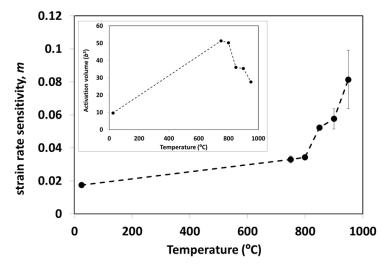
TEMPERATURE DEPENDENCE OF INDENTATION SIZE EFFECTS, PILE-UP AND STRAIN RATE SENSITIVITY IN POLYCRYSTALLINE TUNGSTEN FROM 25-950 ℃

Ben Beake, Micro Materials Ltd. ben@micromaterials.co.uk Adrian Harris, Micro Materials Ltd Jonathan Moghal, Department of Materials, University of Oxford Dave Armstrong, Department of Materials, University of Oxford

Key Words: Indentation size effects; high vacuum; elevated temperature; pile-up; viscoelastic correction.

Elevated temperature nanoindentation measurements were performed on polycrystalline tungsten to 950 °C. Tests were carried out under high vacuum conditions as tungsten oxidizes in air at >500 °C. The temperature dependence of the hardness, elastic modulus, strain rate sensitivity, activation volume and the indentation size effect in hardness were investigated at 25, 750, 800, 850, 900 and 950 °C. Thermal drift assessed from the last 60% of a hold period at 90% unloading was typically ~0.05 nm/s and it did not vary significantly with load or temperature [1]. The hardness measurements were in good agreement with previous determinations by non-depth sensing hot microhardness.



Above 800 °C the hardness of tungsten changes relatively little but more pronounced time-dependent deformation was observed from the temperature, (850 °C, see figure 1), around where Milman et al reported changes to dislocation cell structure occur [2]. The strain rate sensitivity determined by analysis of indentation creep data increased with temperature. Activation volume reached a peak of ~50 b^3 at 750-800 °C (see inset). Decreasing activation volume above 800 °C was due to the increase in strain rate sensitivity.

Fig. 1 Strain rate sensitivity and activation volume vs. T

Although there have been reports of reduced indentation size effects at elevated temperatures, for a bcc metal such as W, lattice resistance depends on T/T_c (where T_c , the critical temperature, at which flow stress becomes insensitive to temperature = 527 °C for W) and so size effects would be expected scale with this relative temperature. Pillar compression tests on W(100) to 400 °C ($T/T_c = 0.84$) have determined much stronger size effects in strength at higher temperatures [3]. In this current study to $T/T_c = 1.56$ significantly stronger indentation size effects in hardness were also found at elevated temperatures.

Elastic modulus measurements determined from standard elastic analysis of the unloading curves were higher than literature values, by ~13% at 750-800 °C and 30-40% at 850-950 °C. The greater discrepancy starts from the point (850 °C) at which more pronounced time-dependent deformation was observed. Time-dependency was therefore accounted for by a viscoelastic compliance correction [4]. After correction values of the elastic modulus agree to within ~1% at 750-800 °C, and to 6% of literature values at 950 °C. The small remaining discrepancy is consistent with the influence of pile-up since AFM measurements show that pile-up is significant in these high temperature indentations and the pile-up height increases slightly over the range 750-950 °C.

- [1] AJ Harris, BD Beake, DEJ Armstrong and MI Davies, Exp Mech (2016) DOI 10.1007/s11340-016-0209-3.
- [2] YV Milman et al, Acta Metal Mater 41 (1993) 2523-2532.
- [3] O Torrents Abad et al, Acta Mater 103 (2016) 483.
- [4] G Feng, A.H.W. Ngan, J Mater Res 17 (2002) 660-668