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Nanoindentation

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Introduction

Indentation testing is a simple method that consists essentially of touching the material of interest whose mechanical properties such as elastic modulus and hardness are unknown with another material whose properties are known. The technique has its origins in Mohs' hardness scale of 1822 in which materials that are able to leave a permanent scratch in another were ranked harder material with diamond assigned the maximum value of 10 on the scale. The establishment of the Brinell, Knoop, Vickers, and Rockwell tests all follow from a refinement of the method of indenting one material with another. Nanoindentation is simply an indentation test in which the length scale of the penetration is measured in nanometres (10^{-9} m) rather than microns (10^{-6} m) or millimetres (10^{-3} m), the latter being common in conventional hardness tests. Apart from the displacement scale involved, the distinguishing feature of most nanoindentation testing is the indirect measurement of the contact area — that is, the area of contact between the indenter and the specimen. In conventional indentation tests, the area of contact is calculated from direct measurements of the dimensions of the residual impression left in the specimen surface upon the removal of load. In nanoindentation tests, the size of the residual impression is of the order of microns and too small to be conveniently measured directly. Thus, it is customary to determine the area of contact by measuring the depth of penetration of the indenter into the specimen surface. This, together with the known geometry of the indenter, provides an indirect measurement of contact area at full load. For this reason, nanoindentation testing can be considered a special case of the more general terms: depth-sensing indentation (DSI) or instrumented indentation testing (IIT).
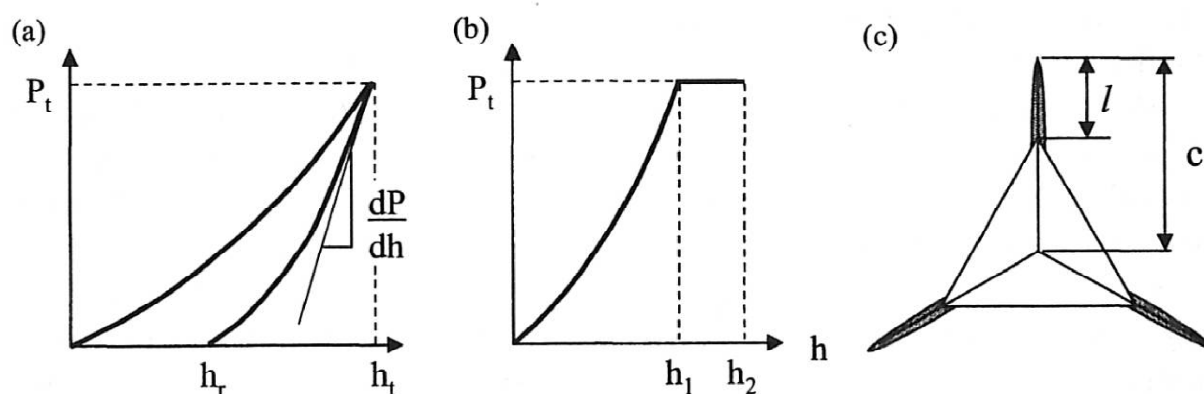
Fig. 1 Load-displacement curves for (a) an elastic plastic solid and (b) a viscoelastic solid for a spherical indenter and (c) cracks emanating from the corners of the residual impression in a brittle material.

It is not only hardness that is of interest to materials scientists. Indentation techniques can also be used to calculate elastic modulus, strain-hardening exponent, fracture toughness (for brittle materials), and viscoelastic properties. How can such a wide variety of properties be extracted from such a simple test, which, in many respects, can be considered a "non-destructive" test method? Consider the load-displacement response shown in Fig. 1. This type of data is obtained when an indenter, shaped as a sphere, is placed into contact with the flat surface of the specimen with a steadily increasing load. Both load and depth of penetration are recorded at each load increment (ultimately providing a measure of modulus and hardness as a function of depth beneath the surface). Following the attainment of the maximum load, in the material shown in Fig. 1 (a), the load is steadily removed and the penetration depth recorded. The loading part of the indentation cycle may consist of an initial elastic contact, followed by plastic flow, or yield, within the specimen at higher loads. Upon unloading, if yield has occurred, the load-displacement data follow a different path until at zero applied load, a residual impression is left in the specimen surface. The maximum depth of penetration for a particular load, together with the slope of the unloading curve measured at the tangent to the data point at maximum load, lead to a measure of both hardness and elastic modulus of the specimen material. In some cases, it is possible to measure elastic modulus from not only the unloading portion, but also the loading portion of the curve. For a viscoelastic material, the relationship between load and depth of penetration is not linearly dependent. That is, for a given load, the resulting depth of penetration may depend upon the rate of application of load as well as the magnitude of the load itself. For such materials, the indentation test will be accompanied by "creep," and this manifests itself as a change in depth for a constant applied load as shown in Fig. 1 (b). An analysis of the creep portion of the load-displacement response yields quantitative information about the elastic "solid-like" properties of the specimen, and also the "fluid-like" or "out-of-phase" components of the specimen properties. In brittle materials, cracking of the specimen may occur, especially when using a pyramidal indenter such as the three-sided Berkovich or the four-sided Vickers indenter. As shown in Fig. 1 (c), the length of the crack, which often begins at the corners of the indentation impression, can be used to calculate the fracture toughness of the specimen material.

More advanced methods can be employed to study residual stresses in thin films, the properties of materials at high temperatures, scratch resistance and film adhesion, and, in some cases, van der Waals type surface forces. In this book, all these issues are examined and reported beginning with a description of the method of test and the basis upon which the analysis is founded. Later chapters deal with the various corrections required to account for a number of instrumental and materials related effects that are a source of error in the measurement, theoretical aspects behind the constitutive laws that relate the mechanical properties to the measurement quantities, recent attempts at formulating an international standard for nanoindentation, examples of applications, and a brief description of commercially available instruments.