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Novel Analysis on the Influence of Tip Radius and Shape of the Nanoindenter on the Hardness of Materials

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Abstract

Nanoindentation is a powerful technique used for assessing mechanical properties at nano/micro-scale. It is used for obtaining material parameters like elastic modulus, hardness, plastic or viscous parameters from experimental readings of indenter load and depth of penetration. Forces involved are usually in the milli or micronewton range and the depth in the order of nanometers. Different kinds of probes can be used for making an imprint into the material surface. Very small material volumes having the order of several tens of nanometers can be accessed with the tip of the nanoindenter and material properties can be evaluated for such a small piece of the materials. Nanoindentation finds application in extracting the elastic and plastic properties of the indented material surface. The effective knowledge of the indenter tip geometry is significant in nanoindentation experimental analysis. A small deviation from the ideal tip geometry affects the accuracy of the result during nanoindentation experiments. In the present investigation, we have studied the influence of tip radius of the indenter on the hardness of for different indenter shapes such as spherical, conical and Berkovich.

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Keywords : Nanoindentation, Elastic modulus, spherical, conical and Berkovich

1. Introduction

The goal of the majority of nanoindentation tests is to extract elastic modulus and hardness of the specimen material from load-displacement measurements. Conventional indentation hardness tests involve the measurement of the size of a residual plastic impression in the specimen as a function of the indenter load. This provides a measure of the area of contact for a given indenter load. In a nanoindentation test, the size of the residual impression is often only a few microns and this makes it very difficult to obtain a direct measure using optical techniques. In nanoindentation testing, the depth of penetration beneath the specimen surface is measured as the load is applied to the indenter. The known geometry of the indenter then allows the size of the area of contact to be determined. The

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procedure also allows for the modulus of the specimen material to be obtained from a measurement of the "stiffness" of the contact, that is, the rate of change of load and depth. In this chapter, the mechanics of the actual indentation test and the nature of the indenters used in this type of testing are reviewed. Nanoindentation has now become a powerful tool to analyze the hardness, elastic properties, dislocation motion, onset of plastic flow in very small volumes, creep resistance and elastic-plastic deformation mechanisms which could be helpful in designing nanodevices [Sheng-Rui Jian et al (2006); Sheng-Rui Jian et al (2008)] and they can be extracted directly from force-displacement measurement. The area of the indent can be indirectly obtained from the depth of penetration and shape of the indenter tip [Arivuoli et al (2000)]. The mechanical properties of these materials are crucial in the device design when reliability issues of the thin film structures are concerned. They exhibit wide applications such as biosensing, fabrication of micromechanical devices, x-ray windows and masks due to their high hardness and Young's modulus. In nanoindentation small loads and tip sizes are used, so the indentation area may only be a few square micrometers or even nanometers. This presents problems in determining the hardness, as the contact area is not easily found. Atomic force microscopy (fig.1) or scanning electron microscopy techniques may be utilized to image the indentation, but can be quite cumbersome.

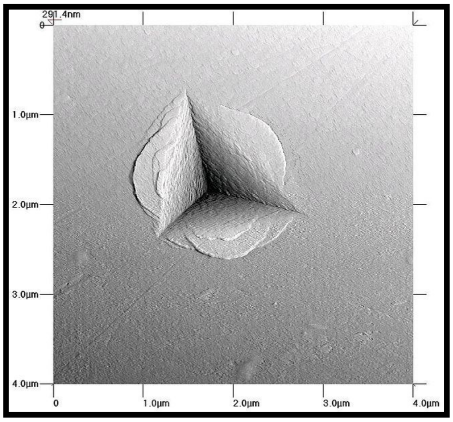


Fig. 1.An AFM image of the residual indent left by a Berkovitch tip during a nanoindentation experiment on a Zr-Cu-Al metallic glass [Jonathan Puthoff et al 2009]

Instead, an intender with a known geometry known to high precision can be employed. Effort is being taken in the present study to identify the influence of tip radius of the indenter on the hardness of materials. The hardness value changes with the three different indenters like berkovich, spherical and conical indenters. The indenter tip radius may provide another reason for the indentation size effect observed in the nanoindentation experiments. The principal components in a nanoindentation experiment are the teat material, the sensors and actuators used to apply and measure the mechanical load and indenter displacement, and the indenter tip. The latter component is conventionally made of diamond formed into a sharp, symmetric shape such as the three sided Berkovich pyramid (figure 2)

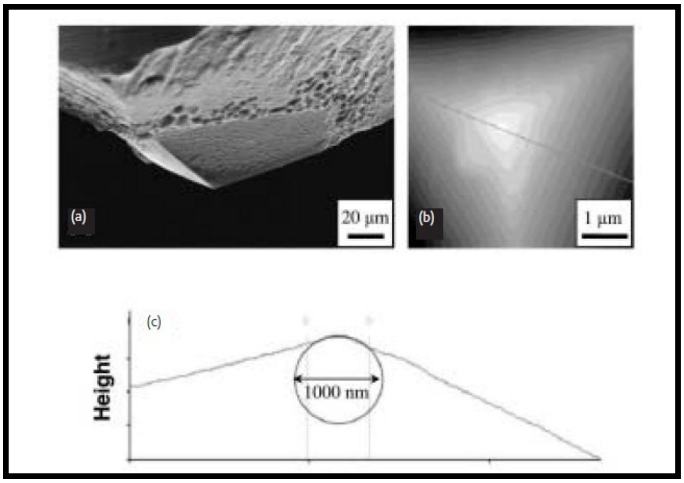


Fig.2 Berkovich diamond geometry commonly used in nanoindentation testing (a)pyramidal diamond tip embedded in braze as observed using a scanning electron microscope (b)Top-down atomic force microscopic image of the tip (c) line scan height profile of the indenter apex geometry along the line marked in (b)

The pyramid shape is chosen at least in part for its nominal geometric self- similarity, which makes for relatively simpler analysis, using the methods of continuum mechanics. Because of the very fine scale of nanoindentation testing, imperfections in the pyramidal tip shape are of paramount importance in analysis and hence much effort has been focused upon methods of characterizing and cataloging tip shapes for more exact quantitative measurements. Of particular relevance in this regard is the nature of the tip apex, which is never atomically sharp and exhibits significant blunting, as shown in the atomic force microscope scan (figure 1c) [Christopher et al (2006)]

2. Types of Indenters

Nanoindentation hardness tests are generally made with either spherical or pyramidal indenters. Consider a Vickers indenter with opposing faces at a semi-angle of $\theta = 68°$ and therefore making an angle $\beta = 22°$ with the flat specimen surface. For a particular contact radius a, the radius R of a spherical indenter whose edges are at a tangent to the point of contact with the specimen is given by $\sin \beta = a/R$, which for $\beta = 22°$ gives $a/R = 0.375$. It is interesting to note that this is precisely the indentation strain1 at which Brinell hardness tests, using a spherical indenter, are generally performed, and the angle $\theta = 68°$ for the Vickers indenter was chosen for this reason. The Berkovich indenter [Berkovich et al (1951)], (a) in Figure.3, is generally used in small-scale indentation studies and has the advantage that the edges of the pyramid are more easily constructed to meet at a single point, rather than the inevitable line that occurs in the four-sided Vickers pyramid. The face angle of the Berkovich indenter normally used for nanoindentation testing is 65.27°, which gives the same projected area-to-depth ratio as the Vickers indenter. Originally, the Berkovich indenter was constructed with a face angle of 65.03°, which gives the same *actual* surface area to depth ratio as a Vickers indenter. The tip radius for a typical new Berkovich indenter is on the order of 50–100 nm. This usually increases to about 200 nm with use. The Knoop indenter, (b) in Figure.3, is a four-sided pyramidal indenter with two different face angles. Measurement of the unequal lengths of the diagonals of the residual impression is very useful for investigating anisotropy of the surface of the specimen. The indenter was originally developed to allow the testing of very hard materials where a longer diagonal line could be more easily measured for shallower depths of residual impression. The cube corner indenter, (**c**) in Figure 3, is finding increasing popularity in nanoindentation testing. It is similar to the Berkovich indenter but has a semi-angle at the faces of 35.26°. Conical indenters have the advantage of possessing axial symmetry, and, with reference to Figure 3, equivalent projected areas of contact between conical and pyramidal indenters are obtained when:

$$A = \pi h_c^2 \tan^2 \alpha \qquad (1)$$

where *hc* is depth of penetration measured from the edge of the circle or area of contact. For a Vickers or Berkovich indenter, the projected area of contact is $A = 24.5h^2$ and thus the semi-angle for an equivalent conical indenter is 70.3°. It is convenient when analyzing nanoindentation test data taken with pyramidal indenters to treat the indentation as involving an axial-symmetric conical indenter with an apex semiangle that can be determined from Eq. 1. Figure gives expressions for the contact area for different types of pyramidal indenters in terms of the penetration depth h_c for the geometries shown in Figure.3

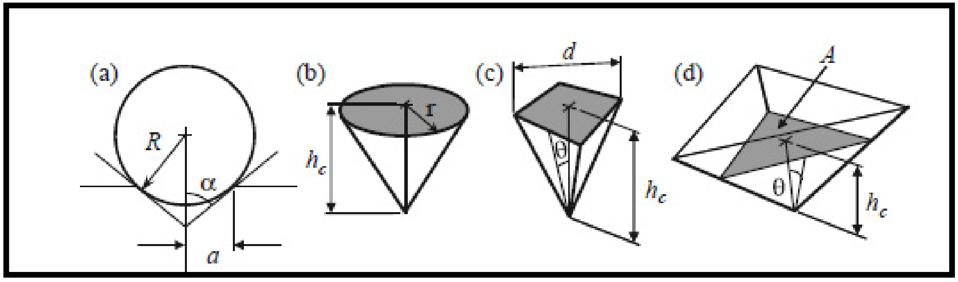


Fig. 3 Indentation parameters for (a) spherical, (b) conical, (c) Vickers, and (d) Berkovich indenters (not to scale)

Spherical indenters are finding increasing popularity, as this type of indenter provides a smooth transition from elastic to elastic–plastic contact. It is particularly suitable for measuring soft materials and for replicating contact damage in in-service conditions.

3. Nanoindentation-The theory Behind

Nanoindentation is an important and popular mechanical experimental technique used in nanomaterial science and nanotechnology. Compared with the other mechanical experimental characterization methods, nanoindentation entails a quite simple setup and specimen preparation. In addition, nanoindentation leaves only a small imprint and can be thus considered as nondestructive. The study of mechanical properties of materials on the nanoscale has gained much attention in recent days, as their properties are size-dependent. The precise analysis of nanomechanical characterization is necessary to use them as structural and functional elements in device fabrication [Joslin et al (1990)]. However, a correct and accurate exploitation of experimental data from nanoindentation tests necessitates a full understanding of the nanoindentation principle and of the assumptions made in carrying out nanoindentation tests. The relationship between the contact area (A) and the depth of indentation (h_i) depends on the exact shape of the indenter and may be experimentally determined by the area function [Joslin et al (1990)], $A= f(h_i)$. The area of contact at full load is determined from the known angle or radius of the indenter. The projected area function $A(h_c)$ for the perfect Berkovich indenter [Oliver et al (1992); Fischer-Cripps et al (2002)] is given by

$$A(h_c) = 24.5h_c^2 \qquad \text{----- (2)}$$

where h_c is the contact depth given by

$$h_c = h_{\max} - \varepsilon P_{\max} S \qquad \text{----- (3)}$$

where P_{max} is the peak indentation load during unloading, ε is a constant that depends on the geometry of the indenter, ε is given as 0.75 and 1 for a Berkovich and spherical indenter, respectively, S is the unloading stiffness and h_{max} is the maximum depth of indentation. The hardness *'H'* is defined as the ratio of maximum load *'P'* to the area of contact *'A'*

$$H = \frac{P_{\max}}{A} \qquad \text{----- (4)}$$

If the tip profile is spherical, the contact area is determined from the nominal radius of the indenter tip and the contact depth [Bell et al (1992)]. Hence, the area is computed from the radius of contact (a) as $A = \pi a^2$. The contact pressure (H) introduced by Meyer called as Meyer's hardness [Meyer et al (1908)] and is given by,

$$H = \frac{P}{\pi a^2} \qquad \text{----- (5)}$$

where *'a'* is the contact radius between the indenter and the sample and it can be given in terms of indenter radius

(*R*) and the indentation depth (*h*), $a^2 = 2Rh_p - h_p^2$ in which h_p^2 can be neglected when the indentation depth is low. Therefore,

$$a^2 = 2Rh_c \qquad \text{----- (6)}$$

Applying the Hertz elastic contact theory [Hertz et al (1882)] to indentation, allows the determination of elastic modulus,

$$E = \frac{3\left(1 - v^2\right)}{4} \frac{P}{R^{\frac{1}{2}} h^{\frac{3}{2}}} \qquad \text{----- (7)}$$

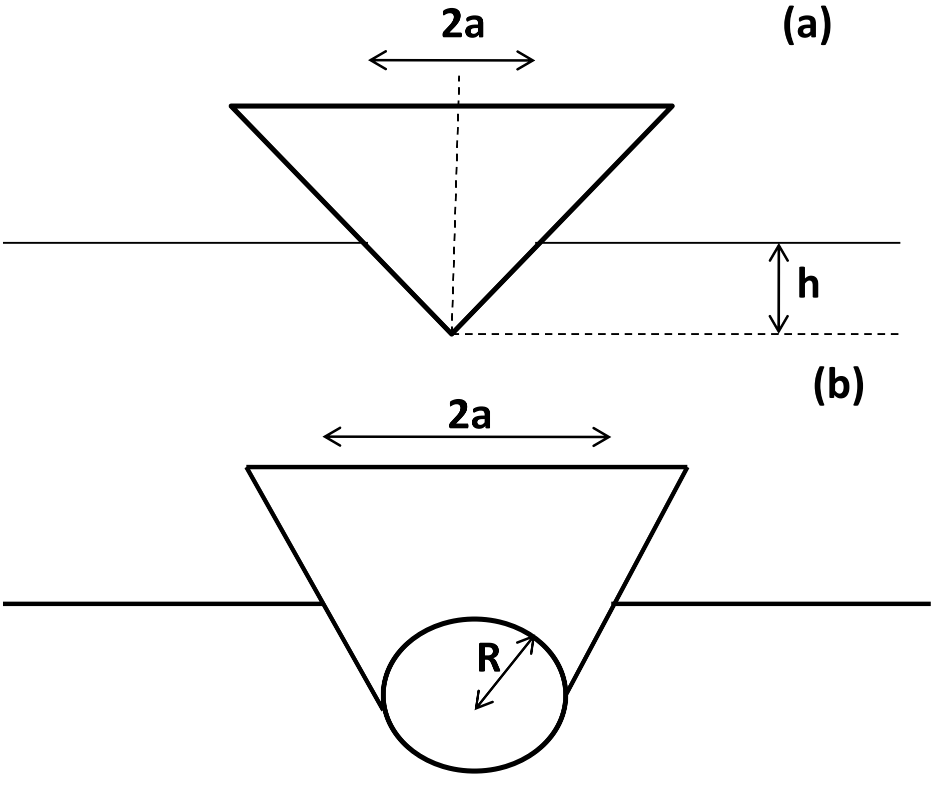
where *P* is the applied indentation load and *R* is the indenter tip radius. In the case of an elastic indenter, $E / (1-v^2)$ in the equation (7) can be replaced by the reduced modulus *E**

$$E^* = \left[\frac{(1 - v_s^2)}{E_s} + \frac{(1 - v_i^2)}{E_i} \right]^{-1} \qquad \text{----- (8)}$$

where '*s*' stands for the sample and *'i'* for the indenter.

4. Nanoindentation - Uncertainty of testing measurements

In atomic force microscopy (AFM) and nanoindentation, optical methods cannot be employed to measure the contact radius directly due to the small contacts. The deviation from the geometry of the indenter tip affects the accuracy of the nanoindentation instrument [Bouzakis et al (2007)]. Apart from this, the reliability of the mathematical model used in interpreting the experimental data, loading and unloading curves which are applied to obtain the mechanical properties of the material under test including hardness, indentation modulus, yield strength, etc also play a vital role in the measurement of uncertainty in nanoindentation testing. The most significant source of uncertainty in nanoindentation measurement is the deviation of the indenter tip from nominal geometry which is widely accepted. The analysis of thin film mechanical properties can be affected by different factors like indentation depth, tip rounding, indenter geometries and coefficient of friction [Tong Hong Wang et al (2007)].



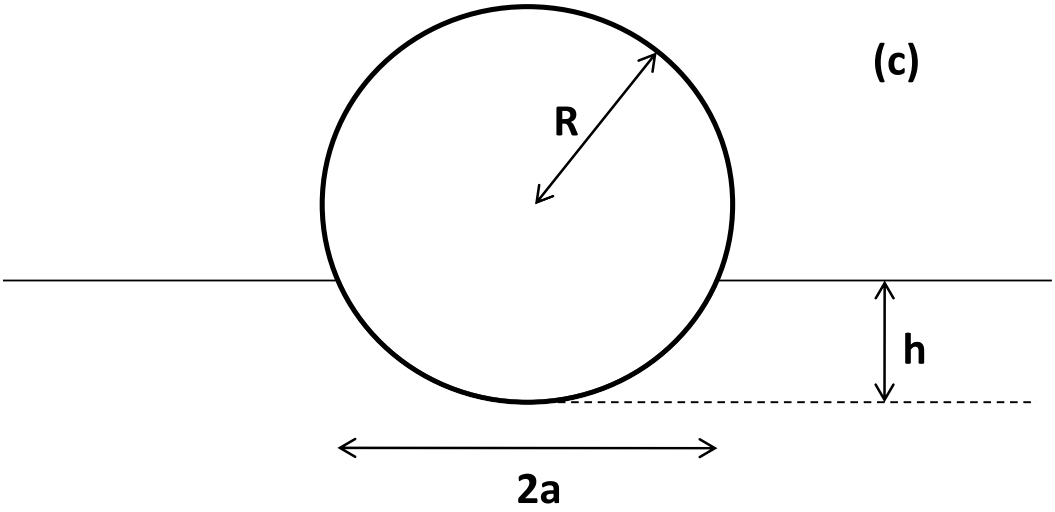


Fig.4 Schematic diagram of different indenters (a) Berkovich indenter (b) Berkovich indenter with worn spherical tip (c) Spherical indenter

5. Nanoindentation - Effect of tip radius

The indenter probe radius can be measured from a number of indents made on a material with the known elastic modulus. It can also be estimated from the two-dimensional images obtained with the help of atomic force microscopy (AFM) or scanning electron microscopy (SEM). As the tip curvature radius is an important parameter in characterizing the mechanical properties, it is necessary to analyze the small deviations in the tip radius geometry. Fig 4 shows the schematic diagram of different indenters (a) Berkovich indenter (b) Berkovich indenter with worn spherical tip (c) Spherical indenter. Generally the indenter tip shape deviates from the ideal tip geometry because of the limitations in the accuracy of the manufacturer. Hence, the indenter tip is not ideally sharp and it could be considered as spherical in some limited region [Bouzakis et al (2007)].The finite element method was used to study the indenter tip radius effect in micro- and nano- indentation hardness and it can be applied to any indentation depth [Qu et al (2004)]. The measured indentation hardness depends not only on the indentation depth but also on the indenter radius [Huang et al (2006)].

6. Indentation size and shape effects

An improved understanding of the indentation size effect (ISE) is the main aim of the recent nanoindentation investigations. Shim et al., [Swadener et al (2002)] reported a new type of indentation size effect depends not on the measured hardness but on the stress necessary to initiate the dislocation plasticity. The authors used ten different spherical indenter tips made out of diamond and sapphire and the tip radii were calibrated by analyzing indentations made in fused silica and sapphire at depths in the range of 5-50 nm. The pop-in data were found to be affected by indenter tip radius and pre-strain. There are two ways in which the initiation of plasticity can be done. The first one is by the activation of mobile dislocations and it happens at low stresses depending on the kind of mechanism of strengthening. The next one is by nucleating dislocations in the dislocation free site which occurs at high stresses near to the theoretical strength of the solid. Hence, the strengths of many materials are greater at small scales when compared with the bulk form. This is well understood by the phenomenon called indentation size effect [Shim et al (2008)] where the hardness value increases with decrease in indenter radius in case of spherical indenters [Weimin Chen et al (2007)] and decrease in depth of indentation in case of conical indenter. Based on the concepts of differential geometry, [Constantinides et al (2007)] proposed a methodology to determine the three-dimensional indenter probe geometry and the method is demonstrated for four cono-spherical indenters with probe radii of the order of 1-10 µm. The non-ideal nature of the probe is also clearly demonstrated in their study. They emphasized the deviation of the radii with manufacturer specification and observed that all the four probes deviate from the assumed spherical geometry. The effects of indenter size and pre-existing dislocation density on the onset of plasticity can be studied with the help of pop-in phenomenon or sudden displacement burst

[Calabri et al (2008)].

7. Young's Modulus *E* and the Hardness *H*

In order to determine mechanical parameters, such as the hardness *H* or Young's Modulus *E*, the tip of the nanoindentor penetrates into the material. During this load-unload cycle the load *P* and the displacement *h* are measured.

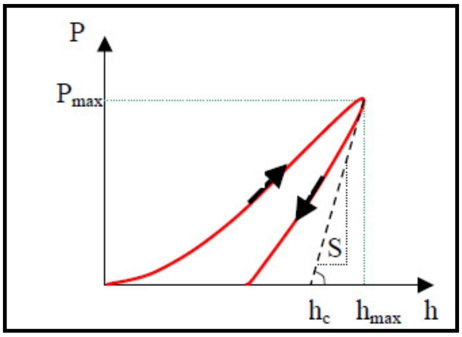


Fig.5 Load *P* versus displacement *h* curve

Later on the hardness *H* can be calculated with the following definition:

$$H = \frac{P_{\max}}{A_c} \tag{9}$$

where P_{max} is the maximum applied load and *Ac* the contact area of the tip on the surface of the material. The contact area can be calculated based on the contact depth h_c Young's Modulus is calculated based on the Sneddon equation:

$$E^* = \frac{\sqrt{\pi}}{2\beta} \cdot \frac{S}{\sqrt{A_c}} \tag{10}$$

where *E** is the composite Young's modulus depending on both Young's modulus and Poisson's ratio of test material and of tip material, *S* is the contact stiffness and β a correction coefficient near to 1 (β=1.034 for a Berkovich tip). Based on the results obtained for *E**, Young's Modulus of the test material *E* can be calculated, where "*i*" is the subscript for the properties of the indenter:

$$\frac{1}{E^*} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i} \tag{11}$$

8. Contact Area (A_c) and Stiffness (*S*)

The calculation of contact area (*Ac)* is based on the measurement of the penetration depth during load *hc*. To take into account the tip defects, Oliver and Pharr have proposed the use of a polynomial function of contact depth [Oliver et al (1992)]:

$$A_c = C_0 h_c^2 + C_1 h_c + C_2 h_c^{1/2} + C_3 h_c^{1/4} + C_4 h_c^{1/8} \tag{12}$$

where C_i are the tip coefficients. C_0 is a value given by the tip supplier; the other coefficients depend on the tip abrasion and have to be determined regularly. The value of *hc* can be determined by the following equation:

$$hc = h - \varepsilon \frac{P}{S} \tag{13}$$

where *h* is the displacement of the tip, *P* is the applied charge and ε a constant (*ε* = 0.75 for a Berkovich tip). The contact stiffness can be calculated by differentiation of the unload part of the experimental curve. This analytical expression has been proposed by [Oliver and Pharr (1992)]:

$$S = \left.\frac{dp}{dh}\right|_{h-h_{\max}} = Bm(h_{\max} - h_f)^{m-1} \tag{14}$$

where h_{max} is the maximal penetration depth, B and m are experimental coefficients determined by interpolation and h_f the depth of the print that remains on the surface after tip removal. A second technique to determine the contact stiffness S has been developed during the last years: the Continuous Stiffness Measurement (CSM). This technique is an important improvement for measuring the contact stiffness. The indenter is driven during loading by superposing a small oscillating force on the primary load signal and the resulting harmonic response is analyzed. The technique is based on an accurate model for the dynamic response of the indentation system [Hay et al (2000); Xiaodong et al (2002)]. As far as S is measured continuously, one can obtain the hardness and Young's modulus as a continuous function of depth.

Conclusion

We have discussed here the effects of the indenter tip radius on the nan indentation hardness. In several important cases, accurate determination of the indenter tip radius is needed for the correct interpretation of the experimental results. We have used the numerical calculations to understand how the hardness value is influenced by the change in indenter tip radius caused by tip rounding effect and also we have discussed the various theoretical calculations to determine the hardness value for different indenter tip radius for three different types of indenters like spherical, berkovich and conical. Experimentally the hardness value increases with increasing tip radius, which can be explained on the basis of indentation size effect.

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