A review of nanoindentation continuous stiffness measurement technique and its applications

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Abstract

Nanoindentation is now commonly used for the study of mechanical properties of materials on the nanoscale. One of the significant improvements in nanoindentation testing is the continuous stiffness measurement (CSM) technique. It offers a direct measure of dynamic 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. Nanoscale damage caused by fatigue is of critical importance to the reliability of ultrathin protective overcoats and micro/nanostructures. The cyclic loading used in the CSM makes the technique useful for the evaluation of nanofatigue. Methodologies of the CSM technique used for the characterization of layered materials and nonhomogeneous composites are reviewed and discussed. Applications of the CSM technique to the measurement of contact stiffness, elastic modulus, hardness, creep resistance, and fatigue properties of the materials used in magnetic storage devices are presented. The nanoindentation CSM technique, in conjunction with nanoscratch and friction and wear tests, can be satisfactorily used for the materials characterization of magnetic storage and microelectromechanical systems (MEMS) devices and should find more application. © 2002 Elsevier Science Inc. All rights reserved.

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1. 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 micro-indentation 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 nanometer range. This has been achieved principally through the development of
Instruments capable of continuously measuring load and displacement throughout an indentation [2,4–6]. 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 [2,8,9]. 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 [2,10], and partly by newly available methods of probing mechanical properties in small volumes [2,5,6]. 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 [11–13], which cannot be measured by conventional indentation tests. With a tangential force sensor, nanoscratch and wear tests can be performed at ramping loads [14–20]. Atomic force microscopes are ideal for imaging of nanometer-scale 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 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 and elastic 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 [1,2,6]. 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 [11,12]. 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 µm made of diamond. This fact limits its application in nanoindentation testing [6].

A recently developed technique, continuous stiffness measurement (CSM) [5,21,22], 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 nonuniform materials, 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 [22]. 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 behavior 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 CSM 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 microelectromechanical systems (MEMS) devices. Discussion on the CSM results in conjunction with nanoindentation scratch and wear data are also presented.

2. Experimental techniques

2.1. 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 [2,5,6]. Fig. 1 shows a typical load–displacement curve and the deformation pattern of an elastic–plastic sample during and after indentation. In Fig. 1, \( h_{\text{max}} \) represents the displacement at the peak load, \( P_{\text{max}} \), \( h_c \) is the contact depth and is defined as the depth of the indenter in contact with the sample under load. \( h_f \) 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 can be obtained at the peak load as

\[
H = \frac{P_{\text{max}}}{A} \quad (1)
\]

where \( A \) is the projected contact area. Measurement of the projected contact area from a load–displacement curve requires the contact depth, \( h_c \), which will be discussed later.

The elastic modulus of the indented sample can be inferred from the initial unloading contact stiffness, \( S = dP/dh \), i.e., the slope of the initial portion of the unloading curve. Based on relationships developed by Sneddon [23] for the indentation of an elastic half space by any punch that can be described as a solid of revolution of a smooth function, a geometry-independent relation involving contact stiffness, contact area, and elastic modulus can be derived as follows

\[
S = \frac{4}{\pi} \sqrt{\frac{A}{E_r}} \quad (2)
\]

where \( \beta \) is a constant that depends on the geometry of the indenter (\( \beta = 1.034 \) for a Berkovich indenter) [5] and \( E_r \) is the reduced elastic modulus, which accounts for the fact that elastic deformation occurs in both the sample and the indenter. \( E_r \) is given by

\[
E_r = \frac{1 - \nu^2}{E} + \frac{1 - \nu_i^2}{E_i} \quad (3)
\]

where \( E \) and \( \nu \) are the elastic modulus and Poisson’s ratio for the sample, respectively, and \( E_i \) and \( \nu_i \) are the same quantities for the indenter. For diamond, \( E_i = 1141 \) GPa and \( \nu_i = 0.07 \) [1,5].

To calculate elastic modulus, \( E_r \), from Eqs. (2) and (3), the contact stiffness and the projected contact area need to be determined from the load–displacement curve. Oliver and Pharr [5] found that the
unloading curve is usually not linear as suggested by Doerner and Nix [24], but is better described by a power law:

\[ P = B(h - h_e)^m \]  

(4)

where \( B \) and \( m \) are empirically determined fitting parameters. The unloading stiffness, \( S \), is then established by differentiating Eq. (4) at the maximum depth of penetration, \( h = h_{\text{max}} \) (i.e., Eq. (5))

\[ S = \left( \frac{dP}{dh} \right)_{h = h_{\text{max}}} = Bm(h_{\text{max}} - h_e)^{m-1}. \]  

(5)

For an indenter with a known geometry, the projected contact area is a function of the contact depth. The area function for a perfect Berkovich indenter is given by

\[ A_c = 24.56h_c^2. \]  

(6)

Indenters used in practical nanoindentation testing are not ideally sharp. Therefore, tip geometry calibration or area function calibration is needed. A series of indentations is made on fused quartz at depths of interest. A plot of \( A \) versus \( h_c \) can be curve fit according to the following functional form (Eq. (7)):

\[ A_c = 24.56h_c^2 + C_1h_c^1 + C_2h_c^{1/2} + C_3h_c^{1/4} + \ldots + C_8h_c^{1/128} \]  

(7)

where \( C_1 \) through \( C_8 \) are constants. The lead term describes a perfect Berkovich indenter, the others describe deviations from the Berkovich geometry due to blunting of the tip [5].

The contact depth can be estimated from the load–displacement data using Eq. (8):

\[ h_c = h_{\text{max}} - \varepsilon \frac{P_{\text{max}}}{S} \]  

(8)

where \( \varepsilon \) is a constant that depends on the indenter geometry (\( \varepsilon = 0.75 \) for a Berkovich indenter) [5].

The above analysis is based on an elastic solution and works well for hard ceramics when sink-in predominates (i.e., the indented material around the indenter is moved below the original surface plane). However, the Oliver–Pharr method can underestimate the true contact area by as much as 50% for soft materials and high loads. Metals such as Al that exhibit a low ratio of the yield stress to elastic modulus and little or no capacity for work hardening generate a pile-up condition (movement of the indented material around the indenter above the original surface plane) under elastic/plastic conditions [6]. This, in turn, leads to overestimations of the hardness and elastic modulus. In general, pile-up error increases with an increasing indentation depth. Although some correction procedures have been proposed, the real contact area measurement requires imaging of indentation impressions.

In practical indentation tests, a Berkovich tip with a radius of about 100 nm is used. Multiple loading and unloading steps are performed to examine the reversibility of the deformation, ensuring that the unloading data used for analysis purposes are mostly elastic. A typical indentation experiment consists of eight steps [2]: approaching the surface, loading to peak load, unloading 90% of peak load, reloading to peak load, holding the indenter at peak load, unloading 90% of peak load, holding the indenter after 90% unloading, and finally, unloading completely. The first hold step is included to remove the influence of creep on the unloading characteristics. The second hold step is
included to incorporate the corrections due to thermal drift. Fig. 2 shows a representative load–displacement curve of an indentation made at 15-mN peak indentation load and the hardness and elastic modulus as a function of indentation depth at various peak loads for Si(100) [20]. The Si(100) exhibits hysteresis in displacement during cyclic loading and unloading, as shown in Fig. 2a. Hysteresis observed in the unloading curve at low loads is due to a pressure-induced phase transformation from its normal diamond cubic form to a β-tin phase. This phase transformation results in a decrease in volume of about 22%, which affects the indentation displacement during loading. The hardness and elastic modulus for Si(100) remain constant at increasing indentation depths, as shown in Fig. 2b. The slight increase in hardness and decrease in elastic modulus at a smaller indentation depth are due to the indenter tip roundness and the surface oxidization of Si(100). To obtain hardness and elastic modulus as a function of indentation depth, several indentation tests at different peak indentation loads have to be performed.

### 2.2. Continuous stiffness measurements

The contact stiffness can be alternatively measured during the loading portion of an indentation test using the CSM technique. The CSM is accomplished by imposing a harmonic force, which is added to the nominally increasing load, \( P \), on the indenter, as shown in Fig. 3. The displacement response of the indenter at the excitation frequency and the phase angle between the two are measured continuously as a function of depth. Solving for the in-phase and out-of-phase portions of the response results in an explicit determination of the contact stiffness, \( S \), as a continuous function of depth [5,21,22].

To calculate the contact stiffness, the dynamic response of the indentation system has to be determined. The relevant components are the mass, \( m \), of the indenter, the spring constant, \( K_s \), of the leaf springs that support the indenter, the stiffness of the indenter frame \( K_f = 1/C_f \), where \( C_f \) is the compliance of the load frame and the damping coefficient, \( C \), due to the air in the gaps of the capacitor plate displacement sensing system. These combined with the contact stiffness, \( S \), produce the overall response as shown in Fig. 4. If the imposed driving force is \( P = P_{os} \exp(i\omega t) \) and the displacement response of the indenter is \( h(\omega) = h_0 \exp(i\omega t + \phi) \), the contact stiffness, \( S \), can be calculated from the displacement signal,

\[
\frac{P_{os}}{h(\omega)} = \sqrt{\left((S^{-1} + K_f^{-1})^{-1} + K_s - m\omega^2\right)^2 + \omega^2 C^2}
\]

or from the phase difference between the force and displacement signals, the phase angle, \( \phi \), is

\[
\tan(\phi) = \frac{\omega C}{(S^{-1} + K_f^{-1})^{-1} + K_s - m\omega^2}
\]

where \( P_{os} \) is the magnitude of the force oscillation, \( h(\omega) \) is the magnitude of the resulting displacement oscillation, \( \omega \) is the frequency of the oscillation, and \( \phi \) is the phase angle between the force and displacement.

![Fig. 3. Schematic of the CSM loading cycle.](image3)

![Fig. 4. Schematic of the dynamic indentation model [5].](image4)
signals. Eqs. (9) and (10) can be solved for the contact stiffness, $S$, and the damping due to the air in the gaps between the capacitor plates $\omega C$ (the damping of the sample itself is regarded to be negligible). The contact stiffness, $S$, and the damping, $\omega C$, can be given by Eqs. (11) and (12), respectively.

$$S = \left[ \frac{1}{h(\omega)} \cos\phi - (K_s - m\omega^2)^{-1} \right]^{-1}$$  \hspace{1cm} (11)

$$\omega C = \frac{P_{os}}{h(\omega)} \sin\phi$$  \hspace{1cm} (12)

Let us discuss the relationship between contact stiffness and indentation contact depth for uniform and nonuniform materials. For a perfect Berkovich indenter, $A$ is a function of contact depth $h_c$, as shown in Eq. (6). Substituting Eq. (6) into Eq. (2), one obtains

$$S = 2\beta \sqrt{\frac{24.56}{\pi} E_r h_c}$$  \hspace{1cm} (13)

From Eq. (3), we know that for a uniform material with a constant elastic modulus value of $E_s$, $E_r$ is a constant, and according to Eq. (13), $S$ is linearly proportional to $h_c$. For a nonuniform material, $E_r$ changes with indentation depth. In this case, the linear relationship between $S$ and $h_c$ does not exist [25]. The schematics of indentations made on uniform and graded materials are shown in Fig. 5. Therefore, the CSM technique can be used to study the mechanical properties of graded materials and multilayered structures by monitoring the change in contact stiffness, elastic modulus, and hardness as a function of indentation contact depth. For most studies, a Berkovich tip and a frequency of 45 Hz are used. The cyclic loading amplitude and the peak indentation load are dependent upon the material to be studied; for example, for magnetic tapes these values are 1.2 and 600 $\mu$N, respectively.

### 2.3. 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 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 indentation 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 [22]. 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 behavior of bulk materials [22] and multilayered solids, including magnetic tapes [26–29]. Fig. 6 shows representative data of the CSM indentation creep test on PTFE. To measure mean stress and contact stiffness the indenter is oscillated at a peak–peak load amplitude of 1.2 $\mu$N and a frequency of 45 Hz during the holding segment. Mean stress is the hardness measured continuously during the CSM during the hold segment. With increasing time, the indentation displacement increases while the mean stress decreases. This indicates that time-dependent deformation occurred for the PTFE.
Fatigue, also called delayed fracture, implies a finite time to failure under any sustained externally applied cyclic stress [30]. 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 behavior of thin films and microbeams can be studied by monitoring the change in contact stiffness because the contact stiffness is sensitive to damage formation [31]. To obtain fatigue deformation and damage, large amplitude oscillations are used. The numbers of cycles can be determined from the elapsed time. Fig. 7 shows the schematic of a fatigue test on a thin-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, \( P_{\text{mean}} \) is the mean load, \( P_{\text{os}} \) is the oscillation load amplitude, and \( \omega \) is the oscillation frequency. Typically, a conical tip with a radius of 1 \( \mu \text{m} \) and an included angle of 60° is used.

2.5. 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

\[
P(t) = P_{\text{mean}} + P_{\text{os}} \sin(\omega t)
\]

Fig. 7. Schematics of a fatigue test made on a thin-film/substrate system by the CSM technique.
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 μm and an included angle of 60° 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 500-μm-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 μm 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 [2,14,15]. 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.

2.6. 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 [30]. 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 = 18 Hz.

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**Fig. 8.** Contact stiffness, elastic modulus, and hardness as a function of contact depth for fused silica, PTFE, and SBR [27].
0.1 Hz, average linear speed = 1.0 mm s\(^{-1}\), normal load = 10 mN, temperature = 22 ± 1 °C, and relative humidity = 45 ± 5% \([2,14,15]\). Wear damage of a tape can be analyzed by LOM imaging of the wear tracks after tests.

3. Applications of the CSM technique

3.1. Uniform and multilayered hard structures

According to Eq. (13), the contact stiffness, \(S\), of a uniform material is linearly proportional to the contact depth, \(h_c\). For a nonuniform material, however, the linear relationship between \(S\) and \(h_c\) does not exist \([25]\). Li and Bhushan \([27]\) conducted CSM tests on fused silica, PTFE, and styrene butadiene rubber (SBR). Fused silica and PTFE are rigid plastic, uniform materials with constant elastic modulus values. SBR is a uniform soft viscoelastic material. Fig. 8 shows CSM results for fused silica, PTFE, and SBR. For fused silica and PTFE, the contact stiffness increases linearly with contact depth, and the elastic modulus and hardness do not change. For the SBR, the contact stiffness increases linearly with contact depth whereas the elastic modulus decreases with contact depth. This suggests that the contact depth calculated from the theory based on rigid plastic materials is not applicable to viscoelastic and viscoelastic–plastic materials, because the time-dependent effect is neglected. Therefore, a new methodology for calculating the contact depth of viscoelastic and viscoelastic–plastic materials that considers time-dependent effects must be developed in the future.

Fig. 9 shows the CSM results of a 1-μm-thick RF-sputtered (SP) Al on Si (Al/Si), a 100-nm-thick RF-sputtered (SP) amorphous carbon on Si (soft a-C/Si), and a 100-nm filtered cathodic arc (FCA) deposited amorphous carbon on Si (hard a-C/Si). For a soft film on a hard substrate such as Al/Si and soft a-C/Si, the contact stiffness increases with an increasing slope while the elastic modulus and hardness increase with contact depth. In the case of the hard a-C/Si, contact stiffness values increases with a decreasing slope whereas the elastic modulus and hardness decrease with contact depth. When the contact depth exceeds 10–20% of the film thickness, a substrate effect on the contact.

Fig. 9. Contact stiffness, elastic modulus, and hardness as a function of contact depth for Al/Si, soft a-C/Si, and hard FCA a-C/Si \([27]\).
stiffness, elastic modulus, and hardness can be observed. For these depths, the elastic/plastic zone expands up to the substrate.

3.2. Multilayered magnetic media

Magnetic media are multilayered structures. Fig. 10a shows the cross-sectional schematics of thin-film rigid disk, metal particle (MP), and metal-evaporated (ME) tapes [32]. The layers used in magnetic media are a few nanometers thick. A thin-film disk is made using an ultrasmooth and flat disk substrate (made of Al–Mg alloy, glass, or glass ceramic) with metallic (Co–X alloy) magnetic films and 3–5-nm-thick diamond-like carbon (DLC) overcoat and 1–2-nm-thick bonded perfluoropolyether lubricant. Magnetic tapes consist of a polymer strip coated with a thin layer of magnetic coating. The

![Cross-sectional schematics of thin-film disk, ME, and MP tapes](image-url)

Fig. 10. (a) Cross-sectional schematics of thin-film disk, ME, and MP tapes and (b) schematic of nonhomogeneous distribution of particles in the MP magnetic coating.
coating consists of either magnetic particles together with nonmagnetic particles dispersed in a polymer binder, as in MP tapes, or it could be a continuous metallic magnetic film (Co–Ni–O or Co–O) vacuum deposited in the presence of oxygen, as in ME tapes. A DLC coating of 8–10-nm thickness is used to improve the tribological performance of ME tapes. The mechanical properties of these magnetic media, which vary as a function of depth because of their multilayer construction, affect their performance. In the case of MP tapes, the magnetic coating can have a nonhomogeneous distribution of particles (Fig. 10b). Recently, Li and Bhushan [26,27,29] and Li et al. [28] successfully applied the CSM technique to measure the mechanical properties of multilayered magnetic media.

### 3.2.1. Thin-film rigid disk

For a magnetic rigid disk with a multilayered structure, the contact stiffness, elastic modulus, and hardness change with indentation depth as illustrated in Fig. 11. From the variations in contact stiffness, elastic modulus, and hardness, we can easily distinguish one layer from another. The elastic modulus values of different layers are comparable, resulting in low interfacial stresses. The underlayer between the magnetic layer and Ni–P layer has a lower

Fig. 11. Contact stiffness, elastic modulus, and hardness as a function of contact depth for a magnetic rigid disk with a multilayered structure [27].

### Commercial MP tape

Fig. 13. Contact stiffness as a function of contact depth for a commercial MP tape measured using both sharp and blunt tips.
elastic modulus, which can be easily detected using the CSM technique. The hardness decreases with contact depth. It has been reported that the hardness values for the magnetic layer and the Ni–P layer are about 10 and 8 GPa, respectively [32]. The hardness data obtained from the CSM technique for the magnetic layer and the Ni–P layer are 9 and 8 GPa, in good agreement with the results reported before for conventional nanoindentation.

Frequent, high-magnitude impacts that occur at the head–disk interface of the modern contact start/stop and load/unload hard disk drives [32] will cause fatal damage. To evaluate impact resistance, the fatigue properties of the coatings must be measured. Although tribological behavior of the head–disk interface has been intensively studied, its fatigue properties have received very little examination. Recently, Li and Bhushan [31] studied the fatigue properties of ultrathin amorphous carbon coatings using the CSM technique. Fig. 12 shows the contact stiffness as a function of the number of cycles for a 20-nm-thick amorphous carbon coating on a silicon substrate cyclically deformed by an

![](image)

Table 1
Calculated values of mean contact pressure and normal approach (interference) using Hertz analysis for an MP tape

<table>
<thead>
<tr>
<th>Tip geometry</th>
<th>Mean contact pressure(^a) (GPa)</th>
<th>Normal approach(^b) (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100-nm radius rigid tip at 10-(\mu)N load</td>
<td>1.91</td>
<td>17</td>
</tr>
<tr>
<td>20-(\mu)m radius rigid tip at 100-(\mu)N load</td>
<td>0.12</td>
<td>14</td>
</tr>
</tbody>
</table>

\(^a\) Mean contact pressure=\((16/WPE^*^2/9\pi^3R^2)^{1/3}\), where \(R\) is the radius of rigid sphere, \(E^* = 1/[(1-\nu^2)/E]\), \(E\) is the elastic modulus, \(\nu\) is the Poisson’s ratio of the tape, and \(W\) is the normal load. The elastic modulus value of the tape of 11 GPa is taken from Fig. 15 and \(\nu = 0.3\) is assumed.

\(^b\) Normal approach=\((9W^2/16RE^*^2)^{1/3}\).

Fig. 14. Contact stiffness, elastic modulus, and hardness as a function of contact depth for a PET substrate and a commercial MP tape [27].
oscillation load amplitude of 8 μN under a mean load of 10 μN at a frequency of 45 Hz. The abrupt decrease in contact stiffness at $0.8 \times 10^4$ cycles indicates fatigue damage has occurred. The nano-fatigue data are of critical importance to the design of magnetic storage devices, and should receive more attention.

3.2.2. Multilayered viscoelastic structures

3.2.2.1. Effect of tip radius. Indenter-tip radius affects the CSM data. The contact stiffness measured using the blunt tip (a conical tip with a 20-μm tip radius) with a peak indentation load of 2 mN and a peak-to-peak load amplitude of 1.5 μN shows the same trend as that measured using the sharp tip (a Berkovich tip with a 100-nm tip radius) with a peak indentation load of 600 μN and a peak-to-peak load amplitude of 1.2 μN, as shown in Fig. 13. The blunt tip, however, gives higher stiffness than the sharp tip. This is probably because the blunt tip encounters more particles than the sharp tip during indentation. The thickness of magnetic coating in an MP tape is about 150 nm, so to measure its properties with certainty the indentation depth should be on the order of 15 nm. Table 1 shows the calculated values of mean contact pressure and normal approach (indentation depth) using Hertz analysis for the tape at loads corresponding to normal approach of about 15 nm. The blunt tip shows a lower contact pressure than the sharp tip. Because the size of debris at the head–tape interface and typical contact spots ranges from a few nanometers to a few microns, a range of tip radii from a sharp tip to a blunt tip may be used. However, the

![Fig. 15. Contact stiffness as a function of contact depth. Elastic modulus, and hardness data at a contact depth of 15 nm for Tapes A, B, and C [27].](image)
ranking of various tapes may not be affected by the tip radius.

3.2.2.2. MP tapes with multilayers having different stiffness ratio. Polyethylene terephthalate (PET) substrate used in the construction of magnetic tapes includes hard particles that may not be uniformly distributed; this leads to graded mechanical properties. MP tapes are of multilayered structures. Data for a PET substrate and a commercial MP tape are shown in Fig. 14. For both specimens, contact stiffness increases with a decreasing slope as contact depth increases. Their elastic modulus and hardness values decrease with contact depth. The elastic modulus values obtained at a shallow indentation depth are the same as those measured from tensile tests for the PET substrate and MP tape [10]. Several factors are responsible for this decrease, and one of these is a creep effect (in the case of polymers). To study this effect, experiments were performed over a range of loading rates, as illustrated in Fig. 14. An increase in loading rate has little effect on the CSM results. This is probably because during indentation, the creep rate is so fast that even a high loading rate cannot suppress the creep effect. Other factors for the decrease in elastic modulus and hardness with depth include a possible graded particle distribution in the PET substrate and MP tape as well as surface hardening resulting from stretching of the substrate during manufacturing [10,33]. For the MP tape, substrate effects become important when the contact depth exceeds 10% of the thickness of the magnetic layer.

The mechanical properties of three recently developed MP tapes, designated as A, B, and C, were measured using the CSM technique [27]. The magnetic layers/underlayers of Tapes A, B, and C were manufactured to be hard/hard, hard/soft, and soft/hard, respectively, by using different binders and cross-linker materials and different formulations. Fig. 15 shows the contact stiffness as a function of time for Tapes A, B, and C [27].
function of contact depth and elastic modulus and hardness at a contact depth of 15 nm for Tapes A, B, and C. Tape A exhibits the highest contact stiffness, elastic modulus, and hardness, followed by Tapes B and C. The higher contact stiffness, elastic modulus, and hardness are attributed to harder binders and stronger bonding between particles and binders in the magnetic layer and underlayer. We can see that the top layer or magnetic layer determines the surface properties.

Fig. 16 shows the CSM indentation creep results for Tapes A, B, and C. Tape A exhibits a slower increase in indentation displacement than Tapes B and C. The latter two show a comparable increase in indentation displacement. The mean stresses of all tapes, on the other hand, decrease with time, indicating that stress relaxation occurred during the hold segment. Tape A exhibits a slower decrease in mean stress than Tapes B and C. The latter two show a comparable decrease in mean stress. The contact stiffness of all samples remains almost constant during the 600-s hold segment. Both Tapes A and B have hard magnetic layers. However, Tape A has a hard underlayer whereas Tape B has a soft underlayer. Tape B shows a lower creep resistance than Tape A, because of the soft underlayer of Tape B. In contrast to Tape B, Tape C has a soft magnetic layer and a hard underlayer. Tape C, however, exhibits a creep resistance comparable to Tape B. This suggests that both magnetic layer and underlayer contribute to the creep resistance for magnetic tapes.

Fig. 17 shows scratch results for Tapes A, B, and C. Tape C shows a higher static friction force (commonly referred as stiction) at the beginning of the scratch and a high coefficient of friction value near the end of the test whereas Tapes A and B show a gradual increase in the coefficient of friction. All samples were damaged by ploughing. Debris was found at the sides of the scratch tracks for both Tapes B and C. The debris is believed to result from the particles pulled out during scratching and its propensity correlates with the coefficient of friction [34]. In addition to the strength of the polymeric structure or binder, the adhesion among the binder, polymeric material, and particles plays an important role in determining scratch resistance. The defects at the polymeric binder and particle interfaces may also greatly affect their scratch resistance. The soft underlayer in Tape B causes the hard magnetic layer to delaminate and break down relatively easily. For Tape C (a soft magnetic layer and a hard underlayer), the scratches tip can more easily penetrate the magnetic layer. This results in a large friction force at the beginning of the scratch and an easy pullout of material with increasing normal load.

Friction and wear tests show the same results as the scratch tests. As shown in Fig. 18, Tape C shows a rapid increase in coefficient of friction from 0.3 to 0.7 whereas Tapes A and B show a slow increase in coefficient of friction. After a sliding distance of about 1 m, the coefficient of friction for Tape C remains constant at 0.7. At the end of sliding, the coefficients of friction for Tapes A and B reach 0.6.
The optical images show that Tape C has the largest wear track; it shows many scratches and lots of debris, followed by Tape B and A. Tape A has the least severe wear. It is believed that the scratches in the wear track result from particle pullout during sliding. These pulled-out particles would accelerate the damage of the tape.

During sliding, the maximum shear stress is located beneath the surface for a coefficient of friction less than 0.3 [30]. The propensity for crack formation depends on the adhesion strength between particles and binder. When the maximum shear stress exceeds the adhesion strength between the particles and binder, cracks occur. These cracks extend during subsequent sliding, resulting in the formation of debris with pulled out particles. This material, in turn, sticks to the sapphire ball and scratches the tape. At this point, the tape fails catastrophically, as indicated by a sudden rise in the coefficient of friction.

Fig. 18. Coefficient of friction as a function of sliding distance for Tapes A, B, and C against a single-crystal sapphire ball and the optical images of wear tracks an debris formed on all samples. The end of the wear track is on the left-hand side of the image [27].

Fig. 19. Bar charts summarizing the elastic modulus, hardness, creep, scratch, and wear results of Tapes A, B, and C. (a) Elastic modulus and (b) hardness data obtained from Fig. 15 at a contact depth of 15 nm, (c) indentation displacements obtained at the end of the 600 s hold in Fig. 16, (d) damage bar chart plotted based on the optical image examination of the scratch and wear tracks as well as debris after tests (0 = no apparent damage, 1 = small damage, 2 = medium damage, 3 = large damage) [27].
friction. Crack formation is reduced in tapes in which particles and binders adhere well to each other. In the case of a soft underlayer, the shear stress in the underlayer is high enough to cause cracks and delamination. A high interfacial stress at the magnetic layer and underlayer adds to the shear stress at the sliding interface and results in delamination of the magnetic layer. Therefore, the selection of underlayer is also very important for MP tape design.

Fig. 19 summarizes the elastic modulus, hardness, creep, scratch, and wear results for Tapes A, B, and C. The elastic modulus and hardness data in Fig. 19(a) and (b) were obtained from Fig. 15 at a contact depth of 15 nm. The indentation displacements at the end of the 600-s hold (Fig. 16) are plotted in Fig. 19(c). The damage bar chart in Fig. 19(d) is based on optical image examination of the scratch and wear tracks as well as debris after tests (0 = no apparent damage, 1 = small damage, 2 = medium damage, 3 = large damage). The results show a good correlation between mechanical properties and scratch/wear damage. Less scratch damage was observed in the tapes that had higher values for elastic modulus and hardness. In this study, Tape A exhibited the most suitable mechanical and tribological properties. The binder and formulation in the magnetic layer and underlayer of Tape A seem to be the most promising for a superior tape.

### 3.2.2.3. Individual layers in MP tapes

Mechanical properties of the individual layers in MP tapes have been studied by using the CSM technique [28]. The double-coated MP tapes are PET or aromatic polyamide (Aramid) substrates coated with a magnetic layer and a backcoat. A nonmagnetic underlayer was used between the magnetic layer and substrate in order to produce a tape with a thin magnetic coating. The three double-coated tapes, designated as A, B, and C have the same size and concentration of magnetic particles and head cleaning agents but have different binders and formulations. The major difference between Tapes A and C are the dispersants used in the coating slurry. The main binders are either polyvinyl chloride (PVC) or polyester–polyurethane, as shown in Table 2. The nonmagnetic underlayer consists of ultrafine nonmagnetic particles. For studying the properties of each layer, individual layers (magnetic layer, underlayer, and backcoat) were directly coated onto a PET substrate. The backcoat consists of ultrafine inorganic particles (generally carbon black and TiO₂) [10,32]. Contact stiffness, as a function of contact depth and elastic modulus and hardness at a contact depth of 15 nm for various double-coated tapes and their individual layers and substrates were calculated, and results are shown in Fig. 20. The contact stiffness for the two substrates, the individual layers, and the double-coated tapes increase with a decreasing slope. Data scatter also increases with increasing contact depth. The double-coated tapes, underlayer, and backcoat have almost the same stiffness, which is higher than that of the individual magnetic layer and substrate individually. Both substrates exhibit almost the same stiffness values at a shallow contact depth whereas the Aramid exhibits a higher stiffness at a deeper contact depth. At a shallow contact depth, the individual magnetic layer has almost the same stiffness value as the other individual layers and double-coated tapes. At a deeper contact depth, the magnetic layer exhibits a lower stiffness value. This is probably because the individual magnetic layer is thicker than the magnetic layer of the double-coated tapes (Table 2) and fewer particles may be packed at the bottom of the individual magnetic layer. The individual magnetic layer

<table>
<thead>
<tr>
<th>Sample</th>
<th>Substrate/ thickness (µm)</th>
<th>Main binder</th>
<th>Formulation</th>
<th>Magnetic layer thickness (µm)</th>
<th>Underlayer thickness (µm)</th>
<th>Backcoat thickness (µm)</th>
<th>Total thickness (µm)</th>
<th>Roughness, Rₐ (nm)</th>
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<td>0.6</td>
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<td>PVC</td>
<td>A</td>
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<td>1.5</td>
<td>0.4</td>
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<tr>
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<td>PVCa</td>
<td>A</td>
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<td>1.5</td>
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<td>6.5</td>
<td>5.6</td>
</tr>
<tr>
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<td>B</td>
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<td>0.4</td>
<td>8.0</td>
<td>5.4</td>
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<td>Tape B</td>
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<td>polyester–polyurethane</td>
<td>B</td>
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<td>1.5</td>
<td>0.4</td>
<td>8.0</td>
<td>5.4</td>
</tr>
<tr>
<td>Tape C</td>
<td>PET/6</td>
<td>PVCa</td>
<td>C</td>
<td>0.15</td>
<td>1.5</td>
<td>0.4</td>
<td>8.0</td>
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<tr>
<th>Sample</th>
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<th>Main binder</th>
<th>Formulation</th>
<th>Magnetic layer thickness (µm)</th>
<th>Underlayer thickness (µm)</th>
<th>Backcoat thickness (µm)</th>
<th>Total thickness (µm)</th>
<th>Roughness, Rₐ (nm)</th>
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<tr>
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<td>polyester–polyurethane</td>
<td>B</td>
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<td>1.5</td>
<td>0.4</td>
<td>8.0</td>
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<tr>
<td>Tape C</td>
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<td>C</td>
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<td>0.4</td>
<td>8.0</td>
<td>5.4</td>
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a Different dispersants were used in the coating slurry.
was directly coated onto the PET substrate, i.e., without an underlayer, resulting in poor particle distribution and weak adhesion between the magnetic layer and substrate. Of the substrates, Aramid exhibits a higher elastic modulus and hardness than PET. Of the individual layers, the individual magnetic layer exhibits the highest elastic modulus and hardness, followed by the underlayer and backcoat. Of the double-coated tapes, Tape C exhibits the highest elastic modulus and hardness, followed by Tapes B and A. The differences in contact stiffness, elastic modulus, and hardness among Tapes A, B, and C result from the different binders and formulations used in their magnetic layers.

Fig. 21 shows the CSM indentation creep results for various double-coated tapes and their individual layers and substrates. The individual magnetic layer exhibits the slowest increase in indentation displacement, followed by the Aramid substrate, the double-coated tapes, the PET substrate, the individual underlayer, and the backcoat. Of the double-coated tapes, Tape C exhibits the slowest increase in indentation displacement, followed by Tapes B and A. In contrast with indentation displacement, the mean stresses of the double-coated tapes, the individual layers, and the substrates decrease with time, indicating that stress relaxation occurred during the hold segment. The double-coated tapes exhibit a slower decrease in mean stress than the other samples, which show a comparable decrease in mean stress. For the double-coated tapes, Tape C exhibits the slowest stress drop, followed by Tapes B and A.

Fig. 22 shows scratch results for various double-coated tapes and their individual layers and substrates. The Aramid substrate exhibits the lowest coefficient of friction, followed by the PET substrate, the underlayer, the double-coated tapes, the backcoat, and the individual magnetic layer. Of the substrates, the PET exhibits a large stiction while the Aramid exhibits a small stiction spike. After the stiction

Fig. 20. Contact stiffness as a function of contact depth and surface elastic modulus and hardness data at a contact depth of 15 nm for various double-coated tapes and their individual layers and substrates [28].
spikes, both substrates show a continuous increase in coefficient of friction with increasing normal load up to 0.9 mN. At 0.9 mN, both substrates exhibit a sudden increase in coefficient of friction, indicating onset of damage. As shown in the optical images, the scratch track on the PET is wider and deeper than that on the Aramid. No debris was found at the sides and ends of the scratches. In the case of the individual layers, the underlayer exhibits a slower increase in coefficient of friction than the magnetic layer and backcoat. The magnetic layer and backcoat show a sudden increase at the beginning of the scratch. This indicates that the magnetic layer and backcoat have a high stiction force. After the stiction spikes, the magnetic layer and backcoat exhibit large variations in the coefficient of friction. The corresponding optical images show that the underlayer has a smooth scratch track whereas the magnetic layer and backcoat have rough scratch tracks. Debris was found at the sides and ends of scratches for the magnetic layer and backcoat. In the scratch track of the backcoat, some bright areas can be seen. This is a sign of delamination of the backcoat from the substrate. The particle pullout and delamination events are associated with large variations in coefficient of friction. Tape A shows a large stiction spike at the beginning of the scratch whereas Tapes B and C exhibit a gradual increase in coefficient of friction. This indicates that Tape A exhibits high stiction force. The coefficient of friction of Tape C is lower than that of Tape B. The optical images show that Tape A has more debris at the sides and ends of the scratch than Tape B. No debris was found near the scratch of Tape C. These data indicate that more particles were pulled out during the scratching of Tape A.

The friction and wear results (shown in Fig. 23) show that both substrates exhibit almost the same coefficient of friction, approximately 0.35, at the
beginning of sliding. With increasing sliding distance, PET shows an increase in coefficient of friction whereas the Aramid shows a decrease. After a sliding distance of about 1 m, the coefficient of friction for both substrates remains constant. The optical images show that PET has a large wear track with several small scratches whereas Aramid does not show any wear track. This indicates that Aramid exhibits lower friction and higher wear resistance than PET. The individual magnetic layer shows a sudden increase in coefficient of friction from 0.4 to 0.8 in the initial stages of sliding and then exhibits large variations in coefficient of friction. The optical image shows that the magnetic layer was severely damaged; it shows a large wear track with many scratches and debris particles. The underlayer and backcoat have comparable coefficients of friction of about 0.35 at the beginning of sliding. With increasing sliding distance, the coefficient of friction of the underlayer increases more slowly than that of the backcoat. The optical images show that the underlayer has a smaller wear track with fewer scratches and less debris than the backcoat. In the case of double-coated tapes, Tape C exhibits the lowest coefficient of friction, followed by Tapes B and A. Tape A shows a rapid increase in coefficient of friction whereas Tapes B and C show a slow increase in coefficient of friction. The optical images show that Tape A has the largest wear
The individual magnetic layer and Tape A's magnetic layer have the same binder and formulation. However, the individual magnetic layer is thicker than Tape A's magnetic layer. We note that the individual magnetic layer has higher friction and lower wear resistance than Tape A. This is probably because the individual magnetic layer was directly coated onto the PET substrate without any underlayer, resulting in poor particle distribution and weak adhesion between the magnetic layer and substrate. During sliding, particle pullout and delamination events can occur more easily.

Fig. 24 summarizes the elastic modulus, hardness, creep, scratch, and wear results of various double-coated tapes and their individual layers and substrates. The elastic modulus and hardness data presented in Fig. 24(a) and (b) were calculated at a contact depth of 15 nm (see Fig. 20). The indentation displacements at the end of the 600 s hold (from Fig. 21) were plotted in Fig. 24(c). The damage bar chart in Fig. 24(d) was plotted based on the optical image examination of the scratch and wear tracks as well as debris after tests (0 = no apparent damage, 1 = small damage, 2 = medium damage, 3 = large damage, 4 = heavy damage). The pause lives for Tapes A, B, and C are also included in Fig. 24(e). In the pause mode test, a tape is placed in a rotary drive and is held stationary. The rotary head then repeatedly traces the same area on a tape surface. This is the most severe wear test used for magnetic tapes. A good correlation exists between mechanical properties, scratch/wear damage, and pause life. Higher mechanical properties generally result in less scratch/wear dam-

Fig. 23. Coefficient of friction as a function of sliding distance for various double-coated tapes and their individual layers and substrates against a single-crystal sapphire ball and the optical images of wear tracks and debris formed on all samples. The end of the wear track is on the left-hand side of the image [28].
age and higher pause life. Although the individual magnetic layer has good mechanical properties, it shows heavy scratch/wear damage because of poor particle distribution and weak interfacial strength. For the substrates, Aramid is more durable than PET. For the double-coated tapes, the scratch/wear damage results are in good agreement with the pause life results. Tape C has been identified as the superior tape because of its better mechanical and tribological properties. The poor mechanical and tribological properties of the backcoat may cause damage to the tape because debris from the backcoat, such as caused by a scratch, can be transferred to the front of the tape and in turn cause scratching of the magnetic layer of the tape. Therefore, improvement in the manufacture of backcoats should receive more attention.

3.2.2.4. MP versus ME tapes. The differences in mechanical and tribological properties between MP and ME tapes is still, to a large extent, unknown [10]. Recent comparisons of the mechanical properties between MP and ME tapes conducted using the nanoindentation CSM technique provide very useful data for designing magnetic tapes [30]. The contact stiffness, elastic modulus, and hardness as a function of contact depth for the PET substrate, MP tape, and ME tapes with and without a DLC coating are shown in Fig. 25. The contact stiffness of all specimens increases with a decreasing slope while the elastic modulus and hardness values decrease. In the case of ME tapes, the DLC coating and metallic film are much harder than the PET substrate. The DLC coating and metallic film act as a stretched elastic membrane under indentation [35]. It can be seen from Fig. 25 that the ME tapes exhibit a more rapid increase in contact stiffness at low contact depth, and then show the same trend as the PET substrate. As per near-surface properties, ME tapes exhibit much higher elastic modulus and hardness than MP tape and PET because of the contribution of the DLC coating and metallic film. The ME tape with a DLC coating exhibits a little higher surface elastic modulus and hardness than the ME tape without a DLC coating.

The CSM indentation creep data (shown in Fig. 26) show that the ME tapes exhibit the smallest creep displacement, followed by the MP tape and PET substrate. In contrast with indentation displacement,
the mean stresses for all specimens decrease with time, indicating stress relaxation. The ME tapes exhibit a more rapid decrease in mean stress than the MP and PET substrate. The contact stiffness for all specimens did not change during the holding segment. The ME tape with a DLC coating exhibits a little higher creep resistance than the ME tape without a DLC coating.

The scratch results (shown in Fig. 27) show that the ME tapes exhibit a lower coefficient of friction at the beginning of the scratch, whereas the MP tape and PET substrate show a large spike, indicating higher stiction force. With increasing normal load, PET exhibits large variations in coefficient of friction whereas the MP and ME tapes show a relatively smooth increase in coefficient of friction. The abrupt increase in coefficient of friction or the first appearance of debris is indicated by the arrows; these events correspond to critical loads. The ME tape with a DLC coating exhibits the highest critical load, followed by the PET substrate, the ME tape without a DLC coating, and MP tape. The DLC coating effectively protects the ME tapes from scratch damage. The optical images of the scratch tracks show that the PET substrate has a smooth scratch track without apparent debris. A wide scratch track with lots of debris at the sides and ends of scratches was observed.

![Contact stiffness, elastic modulus, and hardness as a function of contact depth for the PET substrate, MP tape, and ME tapes with and without a DLC coating.](image1)

![Indentation displacement, mean stress, and contact stiffness as a function of time at a constant load of 30 μN for the PET substrate, MP tape, and ME tapes with and without a DLC coating.](image2)
observed for the MP tape. Both the PET substrate and MP tape exhibit scratch tracks from the start of the scratch. For the ME tapes, scratch tracks start much later than the PET substrate and MP tape. This happens because the DLC coating and metallic film are harder than the magnetic layer of the MP tape. The scratch track for the ME tape without a DLC coating starts much later than the PET substrate and MP tape. This is because the DLC coating and metallic film are harder than the magnetic layer of the MP tape.

Fig. 27. Coefficient of friction as a function of increasing normal load and optical images of scratches and optical images of scratches for the PET substrate, MP tape, and ME tapes with and without a DLC coating [29].

Fig. 28. Coefficient of friction as a function of sliding distance for the PET substrate, MP tape, and ME tapes with and without a DLC coating against a single-crystal sapphire ball and optical images of wear tracks and debris formed on all samples. The end of the wear track is on the left-hand side of the image [29].
starts earlier and exhibits more debris and a wider track than the ME tape with a DLC coating.

Comparison of the coefficient of friction as a function of sliding distance for the PET substrate, MP tape, and ME tapes with and without a DLC coating against a single-crystal sapphire ball with a 3-mm diameter at a normal load of 10 mN, and optical images of wear tracks and debris formed on all specimens are illustrated in Fig. 28. The data show that ME tape with a DLC coating is superior in wear resistance than other tapes. The MP tape exhibits more scratches and debris than the ME tape without a DLC coating. It is believed that the scratches in the wear track for the MP tape result from particle pullout during sliding. In turn, these pulled-out particles accelerate damage of the MP tape. The comparison between the ME tapes with and without a DLC coating shows that the DLC coating does provide protection against wear.

4. Conclusions

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

- The CSM technique probes the mechanical property changes in situ during indentation, and provides more useful information for layered materials and nonhomogeneous (such as graded) composites.
- Time-dependent effect needs to be considered in the application of the CSM for viscoelastic materials. The CSM indentation creep tests can detect creep displacement and stress relaxation at small volumes.
- Load cycles used in the CSM can be used to perform nanoscale fatigue tests. Such tests hold promise for applications in magnetic storage and MEMS devices.
- Fortribological applications, size of debris, and contact spots range from a few nanometers to a few microns. For such applications, a range of tip radii ranging from a sharp tip to a blunt tip may be used.

Future development of the CSM technique requires measurement of contact stiffness over a wide frequency range and development of procedures for viscoelastic materials that take into account viscoelastic deformation.

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References

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