Size dependent strength of bcc metal micropillars: towards high strength surfaces by micropatterning

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Abstract

The size effect in body-centered cubic (bcc) metals was comprehensively investigated through microcompression tests performed on focused ion beam machined tungsten (W), molybdenum (Mo) and niobium (Nb) pillars, with single slip [235] and multiple slip [001] orientations. The relationship between yield strength and pillar diameter as well as the deformation morphologies were found to correlate with a parameter specific for bcc metals, i. e. the critical temperature T_c. This finding sheds new light on the phenomenon of small-scale plasticity in largely unexplored non-fcc metals. This effect may be used in the patterning of surfaces to achieve higher strengths.

Introduction

The mechanical behavior of metals exhibits a size dependence, in which the flow stress is usually inversely proportional to some power of the smallest geometrical dimension [1]. This effect has been convincingly demonstrated for a variety of metals in the micron and nanometer scale. Several mechanical testing techniques measured at e. g. whiskers, nanowires, thin films, etc. have confirmed this effect. Recently several studies have also demonstrated a size effect in single crystal metals via compression testing of focused ion beam (FIB) manufactured micro- and nano-pillars [2-3]. Unlike whiskers, FIB machined small-scale compression pillars are not initially dislocation free. Consequently, near theoretical strengths are not observed in FIB manufactured pillars, even though the strength is significantly higher than in the bulk. These yield stress values, σ_y, have been shown to correlate with pillar diameter, d, according to σ_y μ $d^{-0.6}$ to $d^{-1.0}$ for all face-center cubic (fcc) metals tested to date [2-3].

While many efforts have been made to understand the mechanisms responsible for this effect in fcc metals, both through experimentation and theoretical and simulation-based models, little work has been done on alternative crystal structures, such as body-centered cubic (bcc). The plastic deformation in bcc metals differs fundamentally from that of fcc metals [4]. In bcc metals, the deformation is largely controlled by screw dislocations which, due to the geometry of the glide planes, have non-planar dislocation cores and high Peierls potentials. This intrinsic resistance to their motion can be overcome through thermally activated processes, leading to a strong temperature and strain rate dependence of the flow stress. Although these fundamental differences between the deformation behavior of fcc and bcc metals have been characterized in bulk, it is not well known how the size effect in bcc metals may deviate from that observed in previous fcc studies.

The aim of this work is to study the mechanical properties of bcc metals at the micron and submicron scale and to investigate the effect of the screw dislocation mobility in small dimensions. For this purpose, microcompression tests were conducted at room temperature (RT) on bcc metals with different critical temperatures (Table 1). The critical temperature (T_c) is defined as the temperature at which the flow stress becomes insensitive to the test temperature, i. e. screw and edge dislocations have equal mobility due to thermal activation of the screw dislocations [5]. Below T_c, screw dislocations are less mobile than edge dislocations and their mobility is a function of test temperature T_{test} relative to T_c.

Material	Nb	Ta	Mo	W
Critical temperature T_c [K]	350	450	480	800
Temperature Ratio T_{test}/T_c	0.851	0.662	0.621	0.373

Table 1: Critical temperatures of the bcc metals used in this study; T_{test} = 25 °C in our experiments.

Experimental

Tungsten (W), molybdenum (Mo) and niobium (Nb) samples with multiple slip [001] and single slip [235] orientation were prepared from single crystals by means of electron discharge machining after the orientations of the crystals were determined by Laue diffraction. The sample surfaces perpendicular to either the [235] or [001] direction were mechanically and electrochemically polished. Pillars ranging in diameter from approximately 200 nm to 6 µm were machined with a focused ion beam (FIB) on the surface of both the [001] and [235] single crystal pieces using a DualBeam™ FIB. In general, pillars were cut with a diameter to length aspect ratio of approximately 3:1, and then compressed in load-control with a nanoindenter fitted with a 10 µm flat sapphire punch. Further description of the testing and the data analysis can be found in [6].

Results

Representative compressive stress-strain curves for [001] oriented W, Mo, and Nb pillars for various diameters are shown in Figure 1. The overall shapes of the stress-strain curves are typical for load-controlled single crystal pillar compression and display the stochastic nature of slip in small dimensions. Strength increases markedly with decreasing diameter. Pillars larger than roughly 2 µm exhibit bulk-like flow with a gradual transition from elastic to plastic deformation and relatively little strain hardening. Pillars with smaller diameters exhibit a staircase-like deformation due to strain bursts associated with individual dislocation events. Although the nominal stress values for the materials tested are different, the qualitative features of the stress-strain response are consistent with one another and are similar to observations in fcc single crystalline pillars [2-3].

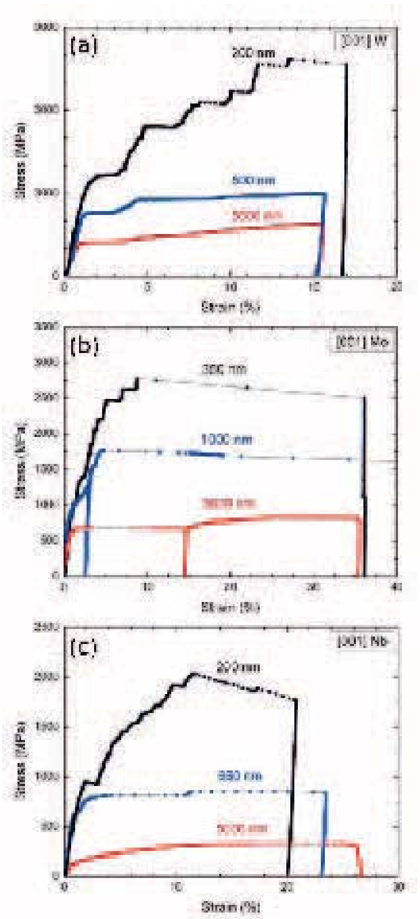

Figure 1: Representative stress-strain curves for [001] (a) W, (b) Mo and (c) Nb pillars over a range of diameters from 200 nm to 6 µm.

Figure 2 shows representative scanning electron microscope (SEM) images of deformed W and Nb pillars with diameters close to 200 nm (2a and b) and 5 µm (2c and d). The deformation morphology of Mo is similar to that of W. Regardless of size, the [001] oriented pillars exhibited slip along multiple systems, as evidenced by the geometry of slip traces in Figure

2, while the [235] oriented pillars exhibited primarily single slip along a preferred slip system [7]. As seen in Figures 2c and d, the larger pillars had a tendency to display slip traces on the pillar surface throughout their length, while pillars with diameters less than approximately 500 nm were more likely to exhibit observable slip deformation closer to the pillar top. For pillars with larger diameters, two different deformation morphologies were identified. In large W and Mo pillars, there are no continuous slip steps on the pillar surfaces. The slip steps appear wavy throughout the sample, as is typically found for bulk bcc metals. Wavy slip planes are consistently more difficult to observe in the Nb pillars, which primarily exhibit localized slip on preferred glide planes with clear slip traces ranging across the pillar surface.

The mechanical size effect of the [001] oriented bcc pillars is shown in Figure 3a, which illustrates the stress at 5 % strain as a function of diameter. For comparison, data from fcc Au [8] and bcc Ta [9] pillars is also shown. The strengths of the bcc metals are higher than those of Au and the relative differences in strength between the different materials decrease with decreasing pillar size. Using a power law fit, slopes of - 0.21, - 0.38, - 0.41 and - 0.48 are obtained for W, Mo, Ta and Nb pillars, respectively. By comparing these slopes with the critical temperatures given in Table 1, it can be seen that the higher the critical temperature, the weaker the size dependence. In Figure 3b the power law exponent determined for the [001] and [235] oriented bcc pillars is plotted as a function of test temperature divided by T_c, to examine the role of screw dislocation mobility on the size dependence. The ratio of T_{test} to T_c is a measure for the thermal activation of the screw dislocations: a larger ratio means higher thermal activation and therefore higher mobility of the screw dislocations. The data points in Figure 3b follow roughly a linear relationship. The extrapolation of the line of best fit to lower critical temperatures yields an exponent of about - 0.6 (represented by the horizontal line in Figure 3b) for $T_c = T_{test}$, which corresponds to the condition where screw and edge dislocations have equal mobility. This value is in agreement with exponents found for fcc metal pillars where screw and edge dislocations have the same mobility at room temperature.

Discussion

The results presented here show that deformation mechanisms in small-scale bcc metals depend on size as well as on critical temperature. All materials tested show an increase in strength with decreasing pillar diameter, indicating that confinement of dislocation processes starts to dominate the deformation; this leads to a transition from continuous, bulk-like to jerky, staircase-like stress-strain behavior and a significant change in the deformation morphology between small and large pillars. The critical temperature of the bcc metals was found to have a strong influence on the deformation morphology of the large pillars. In general, the deformation of bcc metals is controlled by the

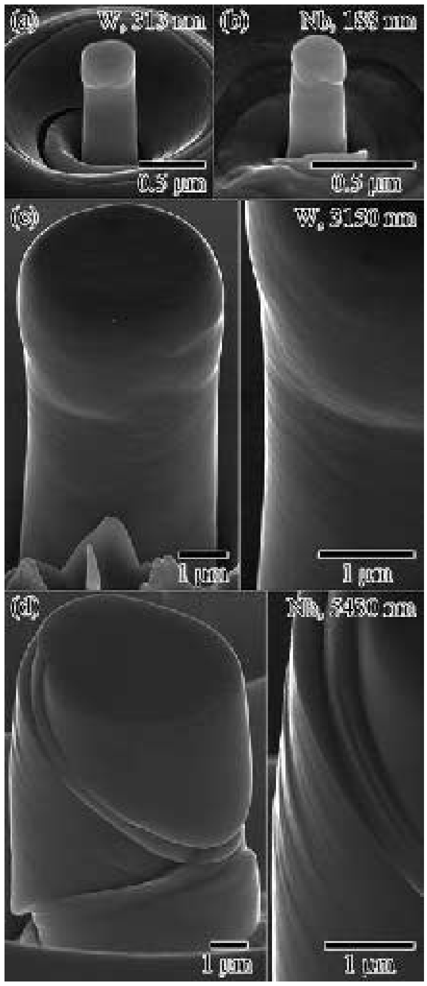

Figure 2: Post-compression SEM images of representative [001] oriented Nb and W pillars: (a) 313 nm W, (b) 188 nm Nb, (c) 4870 nm W, and (d) 5430 nm Nb pillars. Slip traces on the pillar surfaces indicate multiple slip.

motion of long and straight screw dislocations [4; 5]. Their ability to cross-slip between crystallographic planes which intersect along the <111> direction leads to wavy slip steps. The cross-slip of screw dislocations may account for the deformation morphology of the large W and Mo pillars. The well-resolved slip steps of Nb indicate that less cross slip occurred during deformation. For metals with low T_c (Nb) the dislocations may bow out and deviate from pure screw character over considerable length in the pillar. As dislocations of mixed character are confined to specific glide planes, this may lead to localized slip.

Besides the deformation morphologies, the strength values and their size dependence were also found to correlate with T_c (Figure 3). A general correlation of the strength values with T_c is not surprising as the strength of bcc bulk metals at temperatures below T_c is related to the low mobility of screw dislocations. As T_c increases from Nb to W, the mobility of screw dislocations decreases because thermal energy becomes small relative to the height of the Peierls potential, resulting in high stresses for materials with a high T_c. The correlation between size dependence and T_c suggests that the mobility of screw dislocations affects the size scaling. It seems that for temperatures close to T_c, where the influence of the low mobility of screw dislocations becomes negligible, the behavior of bcc also approaches that of fcc metals. The similar behavior of fcc and bcc pillars for test temperatures near T_c indicates that under conditions where

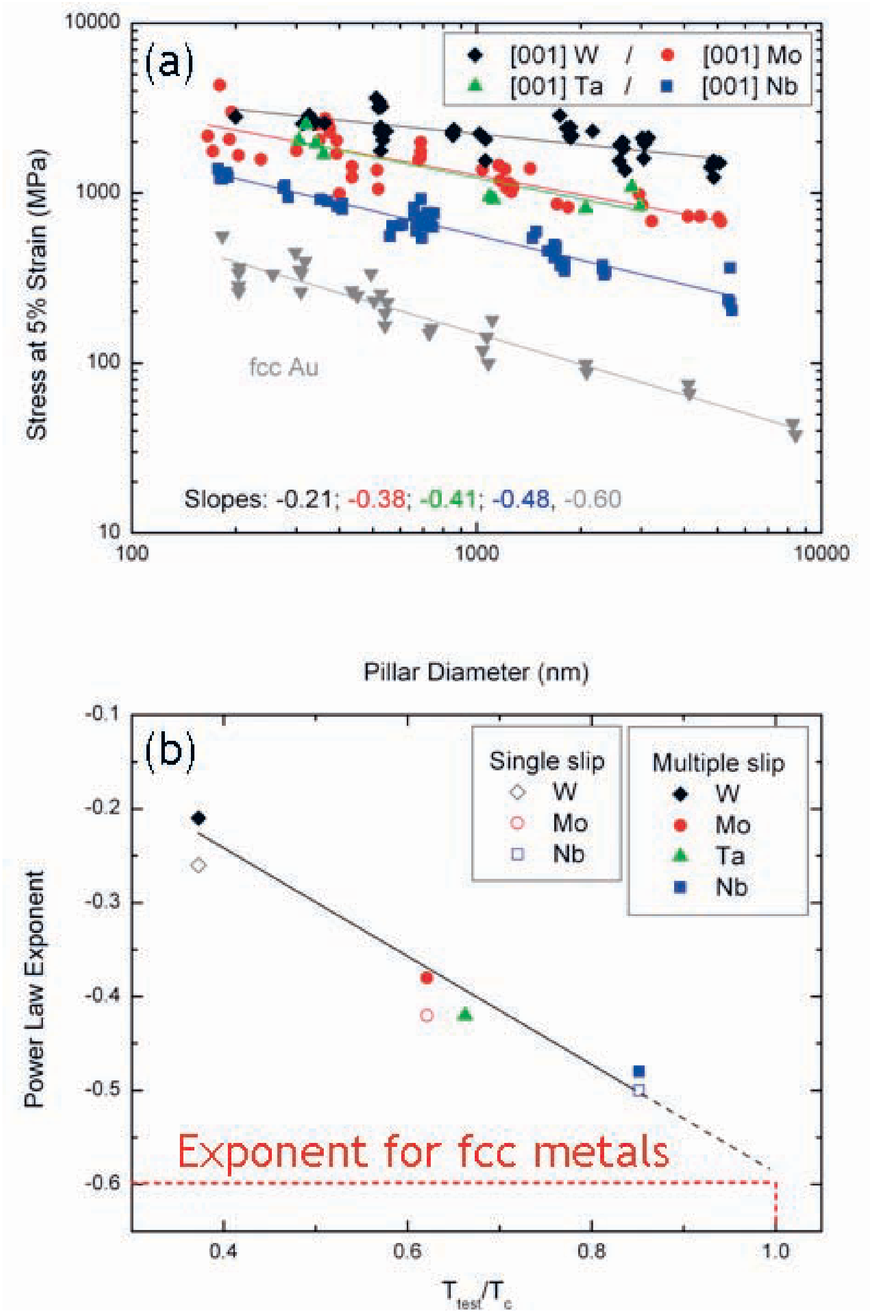

Figure 3: Comparison plots: (a) of stress measured at 5 % strain versus pillar top diameter for all [001] bcc pillars tested (i. e. W, Mo and Nb) as well as Au [8] and Ta [9] pillar data, (b) slope of the line of best fit (exponent) for the size dependence of the [001] and [235] oriented bcc pillars versus normalized test temperature T_{test}/T_c (see text and Table 1).

screw dislocation motion is not the limiting mechanism, the strength of bcc and fcc pillars may be controlled by the same dislocation processes. With increasing critical temperature an increasing effect of screw dislocation mobility becomes apparent, leading to a deviation from fcc behavior that scales with T_{test}/T_c.

Summary and outlook

We have demonstrated that the size dependence of the yield stress differs fundamentally between fcc and bcc metals. In contrast to fcc metals, bcc metals were found to have a weak size dependence, which scales with the critical temperature of the tested material. This difference was attributed to the special role of screw dislocations inherent to the bcc crystal structure. To obtain a better understanding of the associated mechanisms responsible for the size effect, we are currently investigating the influence of internal size parameters on the mechanical properties of small-scale single crystals. For this purpose, microcompression tests have been performed on oxide-dispersion-strengthened (ODS) alloys with an oxide particle spacing smaller than the sample size. Future experiments will be conducted on age hardenable alloys giving the opportunity to change the internal size parameter with respect to the sample size by an appropriate heat treatment. This will enable us to explore the transition from obstacle- to size-controlled deformation in more detail and will give new insights into the microstructure evolution in small dimensions.

Another goal of our group is to transfer these fundamental studies into potential applications. Since small-scale metal structures have superior mechanical properties compared to their bulk counterparts, large-scale micro- or nano-structuring of metal surfaces may offer radical improvements of tribological properties and new functions. Current approaches to make patterned surfaces are expensive because they require multiple steps, in clean and highly controlled environments, on expensive equipment. Therefore, we also investigate new ways for macroscopical micropatterning of metal surfaces.

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