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In situ TEM Investigation of Mechanically Induced Phase Transformations in Nanoscale Composites

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For decades, it has been known that at layer thicknesses approaching a few nanometers, metastable phases can be energetically favored through the constraints imposed by epitaxial growth of thin films. This can result in microstructures that have significantly different mechanical properties than what would be found in the more commonly investigated bulk phases. In this work, we present in-situ HRTEM straining studies of two vapor deposited nanoscale multilayer composite systems: Al-TiN and Mg-Nb.

We conducted HRTEM in-situ straining experiments, with the intent of determining local defect content, defect dynamics, local deformation mechanisms (e.g. stress or plasticity-induced phase transformations), and interface evolution as a function of strain [1]. A particularly intriguing finding is depicted in Figure 1. Al-TiN multilayered composites with a fine interlayer of AlN only a few atom layers thick results in a mixed phase Zinc Blende (ZB) and Wurtzite (WZ) in the AlN layer. Remarkably, under in-situ straining in the TEM (Figure 1), the WZ phase will grow via dislocation glide of Shockley partials at the expense of the ZB phase (Figures 1b-e). The lattice mismatch between the two AlN phases and adjoining materials is different for WZ and ZB phases. The misfit dislocation structure is also different, and must evolve under strain as the ZB phase plastically transforms to the WZ structure.

Magnesium (Mg) alloys, while good lightweight structural material candidates, suffer from low strength and limited ductility at room temperature, due to a lack of available slip systems in hexagonal close-packed (hcp) structures [2, 3]. Improving strength without a concomitant loss of ductility is a hurdle to wider spread application of Mg based materials. Rather than grain refinement [4] or alloying with rare earth elements [5], our approach is to strengthen and improved deformability of Mg alloys through stabilization of the bcc phase of Mg in metal laminates [6, 7].

Since bcc Mg can be stabilized in between bcc/bcc Mg/Nb interfaces when the individual layer thickness is below 5 nm [6, 7], the ductility is improved as bcc Mg has additional active room temperature slip systems as compared to hcp Mg [8]. In-situ TEM mechanical testing is a useful tool for real-time observation of deformation mechanisms at nanometer scales. Our results directly validate the hypothesis that bcc Mg can accommodate large plastic deformation and further reveal that in bcc/bcc Mg/Nb, a reversible bcc-hcp phase transformation occurs during loading and unloading, as shown in Figure 2.

In both of these systems, we demonstrate that plastic deformation can drive phase transformations that are not otherwise seen in bulk, and can contribute to the design of strong, ductile advanced materials.

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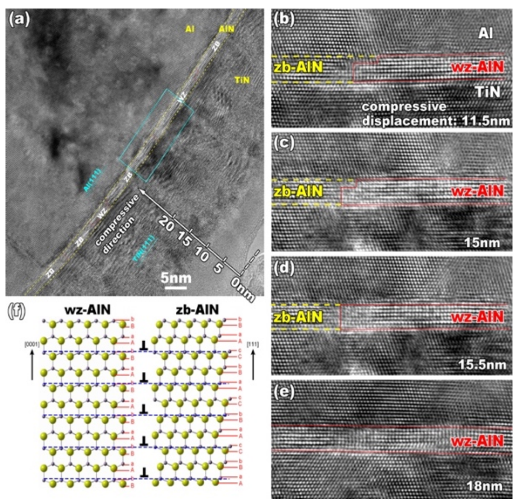

Figure 1. Stress-induced phase transformation in Al-AlN nanolayers. **(a)** one HRTEM image from *in situ* indentation test. WZ and ZB indicate the w-AlN and z-AlN phase. **(b)-(e)** HRTEM images of four progressive snapshots of the region outlined in the blue rectangle in **(a)**, showing that the w-AlN nucleates and propagates towards the right. **(f)** Atomic structures of wurtzite and zinc blende. The phase transformation is realized through the collective glide of Shockley partial dislocations on every two {111} planes of the zinc blende AlN [1].

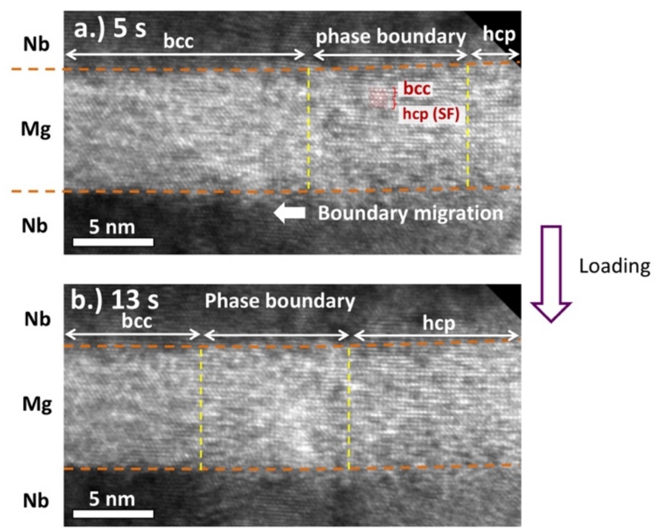

Figure 2. Stress-induced phase transformation in Mg-Nb nanolayers. **(a)** HRTEM image from *in situ* indentation test at t=5 sec. The bcc phase reverts to hcp upon loading **(b** HRTEM image of a mixed hcp-bcc phase boundary region that moves from right to left, transforming bcc Mg to hcp Mg.

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