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Spherical nanoindentation stress–strain curves

Siddhartha Pathak [a], Surya R. Kalidindi [b,*]

[a] Center for Integrated Nanotechnologies, Los Alamos National Laboratory, Los Alamos, NM 87545, USA
[b] George W. Woodruff School of Mechanical Engineering, Georgia Institute of Technology, Atlanta, GA, USA

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ABSTRACT

Although indentation experiments have long been used to measure the hardness and Young's modulus, the utility of this technique in analyzing the complete elastic–plastic response of materials under contact loading has only been realized in the past few years – mostly due to recent advances in testing equipment and analysis protocols. This paper provides a timely review of the recent progress made in this respect in extracting meaningful indentation stress–strain curves from the raw datasets measured in instrumented spherical nanoindentation experiments. These indentation stress–strain curves have produced highly reliable estimates of the indentation modulus and the indentation yield strength in the sample, as well as certain aspects of their post-yield behavior, and have been critically validated through numerical simulations using finite element models as well as direct in situ scanning electron microscopy (SEM) measurements on micro-pillars. Much of this recent progress was made possible through the introduction of a new measure of indentation strain and the development of new protocols to locate the effective zero-point of initial contact between the indenter and the sample in the measured datasets. This has led to an important key advance in this field where it is now possible to reliably identify and analyze the initial loading segment in the indentation experiments.

Major advances have also been made in correlating the local mechanical response measured in nanoindentation with the local measurements of structure at the indentation site using complementary techniques. For example, it has been shown that the combined use of Orientation Imaging Microscopy (OIM, using Electron BackScattered Diffraction (EBSD)) and nanoindentation on polycrystalline metallic samples can yield important information on the orientation dependence of indentation yield stress, which can in turn be used to estimate percentage increase in the local slip resistance in deformed samples. The same methods have been used successfully to probe the intrinsic role of grain boundaries in the overall mechanical deformation of the sample. More recently, these protocols have been extended to characterize local mechanical property changes in the damaged layers in ion-irradiated metals. Similarly, the combined use of Raman spectroscopy and nanoindentation on samples of mouse bone has revealed tissue-level correlations between the mineral content at the indentation site and the associated local mechanical properties. The new protocols have also provided several new insights into the buckling response in dense carbon nanotube (CNT) brushes. These and other recent successful applications of nanoindentation are expected to provide the critically needed information for the maturation of physics-based multiscale models for the mechanical behavior of most advanced materials. In this paper, we review these latest developments and identify the future challenges that lie ahead.

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* Corresponding author. Tel.: +1 404 385 2886.
E-mail address: surya.kalidindi@me.gatech.edu (S.R. Kalidindi).
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1. Introduction

For more than a century, the indentation test has been one of the most commonly employed techniques for characterization of the mechanical properties of a vast range of materials [1,2]. In a typical test, a hard indenter of known geometry is driven into a softer sample by applying a preset load or displacement. The dimensions of the resultant imprint are then measured and correlated to a hardness index number. With the advent of higher resolution testing equipment, it has now become possible to continuously control and monitor the loads and displacements of the indenter as it is driven into and withdrawn from a sample material. Known as nanoindentation (or instrumented indentation testing, or depth sensing indentation), this significantly expands the capabilities of the traditional hardness testing method [3,4]. Instrumented indentation has significant advantages over conventional indentation testing, since it can potentially produce very reliable measurements of stress–strain curves from fairly small indentation depths (of the order of a few nanometers).

The popularity of indentation tests stems in part from its versatility, ease of use (see Fig. 1), and its potential for high throughput. This is in contrast to most of the other currently used methods for interrogating the local mechanical properties at micron and sub-micron length scales that rely largely on testing miniaturized samples in nominally homogeneous deformation/stress modes [5]. For example, the compression testing of micro-pillars produced by removing material around a selected region of interest using a focused-ion beam (FIB) has attracted the recent attention of many researchers [6]. However these techniques typically require tremendous resources in terms of sample preparation, test conditions and operator time, which make their large scale use uneconomical. On the other hand nanoindentation, when aided with proper analysis methods, is capable of producing the desired information at significantly lower effort and cost. Moreover, reliable and quantitative measurement of mechanical degradation of surface layers (e.g., ion-irradiated materials in nuclear applications) is currently only possible with indentation techniques. This high throughput methodology when used in conjunction with structure information measured locally at the indentation site has the potential to become a key tool in efforts aimed at the maturation of physics-based multiscale materials models.

A common limitation in a majority of the indentation analysis methods used today is that the estimation of material properties, such as Young's modulus and hardness, are typically made from the measured unloading segments of load–displacement curves (after some amount of elastic–plastic loading) under the assumption that the unloading segments are predominantly elastic [7,8]. In this approach, the plastic deformation induced during the loading segment is likely to influence strongly the values of the mechanical properties (e.g. hardness) extracted from these experiments. This problem has been recognized since the early 1890s and numerous attempts have been made to measure the 'absolute hardness' of a material [9]. However, quantitative

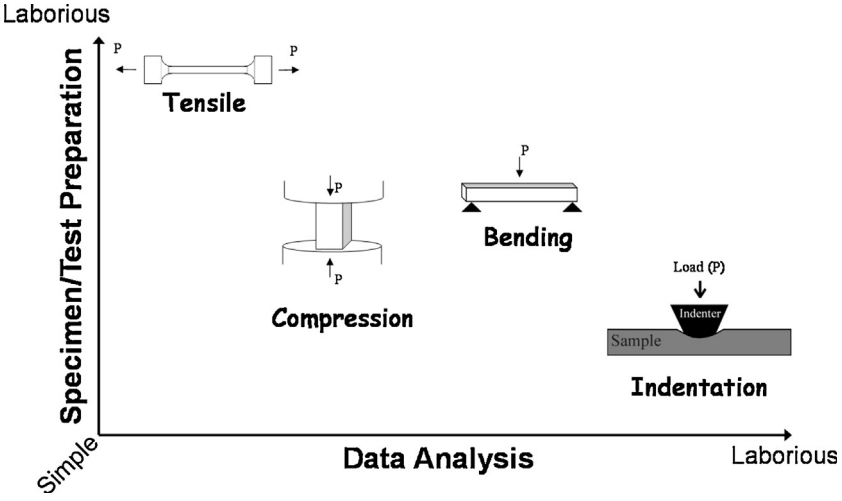

Fig. 1. Various mechanical testing methods for micron to sub-micron length samples.