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SciVerse ScienceDirect
Scripta Materialia 67 (2012) 253–256
Scripta MATERIALIA
www.elsevier.com/locate/scriptamat

Transition in the deformation mode of nanocrystalline tantalum processed by high-pressure torsion

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Received 22 March 2012; revised 24 April 2012; accepted 25 April 2012
Available online 2 May 2012

We present quasi-static room temperature compression and nanoindentation data for nanocrystalline and ultrafine grained tantalum processed by high-pressure torsion. Because bulk samples possess an inherent gradient in properties, microstructures were characterized using site-specific transmission electron microscopy and synchrotron X-ray diffraction. Nanocrystalline Ta shows appreciable homogeneous plastic deformation in compression; however, specimens with the smallest grain sizes exhibit localized plastic deformation via shear bands. Microstructural changes associated with this transition in deformation mode are discussed.

Keywords: Nanoindentation; Nanocrystalline metal; Shear bands; Microcompression; Severe plastic deformation

Grain size (d) refinement to the ultrafine grain (UFG) ($100\ \text{nm} < d < 1000\ \text{nm}$) and nanocrystalline (NC) ($d < 100\ \text{nm}$) levels has been used to strengthen pure metals and alloys [1]. While coarse grained body centered cubic (bcc) metals typically show homogeneous plastic deformation under room temperature quasi-static loading, UFG and NC bcc metals have shown a transition to localized plastic deformation [1,2]. This localized deformation mode will tend to limit the inherent engineering properties, such as the strength and ductility, of this class of materials. Therefore a better understanding of the deformation behavior is needed.

In this letter we present room temperature nanoindentation along with small size, quasi-static compression data for commercial purity tantalum processed by high-pressure torsion (HPT). Because HPT processing yields bulk specimens with an inherent gradient in strain and microstructure, site-specific mechanical and microstructural characterization techniques were used to isolate the response of NC regions found in the disk-like bulk samples. Prior work examined the dynamic mechanical response of similarly processed Ta [3], but the focus here relates the quasi-static response to microstructural characterizations by transmission electron microscopy (TEM) and synchrotron X-ray diffraction (SXRD). NC Ta is considerably stronger than its coarse grained counterparts and shows homogeneous plastic deformation in compression. However, the finest grain sizes present in this HPT disk transition to a localized plastic deformation mode.

Commercial purity disks (H.C. Stark) were processed at room temperature by HPT at a confining pressure of ~5 GPa. The Von Mises [4] equivalent strain (ε_{eq}) is related to the radial position or distance from the disk center (x), the number of turns (N) and thickness (h) as: $\varepsilon_{eq} = \frac{2\pi \cdot N \cdot x}{\sqrt{3} \cdot h}$ [5]. The sample presented here was 1.2 mm thick and ~12 mm in diameter and was deformed to maximum strains approaching 9000% ($N = 5$) [5]. Because the microstructure and properties vary in the HPT disk, throughout this letter microstructural and mechanical analysis will be related back to the position on the bulk sample defined by x.

TEM lamellae were prepared at various positions (x) in the transverse (or tangential) directions using an FEI Nova 600i dual beam FIB with final thinning at 5 kV. TEM was performed in a JEOL JEM-2100F operated at 200 keV. SXRD was done at the Advanced Photon Source at the Argonne National Laboratory in beamline 11-ID-C, with a 115 keV beam and 150 × 150 μm spot size. Peak broadening from SXRD was used to calculate the grain size or coherent domain size.

The Ta disks were lapped flat and parallel and polished to a sub-micrometer final finish. The indentation response was measured using an MTS Nanoindenter XP in continuous stiffness measurement mode at a

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1359-6462/$ - see front matter
http://dx.doi.org/10.1016/j.scriptamat.2012.04.031

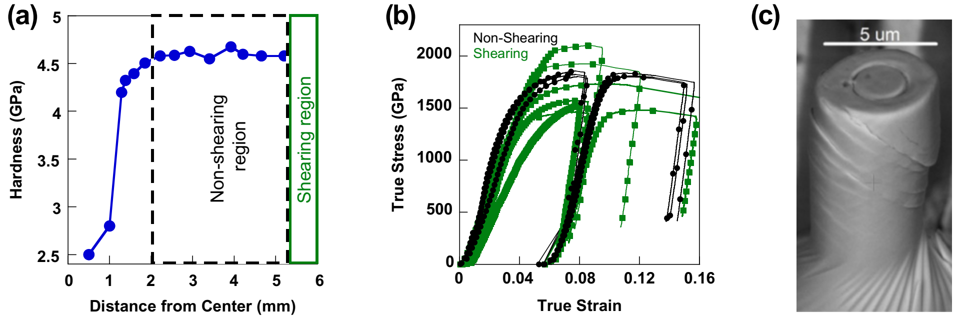

Figure 1. (a) Nanoindentation results taken at different positions x on the HPT Ta disk, showing that hardness (H) increases as x increases until saturation at $x > 2$ mm. (b) True stress vs. strain plots from microcompression performed on specimens in the non-shearing (black) and shearing (green) regions. The non-shearing region shows the slightly increased strength and reduced strain hardening expected for Ta with NC grain sizes compared with coarse grain sizes. Plots from the shearing region also show increased strength and reduced strain hardening, but lack any "pop-in" event to indicate the deformation mode transitioned to shear bands. (c) SEM micrograph of a pillar in the shearing region compressed twice, total strain ~16%, showing the formation of multiple shear bands. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

constant strain rate of 0.05 s^{-1}, to a maximum depth of 2 μm with a Berkovich indenter [6]. Oliver–Pharr hardness (H) [7] was measured to examine property variations throughout the HPT disk. Furthermore, indentations at strain rates ($\dot{\varepsilon}$) of 0.01–0.15 s^{-1} were carried out to derive the strain rate sensitivity (SRS, m) from $m = \partial \ln H / \partial \ln \dot{\varepsilon}$ [8]. Microcompression pillars with diameters ranging from ~5 to 10 μm were fabricated using the "lathe" technique [9] in the FIB operated at 30 keV, with final milling currents of 0.1–1 nA. Micropillars were compressed in the same nanoindenter with a conical 30 μm flat punch at strain rates of ~10^{-4} s^{-1}.

Figure 1a plots the hardness as a function of x for HPT Ta. Measured values of H are lowest near the center of the disk, and quickly increase as a function of x. The hardness saturates at roughly 4.6 GPa and is comparatively constant for $x \geqslant 2$ mm. The Hall–Petch relation has been applied to Ta to relate H (in MPa) to the grain size d (in micrometers) as: $H = 780 + 690d^{-1/2}$ [10]. Prior work showed agreement in estimations using this relation for grain sizes as small as ~50 nm [3]. This relation indicates that grains near the center of the disk are in the UFG regime and are roughly 30–35 nm for $x \geqslant 2$ mm. This NC region of the disk will be the focus of the remainder of this letter and of the investigations using microcompression presented below.

Microcompression experiments were performed throughout the NC region in the HPT Ta, extending from $x \approx 2$ mm to the edge of the bulk sample. Two distinctly different NC regions were identified in the HPT processed disk. In the "non-shearing" region microcompression specimens showed homogeneous plastic deformation. In the "shearing" region microcompresison specimens deformed via localized plastic deformation or shear band formation. Figure 1a schematically shows the boundary for the non-shearing and shearing regions that occurs at roughly $x \approx 5.3$ mm.

The typical true stress–true strain response for non-shearing NC Ta is plotted in Figure 1b (black). The average 0.2% offset yield strength was 1280 ± 95 MPa, which is more than three times that of annealed commercial purity Ta (~400 MPa) and nearly twice that of UFG Ta processed by equal channel angular pressing (ECAP) (~700 MPa) [11]. Micropillars in this non-shearing region all had comparable strengths, in keeping with the hardness results. These plots show initial strain hardening that saturates at a plastic strain of ~5%. Subsequent reloading resulted in slight strain softening at larger strains. Scanning electron microscopy (SEM) analysis of deformed specimens (not presented here) showed homogeneous plastic deformation without failure to compressive strains exceeding ~15%. Prior work showed that HPT Ta [3] and other materials of elastic-perfectly plastic nature are susceptible to plastic buckling [12]. However, no specimens in this work showed evidence of buckling, suggesting very good alignment of the specimen with respect to the loading axis.

As mentioned above, near the edge of the HPT disk ($x > 5.3$ mm) we observed a stark difference in the deformation mode in compression. Specimens in this region consistently showed localized plastic deformation or shear bands. Figure 1b plots the compressive response for shear localizing NC Ta specimens (green). Yield strengths in shearing specimens were more highly variable, with average values of 1385 ± 143 MPa. Figure 1c shows an SEM micrograph of a specimen showing localized plastic deformation. The specimen initially showed a single shear band following a limited plastic strain. When this specimen was reloaded multiple shear bands were evident. SEM measurements indicated that the shear band angle was between 44° and 46° for all pillars tested in this region. Discrete "pop-ins" have been reported for microcompression and nanoindentation tests of metallic glasses [13]. However, no pop-ins were found in the shearing region. This indicates that these shear bands form over longer timescales than those associated with shear bands found in metallic glasses.

TEM and selected area electron diffraction (SAED) were employed to characterize microstructural differences in the non-shearing and shearing regions. Figure 2 shows a TEM micrograph and SAED pattern (inset) for a TEM lamella taken in the transverse orientation at $x \approx 5.9$ mm (shearing region). Qualitatively speaking there are similarities in the microstructures in the non-shearing and shearing regions. HPT processing resulted in elongated grains bearing a similarity to nanocrystalline