LABORATORY EXPERIMENT 3
Plastic deformation
from the micro- to nanoscale

PreLab Questions:

1. Name 2 ways in which instrumented indentation is different from uniaxial mechanical testing of materials.

2. Explain the physical meaning of a material's "hardness" in words.

I. Introduction

There are several examples in materials engineering in which materials are available only in small volumes – either because they result from new, expensive and difficult processing techniques, or because they are designed intentionally to be small in one dimension. An example of the latter is a thin film or structural coating. In such cases, mechanical properties cannot be quantified easily through uniaxial force-displacement testing of structures (e.g., Laboratory Experiment 1) or stress-strain responses of macroscale materials (e.g., Laboratory Experiment 2). One alternative is mechanical testing based on three-dimensional contact between the sample surface and a rigid probe of known geometry and mechanical properties. In this experiment, you will analyze the contact response of several classes of materials via the simple but widely used approach of microscale hardness testing, as well as the more quantitative instrumented indentation testing at the nanoscale. We analyze the data from such experiments using concepts from contact mechanics.

In **hardness testing**, a probe such as a cone-shaped diamond is pushed into a flat, well-polished sample surface to a specific maximum load, held for a specified duration, and then retracted from the surface. Plastic deformation results in a mark (indentation) in the surface, and measurement of the size of that mark indicates the material hardness, which is defined as:

$$H = P_{max}/A_{res} \qquad (1)$$

Figure 1. Hardness indentation for Vickers (square pyramid) indenter geometry. A_{res} is determined by measuring the indentation diagonals.

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http://www.imagemet.com/images/hardness2_Result.jpg

where P_{max} is the maximum load and A_{res} is the projected (2-dimensional) area of the indentation that is observed after unloading (residual) via optical microscopy. As shown in Eq. (1), hardness has units of [N/m^2], but is not physically equivalent to stress because the measured projected area of the indentation is not the actual area normal to the applied load of the 3-dimensional indenter.

One of the early applications of hardness was as an approximate measure of the yield strength σ_y of the material. For materials that undergo plastic deformation primarily via slip (e.g., metals for which dislocation motion requires shear stress), it has been demonstrated empirically and by "slip line theory" that:

$$H \sim 3\sigma_y \qquad (2)$$

In **instrumented indentation**, the experimental approach is quite similar, but the load P and the depth of penetration h of the indenter are measured continuously during loading and unloading (Fig. 1). If $P_{max} < 100$ nN or $h_{max} < 100$ nm, this is termed nanoindentation (but be aware that the term is often used in the literature even when the deformation actually exceeds this range). The result is a load-displacement response that is similar to that obtained in uniaxial mechanical testing, but the loading is multiaxial and thus more complex to analyze in terms of uniaxial mechanical properties such as E and σ_y. Most importantly, the geometry of the indenter dictates that there is a power-law relationship between P and h even during elastic deformation, unlike the linear relationship expected for uniaxial testing of a material in the elastic regime.

Hardness can also be calculated from these data according to Eq. (1), but the advantage of this approach is that the actual projected contact area A can be computed from the $P - h$ response rather than measured for each experiment. For the conical indenter used in this experiment:

$$A \sim 24.5 \, (h_c)^2 \qquad (3)$$

where contact depth h_c is given by a semi-empirical equation that accounts for material sinking in around the perimeter of the indenter. The elastic modulus E is inferred from the instantaneous response of the material upon unloading from P_{max}:

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http://www.nanoindentation.cornell.edu/pictures/schematic_nanoindenter.gif

http://www.nanoindentation.cornell.edu/pictures/DoernerNix_schematic_2.gif

(a)   (b)

Figure 1. Instrumented indentation enables measurement of mechanical properties of small material volumes. (a) Schematic of one type of indenter; (b) Typical load-displacement response, where S = unloading stiffness dP/dh, and h_c is contact depth. Images from http://www.nanoindentation.cornell.edu/.

$$E_r = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A(h_c)}} \qquad (4)$$

where $S = dP/dh|_{Pmax}$ is the tangent to the unloading portion of the $P - h$ response and is also called stiffness S. The area between the loading and unloading portions of the $P - h$ response directly quantifies the amount of work required to plastically deform the material W_p, and therefore the yield strength σ_y can be calculated as a function of W_p.

Note that instrumented micro- and nanoindentation are used for a range of purposes other than mechanical property extraction, including fracture toughness and residual stress determination in physically small samples (e.g., enamel-dentin junction of teeth) as well as dislocation nucleation (which requires high stresses and high displacement resolution).

II. Objectives
The objectives of this experiment are to:
1. Conduct hardness tests on: an amorphous ceramic (glass); 2 ductile metals, an alloy (brass) and a pure metal (copper); 2 polymers (low density polyethylene and ultrahigh molecular weight polyethylene); and a heat-treated metal for which the microstructure and mechanical properties vary as a function of distance from the surface (case-hardened steel).
2. Conduct and analyze nanoindentation as a function of P_{max} for two samples: bulk, pure copper and a thin film of pure copper on silicon. Extract elastic modulus E and indentation hardness H from these data.
3. Explore mechanical properties including elastic, plastic, and fracture behavior for this wide range of materials from the micro- to nanoscale. Understand how structure,

composition, molecular weight and substrate-constraint affect mechanical properties measured in this way.