CSM Instruments
Advanced Mechanical Surface Testing

APPLICATIONS BULLETIN

New Applications of Instrumented Indentation

Temperature-Specific Indentation Testing

At present, the majority of indentation testing is performed at room temperature and this is often sufficient for many applications. However, there is an increasing need to investigate the surface mechanical properties of certain materials at or near their in-service temperatures. Previous development of high-temperature hardness testing [1-4] has shown that the hardness of materials tends to decrease with increasing temperature. In addition, high temperature tests have been used to investigate creep in ceramic materials [4-5], cracking of sapphire [6] and slip lines and dislocations in $MoSi_2$ [7-9].

There are several factors which require consideration when making high temperature indentation measurements:

(i) Thermal drift effects: it is important to enclose the sample as much as possible in order to minimise thermal gradients.

(ii) Indenter: the properties of the indenter material may change significantly during heating and the mounting may be affected by loading at high temperatures.

(iii) Heat transfer between sample and indenter needs to be controlled to prevent large errors in the depth measurement.

The heating/cooling stage being developed at CSM Instruments consists of a sample enclosure which almost completely encapsulates the sample material whilst allowing access for the indenter through a small aperture. Fig. 1 shows the stage mounted on a Micro Hardness Tester which allows high precision lateral positioning under either the measuring head or the neighbouring optical microscope. The high speed of the XY translation tables is a distinct advantage as it permits the user to directly image a residual indentation in a matter of seconds after having performed a test. This is important as many materials will tend to show far greater relaxation after indentation at elevated temperatures.

In its present form, the heating/cooling stage is able to cover ranges of temperature between -35°C and 450°C. The core of the stage is a platinum resistance coil which provides a highly accurate and stable temperature control. The sample is encapsulated by the heating/cooling element and is exposed on the top side through a transparent sapphire window. This window has an access hole for the indenter and also allows easy viewing of the sample surface with the optical microscope. The stage is also equipped with inlets/outlets to allow flushing of the sample with gases to prevent oxidation or condensation. After the indenter has been approached to the sample surface, its tip is locally heated by conduction until it is at the same temperature as the sample. The indentation load-depth measurement can then be carried out with a hold at maximum load. Subsequent analysis of the load-depth curve allows compensation of the thermal drift.

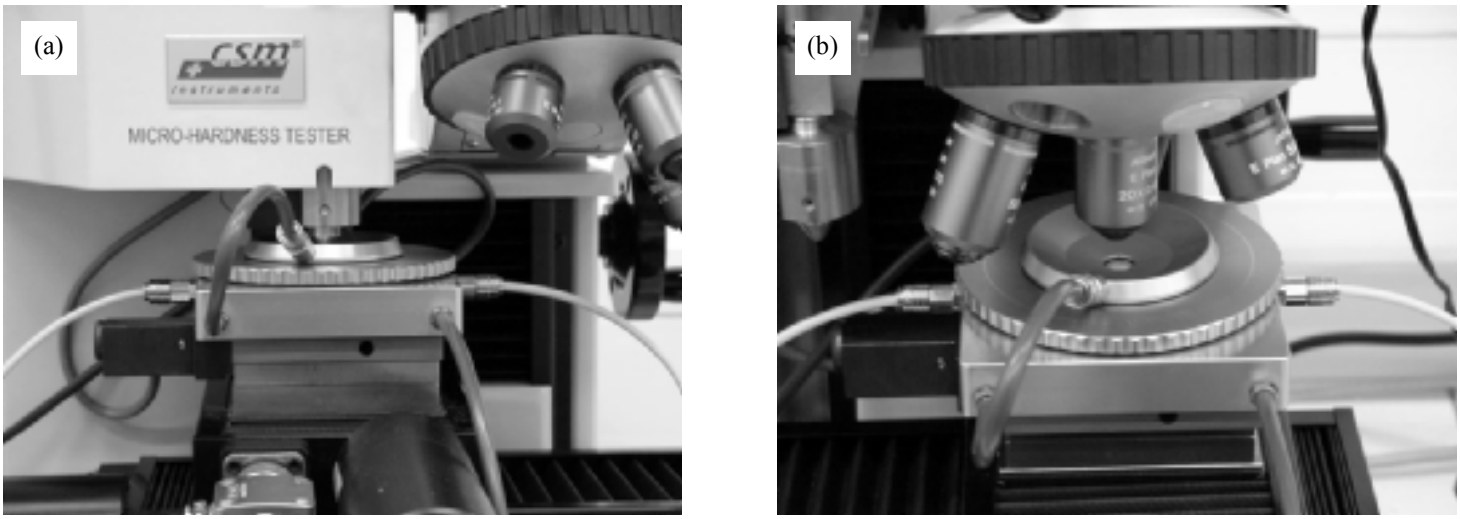

Figure1 : Heating/cooling stage mounted on the Micro Hardness Tester to give an effective sample temperature range of -35°C - 450°C. The sample is mounted in the centre of the stage and is accessed by the indenter through a small aperture (a). Subsequent imaging is performed by displacing the stage under the integrated optical microscope.

The heating/cooling stage in its present experimental form has been used to investigate the effect of heating on the mechanical properties of an AISI 440C steel. A polished steel sample was indented over a range of temperatures from 21to 200 °C with a Vickers indenter mounted on the CSM Instruments Micro Hardness Tester. The heating/cooling stage is shown in use on the instrument in Fig. 1.

At each measurement temperature, the Vickers indenter was approached to the sample surface and maintained there until thermal equilibrium had been achieved, before starting the subsequent load-depth cycle. At the end of the test, the sample was moved under the optical microscope to view the residual indentation and measure the imprint diagonal length. The measurement temperature was maintained during this step to prevent error due to sample cooling and thermal retraction.

The measured imprint diagonals are plotted as a function of sample measurement temperature in Fig 2 and the increase in diagonal length seems to follow an approximately linear relationship over the temperature range measured. Some of the corresponding optical micrographs of residual indentations are shown in Fig. 3 and clearly confirm that the hardness of the material is reduced as the temperature is increased. This corresponds to the expected behaviour of a ductile metal.

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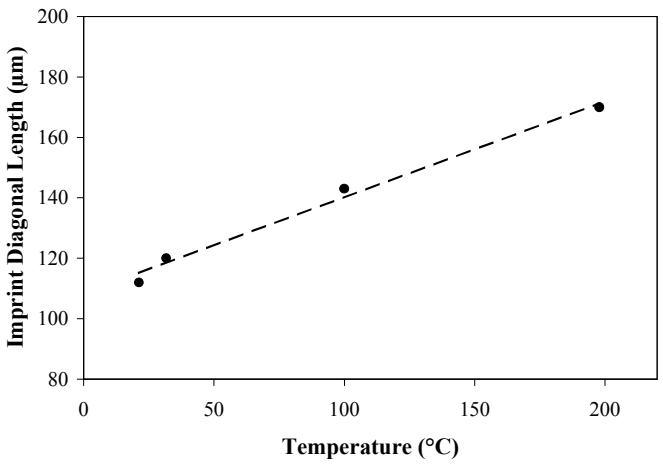


Figure 2 : Measured residual imprint diagonal lengths plotted as a function of sample measurement temperature for the range 21 - 200 °C.

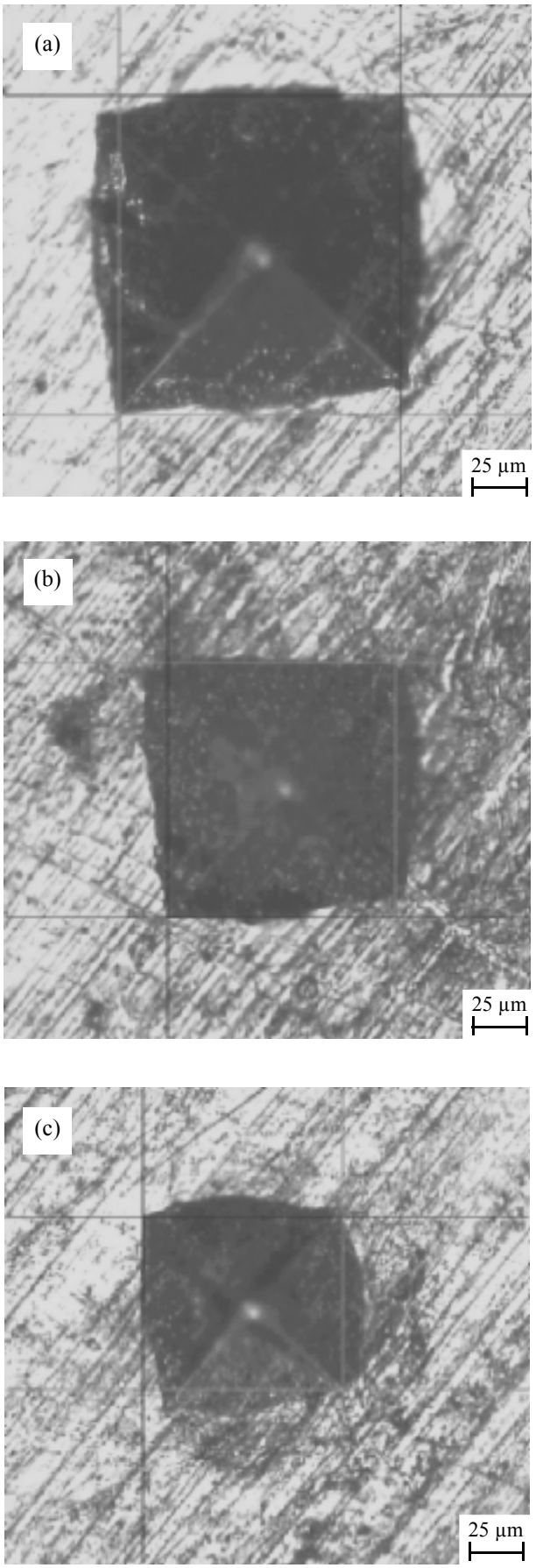


Figure 3 : Residual microindentations made in AISI 440C steel at (a) 200°C, (b) 100°C and (c) 21°C. The applied load in each case was 5 N with a loading rate of 10 N min^{-1} and a pause at maximum load of 5 seconds.

Dynamic Indentation Testing

Dynamic indentation testing (also referred to as Dynamic Mechanical Analysis) is particularly suitable for measuring mechanical properties as a function of depth in the sample for a single load-depth cycle. A small amplitude force oscillation is superimposed onto the applied load signal and the resultant displacement amplitude measured. A typical example of a force sinus and corresponding displacement is shown in Fig. 1. From the phase shift between force and displacement, several mechanical properties (including hardness and modulus) can be measured as a continuous function of depth in a single indentation. Consider a modulated force, p, applied with a frequency, ω, and amplitude p_0:

$$p = p_0 e^{i\omega t}$$

The resulting displacement, h, will have the same oscillation frequency but may have a phase difference, φ, defined:

$$h = h_0 e^{i(\omega t + \varphi)}$$

When using instrumented indentation, the system can be modelled as shown in Fig. 2 and a relationship derived which relates the applied sinus loading to the resulting displacement:

$$\left|\frac{p_0}{h_0}\right| = \sqrt{(S + K_s - m\omega^2)^2 + \omega^2 D^2}$$

In this equation, S is the contact stiffness (dP/dh), K_s is the stiffness of the indenter shaft support springs, m is the mass of the indenter shaft and D is the damping coefficient (instrument parameter). The phase difference, φ, is given by:

$$\tan\varphi = \frac{\omega D}{S + K_s - m\omega^2}$$

Dynamic indentation testing can be used on most materials, including common metals and ceramics. However, it is of particular importance when investigating the mechanical properties of viscoelastic materials such as polymers.

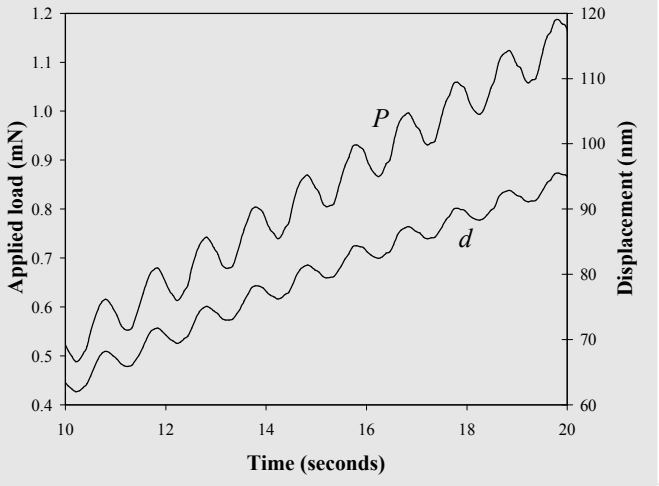


Figure 1 : Measurement made with the CSM Instruments Sinus mode showing the load-depth plot together with a zoomed portion of the loading curve where the force sinus (P) can be seen superimposed on the resultant displacement signal (d).

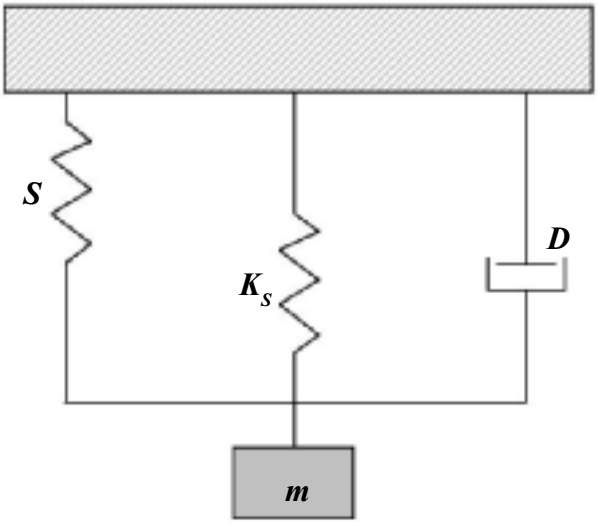


Figure 2 : Schematic representation of a typical dynamic indentation testing system. The mass, m, corresponds to that of the indenter shaft which is supported by two springs which have a stiffness, K_s. D is the damping coefficient of the system and S the contact stiffness.

Viscolelastic materials will show some elastic (energy storage) and some viscous (energy loss) behaviour. The stress, σ, induced in a viscoelastic material subjected to a sinusoidal strain, $\varepsilon = \varepsilon_0 \sin(\omega t)$, will be out of phase by some angle, φ, between 0° and 90° and can be defined:

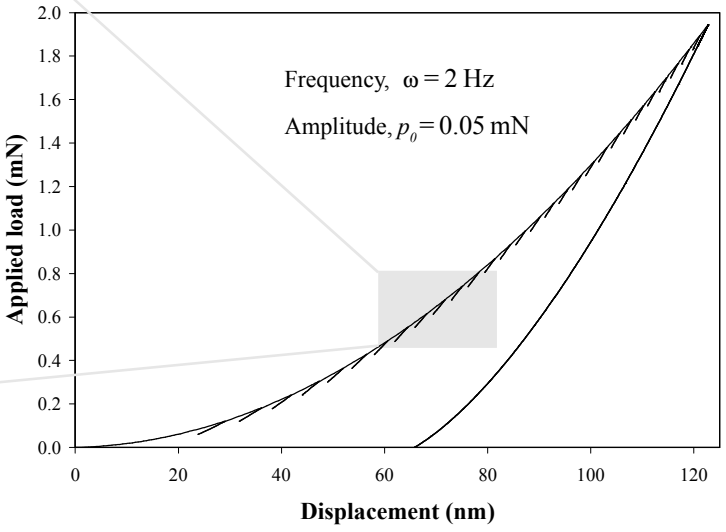
$$\sigma = \sigma_0 \sin(\omega t + \varphi)$$

where σ_0 is the stress amplitude. This can now be rearranged to give:

$$\sigma = \sigma_0 \cos\varphi \sin(\omega t) + \sigma_0 \sin\varphi \cos(\omega t)$$

The first term of this equation represents the in-phase elastic response of the material and the second term the out-of-phase viscous response. The magnitudes of the elastic response ($\sigma_0 \cos\varphi$) and the viscous response ($\sigma_0 \sin\varphi$) can be used to define the storage (E') and loss (E'') moduli:

$$E' = \frac{\sigma_0 \cos\varphi}{\varepsilon_0} \qquad E'' = \frac{\sigma_0 \sin\varphi}{\varepsilon_0}$$



The storage modulus, *E'*, can be regarded as being nearly equal to the stress relaxation modulus. The loss modulus, *E"*, tends to be small in regions where the storage modulus shows a plateau but will increase when a transition occurs. This approach assumes linear viscoelastic behaviour, i.e., the storage and loss moduli are independent of the imposed strain amplitude, ε_0. This assumption is valid for most polymers provided that the strain amplitude does not exceed approximately 0.5 - 1.0%.

Control of the sample temperature is crucial when measuring storage and loss moduli as they are both temperature dependent. For example, a test might consist of maintaining constant frequency while the temperature is ramped over the region of interest. In this way, specific transitions can be monitored, e.g., where the polymer changes from a hard condition to a viscous/rubbery condition (known as the *glass transition temperature, T_g*)

Some typical examples of Sinus mode results are shown in Figs 3 and 4 for a human bone sample. Significant research is presently underway to investigate the effects of modulus degradation in bone and the implications in cracking and implant migration. Both the hardness and modulus can be measured as a function of depth in the material (Fig. 3) and the storage/loss moduli investigated over the same depth range as shown in Fig. 4.

In addition to the aforementioned applications of dynamic indentation testing, the method shows promise for creep testing of materials. In such a test, a constant load is maintained on the indenter and the change in indentation depth is monitored as a function of time. For dynamic creep testing, the indenter is oscillated at a predetermined amplitude and frequency during the holding segment of the load-depth cycle. Time dependent deformation occurs when the indentation displacement increases while the mean stress (measured hardness) decreases.

Fatigue effects in materials can also be studied with dynamic indentation testing. For such tests, large amplitude oscillations are often used to obtain fatigue deformation and damage. Changes in contact stiffness will represent formation of damage and can be monitored until failure occurs. The number of cycles to failure can then be determined from the elapsed time.

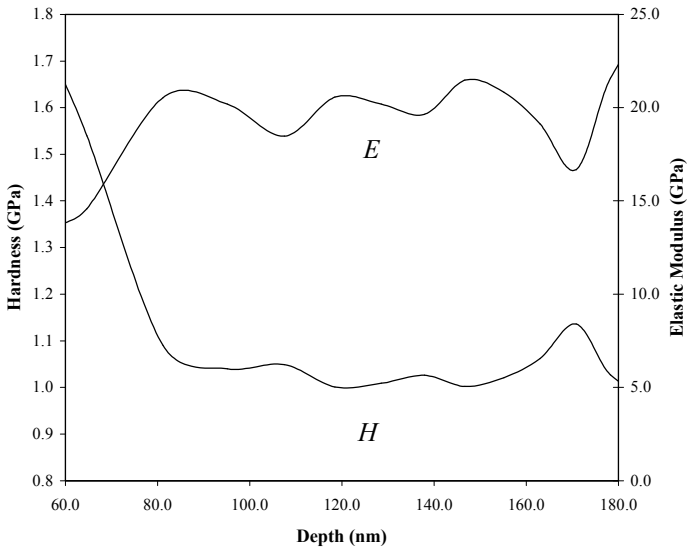


Figure 3 : Sinus mode results for a human bone sample showing measured hardness and elastic modulus as a function of depth. Maximum applied load was 2 mN, with a loading rate of 4 mN/min. and sinus frequency of 1 Hz and amplitude 0.01 mN.

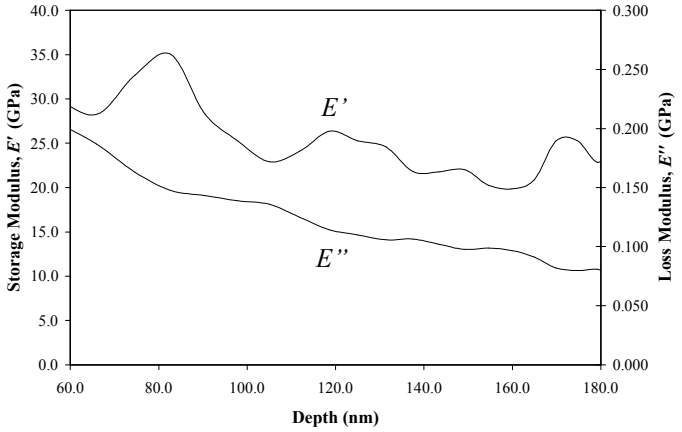


Figure 4 : Sinus mode results for a human bone sample showing both storage (*E'*) and loss (*E"*) moduli as a function of depth. Maximum applied load was 2 mN, with a loading rate of 4 mN/min. and sinus frequency of 1 Hz and amplitude 0.01 mN.

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