

## **Enhanced Mechanical Properties of Microcrystalline, Nanocrystalline, and Nanolaminated Ta-V**

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### **ABSTRACT**

The scaling of microstructure to the nanoscale enhances the physical properties of many materials that can be used in structural applications. A review of recent findings reveals that a ten-fold enhancement in the hardness and nanocrystalline and nanolaminated Ta-V structures is attributable to grain size effects. A Hall-Petch relationship of both hardness and strength with grain size appears in these body-centered-cubic structures from the macro- to nano- scale.

**KEY WORDS:** vapor deposition; strength; hardness; Hall-Petch; nanolaminates; nanocrystalline; and Ta-V

### **INTRODUCTION**

Tantalum (Ta) and Vanadium (V) metal in the form of foils, nanolaminates, and bulk material with nanoscale grain size, are reported (Wei, et al., 2003;2004, Zhang, et al., 2006) to show an increase in strength and microhardness by an order-of-magnitude above fully homogenized materials. The regime of grain size from the micro- to nano-scale is now assessed to examine effects on strength, hardness, and scaling of the Hall-Petch relationship in the body-centered-cubic (bcc) phase of Ta and V.

Ultra-refined microstructures of the body-centered-cubic (bcc) metals, tantalum (Ta) and vanadium (V) are produced using methods of physical vapor deposition. Magnetron sputtering and electron-beam evaporation processes (Jankowski, et al., 2004;2005) prove advantageous to deposit metals with high melt temperatures ( $T_m$ ) as fully-dense, thick coatings. Samples in the form of free-standing foils are characterized in the as-deposited condition and subsequent to high-temperature vacuum anneal treatments to assess stability and quantify the kinetics of diffusion for grain growth. Sample strength is measured under uniaxial tension using a pull tester, and hardness is measured by Vickers microindentation.

To assess structural origins of enhancements at the nanoscale, three basic structures are considered: a single layer of each metal is continuously sputter deposited, i.e. Ta, V; each metal is deposited in layered form by shuttered interruption, i.e. Ta-Ta, V-V; and a

composition modulated nanolaminate is produced by alternating deposition between elements, i.e. Ta-V; V-Ta. For reference, the first metal cited is the first layer deposited. Nanoscale features that are parallel versus perpendicular to the growth plane of the films, i.e. the relative effects of grain size versus the layer pair spacing, will be evaluated as affects on hardness as measured using nanoindentation.

### **EXPERIMENTALS**

#### **Synthesis and Processing**

Thick foils are prepared by electron-beam evaporation and magnetron sputtering. Coatings of 10-50  $\mu\text{m}$  are deposited onto sheets of muscovite. The substrates are subsequently peeled away to yield free-standing metal foils. The sputter deposition of the coatings utilizes planar magnetrons of 6.35-7.62 cm diameter. The sputter sources are operated in the dc mode using an ultra-high purity, working-gas of Argon from the boil off of liquid Argon. The control of substrate temperature and the energetic sputtered neutral (Thornton, 1974;1986) are key to the deposition of fully dense coatings. A Ta platen is resistively heated onto which the muscovite substrates are clamped. The coating temperature is measured using a thermocouple on the substrate side exposed to the deposition flux. The deposition temperature for the sputter deposits is less than 800 K, whereas heating up to 1500 K is used for evaporated coatings. For sputtering, the source to substrate separation is kept minimal at <8 cm, just beyond the hot electron sheath above the 0.9999 pure target, to provide the condition (Westwood, 1978, Somekh, 1984) for energetic sputtered neutrals.

Thin film nanolaminates are prepared by magnetron sputter deposition in a cryogenically-pumped vacuum chamber to a  $2 \cdot 10^{-5}$  Pa base pressure. The sequenced deposition of the metals produces a nanolaminate with  $N$  layer pairs. The layer thickness is controlled by the exposure time of the Si substrate wafer to the magnetron sputter source. The composition modulated nanolaminate is deposited so as to contain equal layer thicknesses of each chemical species. The Ta and V layers are deposited from greater than 0.999 pure target materials. The planar magnetrons are operated in the dc mode with a discharge cathode potential of  $215 \pm 2$  Volts and  $225 \pm 2$  Volts for Ta and V, respectively. The source-to-substrate separation is 7.3 cm and the