

Grain Size Effect on the Micro-Hardness of BCC Metal Vapor Deposits

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Abstract

The physical vapor deposition methods of evaporation and sputtering are used to prepare foils of the body-centered-cubic metals, vanadium and tantalum. A two-fold increase in the micro-hardness is measured as the grain size decreases to the sub-micron scale. The micro-hardness of vanadium increases to 2.7 GPa and for tantalum to 2.9 GPa.

Introduction

The preparation of body-centered-cubic (bcc) metals in the form of thin foils is of growing interest for laser-material interaction studies at high ($>10^5 \text{ s}^{-1}$) strain rates.[1] Physical vapor deposition methods can be used to prepare metal foils. The structure of the vapor deposit is dependent on the energetics of the synthesis process as described in various zone-growth models.[2-4] Using these growth models as a guide, foils of vanadium (V) and tantalum (Ta) are prepared using magnetron sputtering and electron-beam evaporation. The objective is to prepare fully dense foils with a range of grain sizes, from the submicron to the millimeter. In addition to measuring the micro-hardness of the V and Ta foils, the microstructure is characterized with optical and electron microscopy as well as with Bragg and Laue x-ray diffraction.

Experimental Methods

Synthesis

Foils that are 1-100 μm thick are deposited onto substrates including silicon, sapphire, mica, and various metals. The evaporation method utilizes a thermionic, self-accelerated electron gun. A 10 kV, 50-300 mA electron-beam melts the solid metal to form a molten pool in a 7, 15 or 40 cm^3 water-cooled hearth. To verify full density of the deposit, as measured by weight and volume, the evaporation process requires elevated substrate temperatures relative to the melt point (T_m). Fine grained columnar

deposits are produced at $(0.3-0.5) \cdot T_m$, whereas large grain deposits are produced at $>0.5 \cdot T_m$. [3] The evaporated coatings are deposited over a temperature range of 500-1150 °C. Through evaporation, single crystals are produced with lateral dimensions in excess of several millimeters square. Planar-magnetron sputter deposition utilizes an ionized working gas, such as argon, accelerated to a high potential cathode to sputter the metal target. The magnetron sputtering process relies on control of both gas pressure and substrate heating to produce fully dense foils. [4] A cathode voltage of 200-350 V is used, along with resistive heating to yield a temperature of 250-450 °C for the sputtered foils.

Characterization

The coatings are prepared using standard metallographic preparation and etching procedures for imaging in cross section. Optical micrographs are taken at 200-1000 magnification to reveal the growth morphology of the coatings. The growth texture of the coating is determined through Bragg diffraction in the $\Theta/2\Theta$ mode using Cu $K\alpha$ x-ray radiation. Coherent scattering from large grains produces identifiable Laue patterns in back reflection utilizing white x-ray radiation from a tungsten filament. Selected-area diffraction patterns of samples thinned for examination in plan view using transmission electron microscopy reveal the polycrystalline in-plane nature of the fine grain samples. The Vickers micro-hardness (H_v) is measured using a pyramidal indent and a 5, 10, and/or 25 gf load on samples as prepared in cross section. A typical indent in the V and Ta cross-sections is 5-10 μm in size.

Results

Fine grain deposits of Ta and V sputter deposits are shown in Fig. 1. The microstructure of the evaporative and sputter deposits is characterized by tapered crystallites. The crystallites are elongated along the growth direction to form the familiar, columnar-type grain structure. [3-4] The crystallite size is determined using the linear intercept method. The average crystallite width is measured from the

cross-section view of the polycrystalline structure. The crystallographic orientation of the V and Ta deposits is (110) as determined using Bragg diffraction. A typical result (seen in Fig. 2) for a sputtered Ta foil reveals only the (110) growth orientation. A Laue pattern generated in back reflection from a millimeter crystallite-size region of a high-temperature evaporative deposit of V is seen in Fig. 3. The (110) orientation is seen from the Laue image as well as from Bragg diffraction scans.

The hardness results are plotted (in Fig. 4.) as a function of inverse square-root crystallite size ($d^{-0.5}$) to determine the grain boundary effect using the Hall-Petch relationship

$$\Delta H_g = H_g - H_o = k \cdot d^{-0.5} \quad (1)$$

where H_g is the hardness caused by grain size effects, d is the crystallite width, and k is a material constant. Values of k^V equal to 4.3 GPa- $\mu\text{m}^{0.5}$ and H_o^V equal to 1.1 GPa are determined for V using eqn. (1) over the (Fig. 4.) data range of $0.75 < d^{-0.5} < 1.15$. The micro-hardness values reach a lower plateau at $d^{-0.5} < 0.7 \mu\text{m}^{-0.5}$ since the pyramidal indenter deforms just a few grains at most. A two-fold increase in H_g from H_o is found for V and Ta as d decreases to $\sim 0.5 \mu\text{m}$.

Discussion and Summary

The micro-hardness values measured for the vapor deposited V and Ta foils with the larger crystallite sizes are in agreement with established H_o values reported for bulk materials. The H_o^{Ta} of electron-beam melted Ta is 1.08 GPa whereas the equivalent H_o^V of an electron-beam V ingot is 1.27 GPa (72 HRB) and that of vacuum-annealed V wire is 1.38 GPa (48 HRA).[5-6] A Hall-Petch behavior is observed as the hardness of the V and Ta vapor deposits increases two-fold when the crystallite size decreases to $\sim 0.5 \mu\text{m}$. The observed variation in micro-hardness indicates the need to evaluate the potential for grain size effects on mechanical strength. Tensile tests of vapor deposited V and Ta foils will be performed to quantify the grain size effect on strength and ductility.

Acknowledgment

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Figures

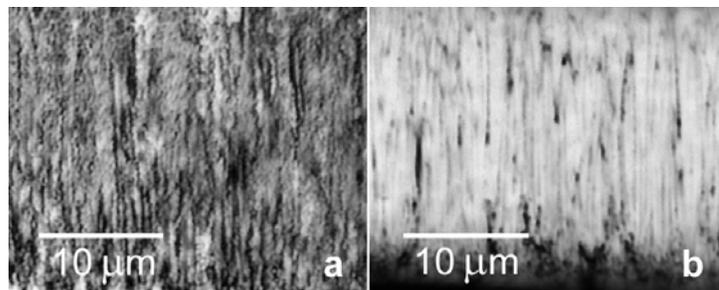


Figure 1. Optical micrographs of sputter deposited (a) Ta and (b) V foils as viewed in cross-section.

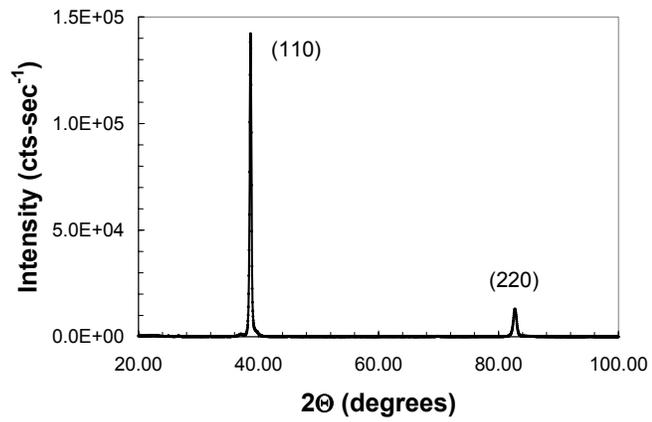


Figure 2. Cu $K\alpha$ diffraction scan in the $\Theta/2\Theta$ mode of a sputter deposited Ta foil.

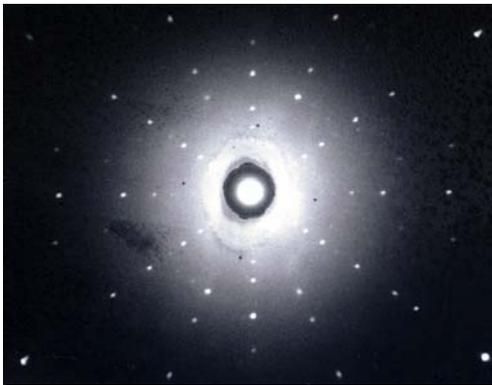


Figure 3. A Laue back reflection pattern taken from the surface of an evaporative deposited V foil.

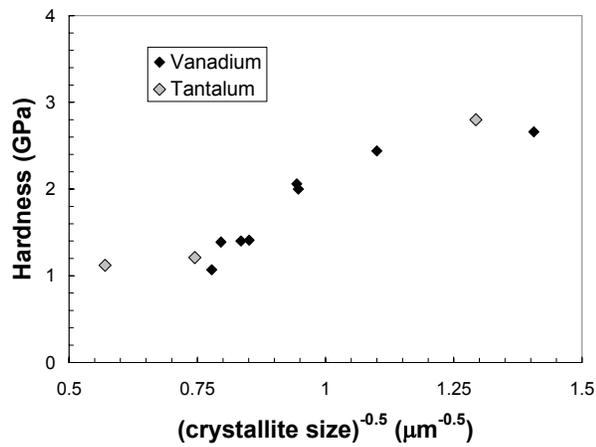


Figure 4. The variation of Vickers micro-hardness (GPa) with inverse square-root crystallite size ($\mu\text{m}^{-0.5}$) for vapor deposited Ta and V foils.

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