

Exploring nanomechanics with high-pressure techniques

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ABSTRACT

For around three decades, high-pressure techniques have been used to study nanomaterials. In most studies, especially the early ones, x-ray diffraction and Raman and infrared spectroscopy were used to investigate the structural transition and equation of state. In recent years, the exploration has been extended to the plastic deformation of nanomaterials by using radial diamond-anvil-cell x-ray diffraction and transmission electron microscopy. Compared with the traditional techniques, high-pressure techniques are more advantageous in applying mechanical loads to nanosized samples and characterizing the structural and mechanical properties either *in situ* or *ex situ*, which could help to unveil the mysteries of mechanics at the nanoscale. With such knowledge, more-advanced materials could be fabricated for wider and specialized applications. This paper provides a brief review of recent progress.

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I. INVESTIGATING PLASTIC DEFORMATION OF NANOMATERIALS

A. Probing lower size limit of dislocation activity

Many different reports suggest that below a certain size scale, other mechanisms such as grain rotation, grain boundary sliding, and diffusion would take over from dislocations. However, many controversies and debates persist because of the lack of *in situ* examinations. In 2012, Chen *et al.*¹ studied the deformation texturing of nickel with radial diamond anvil cell (rDAC) x-ray diffraction (XRD), having found that dislocation activity can be extended down to 3 nm (Fig. 1). This new finding inspired more exploration and has advanced high-pressure nanomechanics considerably.

B. Probing grain rotation at the nanoscale

Grain rotation is one of the plastic deformation mechanisms of powdered and polycrystalline materials. Although it is feasible to observe grain rotation at the microscale, it is difficult to probe it *in situ* at the smaller nanoscale.² To overcome technical difficulties, Zhou *et al.*³ used Laue x-ray microdiffraction to track grain-rotation markers in nickel nanocrystals. On average, markers of the same size in the same stress environment should rotate by the same amount, but surprisingly the markers in the 70-nm nickel media were found to

rotate the most, thereby challenging the traditional belief that smaller grains rotate more and faster. The reversal in the size dependence of grain rotation is thought to arise from the mechanism transition between deformation mediated by grain boundary dislocation and that mediated by grain interior dislocation. This new finding could be useful for developing nanotechnology such as material fabrication, especially when abnormal grain growth mediated by oriented alignment is involved. With this knowledge, melt-free growth of bulk-sized single crystals could be possible.

C. Revealing ductility of nanoceramics

Ceramics are usually brittle, but nanoceramics are much less so. The traditional belief is that the improved ductility arises from the surface diffusivity of nanoceramics. Recently Chen *et al.*⁴ observed substantial texturing in pressurized nanoceramic MgAl₂O₄. The observed texturing indicates that dislocation-associated mechanisms, usually suppressed in ceramics at room temperature, become operative in nanoceramics, thereby making nanoceramics ductile. This finding suggests that room-temperature ductility of ceramics could be achievable and that making machinable ceramic parts may be possible.

Silicon is also a brittle material. Zeng *et al.*⁵ used rDAC XRD to probe *in situ* the deformation of silicon nanoparticles. They found

that pressure-induced phase transitions account for the large plasticity, which is quite interesting. Actually, in a general sense, phase transitions are coherent plastic deformation. The change of atomic positions and mass density between different phases could increase the ductility of materials. Their results suggest that the plastic deformation of materials with and without phase transition involved can be quite different. This could be an interesting topic in plasticity, demanding more exploration.

II. ACHIEVING ENHANCED STRENGTHENING IN NANOMETALS

In conventional mechanics, the strength of metals usually increases with decreasing grain size, following the Hall–Petch relationship. To test this law at the nanoscale, Yang *et al.*⁶ examined the yield strength of nonhydrostatically compressed tungsten with synchrotron XRD techniques. They reported a great enhancement of yield strength for the tungsten nanocrystals. The Hall–Petch effect

does not fail even down to 10 nm. The yield strength of 10-nm tungsten is around 3.5 times larger than that of the micrometer-grained tungsten.

Some MD simulations suggest size softening below a size between 10 nm and 15 nm due to the transition of plastic deformation mechanisms. However, there have been no direct experimental measurements of the strength of nanograins below 10 nm–15 nm because of the technical limitation in traditional mechanical measurements at such small nanoscales. Zhou *et al.*⁷ investigated with rDAC XRD and found the yield strength of a 3-nm-grain-size sample to be 10 times that of a common commercial nickel material (Fig. 1). That work was the first experimental observation of the strength of a nanometal with a grain size below 10 nm and also the first report on grain-size strengthening in sub-10-nm pure metal. It showed that rDAC XRD has advantages over transmission electron microscopy (TEM) in determining material strength, although TEM has its own advantages in defect and microstructure analysis. However, rDAC XRD characterizations

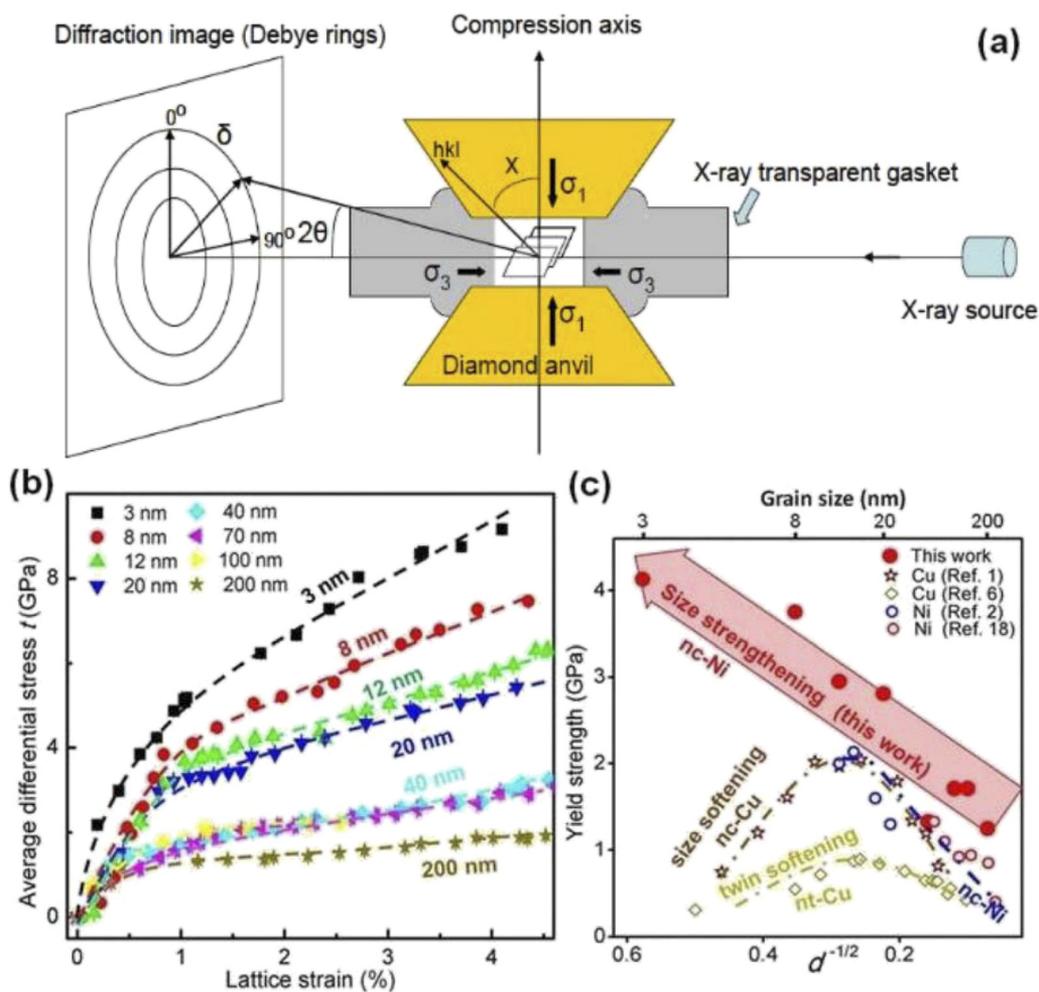


FIG. 1. (a) Schematic of experimental setup for radial diamond-anvil-cell x-ray diffraction.¹ (b) Stress–strain curves of nickel nanocrystals with different grain sizes.⁷ (c) Size strengthening extends down to 3 nm.⁷

should be extended to overlap with the traditional characterizations for convincing calibrations.

III. HIGH-PRESSURE FABRICATION

The above results emphasize the importance and effectiveness of rDAC XRD characterizations in assessing the mechanical performance of nanomaterials as well as introducing a new fabrication method. The strength of materials can be measured reproducibly for grain sizes of a few nanometers. Thus, rDAC XRD techniques are expected to play an important role in the research and development of nanomaterials.

The knowledge achieved from high-pressure plastic deformation and strengthening studies could provide a good guide to the fabrication of materials with exceptional performance. Tian and colleagues^{8,9} found that Hall–Petch hardening extends down to sub-5-nm twin thickness in cubic boron nitride and diamond, and they achieved twice the hardness of single-crystal counterparts. By introducing hierarchical nanostructures, they succeeded recently in making a diamond composite with exceptional toughness.¹⁰ More insights could be achieved if rDAC XRD studies are made. With more knowledge about nanomechanics available, it can be expected that bulk-volume nanometals with ultra-high strength and nanoceramics with ultra-toughness can be fabricated for a wide range of applications.

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