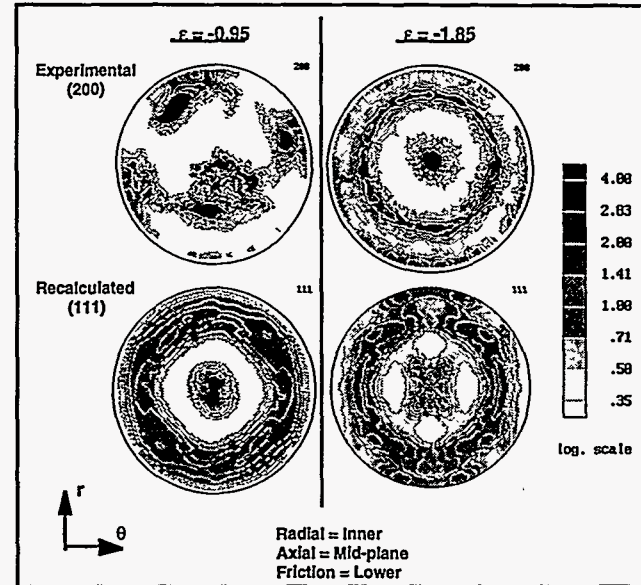


Figure 12: {001} and {111} pole figures from the annealed and upset-forged Ta-bar as a function of upset strain.

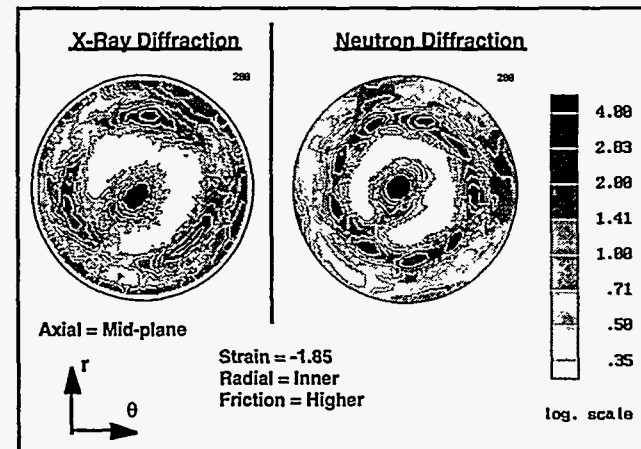


Figure 13: Comparison of textures measured using X-rays and Neutrons for the Ta-Bar upset forged to $\epsilon=1.85$.

Upset Forging Experiments - Mechanical Properties

Four batches of compression samples EDM cored from discs with prestrains of 0, 0.42, 0.95, and 1.85 were tested at room temperature at a strain rate of 10^{-3} s^{-1} . The reload stress-strain responses after off-setting for the corresponding amount of prestrain are shown in Figure 14. It is seen that the reproducibility in the stress-strain response is diminished after pre-straining. This scatter is

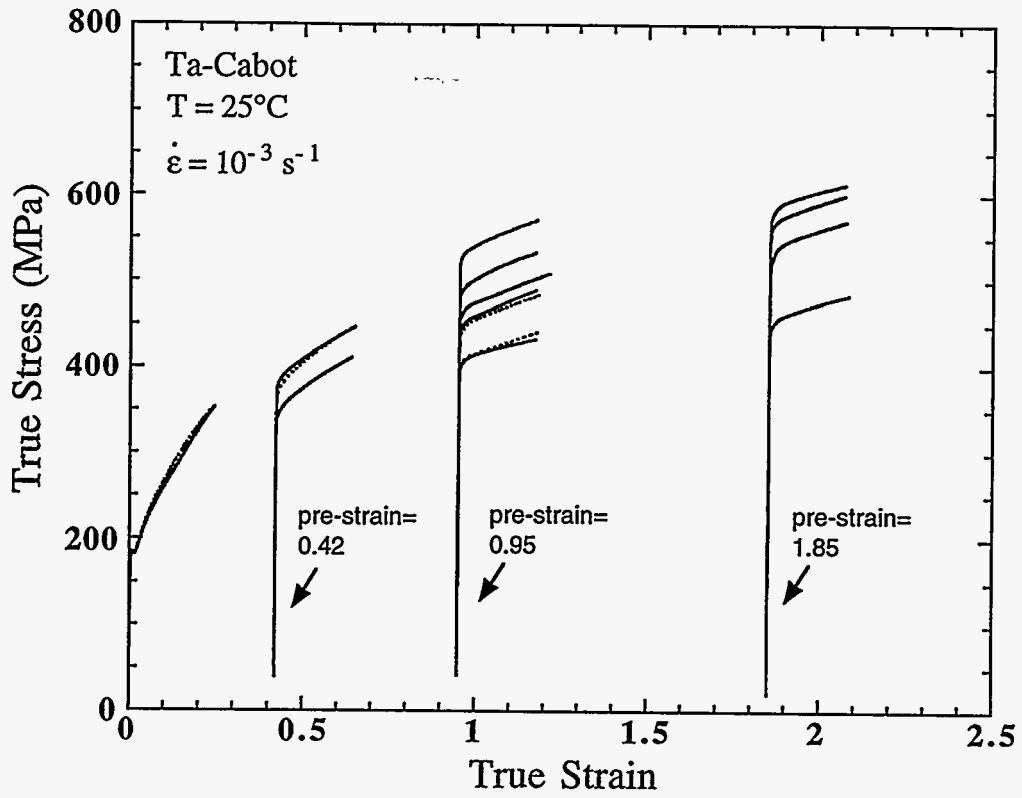


Figure 14: Stress-strain response of Ta-bar as a function of upset-forging strain.

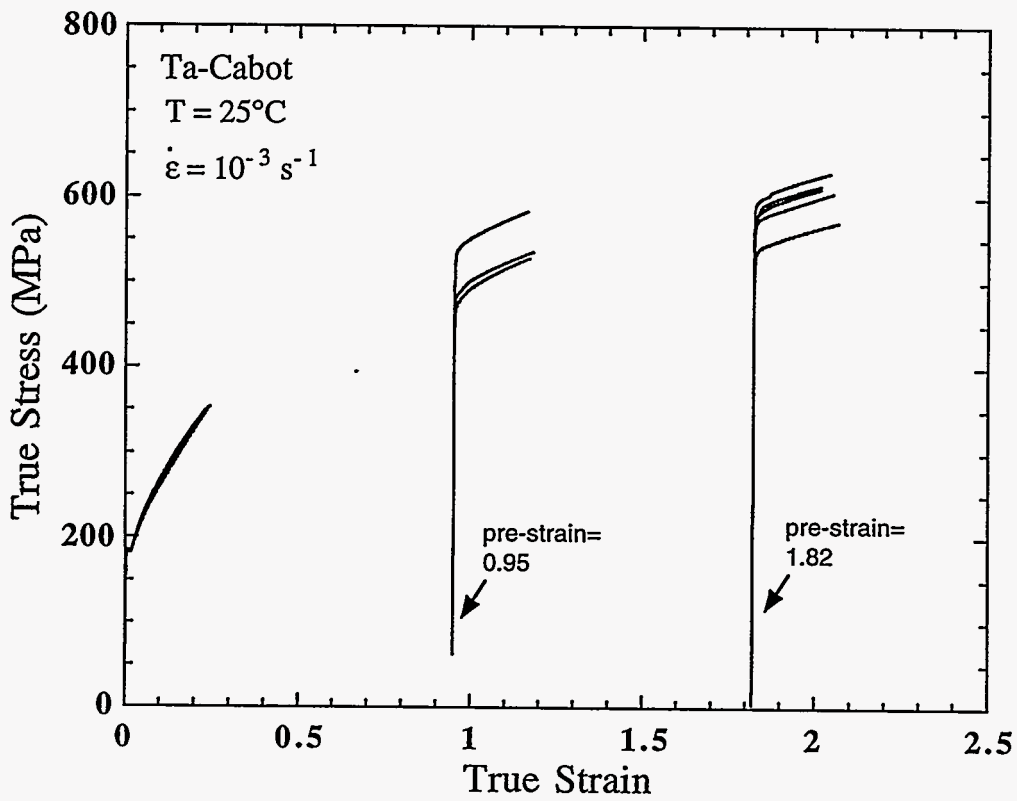


Figure 15: Stress-strain response of Ta-bar as a function of upset-forging strain using increased lubrication.

much larger than would be seen if it was due to differences in the instrumentation used in the experiment setup. The non-homogeneities in the Cabot Ta-bar documented in the previous sections have clearly identified the existence of banding in the microstructure and a texture gradient in this material. The degree of inhomogeneity is seen to be magnified following upset forging to the highest strain (1.85). The exact sample locations in the upset-forged plate were marked in order to correlate the measured properties to the upset forging process which is nominally an axi-symmetric operation to the workpiece. The strength of the Ta was found to in general increase inward from the circumference for the forged plate prestrained to 1.85.

The question was raised if the scatter observed in the reload mechanical response could be a reflection of friction contributions during the forging. Accordingly, a second batch of cylinders sectioned from the annealed Ta bar were upset forged using a constant forging rate and more frequent lubrication to systematically study the influence of friction and strain rate on the large-strain mechanical response. Reloading samples from these more-controlled forged discs revealed that the scatter in the mechanical properties remained as seen in Figure 15. The degree of scattering does however seem to have decreased based on the albeit fewer number of tests performed. This reduced level of scatter in the mechanical properties is consistent with the observation of a more consistent microstructure and texture observations discussed earlier. The lowest strength exhibited in the discs upset with the increased level of lubrication were found to be from regions near the disc circumference. The samples located within the central half of the diameter of the forged discs with increased lubrication showed much smaller, although still present, scattering in the reload strength. This finding is consistent with the better reproducibility of the microstructure in this area of the forging and the most controlled forging stress-state maintained in this area of the forged discs.

Summary

The current study of the influence of large-strain upset forging on the mechanical properties and texture evolution of tantalum bar indicates:

- a) Ta-bar material can exhibit pronounced microstructural and textural inhomogeneity in the form of asymmetric macroscopic banding. This texture gradient is thought to be due to insufficient or variable radial reductions per pass imparted during forge break-down and/or round rolling of the bar.
- b) Microstructural and textural banding can lead to radial strain gradients, associated inhomogeneity in deformation texture and resultant non-uniform recrystallization textures, and scatter in the mechanical property behavior of the Ta-bar material following cold work.
- c) Axial forging to strain levels of $\epsilon > 1.75$ can produce more in-plane isotropy in Ta-bar material possessing an initially banded inhomogeneous microstructure / texture.

Careful control of both the forging strain rate and lubrication are required to minimize the scatter in the mechanical properties of upset-forged Ta-bar.

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