Fracture at the Nanoscale

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Abstract:

The objective of this research was to develop a micro-electro-mechanical systems (MEMS)-based tensile and fracture testing device for ultra thin specimens *in situ* in the transmission electron microscope (TEM). In particular, we used nanofabrication and innovative design principles to miniaturize a mechanical testing device to a 3 × 5 mm size.

The technical contribution of this research is a unique experimental technique that can perform tensile/fracture testing of nanoscale materials with virtually no restriction on specimen thickness/size. The scientific contribution of this research is that the fundamentals of fracture at extreme (1-10 nm) length-scales will be explored, both qualitatively and quantitatively. Using this technique, we studied the mechanics of fracture in ultra thin films of titanium-titanium nitride. Our experimental results indicate that the titanium-titanium nitride interface is quite strong and that there is a lack of a plastic zone traveling ahead of the crack tip, which is typically seen in bulk materials.

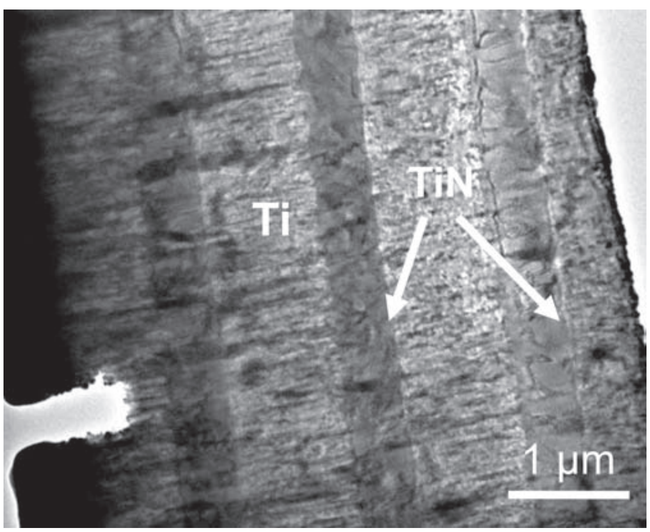

Figure 1: Titanium-titanium nitride multilayer system.

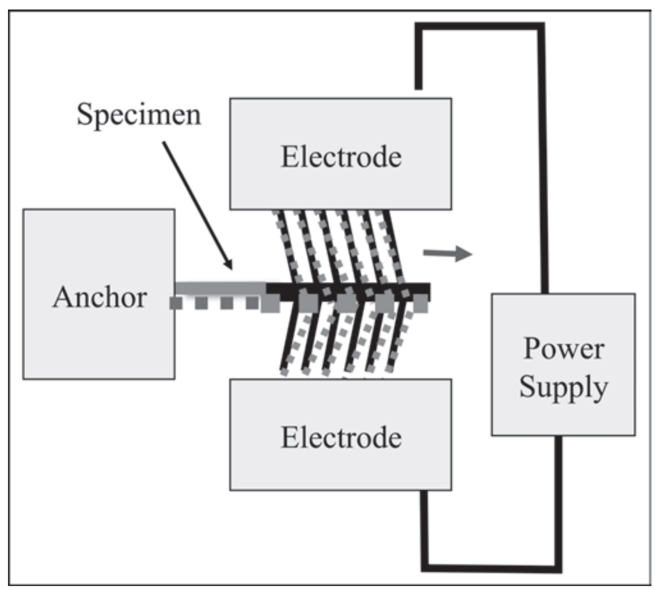

Figure 2: Tensile testing device schematic.

Introduction:

Advanced coating systems include alternating layers of hard and tough materials to achieve these two mutually exclusive properties. These layers can be only a few nanometers thin, and their interfaces make the deformation and fracture mechanics difficult to ascertain by conventional laws. Since transmission electron microscopes (TEM) visualize such small microstructures, we aimed to perform fracture testing of nanoscale thin sections inside the TEM. Our system was comprised of titanium and titanium nitride (Ti-TiN) (Figure 1), which is used as erosion resistant coatings in turbine engine compressor airfoils.

Experimental Procedure:

The tensile testing device design (Figure 2) featured bent beam thermal actuators to apply the stress on a freestanding specimen. Current would be passed through the electrodes and Joule heating would expand the beams, applying tensile stress on the specimen. Force sensors measure the applied stress and displacement sensors measure strain.

The devices were fabricated by deviceside and backside patterning of silicon-on-insulator (SOI) wafers using photolithography. Both sides of the wafer were then etched with deep reactive ion etching. The devices were released with hydrofluoric vapor etching.

The Ti-TiN specimen was milled with an ion beam from bulk substrate in focused ion beam/scanning electron microscope. The specimen was then integrated onto the tensile testing device with the Omniprobe nanomanipulator and platinum was deposited as a glue to hold the specimen to the sample. A notch was milled into the specimen as an initial point for crack formation.

Results and Conclusions:

We hypothesized that the crack would initiate at the notch which was milled into the sample with the focused ion beam. The crack should have propagated through the titanium layer, but arrested at the Ti-TiN interface since the boundary between the two would require higher energies in order to be crossed. If more energy in the form of increased stress was supplied, then the interface could be breached and the crack cold propagate through the next layer.

In our experiment, the crack initiated at a point below the notch and propagated through the Ti layer (Figure 3). No arrestation at the interface was noted and the crack traveled into the TiN layer (Figure 4). The crack was arrested in the TiN layer when the platinum glue holding the specimen to the device failed and stress was no longer being applied to the specimen.

Although the crack was not arrested at the interface as we had predicted, that the crack crossed the interface rather than traveling along the interface suggests that it is quite strong. In our experiment, we also noted the absence of a plastic zone ahead of the crack tip. In bulk materials, dislocations are typically noted traveling ahead of the crack tip. This demonstrated an interesting difference between fracture behavior in bulk and nanoscale materials.

Future Work:

Further research in Ti-TiN multilayer systems should be completed, including the effect of varying thicknesses of the two materials. Other areas of research include integrated specimen and device processing, which would eliminate the most challenging step—integrating the device and the specimen, as well as fracture testing using the described techniques on a wide range of other materials.

Acknowledgments:

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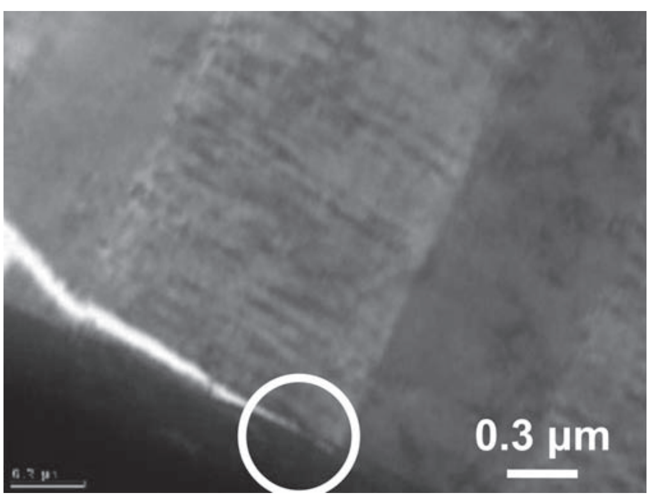

Figure 3: Crack propagating through titanium layer.

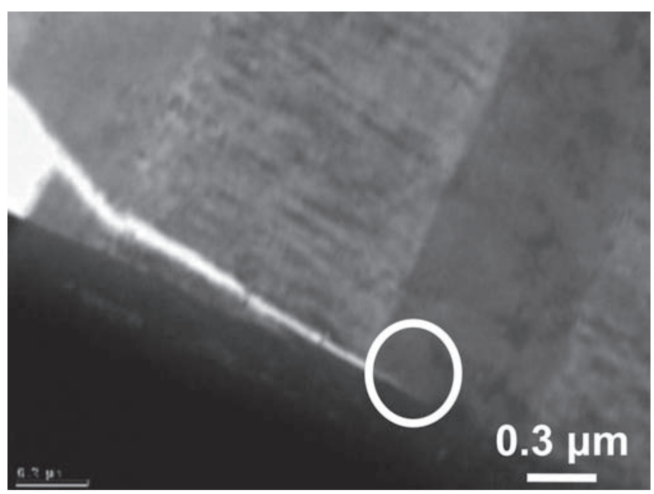

Figure 4: Crack propagating through titanium nitride layer.