DEFORMATION AND FRACTURE MECHANISMS IN NANOCOMPOSITE AND NANOLAMINATE THIN FILMS REVEALED THROUGH COMBINATORIAL DESIGN AND NANOMECHANICAL TESTING

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We've integrated an atomic layer deposition (ALD), a physical vapor deposition (PVD) and a nanoparticle inert gas condensation (NP) deposition system into a single vacuum chamber. This combined system allows for PVD sputtering of micrometer thick films and incorporation of size filtered nanoparticles and/or controlled deposition of mono-layer highly conformal film coatings within a multilayer structure. In this way, unique model thin film microstructures can be architectured. We designed three thin films to understand the basic mechanism of plasticity and fracture in thin films: a) Al_2O_3 oxide films were deposited on combinatorial libraries of the ternary noble metal alloys with full compositional range to understand interfacial adhesion between oxide and noble metal alloys b) monosized tungsten nanoparticles were deposited at the interface of Cu/Ni multilayers to understand how thin film hardness and thermal stability can be engineered, c) ultrathin monolayers of Al_2O_3 layers were sandwiched between sputtered Al layers and micropillar compression was used to understand dislocation transmission and fracture across ultrathin ceramic layers.
<u>Combinatorial libraries</u>: By specifically programming the movement of shutters above the sample during PVD it is possible to create multilayered thickness gradients of three different materials, which can then be annealed to create films with full compositional range of a ternary phase diagram. The AlCuAu alloy consisted of multiple phases and intermetallics across the wafer; whereas the AuAgPt alloy consists of a solid-solution. Both alloys were then coated with a 500 nm thick layer of Al_2O_3, deposited using ALD, to survey the effect of composition on the adhesion probed by nanoindentation. Highest adhesion occurred in a two phase, Cu rich composition.
<u>Nanoparticles at interfaces</u>: The addition of nanoparticles to the interfaces in a nanolaminate, see Fig 1a, was found to increase the strength of the Cu/Ni film by more than 1 GPa above that of particle-free multilayers. However, at higher particle densities the hardness begins to decrease again, indicating there is likely an ideal particle concentration that would lead to the highest increase in hardness. This was attributed to competing mechanisms where the particles act both as dislocation sources and barriers to dislocation transmission. Initial in-situ XRD heating experiments show that the W nanoparticles can dissolve into the Ni layer to create a solid-solution Ni-W layer and react with oxygen, which could potentially create additional hardening in the material.
<u>Ultrathin ceramic films in metal multilayers:</u> 250 nm thick Al films have been stacked with varying Al_2O_3 interlayers (from 1 to 10 nm, in 1 nm increments, see Figure 1b.), showing high strength with a brick-and-mortar structure. 1 nm thick Al_2O_3 has been demonstrated to be sufficient for interrupting grain growth. Microcompression pillars were formed by FIB for investigation of mechanical properties. Very high yield strength was observed in the 600 MPa range. An initial TEM study of deformed samples showed that the deformation after compression is homogeneous and no obvious shearing was observed. The Al_2O_3 interlayers seems act as a dislocation sink, leading to no work-hardening and there seems no indication of dislocation accumulation at grain boundaries.

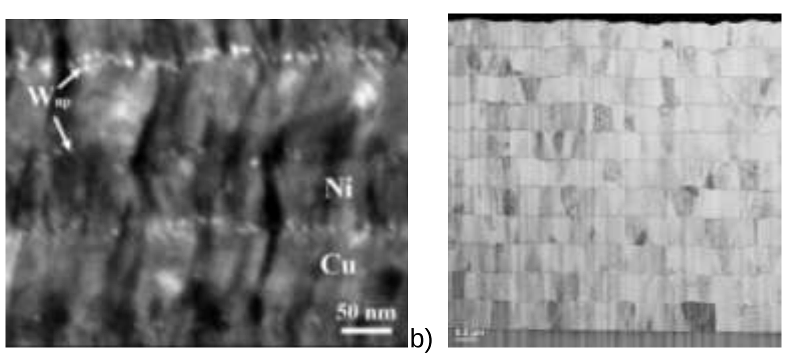
a)  b)

Figure 1 – TEM cross-section of a) tungsten NP at interface of Cu/Ni multilayers and b) an Al-Al_2O_3 multilayer thin film system