CSM Instruments
Advanced Mechanical Surface Testing

APPLICATIONS BULLETIN

Overview of Mechanical Testing Standards

Indentation Test Standards

The International Organisation for Standardisation (ISO) has produced an international standard ISO 14577 [1] which can be applied to instrumented indentation testing. The calculation methods used by the CSM Instruments Nano- and Micro Hardness Testers include the methods described in this standard.

The following section is a summary of the material property parameters which can be calculated from a standard indentation load-depth curve following the methodology defined in ISO 14577 . The determination of properties is divided into three ranges:

(i) Macro range:	2 N < F < 30 kN
(ii) Micro range:	2 N > F; h > 200 nm
(iii) Nano range:	h < 200 nm

The indentation test can be controlled either in Force, F, or in depth, h. Figs 1 and 2 show the main parameters required when analysing a typical load-depth curve.

(1) Indentation Hardness (H_{IT})

The indentation hardness, H_{IT}, is defined as the mean contact pressure and is given by:

$$H_{IT} = \frac{F_{max}}{A_p}$$

where F_{max} is the maximum load and A_p is the projected contact area at that load.

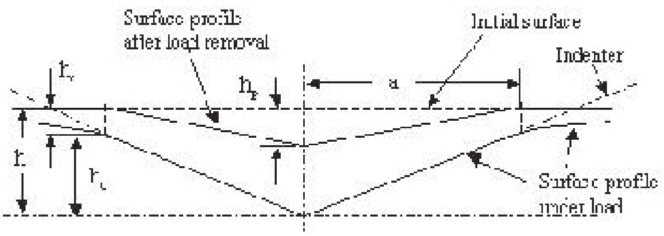

Figure1 : Schematic representation of the indentation process where h_p is the plastic depth, h_c is the contact depth, h_s is the displacement of the surface at the perimeter of contact and *a* is the radius of the contact circle defined by the indenter.

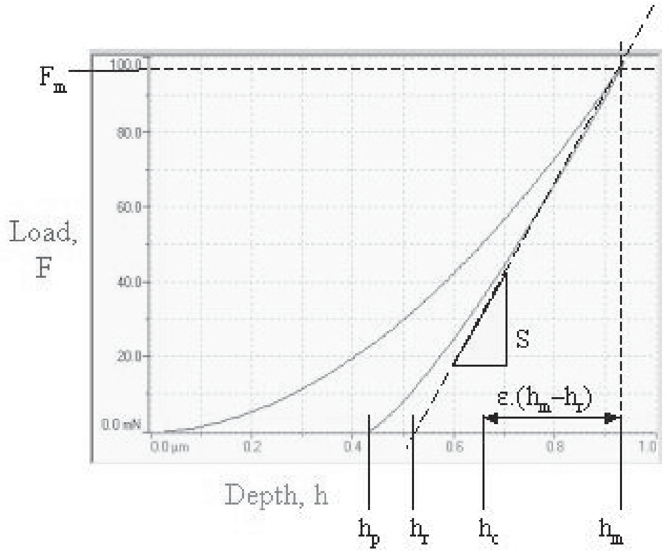

Figure 2 : Typical load-depth curve from a nanoindentation test showing the parameters defined in Fig. 1 as well as the stiffness, *S*, calculated from the tangent of the unloading curve.

(2) Martens (Universal) Hardness (HM)

The Martens hardness, *HM*, is defined as the maximum applied load, F_{max}, divided by the contact area, A_s, at that load:

$$HM = \frac{F_{max}}{A_s}$$

The Martens hardness is defined for Vickers and Berkovich indenter geometries but not for spherical or Knoop indenters. It has also been referred to previously as the 'Universal hardness' (*HU*) [2]. For a perfect Berkovich indenter, the Martens hardness is given by:

$$HM = \frac{F}{A_s(h)} \approx \frac{F}{26.43 \cdot h^2}$$

where

$$A_s(h) = \frac{3 \cdot \sqrt{3} \cdot \tan(\acute{a})}{\cos(\acute{a})} \cdot h^2$$

The parameter α refers to the face angle of the indenter (65.03° in this case) and *h* is the penetration depth.

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The Martens hardness should be recorded with the symbol HM followed by the exact test conditions used:

(i) Indenter geometry (if not Vickers)
(ii) Test force (in Newtons)
(iii) Time for the application of the test force (in seconds)
(iv) Number of steps until the maximum load is reached if the load is not applied continuously (CSM instruments use continuous loading)

Examples:

HM 0.5/20/20 = 8700 N/mm^2

The Martens hardness is 8700 N/mm^2, determined with a test load of 0.5 N, applied over 20 seconds in 20 steps. If a Berkovich indenter had been used, then this would become:

HM (Berkovich) 0.5/20/20 = 8700 N/mm^2

(3) Indentation Modulus (E_{IT})

The indentation modulus, E_{IT}, is calculated from the slope of the tangent of the unloading curve, whether using a linear fit to the initial unloading data or a power-law fit [4 - 5]:

$$E_{IT} = \frac{1-n_s^2}{\dfrac{1}{E_r} - \dfrac{1-n_i^2}{E_i}}$$

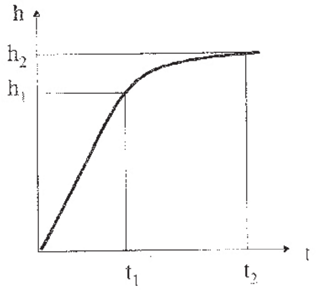
where E_i is the elastic modulus of the indenter (1141 GPa for diamond), ν_i is the Poisson's ratio of the indenter (0.07 for diamond) and ν_s is the Poisson's ratio of the tested sample. The reduced modulus, E_r, which is calculated from the indentation data is defined as:

$$E_r = \frac{\sqrt{p} \cdot S}{2 \cdot b \cdot \sqrt{A_p(h_c)}}$$

(4) Indentation Creep (C_{IT})

The indentation creep, C_{IT}, can be defined as the relative change of the indentation depth whilst the applied load remains constant:

$$C_{IT} = \frac{h_2 - h_1}{h_1} \cdot 100$$



The load is applied until a fixed val

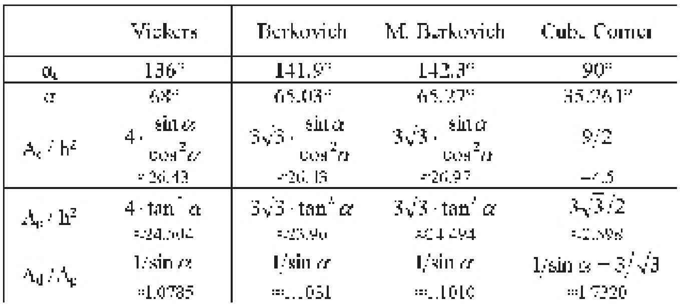
value is then maintained until time t_2 is reached and h_2 - h_1 signifies the relative change of the indentation depth. Creep is displayed as a percentage, for example:

C_{IT} 0.08/30/60 = 4.3 %

The creep is 4.3 % determined with an applied load of 80 mN which was applied in 30 seconds and maintained constant for 60 seconds. Note that C_{IT} is not expressed as a displacement versus time (µm/sec).

Indenters

The indenter used for the indentation test should be calibrated independently of the indentation instrument by a direct optical method and the calibration certificate should include the relevant geometrical measurements. A summary of the 4 most commonly used pyramidal indenter geometries is given in Fig. 3. There is often confusion between the two types of Berkovich geometry; the standard Berkovich has the same ratio of actual surface area to indentation depth as a Vickers indenter. The modified Berkovich indenter has the same ratio of projected area to indentation depth as the Vickers indenter.



	Vickers	Berkovich	M. Berkovich	Cube Corner
α_t	136°	141.9°	142.3°	90°
α	68°	65.03°	65.27°	35.2641°
A_d / h^2	$4 \cdot \frac{\sin\alpha}{\cos^2\alpha}$ ≈26.43	$3\sqrt{3} \cdot \frac{\sin\alpha}{\cos^2\alpha}$ ≈26.43	$3\sqrt{3} \cdot \frac{\sin\alpha}{\cos^2\alpha}$ ≈26.97	9/2 ≈4.5
A_p / h^2	$4 \cdot \tan^2\alpha$ ≈24.504	$3\sqrt{3} \cdot \tan^2\alpha$ ≈23.96	$3\sqrt{3} \cdot \tan^2\alpha$ ≈24.494	$3\sqrt{3}/2$ ≈2.598
A_d / A_p	$1/\sin\alpha$ ≈1.0785	$1/\sin\alpha$ ≈1.1031	$1/\sin\alpha$ ≈1.1010	$1/\sin\alpha = 3/\sqrt{3}$ ≈1.7320

Figure 3 : Summary of angle and area data for the 4 most commonly used indenter geometries, where α_t is the total included angle, α is the angle between the axis of the pyramid and its faces, A_d is the real (or 'developed') contact area and A_p is the projected contact area.

Indenters my gradually become contaminated by adherence of foreign matter from tested samples. Diamond indenters can be effectively cleaned by gently pushing them into a block of high-density polystyrene foam followed by a rinse in isopropyl alcohol. The cleanliness of an indenter may be checked by carefully inspecting it under an optical microscope with a magnification greater than 400x.

The indenter area function is usually plotted as a graph of true contact area as a function of the contact depth, h_c, and can be determined by direct measurement (e.g. using a Scanning Force Microscope) or by an indirect method [5].

For a more complete explanation of ISO 14577, see ref. [6]. Other related standards are DIN 50359 [2] and DIN ISI 4516 [3]. CSM Instruments is currently participating in the finalisation of a new ASTM standard for instrumented indentation testing.

[1] ISO/FDIS 14577-1:2002; Metallic materials - Instrumented indentation test for hardness and materials parameters, ISO Central Secretariat, Rue de Varembé 1, 1211 Geneva, Switzerland

[2] E DIN 50359; Testing of metallic materials - Universal hardness test

[3] DIN ISI 4516; Metallic and similar layers - Micro hardness test after Vickers and Knoop, 1988

[4] M. F. Doerner and W. D. Nix, J. Mater. Res., 1 [4] (1986) 601-609

[5] W. C. Oliver and G. M. Pharr, J. Mater. Res., 7 [4] (1992) 1564-1583

[6] A. C. Fischer-Cripps, *Nanoindentation*, Mechanical Engineering Series, Springer-Verlag 2002, ISBN 0-387-95394-9

Tribology Test Standards

The two main standards referring to the pin-on-disk Tribometer are DIN 50 324 [1] and ASTM G 99 - 95a [2]. With the linear reciprocating option (see App. Bull. No. 8, July 1998), ASTM G 133 - 95 covers the standard method for sliding wear of a linearly reciprocating ball-on-flat contact.

Both ASTM standards determine the amount of wear by measuring appropriate linear dimensions of both specimens (ball and disk) before and after the test, or by weighing both specimens before and after the test. In practice, linear measures are often preferred since mass loss is often too small to measure precisely. The standard ball-on-disk setup is shown in Fig. 1 with the parameters required to calculate wear.

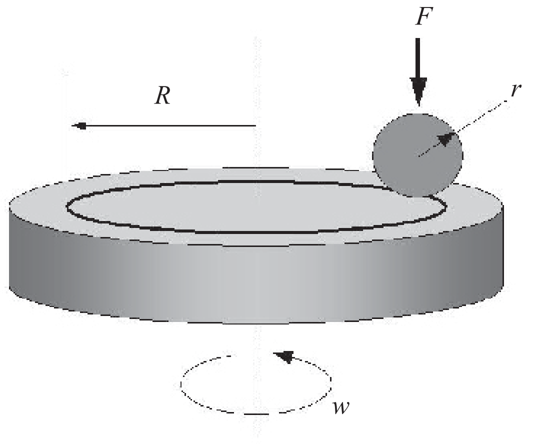


Figure 1 : Typical ball-on-disk setup where *F* is the normal force applied on the ball, *r* is the ball diameter, *R* is the radius of the wear track and *w* is the rotational speed of the disk.

The material pair which is tested will dictate whether the ball, the disk or both, will become significantly worn. An example of each case is summarised in Fig. 2. Assuming that there is no significant disk wear, the volume loss of the spherical ball (or spherical-ended pin) is given by:

Pin volume loss = $(\pi h/6)[3d^2/4 + h^2]$

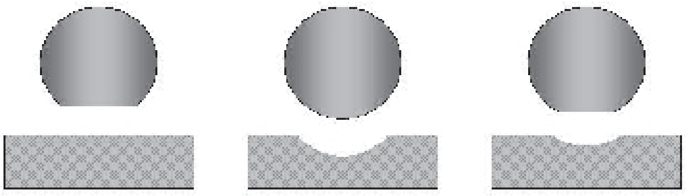
where $h = r - [r^2 - d^2/4]^{1/2}$ (height of material removed)
d = wear scar diameter
r = ball radius

Assuming that there is no significant pin wear, the volume loss of the disk is given by:

Disk volume loss = $2\pi R[r^2 \sin^{-1}(d/2r) - (d/4)(4r^2 - d^2)^{1/2}]$

where R = wear track radius
d = wear track width

The disk volume loss calculated in this manner may have certain error due to variations around the wear track, accumulations of debris and plastic deformation. A stylus profilometer is often used to measure the cross-sectional area of the wear track in several places around the track. This provides a more accurate measure of the disk volume lost when multiplied by the track length.



(a) (b) (c)

Figure 2 : Three possible situations for differing wear resistance of ball and flat disk specimens; (a) only the ball wears, (b) only the disk wears, and (c) both ball and disk wear.

When using the linear reciprocating option (Fig. 3), constant velocity conditions are not maintained but the test is useful for simulating contacts between materials where there is a periodical reversal in the direction of relative sliding. The wear resulting from this mode of operation may significantly differ from that produced by the same materials sliding continuously in only one direction (unidirectional sliding). The wear volume of the linear track (V_{track}) is given by:

$$V_{track} = A \times L$$

where A = average cross-sectional area of the track
L = length of the stroke

In most cases, the width and depth of the wear track on the disk will be relatively uniform along its length. If the areas of the three initial profiles differ by less than 25%, three profiles are sufficient for the calculation of *A*.

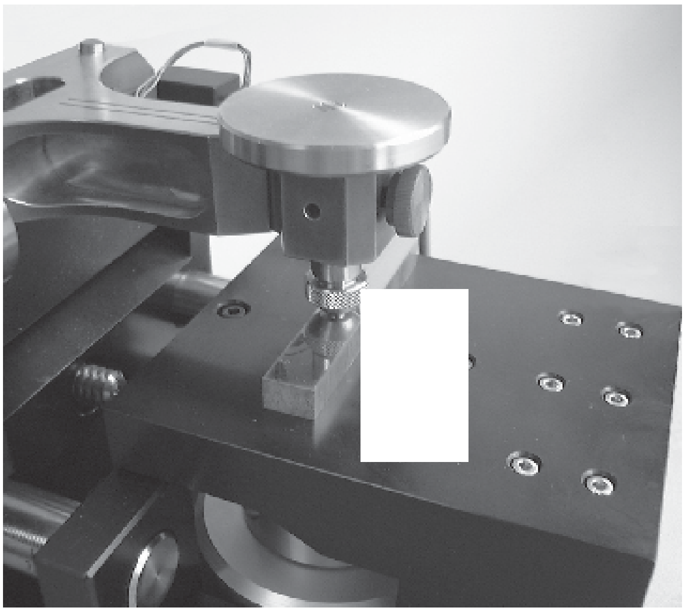


Figure 3 : Standard pin-on-disk Tribometer being used with the linear reciprocating option. Linear movement direction is shown by the arrow.

[1] DIN 50 324; Testing of friction and wear

[2] ASTM G 99 - 95a; Standard test method for wear testing with a Pin-on-Disk apparatus

[3] ASTM G 133 - 95; Standard test method for linearly reciprocating ball-on-flat sliding wear

Scratch Test Standards

Owing to the many variable parameters involved in scratch testing, it is quite a challenge to be able to adequately standardise the method. One recent approach by the European Commision [1] has focussed on one class of coating material (ceramics) in order to reduce the vast numbers of possible critical failure modes. The standard corresponds to the measurement ranges of both the Micro Scratch Tester and the Revetest, and covers three different scratching procedures:

(1) Progressive load scratch test (PLST)

This corresponds to a load ramp applied to the indenter during defined displacement of the sample beneath it. Standard operating parameters of 100 N/min (loading rate) and 10 mm/min (lateral displacement speed) are recommended in order to minimise the number of test specific parameters. The first scratches are used to define the highest critical load (HL_c) and subsequent scratches can then be limited to HL_c + 10 N to prevent unnecessary wear of the indenter tip.

(2) Constant load scratch test (CLST)

In this testing mode the normal load is increased in increments between successive scratches carried out under constant load at different locations on the sample surface. The recommended standard operating parameters are 10 mm/min (lateral speed) and 10 mm scratch length. It is also recommended that one fifth of the critical load (determined by progressive load mode) be used for the indenter load step size between consecutive tests.

(3) Multipass scratch test (MPST)

Multipass scratch testing corresponds to repeated scratching under a constant sub-critical load within the same scratch track. The standard operating parameters are the same as in the CLST mode and it is recommended to use 50% of the critical load (determined by progressive load mode) and determine the number of scratches until failure occurs. Depending on the mechanical response of the tested sample, it may be necessary to adjust the applied load by reducing it (for better discriminating capacity) or increasing it (to obtain results in an acceptable time-scale).

The EN 1071 standard recommends that the PLST mode be used as a first order assessment of critical loads corresponding to major coating damage failure, while the CLST mode allows statistical damage analysis of coatings along their surface. The MPST mode subjects the coated surface to a low-cycle fatigue type contact, which is considered to better simulate real working conditions encountered by most coated components in service.

In most cases, the CLST operation mode allows better discrimination between better or poorer adhesion properties than does the PLST. However, the former is significantly more time-consuming than the latter. The MPST mode has been shown to better rank brittle coatings in terms of their adhesion properties.

EN 1071 includes a comprehensive atlas of scratch test failure modes, some examples of which can be found in App. Bull. No. 14 (April 2000). This inventory of the major scratch test failure modes classifies them into plastic deformation and different forms of cracking, spallation and coating perforation events. It is not a fully comprehensive catalogue, but it is a first step in the standardised reporting of scratch test critical loads.

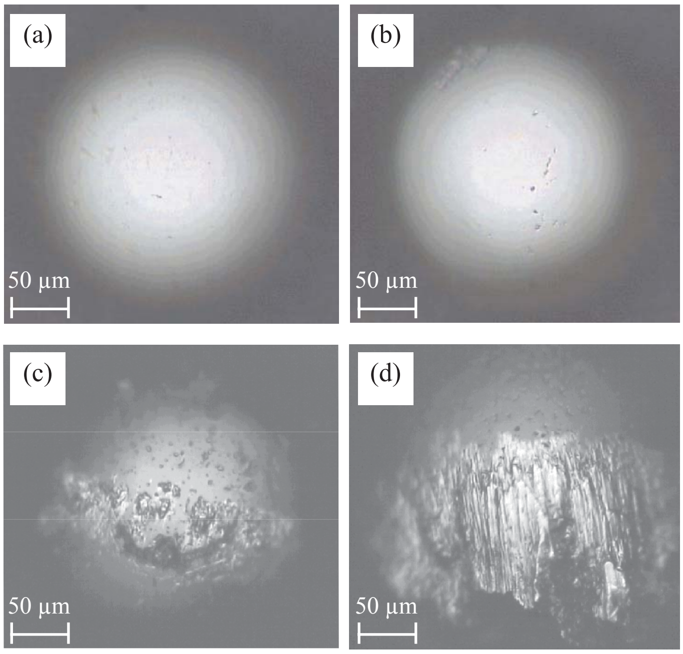


Figure 1 :A selection of 200 µm radius diamond indenters viewed through an optical microscope at 200x magnification. A new undamaged indenter is shown in (a) and a slightly worn (but acceptable) indenter in (b). Note the ring crack damage in (c) and the catastrophically worn tip shown in (d).

[1] EN 1071 - 3; Advanced technical ceramics - Methods of test for ceramic coatings - Part 3: Determination of adhesion and other mechanical failure modes by a scratch test.

CSM Instruments

This Applications Bulletin is published quarterly and features interesting studies, new developments and other applications for our full range of mechanical surface testing instruments.

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