Superhard Nanostructured Films

NSF NIRT Grant 0103169

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Evidence from a number of studies suggests that it should be possible to produce nanocrystalline composites that are *superhard*—possessing hardness rivalling diamond—and that also possess combinations of other properties that make them superior to diamond for applications requiring friction- and wear-resistance. Such superhard nanostructured films promise revolutionary improvements for wear protection for applications such as high-speed machining as well as in the emerging field of miniaturized moving parts in microelectromechanical systems (MEMS).

This project [1] involves both a practical approach to synthesizing such materials, based on plasma synthesis and deposition of nanoparticles, and fundamental studies whose goal is to relate the nanoscale structure of these materials to their mechanical properties. The interdisciplinary research team includes expertise in fracture micromechanics, characterization of structure and interfaces, plasma science and technology, aerosol science and technology, and MEMS fabrication. In addition, the Minnesota group is collaborating with colleagues at Los Alamos (M. Baskes) and Sandia (M. Horstemeyer) National Laboratories who are leading experts on atomistic simulations of small-volume deformation.

Two different but related types of deposition are being studied: deposition of continuous nanoparticle films, using a method known as hypersonic plasma particle deposition (HPPD), and the use of focused, tightly collimated beams of nanoparticles to accomplish microfabrication. In HPPD [2] nanoparticles of desired composition nucleate in a reactive thermal plasma and are then accelerated in a hypersonic expansion, causing them to deposit onto a substrate by high-velocity inertial impact to create a dense, nanostructured film. This process has been used to deposit silicon carbide films at rates exceeding 1 μm/s, with measured hardness up to ~39 GPa, above that of conventional silicon carbide. Current efforts are focused on the deposition of nanocomposite films, where the composites include various combinations of the carbides and nitrides of boron, silicon and titanium.

In focused particle beam deposition (FPBD) [3], the substrate is replaced by a series of aerodynamic lenses, which focus the particles to a narrow beam. The Minnesota group is exploring the use of these beams for microfabrication, as suggested by Figure 1. If a substrate is scanned across the particle beam one deposits "lines" of nanoparticles, as shown in Figure 2. These lines are over 10 mm long. The line on the left has a central height of 5 μm, and is 35 μm wide at its half-height.

While the deposition of such lines will be crucial for the synthesis of two- and three-dimensional microdevices made out of nanoparticles, they have also proven serendipitously useful for the characterization of individual nanoparticles. As one moves away from a line one finds a

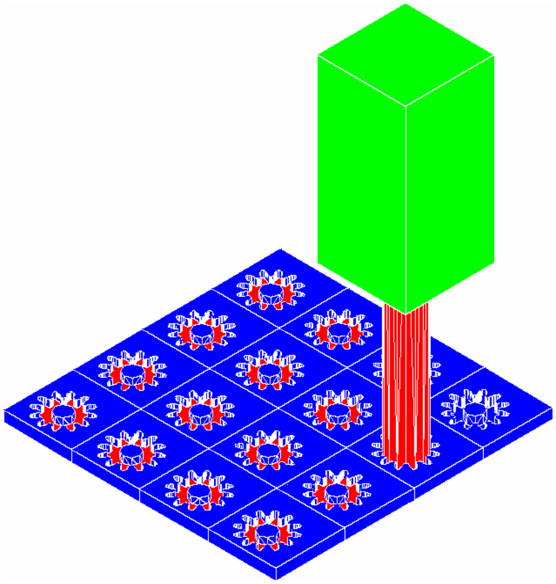

Figure 1. Fabrication of MEMS gears using focused nanoparticle beams.

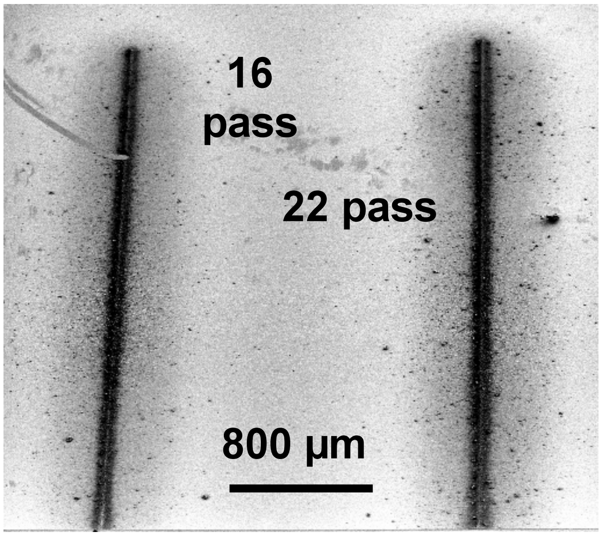

Figure 2. Lines deposited by scanning across focused beam of silicon nanopartiples.

decreasing density of particles, and finally one finds isolated particles that can be imaged and characterized. Examples of such characterization are shown in Figures 3–6. In Figure 3 the elemental composition of a single silicon particle was examined using electron energy loss spectroscopy, with spectra obtained at 1-nm intervals [4]. This technique clearly resolves the oxide surface layer. Figure 4 shows a bright-field high-resolution transmission electron microscope (HRTEM) image [4] of microtwins in a single β-SiC particle. The changes in atomic stacking are evident across the twin boundaries.

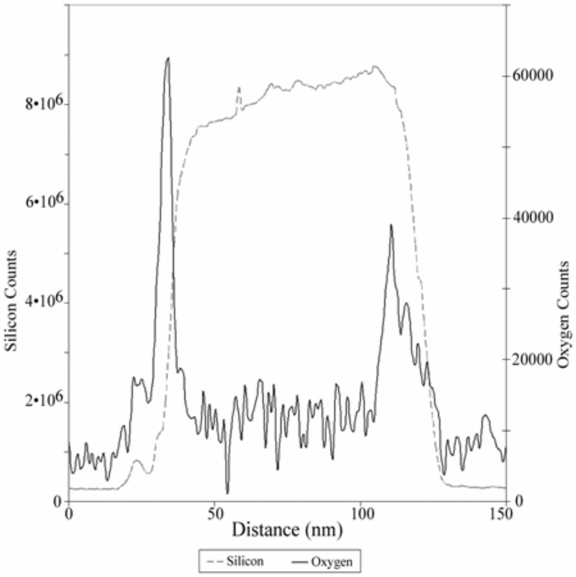

Figure 3. Spatial profiles of electron energy loss spectra across a single silicon particle.

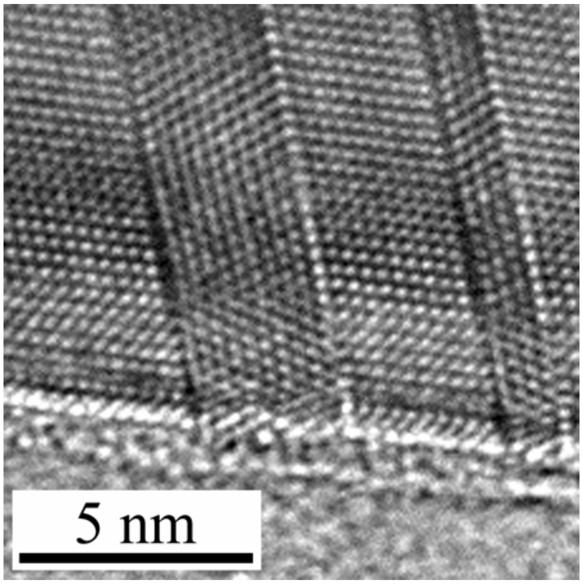

Figure 4. Bright field HRTEM image of microtwins in a β-SiC particle.

The mechanical behavior of nanostructured materials is currently of considerable interest. The ability to probe individual nanoparticles using nanoindentation provides a window into mechanical response at small length scales. Figure 5 shows a series of load-displacement curves obtained by nanoindenting a single nanoparticle deposited by FPBD onto a sapphire substrate [5]. The particle is successively compressed with each load cycle, until finally it fractures. From these studies one can determine “hardening curves”, as seen in Figure 6 for several particles of various initial sizes [5]. Hardness is seen to increase as particle size decreases. Hardness values several times higher than that of bulk silicon (12 GPa) were measured for these nanoparticles. To our knowledge, these are the first reported nanoindentation studies of individual nanoparticles.

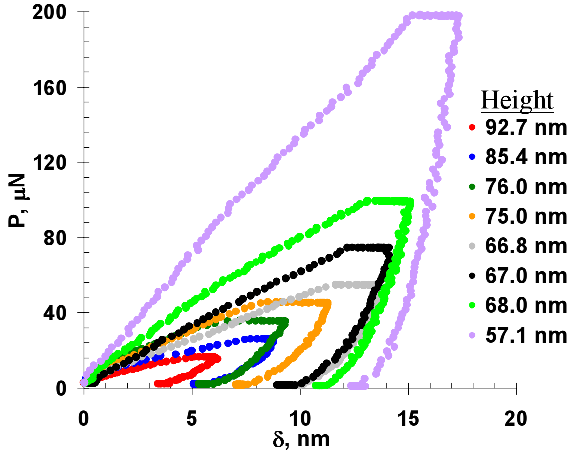

Figure 5. Successive load-displacement curves of a silicon particle with an initial diameter of 92.7 nm.

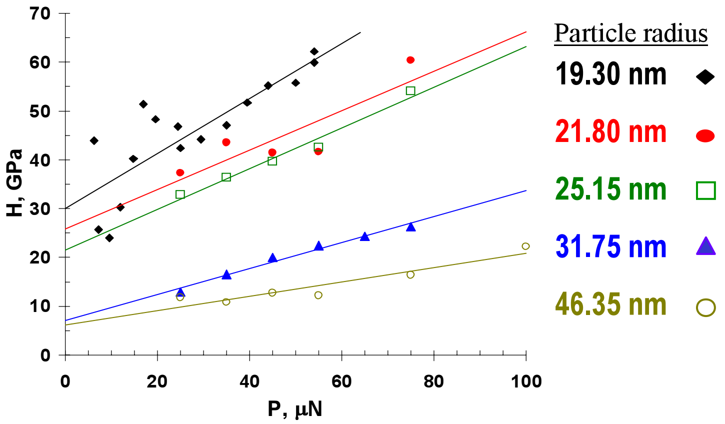

Figure 6. Hardening curves for silicon particles of various initial sizes.

References

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