

APPLICATIONS BULLETIN

The Influence of the Rockwell Indenter Tip Geometry on the Progressive Load Scratch Test

//// Introduction:

Over the last 30 years, CSM Instruments has established itself as the world leader in Scratch Testing. In a continuing effort to improve the accuracy and reproducibility of the method, CSM Instruments remains actively involved in several standardization committees.

Many companies are now using the scratch test as a dedicated instrument for quality control and therefore require suitable ASTM and ISO standards which are robust and assure reproducibility.

In order to conform to these new standards, CSM Instruments has developed a new procedure to verify the geometry of Rockwell indenters with much tighter tolerance than that specified in the current standards. This Applications Bulletin summarizes some recent work which has been carried out to characterize the tip geometries of 4 different batches of Rockwell diamond indenters manufactured by 4 different suppliers. The results presented will confirm that the tolerances specified by current standards are insufficient for obtaining sufficient reproducibility in the scratch test method.

//// Scratch Standardization

At present, all scratch test standards recommend verification of the indenter following the requirements of ISO 6508-2 which details the Rockwell hardness test. For example, ISO 20502 states:

> **ISO 20502: Fine ceramics (advanced ceramics -advanced technical ceramics) - Determination of adhesion of ceramic coatings by scratch testing**

Points 4.2 Diamond stylus

This consists of a rigidly mounted diamond normally having a Rockwell C geometry in accordance with the requirements of ISO 6508-2. The stylus shall be inspected regularly to check for contamination and changes in geometry. If damage is observed at 200x or lower magnification then the stylus shall be changed, and if either damage or contamination is observed, the test results since the last inspection shall be disregarded. If the friction force increases at a constant normal force during operation, this is a presumption of contamination of the stylus.

NOTE 1 Uncertainties in the Rockwell C stylus tip shape and manufacturing defects are a major source of error for the scratch test method. The use of an imperfect stylus may result in different values of critical normal force when the stylus is rotated in its holder. The control of stylus shapes is imperative, in the as-received condition as

well as during usage, to detect wear at the tip. Wear usually occurs in the form of ring cracks or crater wear, which is easily visible under a reflected-light microscope (magnification > 100x).

NOTE 2 A certified reference material (BCR-692) has been developed and is available from the Institute of Reference Materials and Measurements, European Commission Joint Research Centre. This material, a diamond-like carbon coated substrate, presents three repeatable failure events at known critical normal-force intervals, and is available for verification purposes. This can provide a good indication of overall performance, including stylus condition and calibration.

> ISO 6508-2:

Metallic materials -- Rockwell hardness test -- Part 2: Verification and calibration of testing machines

Point 4.3 Verification of the indenters

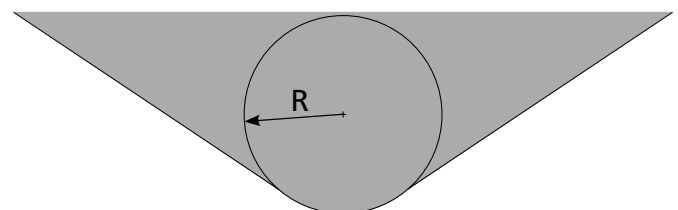
4.3.1.1.1 The surface of the diamond cone and spherical tip shall be polished for a penetration depth of 0.3 mm and shall blend in a truly tangential manner. Both surfaces shall be free from surface defects.

4.3.1.1.2 The verification of shape of the indenter can be made by direct measurement or by a measurement of its projection on a screen. The verification shall be made at no less than four equally spaced sections.

4.3.1.1.3 The diamond cone shall have an included angle of $120^\circ \pm 0.35^\circ$

4.3.1.1.4 The angle between the axis of the diamond cone and the axis of the indenter holder (normal to the seating surface) shall not exceed 0.5°

4.3.1.1.5 The tip of the indenter shall be spherical. Its radius shall be determined from single values, measured in the axial section planes defined in 4.3.1.1.2 The distance between the concentric circles shall be not more than 0.004 mm. Each single value shall be within 0.2 ± 0.015 mm. The mean value out of at least four single values shall be within **(0.2 \pm 0.01 mm)**



To resume, the standards acceptance values are:

Radius [R]: 200 ± 10 μm

Angle: $120^\circ \pm 0.35^\circ$

Profile deviation: ± 2 μm

//// Evaluated Diamond Indenters:

In this study, we have analyzed 4 similar sets of diamond indenters from 4 different manufacturers:

Indenter A: from UNITED STATES

Indenter B: from EUROPE

Indenter C: from JAPAN

Indenter D: from EUROPE



Fig 1: An example of a Rockwell diamond indenter from each of the 4 suppliers

//// Tested Samples

For evaluation of the indenters, two well-known sample materials were chosen:

> Titanium Nitride (TiN): 4 μm TiN on High Speed Steel (HSS) substrate.

This is the CSM Instruments internal standard material

> Diamond-like Carbon (DLC): 2 μm DLC on High Speed Steel (HSS) substrate.

This is the BCR-692 certified reference material

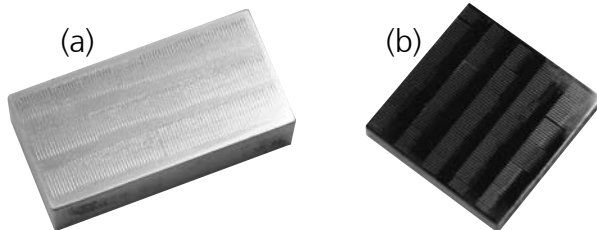


Fig 2: Examples of the TiN Sample (a) and the DLC Sample (b), each containing more than 300 scratches

//// CSM Instruments internal indenter verification

The two methods used to measure Rockwell indenter geometries are Chromatic Confocal imaging and knife profilometry.

The Confocal method allows the entire indenter tip to be imaged in three dimensions without any contact of the surface. The tip radius, sphericity and surface roughness can be easily extracted from the resultant 3D image.

The knife profilometer provides a line profile of the indenter tip and is a contact measurement.

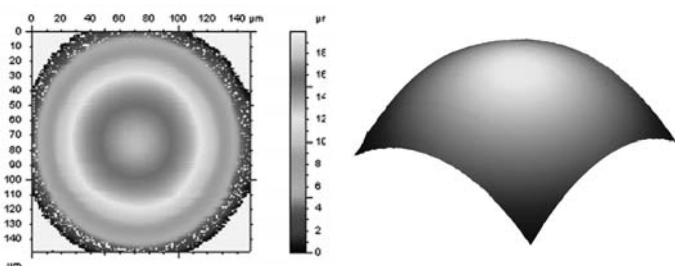


Fig. 3: Confocal 3D topography image of a Rockwell diamond indenter

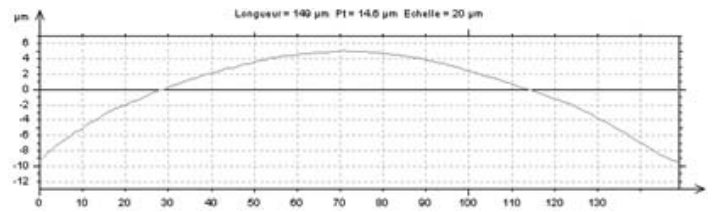


Fig. 4: Knife profilometer line-scan of a Rockwell diamond indenter

Experience has shown that the indenter is the most critical component of the scratch test method.

This is why the CSM Instruments internal quality check is far more rigorous than the tolerances of ISO 6508-2 (standard CSM tolerances are Radius: $R \pm 4\%$; Angle: $120^\circ \pm 0.3^\circ$).

The main limitations of simply measuring a radius and an angle are that these two parameters do not give a measure of the "sphericity" of the tip. This is illustrated by the two indenter tip profiles shown in Fig. 5. Both of these profiles conform to the tolerances of ISO 6508-2, but it is clear that the example in Fig. 5 (a) is sharper than that in Fig. 5 (b). Such geometrical differences would translate into significantly different contact pressures during a scratch test.

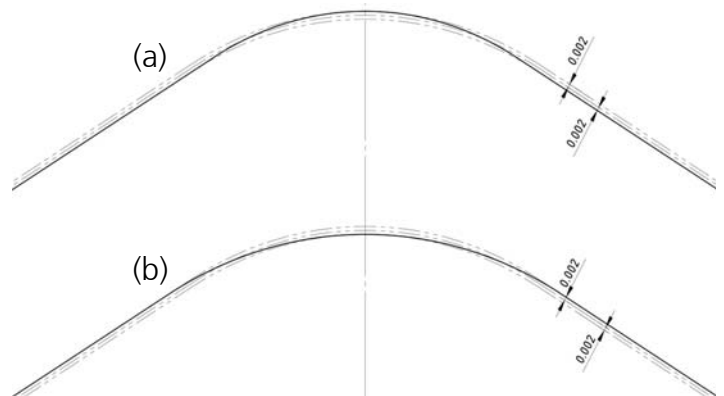


Fig 5: Indenter tip profiles for two different diamond indenters

Another limitation of the existing standards is the range over which the indenter radius is measured. The portion of the indenter tip over which the radius is extracted has a major influence on the actual radius value. Fig. 6 shows 3 different measurement portions (A, B and C) on a line profile. On a typical Rockwell indenter, each such portion will provide a very different radius value.

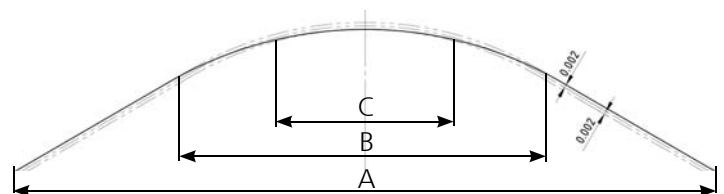


Fig 6: Example of how the measurement portion affects the measured indenter radius

In contrast to the Rockwell hardness test, the scratch test rarely exceeds a scratch width of 20 μm , so it makes no sense to measure the indenter radius from a line profile which extends to a width of $> 100 \mu\text{m}$.

The examples in Fig. 7 summarize line profiles made on indenters A and D. The maximum lateral range of the Confocal microscope is 140 μm and if the radius is extracted over this

range, both indenters seem to conform to the ISO 6508-2 tolerances. However, if smaller measurement ranges are used (in this example, 100 μm , 71 μm and 45 μm) both indenters are no longer within tolerance.

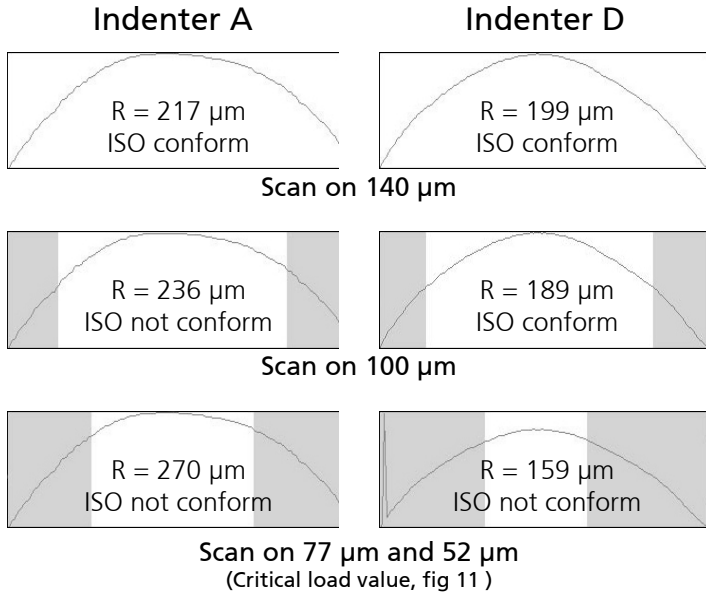


Fig 7: Radius (R) comparison on indenters A and D

The influence of the radius on the scan diameter is also interesting. The graph in Fig. 8 shows the measured radius plotted against the corresponding diameter for indenters A and D. If we compare the radius values for each indenter for the same scan diameter of 40 μm , the difference between the two indenters exceeds 250% (321 μm for indenter A and 138 μm for indenter D). Although both these indenters conform to the ISO tolerances, their scratch test performance will be very different!

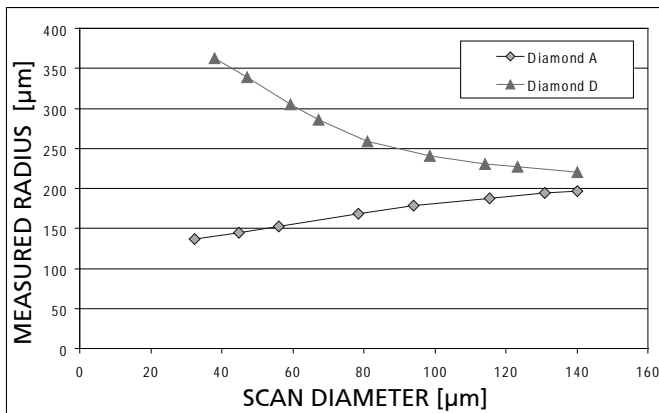


Fig 8: The influence of the radius versus the scan diameter is significantly different on the indenter A and D

//// Scratch Testing

To verify the influence of the Rockwell indenter tip geometry on the progressive load scratch test, a series of 10 identical scratches were performed on the two samples (TiN and DLC) with all the indenters (A, B, C, D).

The scratch test parameters were:

Load: 1 - 100 N

Loading rate: 100 N/min

Scratch length: 5 mm

Scratch speed 5 mm/min

Note: Only the main critical failure point (corresponding to chevron cracking along the edges of the scratch track) was taken for this investigation.

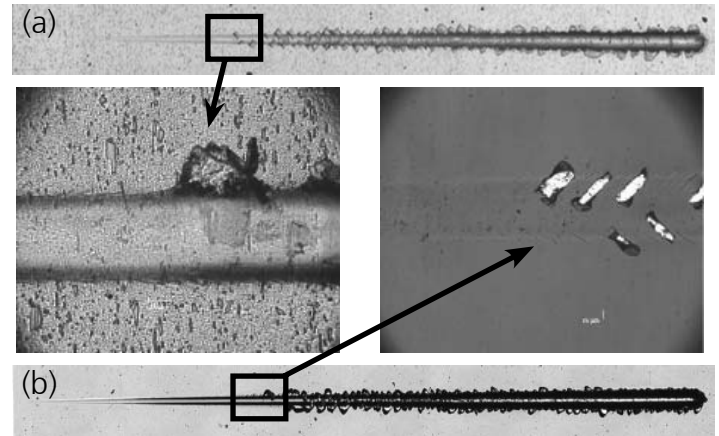


Fig 9: Measured critical failure points on TiN (a) and DLC (b) samples

The critical failure load values show a large variation between the 4 different indenters, as summarized below:

> For the TiN Samples

> Indenter A : 37.71 N (STDV 0.62; n=2)

> Indenter B : 31.50 N (STDV 2.85; n=4)

> Indenter C : 36.32 N (STDV 2.48; n=4)

> Indenter D : 31.94 N (STDV 2.20; n=4)

> For the DLC samples

> Indenter A : 21.70 N (STDV 1.41; n=2)

> Indenter B : 15.95 N (STDV 2.82; n=4)

> Indenter C : 15.86 N (STDV 3.35; n=4)

> Indenter D : 11.70 N (STDV 0.29; n=4)

These huge differences in critical failure load are solely due to variations in the indenter tip geometry ! For a Rockwell indenter of radius 200 μm , the conical portion of the indenter (beyond the spherical apex and with included angle of 120°) is never reached during the scratch test and so bears no influence.

Another way to visualize the differences between certain indenters is to compare the scratch width at the same point along a scratch made with each. Fig. 10 shows the significant difference in scratch width when measured at the critical failure point on the TiN sample with indenters A and D. Such different scratch widths are also seen to cause differences in the way that the coating fails, due to the variation in localized contact pressure.

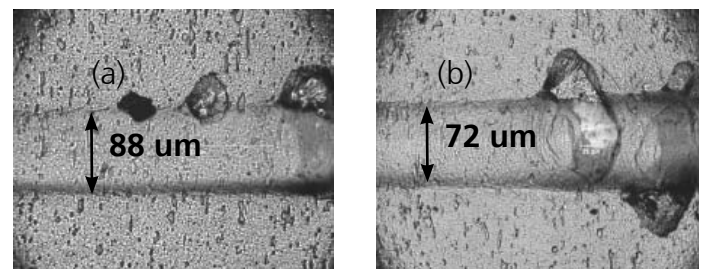


Fig: 10 Scratch width at critical failure point on TiN with indenters A (a) and D (b)

The same differences can be seen for scratch tests performed on the DLC sample, as shown in Fig. 11.

This certified reference material has a critical failure load of 17.00 +/- 2 N whereas in this example indenter A fails the coating at 21.7 N and indenter D at 11.7 N, both significantly exceeding the accepted tolerance. In addition, the mode of failure seems different between these two indenters.

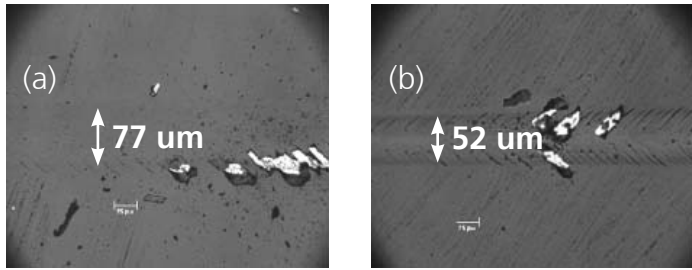


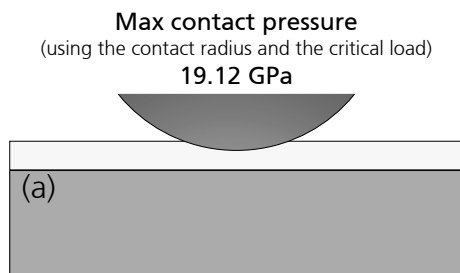
Fig. 11 Scratch width at critical failure point on DLC with indenters A (a) and D (b)

//// Pressure comparison

Finite Element Analysis can be used to calculate the exact contact pressure between an indenter and the surface, using the residual scratch width at failure (measured by optical microscopy), the indenter radius (calculated from Confocal image) and the critical failure load.

Taking into account these three variables results in almost identical values of contact pressure which confirms that the scratch test is a very accurate tool for adhesion measurement of hard coatings, despite limitations in indenter geometry.

> DLC with indenter A (mean on 2 tips)



> DLC with indenter D (mean on 4 tips)

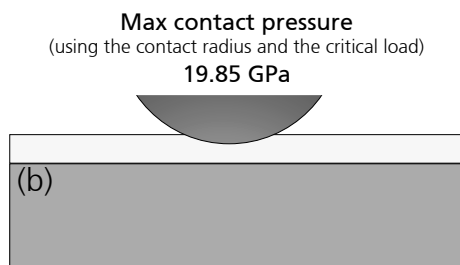


Fig 12: Comparison of contact pressure at critical failure point on DLC with indenters A (a) and D (b)

These results confirm that scratch test critical failure load values can actually be corrected if the exact indenter tip geometry is known.

In light of the findings of this study, CSM Instruments is committed to improving the existing ISO and ASTM standards

by reducing the acceptable tolerances on Rockwell diamond indenters. Whilst awaiting improvements to the standards, CSM Instruments will continue to collaborate with its suppliers in order to provide the most accurate and reproducible indenter geometries available commercially.

The rigorous internal quality control protocols are now applied to all indenters arriving from suppliers, to ensure that only the best indenters are sold to customers, backed up by full certification which includes a 3D confocal scan of the indenter tip and calculation of radius.

//// Conclusions

> CSM Instruments has now established itself as a leader in the routine characterization of Rockwell scratch indenters, using a calibrated Confocal microscope in an automated way.

> The BCR 692 certified reference sample is only useful if highly accurate indenter geometries are used with good sphericity, otherwise significant variations in critical failure load are observed. This material seems to be more susceptible to indenter differences due to its high hardness and modulus.

> This Applications Bulletin is only a preliminary study of an ongoing effort to improve the quality of commercially available Rockwell indenters and will hopefully encourage suppliers to join the project and improve characterization methods.

> The ISO 6508-2 standard is far too broad and inaccurate when applied to indenters used for scratch testing. This suggests that a dedicated Rockwell indenter standard for scratch testing would be more appropriate.

> The 120° cone angle of Rockwell indenters is insignificant for scratch testing.

> Further studies of indenter properties should include the influence of surface roughness, the wear rate and minimum acceptable lifetime.



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

Editor Gregory FAVARO
Dr Nicholas X. RANDALL

Should you require further information, please contact:

CSM Instruments
Rue de la Gare 4
CH-2034 Peseux
Switzerland

Tel: + 41 32 557 5600
Fax: +41 32 557 5610
info@csm-instruments.com
www.csm-instruments.com