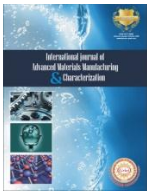

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Relevance of Nanoindentation Experiments in Materials Research-A Review

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ABSTRACT

Nano indentation is now commonly used for the study of mechanical properties of materials on the nanoscale.. It offers a direct measure of contact stiffness during the loading portion of an indentation test and, being somewhat insensitive to thermal drift, allows an accurate observation of small volume deformation. Nano scale damage caused by fatigue is of critical importance to the reliability of ultrathin protective overcoats and micro/nanostructures. The cyclic loading used in the nanoidentation makes the technique useful for the evaluation of nanofatigue. Methodologies of the nanoindentation technique used for the characterization of layered materials and nonhomogeneous composites are reviewed and discussed. Applications of the nanoindentation experiments to the measurement of contact stiffness, elastic modulus, hardness, creep resistance, and fatigue properties of the materials are presented. The nanoindentation in conjunction with nanoscratch and friction and wear tests, can be satisfactorily used for the materials characterization of magnetic storage and micro electromechanical systems (MEMS) devices and should find more application

Introduction

Indentation has been the most commonly used technique to measure the mechanical properties of materials because of the ease and speed with which it can be carried out. At the beginning of the 20th century, indentation tests were first performed by Brinell, using spherical and smooth balls from ball bearings as indenters to measure the plastic properties of materials [1,2]. The Brinell test was quickly adopted as an industrial test method soon after its introduction and prompted the development of various macro- and microindentation tests [3]. Traditional indentation testing involves optical imaging of the indent. This clearly imposes a lower limit on the length scale of the indentation. During the past two decades, the scope of indentation testing has been extended down to the nanometre range. This has been achieved principally through the development of instruments capable of continuously measuring load and displacement throughout an indentation [2,4]. In recently developed systems, loads as small as a nanonewton and displacements of about 0.1 nm can be accurately measured. On the other hand, the recognition in the early 1970s that elastic modulus could potentially be measured from an indentation load–displacement curve [7]greatly promoted the development of instrumented indentation testing methodologies. In recent years, the study of mechanical properties of materials on the nanoscale has received much attention, as these properties are size-dependent [2]. These studies have been motivated partly by the development of nanocomposites and the application of nanometer thick films for miniaturization of engineering and electronic components [2], and partly by newly available methods of probing mechanical properties in small volumes [2,5]. The nanoindenter is maturing as an important tool for probing the mechanical properties of small volumes of material. Indentation load–displacement data contain a wealth of information. From the load–displacement data, many mechanical properties such as hardness and elastic modulus can be determined without imaging the indentations [2,5]. The nanoindenter has also been used to estimate the fracture toughness of ultrathin films [9–11], which cannot be measured by conventional indentation tests. With a tangential force sensor, nanoscratch and wear tests can be performed at ramping loads [12]. Atomic force microscopes are ideal for imaging of nanometerscale indents, providing useful information about nanoindentation deformation and cracking. When an indentation system is used in conjunction with an atomic force microscope, in situ imaging can be obtained [8]. With the rapid development of instruments and analytical procedures, more material properties will be measured or estimated using nanoindentation in the near

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future. Diamond is the most frequently used indenter material, because its high hardness and elastic modulus minimize the contribution of the indenter itself to the measured displacement [2]. For probing properties such as hardness andelastic modulus at the smallest possible scales, the Berkovich triangular pyramidal indenter is preferred over the four-sided Vickers or Knoop indenter because a three-sided pyramid is more easily ground to a sharp point [1,2]. Another three-sided pyramidal indenter, the cube corner indenter, can displace more than three times the volume of the Berkovich indenter at the same load, thereby producing much higher stresses and strains in the vicinity of the contact and reducing the cracking threshold. This makes this indenter ideal for the estimation of fracture toughness at relatively small scales [9]. The spherical indenter initiates elastic contact and then causes elastic–plastic contact at higher loads. This indenter, then is well suited for the examination of yielding and work hardening. However, it is very difficult to obtain a precise sphere with a diameter of less than 100 mm made of diamond. This fact limits its application in nanoindentation testing [6]. A recently developed technique, continuous stiffness measurement (CSM) [5], offers a significant improvement in nanoindentation testing. The CSM is accomplished by imposing a small, sinusoidally varying signal on top of a DC signal that drives the motion of the indenter. By analyzing the response of the system by means of a frequency specific amplifier data are obtained. This allows the measurement of contact stiffness at any point along the loading curve and not just at the point of unloading as in the conventional measurement. The CSM technique makes the continuous measurement of mechanical properties of materials possible in one sample experiment without the need for discrete unloading cycles, and with a time constant that is at least three orders of magnitude smaller than the time constant of the more conventional method of determining stiffness from the slope of an unloading curve. The measurements can be made at exceedingly small penetration depths. Thus, this technique is ideal for mechanical property measurements of nanometer thick films. Furthermore, its small time constant makes it especially useful for measuring the properties of polymeric materials. In nonuniformmaterials,such as graded materials and multilayers, the microstructure and mechanical properties change with indentation depth. Continuous measurements of mechanical properties of these materials during indentation are greatly needed. Utilizing the CSM technique, creep measurements on the nanoscale can be performed by monitoring changes in displacement and stress relaxation. Because the CSM is carried out at frequencies greater than 40 Hz, it is less sensitive to thermal drift . Also utilizing the CSM technique, load cycles of a sinusoidal shape at high frequencies allow the performance of fatigue tests at the nanoscale. The fatigue behaviour of thin films and microbeams can be studied by monitoring the change in contact stiffness because the contact stiffness is sensitive to damage formation.

The purpose of this review paper is to present the recent work on the nanoindentation technique and its applications. Emphasis is placed on the CSM analytical methodologies and how they can be used to study hardness, elastic modulus, creep, and fatigue properties for layered materials and nonhomogeneous composites, especially those designed for use in magnetic storage and micro electromechanical systems (MEMS) devices. Discussion on the CSM results in conjunction with nanoindentation scratch and wear data are also presented.

Experimental techniques

Hardness and elastic modulus measurements

The two mechanical properties measured most frequently using indentation techniques are the hardness, H, and the elastic modulus, E. As the indenter is pressed into the sample, both elastic and plastic deformation occurs, which results in the formation of a hardness impression conforming to the shape of the indenter. During indenter withdrawal, only the elastic portion of the displacement is recovered, which facilitates the use of an elastic solution in modeling the contact process [2,5]. Fig. 1 shows a typical load–displacement curve and the deformation pattern of an elastic–plastic sample during and after indentation.

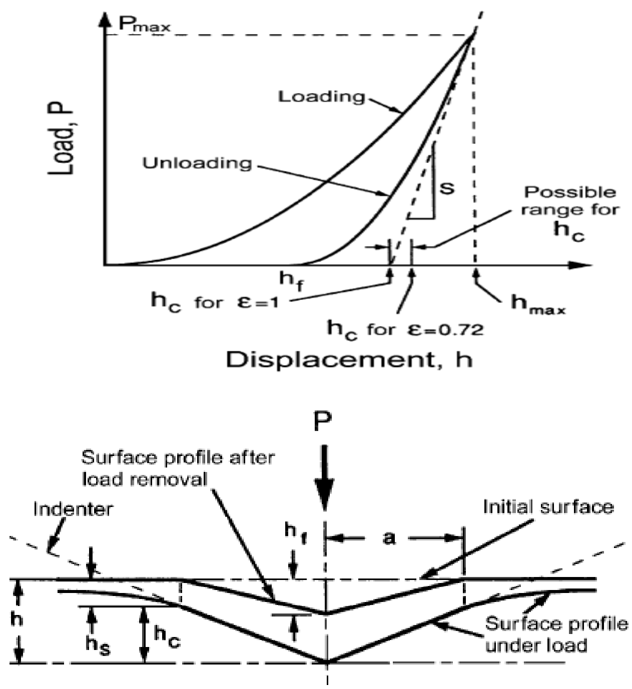

Fig 1. Typical Load displacement curve and the deformation pattern in sample

In Fig.1, h_{max} represents the displacement at the peak load, P_{max}. h_c is the contact depth and is defined as the depth of the indenter in contact with the sample under load. hf is the final displacement after complete unloading. S is the initial unloading contact stiffness. Nanoindentation hardness is defined as the indentation load divided by the projected contact area of the indentation. It is the mean pressure that a material can support under load. From the load–displacement curve, hardness and youngsmodulus can be obtained at the peak load as

$$S = \frac{dp}{dh} = \alpha m(h_{max} - h_f)^{m-1} \qquad (1)$$

variables*m*, *h f*and α are best-fit constants, The contact height, *hc*, is the depth over which the diamond makes contact with the material

$$h_c = h - \varepsilon \frac{P_{max}}{S} \quad (2)$$

Where 'h' is the total penetration depth into the sample and 'ε' is a geometric constant based on the indenter geometry; ε = 0.75 for a pyramidal indenter, Once the contact depth has been determined it is then possible to calculate the contact area, *Ac*. For an ideal Berkovich indenter, the area function is $A_{proj} = 24.56\ h_c^2$ The reduced modulus, *Er*, is determined by

$$E_r = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A_{proj}}} \quad (3)$$

Indentation modulus or specimen modulus Es is determined by

$$\frac{1}{E_r} = \left[\frac{1 - \nu_i^2}{E_i} + \frac{1 - \nu_s^2}{E_s} \right] \quad (4)$$

E_r is the reduced modulus and ν_s is the Poisson ratio of the specimen ν_i and E_i denote the Poisson ratio and young modulus of the indenter. Hardness of the specimen is the resistance to the local deformation, as per Oliver it is expressed as the maximum indentation load 'P_{max}' upon the projected contact Area 'A_{proj}'.

$$H = \frac{P_{max}}{A_{proj}} \quad (5)$$

Creep measurements

For metals and ceramics at elevated temperatures and for polymers under most conditions, time-dependent deformation occurs under application of load. This phenomenon is termed creep. One of the promising applications of the Nanoindentation CSM technique is indentation creep testing. In an indentation creep test, a constant load is applied to the indenter and the change in indentation depth (size) is monitored as a function of time. Compared to conventional tensile creep tests, the CSM Nanoindentation creep experiments are particularly useful as they simulate creep resulting from asperity contact. The CSM technique gives a direct measure of mean stress and contact stiffness, and being insensitive to drift, allows the accurate observation of creep in small indents to be carried out over a long time period [16]. The depth-sensing nanoindenter used in this study can measure indentation depth in situ. For an indenter with known geometry, indentation size can be calculated from the indentation depth. Mean stress is defined as the indentation load divided by the projected contact area of the indentation. The CSM technique has been used to study the creep behaviour of bulk materials [16] and multilayered solids, including magnetic tapes.

Fatigue measurements

Fatigue, also called delayed fracture, implies a finite time to failure under any sustained externally applied cyclic stress [17]. Nanoscale fatigue has been studied rarely in the past because of lack of specialty instruments. The CSM technique provides force cycles of a sinusoidal shape at high frequencies that can be used to perform nanoscale fatigue tests. The fatigue behaviour of thin films and micro beams can be studied by monitoring the change in contact stiffness because the contact stiffness is sensitive to damage formation [17]. To obtain fatigue deformation and damage, large amplitude oscillations are used. The numbers of cycles can be determined from the elapsed time.

Fig. 2 shows the schematic of a fatigue test on athin-film/substrate system using the CSM technique. Force cycles are applied to the film, resulting in a cyclic stress. P(t) is the cyclic load, Pmean is the mean load, Pos is the oscillation load amplitude, and w is the oscillation frequency

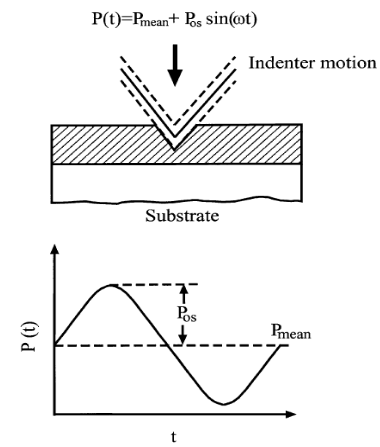

Fig 2: Schematics of a fatigue test made on a thin-film/ substrate system by the Nanoindentation technique.

Typically, a conical tip with a radius of 1 mm and an included angle of 60 is used.

Scratch measurements

Scratch testing is commonly used in materials science and tribology to characterize materials for scratch and wear resistance. In a scratch test, a sharp tip is moved over the surface of a test material at a constant or ramp-up load. Scratch depth at a given load or the load at which material fails catastrophically, is a measure of scratch and wear resistance.

Most of the commercial nanoindenters have a scratch option. A conical diamond indenter is preferred over the Berkovich because a three-sided pyramidal indenter is difficult to align along the scratch direction. In practical scratch tests, a conical diamond indenter with a tip radius of 1 mm and an included angle of 60^0 is drawn over the sample surface. The load is ramped up until substantial damage occurs. The coefficient of friction is monitored during scratching. Typically, a 500μmm-long scratch is made by translating the sample while ramping the load on the conical tip over a range of 0–2.5 mN. The translation speed is typically 5 μmm s-1. A typical scratch experiment consists of three steps: (1) approaching the surface, (2) translating the sample at ramping loads, and (3) final unloading of the tip. Scratch-induced damage is monitored by in situ tangential (friction) force measurements and by light optical microscopy (LOM) imaging of the scratches after tests [2,]. By using a diamond tip to scratch a magnetic tape, the situation is very similar to that of debris and asperities scratching the tape at the head–tape interface. Nanoscratch data on magnetic tapes and their individual layers will be presented and discussed later in conjunction with the CSM data.

Friction and wear measurements

Coefficient of friction and wear need to be minimized for most sliding applications. In order to minimize test duration, accelerated friction and wear tests are conducted [17]. One of the commonly used accelerated test apparatus is a ball-on-flat tribometer under reciprocating motion. For example, a sapphire ball with a 3-mm diameter and a surface finish of about 2 nm RMS is fixed in a stationary holder. The sample is mounted to a flat screw type X–Y stage, which is driven in a reciprocated motion by a DC motor. The load on the stationary component is applied by lowering the beam to which the ball is fixed against the sample. Normal and frictional forces are measured with semiconductor strain gages mounted on a crossed-I beam structure and the data are digitized and collected on a personal computer. Typical test conditions are as follows: stroke length = 2.0 mm, frequency =0.1 Hz, average linear speed = 1.0 mm s^{-1}, normal load = 10 mN, temperature = 22 ± 1 ^{0}C, and relative humidity = 45 ± 5% [2]. Wear damage of a tape can be analyzed by LOM imaging of the wear tracks after tests.

Fracture toughness measurement

When the loading forces are larger than 200 mN, , the discrete displacement bursts during nanoindentation experiments with large applied force are most likely caused by micro-cracking, The micro-cracks become more distinct along with
increased crack lengths at all three corners of the Berkovich indentations and can be easily observed at high maximum loads. However, the severe chipping or damage that usually occurs in very brittle materials Based on the crack length and the maximum loads, the fracture toughness of material can be measured by Berkovichnanoindentation using the Eq (6).

$$K_c = x_v \left(\frac{a}{l}\right)^{\frac{1}{2}} \left(\frac{E}{H}\right)^{\frac{2}{3}} \frac{P}{C^{\frac{2}{3}}} \qquad (6)$$

(where P is the maximum indentation load, c is the crack length, E is the Young’s modulus and H is the hardness.)

Conclusions

In this paper, the nanoindentation technique and its methodologies are reviewed. Applications of the nanoindentation technique to the measurement of contact stiffness, elastic modulus, hardness, creep resistance, and fatigue properties of the materials used in materials research and discussed in conjunction with the data of nanoscratch, and friction and wear tests,we conclude the following:

- The Nanoindentation technique probes the mechanical property changes in situ during indentation, and provides more useful information for layered materials and nonhomogeneous (such as graded) composites.

- The CSM indentation creep tests can detect creep displacement and stress relaxation at small volumes.

- Load cycles used in the nanoindentation CSM can be used to perform nanoscale fatigue tests. Such Tests hold promise for material research applications

References

1. Corbin ND. J Eur Ceram Soc 1989;5:143.
2. Cheng JP, Agrawal D, Zhang YJ, Roy R. J Mater SciLett 2001;20:77.
3. Miller L, Kaplanw WD. J Am Ceram Soc 2008; 91:1693.
4. McCauley JW, Patel P, Chen MW, Gilde G, Strassburger E, Paliwal B, et al. J Eur Ceram Soc 2009;9:223.
5. Paliwal B, Ramesh KT, McCauley JW, Chen MW. J Am Ceram Soc 2008; 91:3619.
6. Vaughn BAM, Proud WG, Field JE. Cavendish laboratory report. SP/1092; 2001.
7. Paliwal B, Ramesh KT, McCauley JW. J Am Ceram Soc 2006; 89:2128.
8. Sekine T, Li X, Kobayashi T, Yamashita Y, Patel P, McCauley JW. J ApplPhys 2003; 94:4803.
9. McCauley JW, Wilantewicz TE. In: Proceedings of the 26th army science conference, Orlando, Florida, ADM002187; December 1–4,
10. 2008.
11. Chen MW, McCauley JW, Dandekar DP, Bourne NK. Nat Mater 2006;5:614.
12. Lundberg P, Lundberg B. Int J Impact Eng 2005; 31:781–92.
13. Bourne NK. Shock compression of condensed matter. In: AIP
14. conference proceedings, vol. 1195; 2009. p. 993.
15. Scholz T, Schneider GA, Munoz-Saldana J, Swain MV. ApplPhysLett 2004; 84:3055.
16. Page TF, Oliver WC, McHargue CJ. J Mater Res 1992; 7:450.
17. Page TF, Riester L, Hainsworth SV. Mater Res SocSymp Proc1998; 522:113.
18. Jang J, Pharr GM. Acta Mater 2008; 56:4458.

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