SIZE MATTERS: Mechanical Response of Nano-Sized Metallic Systems

Julia R Greer*, Dongchan Jang, Jie Lian, Andrew Jennings, and Robert Maass
*Division of Engineering and Applied Sciences, California Institute of Technology
Pasadena, CA USA

Summary Useful properties of structural materials are generally governed by their bulk microstructure. For centuries, the improvements in structural materials relied heavily on processing, which in turn, dictated the resulting microstructure and properties. With the current rapid shift towards interdisciplinary materials science, significant improvements have been enabled by the development of microstructure-property relationships specifically by introducing a new design parameter, **size**. As we enter a revolutionary era in materials science, where specific material properties are attained through not only material but also architecture control of its constituents, often with micron- or sub-micron dimensions, it is imperative to develop a thorough understanding of material mechanical deformation properties at the appropriate scale.

The scope of our research focuses on the problems of unraveling the physical origins of size-dependent mechanical properties in nano-scale solids, where the presence of surfaces causes the emergence of unexpected deformation mechanisms in response to mechanical deformation. It has been shown, for example, that in single crystals strengths increase in a power law fashion with sample size reduction once micron scale is reached, and therefore can no longer be inferred from the bulk response or from literature. They are thought to arise from the distinct defect behavior that emerges as a result of reducing material dimensions to the nano-scale and manifest themselves by causing unusual mechanical properties. While these studies provide a powerful foundation for the fundamental deformation processes operating in these materials at small scales, they are a far reach from representing real materials used in structural applications, whose microstructure is often complex, containing boundaries and interfaces. In fact, both homogeneous (grain boundaries, twin boundaries, etc.) and heterogeneous (i.e. phase boundaries, precipitate-matrix boundaries, and even free surface) interfaces in size-limited features are crucial elements in the structural reliability of most modern materials. Establishing the link between the observed mechanical properties and microstructural evolution remains a grand challenge, and establishing a more quantified, predictive relationship between the two is a key focus in Greer's group.

Our key research thrusts lie in the development of innovative experimental approaches that enable *assessing* mechanical properties of nano-sized solids and *explaining* them through evolved *microstructure*. One approach involves fabrication of nano-scale samples with different initial microstructures (i.e. containing boundaries, interfaces, interphases, dislocations, etc.) ranging from below 50 nm to ~ 1 micron by using ion- and electron-beam assisted patterning, lithography and electroplating. In order to test their strengths in uniaxial tension and compression we built a unique nano-mechanical deformation instrument with simultaneous electrical measurement capability, SEMentor, comprised of Scanning Electron Microscope (SEM) and nanoindenter. In this talk we will describe how this *in-situ* technique has helped to address three specific classes of problems:

1. Hierarchical micro-lattice materials. Micro-trusses with periodic cellular architecture were fabricated using a process that enables precise control over architecture at three levels of hierarchy at three distinct length scales. Lattice unit cell (~mm-cm), hollow tube lattice member (μm – mm) and hollow tube wall thickness (~nm - μm) can be controlled independently, enabling the design of micro-lattice materials with unprecedented properties. We achieve hollow tube wall thicknesses <200 nm, resulting in metallic micro-lattices with densities as low as 0.9 mg/cc, as shown in Figure 1. In these lattice-type structures, as the truss member thickness is reduced to micron- and sub-micron levels, the interactions between the external dimensional parameter, film thickness and the microstructural parameter, grain size can drastically alter the material's mechanical strength. We report the compressive behavior of vertically oriented nanocrystalline Ni hollow truss members with two different wall thicknesses, i.e. 500 nm (thick) and 150 nm (thin). We show remarkably distinct collapse modes of thick and thin samples. The compressive strength of the thin sample is significantly lower than the thick sample and the theoretical prediction. While the thick sample collapses in brittle manner, via a single strain burst, resulting in a diamond-shaped, completely-crushed topology, the thin sample shows a gradually collapse, via a series of small, discrete strain bursts, with only the top portion deformed.

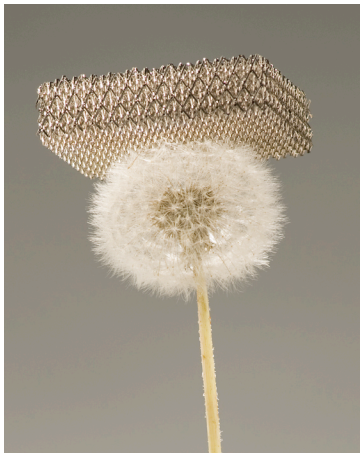

Figure 1. Photo of ultra-light metallic micro-lattice on dandelion (reprinted with permission from [1])

2. Size effects in plasticity of crystalline metals. Experimental results on uniaxial deformation of single crystals present us with an intriguing mystery: in a striking deviation from classical mechanics, we and others have demonstrated a "smaller is stronger" phenomenon in face-centered cubic (fcc), body-centered cubic (bcc), and hexagonal close-packed single crystals manifested by the significant (~50x) increase in strength as material size is reduced to below 100nm. Further,

very recently the group performed nano-mechanical tests on smallest-ever fcc nano-pillars and showed the emergence of strain rate sensitivity in fcc nano-crystals (not present at bulk scale), distinctly illustrating the transition in plasticity mechanism to dislocation nucleation control. Expanding these studies towards boundary-containing materials, we discovered that nanocrystalline metals exhibit the opposite trend: "smaller is weaker" while bi-crystalline metals remain unaffected, i.e. exhibit an identical size effect to their single crystalline counterparts [1]. Perhaps most intriguingly, nano-twinned Cu nano-pillars appear to attain highest tensile strengths reported for Cu and exhibit twin boundary-spacing dependent failure mode transition, as shown in Figure 2. Unlike their bulk counterparts, the deformation in these nano-sized metallic crystals is discrete, with many intermittent strain bursts, and therefore cannot be modeled by using continuum theories. We discuss some of the existing theories attempting to explain size-dependent, discrete stress-strain signatures based on dislocation activity. We also characterized stochastic behavior and discuss it in the framework of Mean-Field Theory [3], as shown by a scaling-collapse of the stress-dependent slip size distributions in Figure 3.

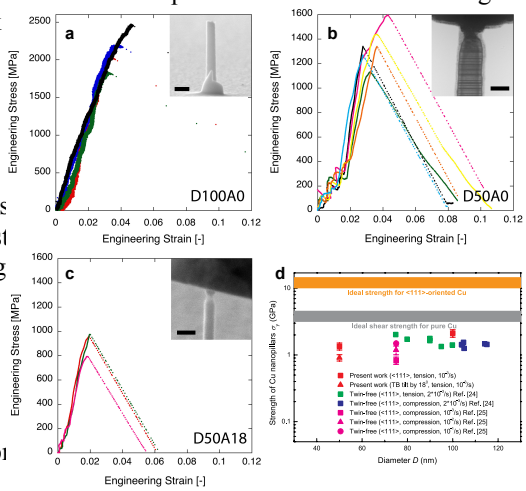

Figure 2. Tensile stress-strain curves and morphology in nano-twinned Cu nano-pillars with different twin spacings [2]

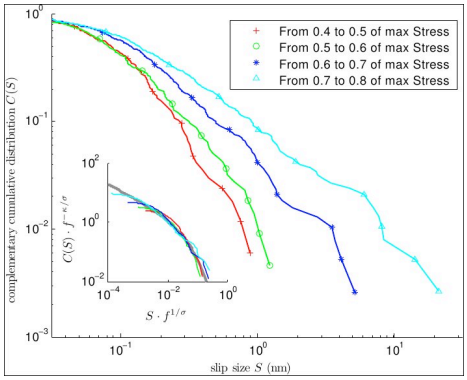

Figure 3. Stress-binned cumulative histogram C(S,τ) of slip sizes S as a function of applied stress τ [3].

3. Mechanical behavior of nano-scale amorphous glasses. Beyond crystalline metals, we will convey some results of our investigations on small-scale response of metallic glasses (amorphous metallic alloys). A common understanding of metallic glasses is their failure via catastrophic shear band formation. Our group discovered that these materials attained superior strength and plastic deformability of ~ 25% (unprecedented at that point) when reduced to ~100nm. Further, we show that to utilize this size-induced ductility towards useful applications, thin layers of metallic glasses can be combined with either crystalline metals or polymers into nanolaminates, thereby retaining the high strength and non-catastrophic deformation.

References

[1] Schaedler, et al.: Ultralight Metallic Microlattices. *Science* **334**:962, 2011

[2] Jang, D., Li, X., et al.: Nanotwinned nanopillars: fabrication and deformation Mechanisms (submitted) 2011

[3] Friedman, N. et al.: Statistics of dislocation slip avalanches in nano-sized single crystals explained by a simple mean field model (submitted) 2011

[1] For the particular boundary studied