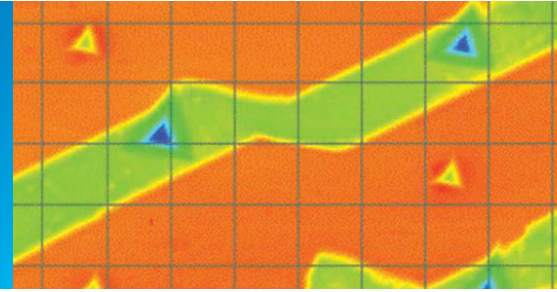KLA+

Instrumented Indentation Testing with the KLA Nano Indenter® systems

Introduction

The scale of materials and machined components continues to decrease with advances in technology making traditional test systems increasingly more difficult to use for determining mechanical properties. For this reason, instrumented indentation testing (IIT) or depth sensing indentation (DSI) testing is becoming the technique of choice for determining mechanical properties of materials on the micro and nano scales. Based on research by Sneddon, W.C. Oliver and G.M. Pharr published a landmark paper on IIT in 1992, which laid the foundation for much of the ongoing research and development in the fields of materials science and engineering. The instrument used for this seminal paper was the first design of what became the KLA Nano Indenter® systems, including Nano Indenter® iNano, Nano Indenter® iMicro, Nano Indenter® G200.

Instrumented indentation testing is similar to a hardness test in that a rigid probe is pushed into the surface of a material. Traditional hardness tests return one value of hardness at a single penetration depth or force. For most techniques, the calculation of this single measurement value requires the area of the residual hardness impression to be measured either optically or by microscopy. IIT is an improvement to traditional methods because there is no need to measure the area of the residual impression. With instrumented indentation testing, the area of contact is calculated from the load displacement history, which is recorded continuously throughout the experiment.

The two most common measurements made with IIT are Young's modulus E, and hardness H. Young's modulus can be thought of as the stiffness of a material or the material's resistance to elastic deformation. In tensile testing, Young's modulus is calculated as the slope of the stress-strain curve when the deformation is elastic. Young's modulus is an intrinsic property of a material; the only way to change E is to change the atomic structure of the material. Hardness is directly proportional to yield stress and is generally smaller by a factor of about three. Hardness is not an intrinsic property because hardness can be altered by cold-working, heat treating and other means. Elastic modulus and hardness are important to design engineers because they deliver information on how a material will behave under various stresses and strains. IIT has also been used to calculate complex modulus of polymers and fracture toughness in ceramics and glasses.

Typical Test Method

Figure 1 shows a common force-time history for an IIT test. The numbers for each segment correspond to the descriptions below:

0. The indenter approaches the test surface until contact is realized. Contact is determined by an increase in stiffness relative to the indenter column's support springs. Approach rate and stiffness increase criteria are usually user-specified.

1. The indenter is driven into the surface until the maximum force or penetration depth is reached. The rate at which the indenter is pushed into the material and the displacement or force limit is user-defined.

2. The force applied to the material is held constant for a period of time determined by the user. This dwell time is implemented for materials that experience small amounts of creep. At the end of the dwell time, creep should be negligible.

3. The indenter is withdrawn from the material at a rate equivalent to the loading rate until the force reaches 10% of the peak force.

4. The force applied to the material is held constant for a user-specified period. This test segment is used to determine the drift rate or thermal drift experienced by the material. If the drift rate is small in relation to the overall penetration depth, this segment is not required.

5. The indenter is withdrawn from the sample.

Analysis

One of the main benefits of IIT is the calculation of material properties without the need to measure the contact area directly once the indenter is withdrawn from the material. In IIT, as the indenter is pushed into the material, both the load and displacement increase and upon unloading the load and displacement decrease as the indenter is withdrawn. Other
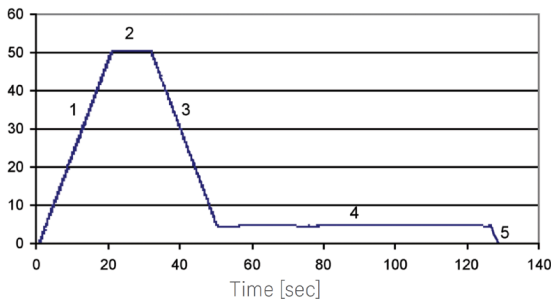
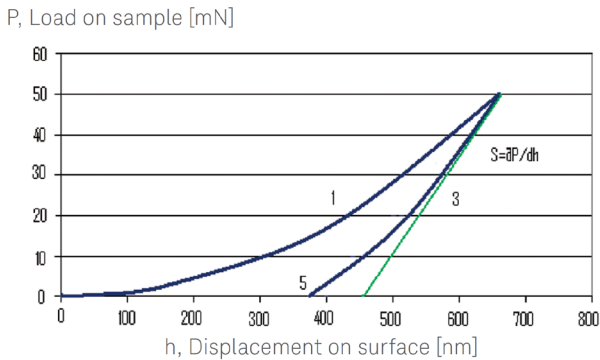**Figure 1. Force-time history for typical IIT test.**

Figure 2. Typical force-displacement curve for an instrumented indentation test. The numbered segments correspond to the test segments in Figure 1; data in segments 2 and 4 are plotted, but are not distinguishable as a function of displacement. Stiffness is determined from the slope of the tangent of the unloading curve (green line).

methods, such as a Rockwell hardness test also rely on measuring indentation depth, but IIT has the distinct advantage that the indentation depth is continuously measured across the full range of loading and unloading. For this reason, a wide range of measurements are available in IIT that cannot be acquired in traditional indentation tests.

Theory

As detailed in the work of Oliver and Pharr,[1] several parameters are needed for the calculation of the modulus of elasticity and hardness of a sample. The first parameter in the calculation stream is contact stiffness, S. Stiffness is the relationship between force and displacement when the indenter just begins to withdraw from the material. At this point the material response is entirely elastic. For the unloading segment, the dependence of P on h is given by the equation

$$P(h) = \alpha(h - h_f)^m$$

Eq. 1

Where h_f is the depth of the residual impression after the probe has fully withdrawn from the sample. The variables m, h_f and α are best-fit constants found by fitting Equation 1 to the data in the unloading segment of an IIT test on a reference material. Once α, m and h_f have been calculated, Equation 1 is differentiated with respect to displacement, and is then evaluated at the maximum displacement, yielding

$$S = \frac{dP}{dh}\bigg|_{h=h\ max} = \alpha m \big(h_{max} - h_f\big)^{m-1}$$

Eq. 2

When the indenter's probe is forced into the sample as shown in Figure 3, the material deforms in the vicinity of contact. The contact height, h_c, is the depth over which the diamond makes contact with the material. Consistent with the Oliver-Pharr model, Figure 3 assumes "sink-in" around the indentation. In Figure 3 there are three displacements that must be accounted for: total penetration depth, h; displacement of the sample surface, h_s; and contact depth, h_c. From the figure, contact depth may be calculated by

$$h_c = h - h_s$$

Eq. 3

From Sneddon's solution, the deflection of the sample surface is calculated as

$$h_s = \lambda \frac{P}{S}$$

Eq. 4

For pyramidal, conical, and spherical indenters, λ is 0.75. Thus, contact depth is calculated from

$$h_c = h - 0.75 \frac{P}{S}$$

Eq. 5

Note: What differentiates nanoindentation from traditional tests is Equation 5, which was developed by Oliver and Pharr. This equation is important because it allows for the calculation of contact depth (which is needed for the calculation of contact area) strictly from the load-displacement history; there is no need to optically measure or image the residual impression.

Once the contact depth has been determined it is then possible to calculate the contact area, A_c.
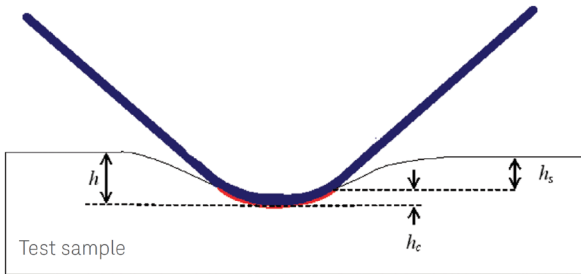
$$A_c = f(h_c)$$

Eq. 6

Figure 3. Probe-sample contact. Red outline is real contact area.

The exact form of Equation 6 is termed the area function. This function is dependent on the geometry of the probe used for testing. The most common indenter is the Berkovich tip, which is a three-sided pyramid and is usually made of diamond. For an ideal Berkovich indenter, the area function is

$$A_c = 24.56h_c^2$$

Eq. 7

For indentation depths less than 2μm the area function is written as

$$A_c = 24.56h_c^2 + Ch_c$$

Eq. 8

Equation 8 is used to account for rounding of the tip, which becomes increasingly important as indentation depth decreases. For a relatively new Berkovich indenter, the constant, C, has a value of 150nm or less. The probe will begin to wear over time and the degree of wear will depend on testing frequency and type of materials tested. As the probe wears, rounding of the apex will increase and it will be necessary to perform a calibration of the probe area function.

The reduced modulus, E_r, is determined by

$$E_r = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A}}$$

Eq. 9

The indentation modulus, E_{IT}, is then calculated as

$$E_{IT} = (1 - v_s^2) \left[\frac{1}{E_r} - \frac{1 - v_i^2}{E_i} \right]^{-1}$$

Eq. 10

Or,

$$E_{IT} = (1 - v_s^2) \left[\frac{2}{S} \sqrt{\frac{A}{\pi}} - \frac{1 - v_i^2}{E_i} \right]^{-1}$$

Eq. 11

where the subscripts s and i correspond to sample and indenter, respectively. Although the calculation of Young's modulus requires knowledge of the sample's Poisson's ratio, the dependence on υ is weak. Uncertainty analysis shows that with an uncertainty of 40% in Poisson's ratio, there is only a 5% uncertainty in Young's modulus. If the Poisson's ratio is unknown the rule of thumb is: 0.2 for glasses and ceramics, 0.3 for metals, and 0.4 for polymers. Indentation hardness, H_{IT}, is defined as the ratio of the maximum load to the contact area at that load

$$H_{IT} = \frac{P_{max}}{A(h_{c(max)})}$$

Eq. 12

As seen from Figure 4, nanoindentation is a valuable tool for evaluating the mechanical properties of a wide range of materials.
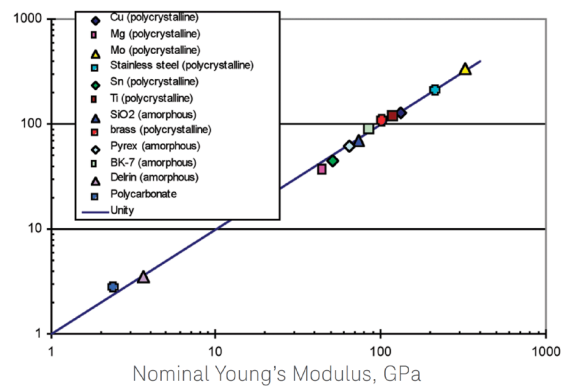

Figure 4. Indentation modulus vs. nominal modulus for various materials.

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Summary

Instrumented indentation testing with the KLA Nano Indenter® systems employs the Oliver-Pharr method and allows mechanical properties characterization on the micro- and nanoindentation scales. With the Oliver-Pharr method, the load-displacement history is measured and recorded throughout the experiment. This allows for a wealth of information to be extracted from a single experiment. Although Young's modulus and hardness are the two most sought-after values in IIT, many other properties such as complex modulus, stress exponent for creep, and fracture toughness can be calculated with all KLA Nano Indenter® systems. These properties may be calculated using standard test protocols already embedded in the software or by using the software wizard to aid in scripting a new test protocol.

Reference

1. W.C. Oliver and G.M. Pharr: An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. J. Mater. Res. 7, 1564 (1992).