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Size effects on strength and plasticity of vanadium nanopillars

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A size effect study was conducted on [0 0 1] oriented vanadium nanopillars that were synthesized from both a thin film and a bulk crystal. The results indicate that a size-dependent deformation behavior exists for vanadium; the smaller nanopillars displayed discrete strain bursts and higher stresses during deformation. The size effect exponent is found to be 0.79, and the results are compared with previous reports on other body-centered cubic (bcc) metals: Nb, Ta, Mo and W.

Keywords: Bcc metal; Size-dependent plasticity; Microcompression; Nanopillar

Mechanical properties of small scale structures are of technological importance in various applications, including microelectronic devices, and MEMS/NEMS devices. Since the microcompression methodology was introduced by Uchic et al. [1], there has been an explosion of research on strength and plasticity of metallic nanopillars made out of face-centered cubic (fcc) single crystals [1–3]. Through extensive study of fcc nanopillar compression, there is wide agreement on the basic results: the pillars display discrete flow behavior with frequent strain bursts and an increase in the yield strength (σ^{YS}) with decrease in the sample diameter (d) according to $\sigma^{YS} \propto d^{-n}$. The size effect exponent n ranges between 0.6 and 1.0 for fcc metals. However, interpretation of the observed behavior is still under debate.

There are two dominant competing theories that aim to explain the observed size-dependent properties in fcc nanopillars. One is the single-armed source theory, as proposed by Parthasarathy et al. [4], and the other is the dislocation starvation/nucleation theory, as proposed by Greer et al. [2]. In both single-armed source and starvation theories, it is important to understand the dislocation multiplication process responsible for the observed plasticity. The multiplication process is achieved by continued operation of a truncated source or by surface nucleation in the single-armed source and starvation theories, respectively. In this regard, studying body-centered cubic (bcc) metals and comparing the results to those for fcc metals might provide insight into the operative multiplication processes. BCC metals have higher Peierls stresses and exhibit easier cross-slip of the slower screw dislocations compared to fcc metals. These factors may result in more dislocation interactions that, in turn, may cause more dislocations to be retained in the nanopillar during deformation. In a recent study, Weinberger and Cai showed by dislocation dynamics (DD) simulation that the slower screw components of dislocations are retained during deformation and can cross-slip to form a loop that eventually divides into two separate dislocations [5]. Their work indicated that the multiplication of dislocations in bcc pillars may occur through this mechanism. Weinberger and Cai reported that this mechanism occurs above a critical applied compressive stress, and that this critical stress is pillar size dependent; the larger the pillar dimension, the smaller the critical stress for dislocation multiplication.

More recently, a significant body of results for bcc nanopillars has been emerging. Bei et al. have shown that their alloyed Mo pillars, uniquely synthesized from directional solidification, deform at close to the theoretical strength and do not display any size-dependent strength [6]. On the other hand, Nb, Ta, Mo, and W nanopillars synthesized using focused ion beam milling show a size effect that differs for the different metals [7–13]. Molybdenum nanopillars showed size-dependent strength with n varying between 0.3 and 0.5 [7–9,12]. However, the size effect exponent for Nb was observed to be higher ($n = 0.48$–1.06) than that for Mo nanopillars [10,11,13]. Kim et al. and Schneider et al. proposed that the difference is due to the different Peierls stresses for these metals at room

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temperature [10,13]. It is well known that low temperature deformation in bcc metals is strongly dependent on temperature, and there is a critical temperature above which the screw dislocations can glide easily due to thermal activation over the Peierls barriers [14,15]. This critical temperature for Nb is 350 K, whereas for Mo the critical temperature is much higher, 480 K [15,16]. Schneider et al. and Kim et al. explored two additional bcc metals (Ta, W in addition to Nb, Mo), and they both reported that the size-dependent strength depends on the critical temperatures for each of these materials [11,13]. Schneider et al. reported that the exponent scales well with T_c, while Kim et al. reported that the size exponents are approaching those of the fcc metals when tested at or above T_c but do not scale with T_c.

In the present study, we report on the size-dependent strength of vanadium single crystal nanopillars. This study of vanadium provides useful insights into the deformation mechanisms of bcc pillars in two respects. First, vanadium offers a unique opportunity to explore in situ TEM compression testing to reveal the dislocation mechanisms that are responsible for the observed size effects in bcc crystals. Vanadium is a good choice for this purpose because it is relatively electron transparent due to its low atomic number, compared to other bcc metals such as Mo, Nb, and Ta. Although in situ TEM testing of nanopillars is a powerful technique, a major disadvantage of the technique is that a wide range of pillar sizes cannot be studied because the sample dimensions must be limited to achieve electron transparency. Therefore, we have conducted ex situ microcompression tests on *V* nanopillars to obtain a more complete picture of size effects for *V*. Another objective of this work is to compare the size effect study of the V, which has $Tc = 380$ K [17], to those for other bcc metals with different T_c to potentially shed light on to the T_c dependent size-dependent plasticity.

Two vanadium samples were used for the microcompression tests. The first sample was a bulk single crystal of vanadium with a (1 0 0) surface that had been electro-polished and well-annealed to remove the surface defects arising from the polishing process. The second sample was a vanadium thin film that had been grown epitaxially onto a (1 0 0) MgO substrate using e-beam evaporation. The (1 0 0) MgO substrate was initially subjected to a light ion etch (50 V for 1 min with Ar ions) to remove any surface contaminants. Vanadium was then evaporated onto the substrate at 650 °C at a deposition rate of 1 Å s^{-1}. The growth process was monitored using reflection high energy electron diffraction (RHEED) to ensure epitaxy, and X-ray diffraction was used to confirm the epitaxy after the film had been deposited. The total thickness of the vanadium film was 1.2 μm. Nanopillars were then machined using the focused ion beam (FIB) from both of these samples to produce a wide range of sample diameters, all of which were kept close to a length to diameter ratio of 3:1. Thin film pillars were limited in length by the film thickness, but they have the advantage that the length of the pillar can be determined precisely. The bulk sample, on the other hand, was useful for producing a wide range of sample sizes. The FIB synthesized pillars were then deformed using two different nanoindentation systems, the Agilent NanoXP and the Hysitron Ti-750, both fitted with a diamond flat punch tip. Compression tests were performed at a constant nominal displacement rate of 2 nm s^{-1}. Since bcc materials are known to be strain-rate-dependent, a few tests at nominally constant strain rates were also performed.

A series of scanning electron microscopy (SEM) images of the *V* pillars made from both the bulk single crystal and the thin film, before and after deformation, are shown in Figure 1. These images clearly indicate that there are multiple slip events that lead to inhomogeneous deformation behavior. This behavior is more apparent for the sub-micrometer scale pillars compared to the larger pillars. The SEM image in Figure 1b shows a thin film nanopillar after compression. From this image, the angle between the top surface of the pillar and the slip plane was calculated to be 40.4°. This is in approximate agreement with {1 1 0} slip of a (0 0 1) orientated bcc pillar.

The conventional constant volume, homogeneous deformation model was used to calculate the true stress–true strain curves. It should be noted that the computed true stresses and true strains are only approximate since the deformation geometry is not uniform. However, we minimize the error by working with stress vs. strain up to 5% strain where inhomogeneous deformation is minimized. First, assuming that the pillar deforms homogeneously during compression such that the pillar becomes shorter and wider uniformly, the instantaneous cross-sectional area at the top during compression was estimated using

$$A_0 L_0 = A_p L_p \rightarrow A_p = A_0 L_0 / L_p \quad (1)$$

where A_0, L_0 are initial top cross-sectional area and length of the sample, and A_p, L_p are the top area and length of the sample during deformation. To determine L_p, the plastic displacement was first defined by subtracting the compliances associated with Sneddon sink-in and compression of the substrate pedestal:

$$u_p = u_{\mathrm{tot}} - u_{\mathrm{Sneddon}} - u_{\mathrm{Subs}} \quad (2)$$

where

$$u_{\mathrm{Sneddon}} = \frac{P}{k_{\mathrm{Smed}}} = \frac{P}{2E_{\mathrm{Subs}}/\left(1 - v^2_{\mathrm{Subs}}\right) * \sqrt{A_{\mathrm{bottom}}/\pi}},$$
$$u_{\mathrm{Subs}} = \frac{P}{k_{\mathrm{Subs}}} = \frac{P}{E_{\mathrm{Subs}} A_{\mathrm{bottom}} / L_{\mathrm{Subs}}} \quad (3)$$

The Sneddon compliance arises due to the elastic punching effect of the pillar (pedestal), and the substrate compliance refers to the MgO pedestal's finite compliance for the pillars made from the thin film. For the case of the

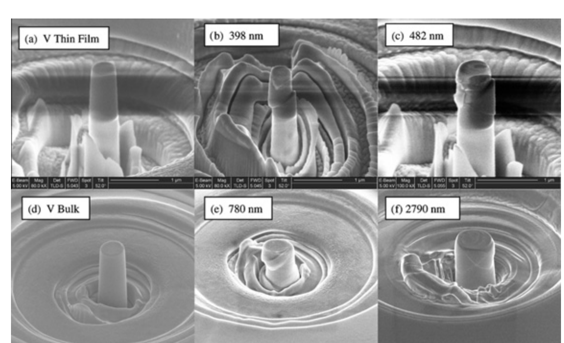

Figure 1. Before and after deformation SEM images of *V* pillars. (a)–(c) are from *V* thin film and (d)–(e) are from bulk *V*.