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Instrumented Indentation to Characterize Mechanical Behavior of Materials

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Abstract

The recent emergence and proliferation of the surface force apparatus and computational techniques for simulating tip-surface interactions have led to the appearance of a new form of mechanical characterization of materials tribology and contact mechanics: the so-called Depth Sensing Indentation. Indentation experiments and analysis are widely used in characterizing the mechanical properties of materials. It has two inherent advantages: it is a low destructive test, the specimen is neither fractured nor excessively deformed, and only a small amount of material is needed.

Through this work several applications involving different features of materials characterization via instrumented indentation are shown.

Keywords: depth sensing indentation, hardness, reduced elastic modulus, mechanical behavior of materials.

1. Introduction

Instrumented indentation was developed in the 70s. This new machine continuously records the applied load and displacement of the indenter tip during a test, enabling the determination of a valuable of mechanical properties. These systems have been widely used to characterize hard inorganic engineering materials such as metals and ceramics but also thin films, hybrid coatings, polymers, soft and biological materials and semiconductors. Nanoindentation has two inherent advantages: it is a low destructive test, the specimen is neither fractured nor excessively deformed, and only a small amount of material is needed. This technique uses very low loads and accurately measures very low displacement values, so local surface mechanical properties can be evaluated. With this technique it is possible to determine elastic modulus, time dependence of soft materials, fracture toughness, interfacial adhesion and plasticity of metals.

Besides, by using the inverse problem technique, which involves introducing known properties into a numerical model and recovering them by an appropriate algorithm, it is possible to extract complex stress field mechanical constitutive equations.

2. Equipment

Instrumented indentation experiments as well as nanoscratch and nanowear tests are currently being carried out at INTEMA in a Triboindenter Hysitron equipped with a Scanning Probe Module (SPM) and Multirange Nanoprobe device (MRNP). The equipment is shown in Figure 1.

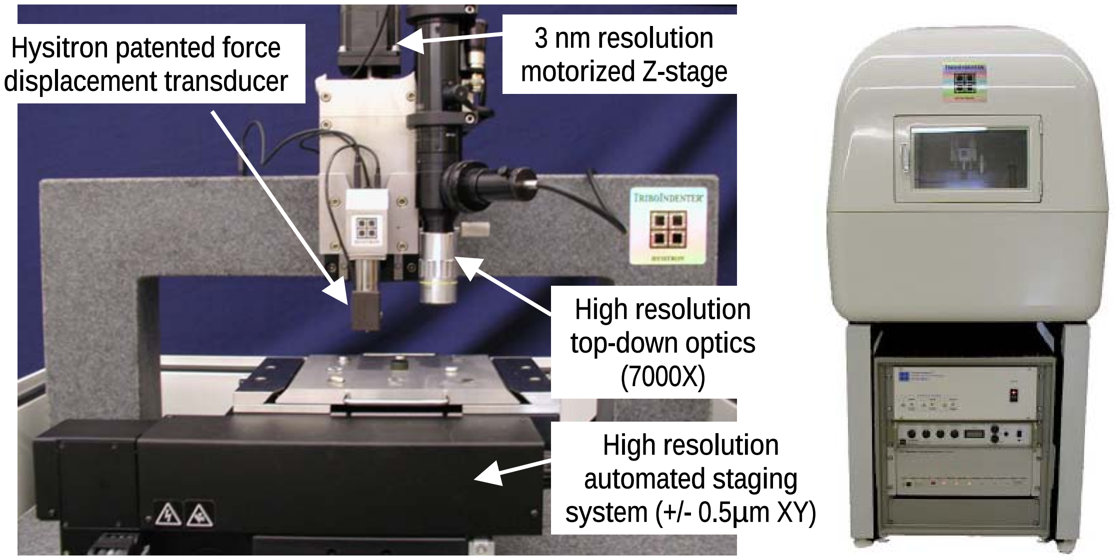


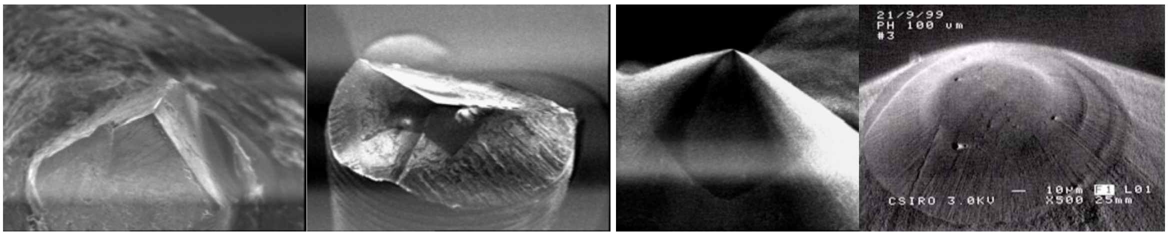
Figure 1. Triboindenter Hysitron

It works using both 1D and 2D transducers. The main specifications of the available measurement capabilities are detailed in Table 1. The equipment is able to perform indentation loading-holding-unlading cycles under load or displacement controlled conditions (1D and MRNP), scratch tests under constant normal load or applying a ramp normal load (2D) and wear experiments using the SPM module (similar to AFM).

Table 1. Details of measurement capability and accuracy of the Triboindenter Hysitron

Transducer	1D (Z axis)	2D (X axis)	MRNP(Z axis)	SPM (X,Y,Z)
Maximum Force	10mN	2mN	2N	90µN
Load Resolution	1nN	3µN		
Load Noise Floor	100nN	10µN		
Maximum Depth	5µm	15µm	60 µm	3µm
Disp. Resolution	0.04nm	4nm		
Disp. Noise Floor	0.2nm	10nm		
Maximum Scann size	-	-	-	80x80 µm^2
Scann rate	-	-	-	0-3Hz

The tips are generally made of diamond and have different defined geometries as shown in Figure 2. The selection of the tip depends on the test, material and desired equivalent strain.



Cube corner *Berkovich* *Conical* *Spherical*

Figure 2. Geometry of available tips for depth sensing indentation experiments.

3. Applications

2.1 Elastic Modulus and Hardness by the Oliver-Pharr approach from indentation load-depth curve

The analysis for the tip area calibration and the calculation of reduced elastic modulus (E_r) and universal hardness (H) is generally conducted using the approach outlined by Oliver and Pharr (O&P) [1]. This method is based on the assumption that the material behavior during unloading is purely elastic. According to O&P proposal (Figure 3), the unloading part of the recorded load-depth (P-h) curve was fitted through a power law function:

$$P = A(h - h_f)^m \tag{1}$$

and the contact stiffness (S) was calculated from slope of unloading curve as:

$$S = \left.\frac{dP}{dh}\right|_{h_{\max}} = mA(h_{\max} - h_f)^{m-1} \tag{2}$$

where A and m are the power law function fitting parameters, h_f is the residual penetration depth and $h_{\max}$ was taken as the maximum penetration depth achieved after the holding period.

The contact depth h_c was calculated by:

$$h_c = h_{\max} - \varepsilon \frac{P_{\max}}{S} \tag{3}$$

being ε a tip geometry factor, usually taken as 0.75.

Reduced elastic modulus (E_r) and hardness (H) were then calculated as:

$$E_r = \frac{S\sqrt{\pi}}{2\sqrt{A_c}} \tag{4}$$

$$H = \frac{P_{\max}}{A_c} \tag{5}$$

where A_c is the actual contact area. To account for the non-ideal shape of the tip, A_c is often fitted to a polynomial function of h_c using a series of indentations performed on a standard material (of known E), typically fused-quartz.

$$A_C = C_0 h_c^{\,2} + C_1 h_c + C_2 h_c^{\,1/2} + C_3 h_c^{\,1/4} + C_4 h_c^{\,1/8} + C_5 h_c^{\,1/16} + ... \tag{6}$$

For indentation depths larger than 6µm, A_c is generally assumed to be the ideal one (ie. for the Berkovich tip, A_c=24.5h_c^2).

E_r is directly related to the Young modulus of the material by:

$$E_r = \left[\frac{1-\nu^2}{E} + \frac{1-\nu_i^{\,2}}{E_i}\right]^{-1} \tag{7}$$

being E_i and ν_i are the Young modulus and the Poisson´s ratio of the indenter (1140GPa and 0.07 for diamond tips) while E and ν are the sample properties.

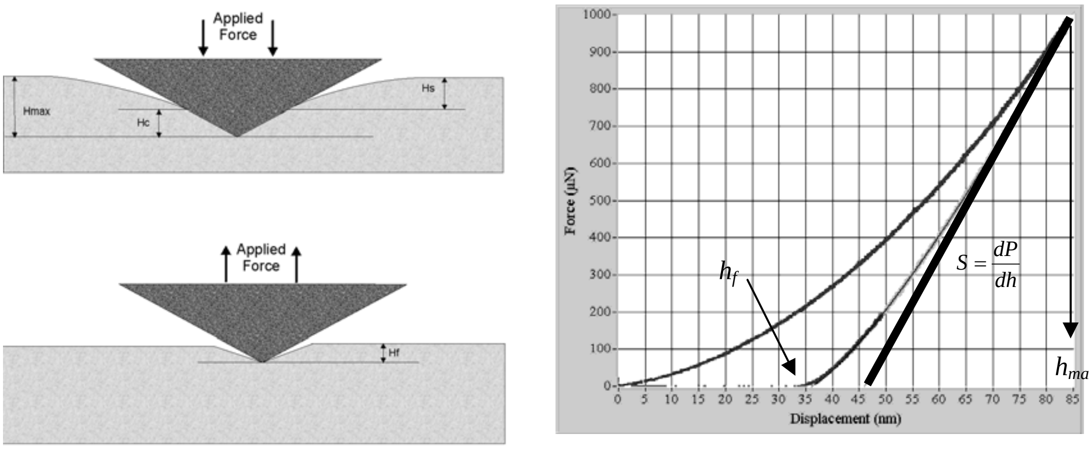


Figure 3. Scheme of the Oliver-Pharr approach for *E* and *H* determination.

The O&P approach considers that the material surface exhibits sinking-in during indentation. If the evaluated materials tend to pile-up or to excessively sink-in, so the application of the O&P approach is limited. Errors in the evaluation of A_c can lead to large errors in E and H values as schematically shown in Figure 4. Piling-up effect leads to overestimation of material properties.

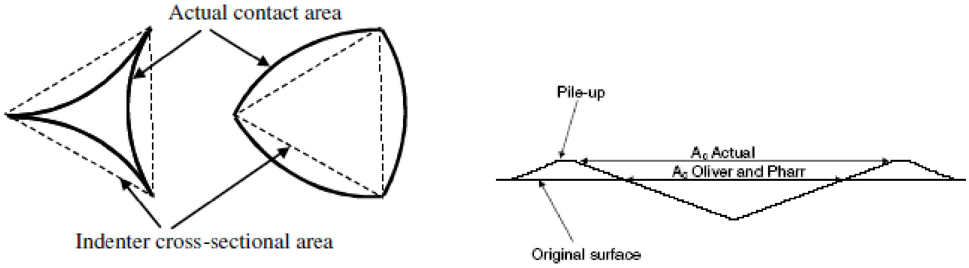


Figure 4. Comparison of the actual contact area and the one determined by the Oliver-Pharr approach

Inadequate surface roughness can also lead to uncertainties in the contact area evaluation. It is recommended maximum indentation depths larger than 10 times the surface roughness (*Ra*).

Joslin and Oliver [2] proposed an alternative method to analyze nanoindentation data for samples that are less than ideal surfaces because they are rough or because material tends to pile-up. By combining equations (4) and (5), it emerges that:

$$\frac{P_{\max}}{S^2} \propto \frac{H}{E_r^{\,2}} \qquad (8)$$

The ratio of the maximum load to the stiffness squared parameter, P_{max}/S^2, is a mechanical property that describes material's resistance to plastic deformation. When the indenter is forced a certain distance beyond the initial contact point, the interference between the indenter and the specimen is accommodated in two ways: elastic deformation and plastic (or permanent) deformation. For a given hardness, the lower the modulus, the greater the elastic accommodation and the smaller the permanent damage to the specimen when the indenter is removed. On the contrary, for a given modulus, if the hardness is increased, the plastic strain is reduced. The P_{max}/S^2 ratio is a directly measurable experimental parameter that is independent of the contact area provided the hardness and elastic modulus do not vary with depth.

The application of the O&P approach is also limited for materials that exhibit time dependent behavior. These materials tend to creep during indentation and often the unloading curve exhibits a "nose". The usual experimental way to diminish indentation creep effects during unloading is applying trapezoidal loading functions (instead of triangular loading functions) with long holding times at maximum load and high unloading rates [3]. If such requirements can not be achieved, reliable properties can be obtained applying a simple post-experiment data procedure proposed by Ngan et al [4]. According to this method, a corrected elastic stiffness, S_e, was determined as:

$$\frac{1}{S_e} = \frac{1}{S_u} + \frac{\dot{h}}{\dot{P}_u} \qquad (9)$$

where S_u is the apparent contact stiffness at the onset of unloading, $\dot{h}$ is the tip displacement rate at the end of the load hold just prior to unloading and $\dot{P}_u$ is the unloading rate. The calculated S_e value replaced S in Eq. 3 and 4 for E_r and H calculations.

2.2 Creep properties

Depth sensing indentation can be used to determine visco-elastic properties of materials. Creep within the sample can occur under indentation loading and manifests itself as a change of the indentation depth with a constant test force applied (Figure 5). This behavior is typical of polymers and metals at high temperatures. Creep experiments are carried out using trapezoidal indentation cycles, in which the load is applied instantaneously (in practice a ramp load of about 1s is achieved). The relative change of the indentation depth is referred to as the creep of the material [5]. The precise mechanisms of creep depend upon the material being indented and so several models are available in literature to interpret creep data and obtain creep compliance *J(t)* and relaxation parameters [6].

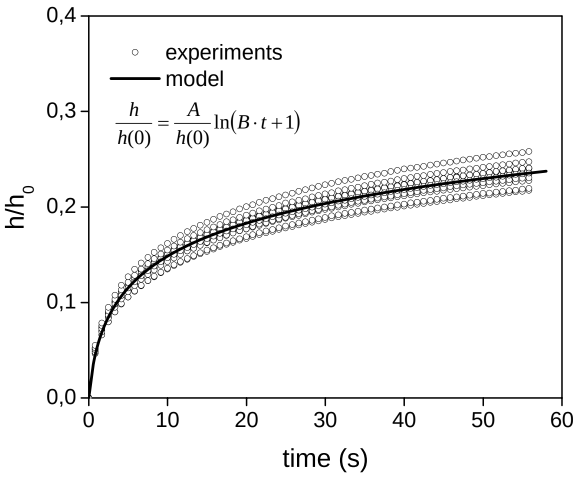


Figure 5. Relative change of tip penetration depth during the holding period of the indentation cycle for a polymeric sample.

2.3 Fracture Toughness

Depth sensing indentation can be used to evaluate the fracture toughness of materials and interfaces in a similar manner to that conventionally used in larger scale testing. During loading, tensile stresses are induced in the specimen material as the radius of the plastic zone increases. Upon unloading, additional stresses arise as the elastically strained material outside the plastic zone attempts to resume its original shape but is prevented from doing so by the permanent deformation. Different types of cracks are developed. For sharp (Berkovich and Vickers) indenters, surface cracks are observed at the corners of the imprints. Fracture toughness parameter, K_{Ic}, can be found from [5]:

$$K_{IC} = \chi\left(\frac{E}{H}\right)^{1/2}\frac{P}{c^{3/2}} \tag{10}$$

where χ is a calibration factor and c is the length of the crack (as measured from the center of the indentation to the crack tip using SPM, AFM or SEM images) as shown in Figure 5, and P is the maximum applied load.

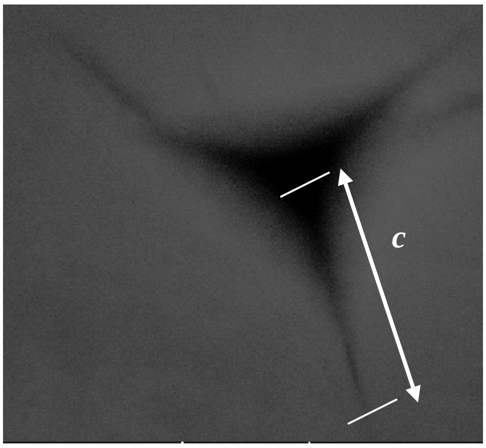


Figure 5. Example of measurement of crack length parameter (*c*) on a glass surface sample.

2.3 Stress-Strain curves, yield stress and strain-hardening exponent

The stress field under any indenter is inhomogeneous. It is characterized by representative stress and strain quantities. The mean contact pressure is suitable for representative stress while the expression for representative strain depends on the indenter shape. As the stress state beneath indenter is triaxial, the stress-strain curve based on indentation tests is derived in terms of representative stress-strain coordinates. As long as the equivalent stress is lower than the yield strength of the material, the relationship between the equivalent stress and representative strain is linear and can be evaluated using the Hertz solution. Several models are proposed to account for elasto-plastic behavior, full plastic flow and visco-plastic behavior. Using the inverse method combined with finite element simulations, the material constitutive equations as well as yield stress and strain-hardening exponents can be obtained [6].

3. Conclusions

Depth sensing indentation techniques enables the determination of many important material characteristics, such as elastic modulus and hardness, fracture toughness, yield strength, stress-strain curves and time dependency. Even if models and methods are derived for very complex analysis, they are expressed by simple equations. However, one must be aware of the limited validity of each particular model in order to choose the most appropriate one.

Depth sensing indentation combined with FEA constitutes nowadays a powerful tool able to obtain a valuable of mechanical characteristics not only for hard engineering homogeneous materials but complex materials like MEMS, thin coating/substrate systems, multilayered structures, soft materials and surface graded samples.

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