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AN AREA FUNCTION MEASUREMENT METHOD BASED ON INDENTER GEOMETRY FOR SMALL-DEPTH NANOINDENTATION

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Abstract: The measurement methods of area function that are commonly used in nanoindentation are summarized and analyzed. An area function determination method based on the geometry of indenter tip for very small indentation depth is proposed. It combines the advantages of the area function measurement method based on measurement of standard sample and that based on indenter geometry. Experiments have been carried out to examine the effect of the area function. It is shown that this area function measurement method can improve the measurement accuracy of indentation hardness and reduced modulus when the indentation depth is very small.

Keywords: Area function, Nanoindentation, Indentation hardness, Reduced modulus, Measurement.

1. INTRODUCTION

Nanoindentation is an important technique for measuring the mechanical properties of materials at micro- and nano-scale. Various nanoindentation instruments have been widely used in determination of indentation hardness, reduced modulus and other mechanical properties of material. The basic principle of nanoindentation test is to perform indentation on the sample surface using an indenter with known shape, measure the load applied on the indenter and the contact depth between the indenter and the sample continually during the indentation, and then determine the indentation hardness and reduced modulus of material according to the test results [1]. The relationship between the load and displacement of indenter in a nanoindentation test is represented schematically in Fig. 1. By analyzing the unloading curve, the indentation hardness, H, and reduced modulus, E_r, can be obtained according to the following equations.

$$H = \frac{P_{\max}}{A} \qquad (1)$$

$$E_r = \frac{\sqrt{\pi}}{2\beta}\frac{S}{\sqrt{A}} \qquad (2)$$

where $P_{\max}$ is the maximum load, A is the projected contact area between the indenter and material, $S = \left(dP/dh\right)_{h=h_{\max}}$ is the stiffness of unloading data at maximum depth and β is a constant related to the indenter geometry. It is shown in the above equations that the projected contact area is one of the most important parameters in the measurements of indentation hardness and reduced modulus by the nanoindentation method. The relationship between the projected contact area and the contact depth, which is the distance from the tip of the indenter, is expressed by the area function. Nanoindentation test are generally made with pyramidal or spherical indenters. The geometry of indenter tip may deviate from the designed shape because of machining error and wearing in application. Typically, there exists a radius of curvature on the tip of a pyramidal indenter. Since the indentation depth is generally small in nanoindentation test, the effect of tip shape deviation on the projected contact area is significant [2]. In order to ensure the accuracy of nanoindentation test results, the accurately determination of indenter area function is necessary, and how to realize this has attracted wide attention of researchers [3-9].

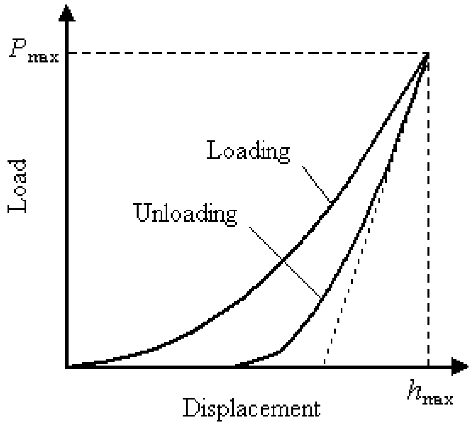

Fig. 1 Schematic representation of load-displacement curve of nanoindentation test

In this paper, the commonly used measurement methods of area function are reviewed. On the basis of analyzing the advantages and disadvantages of different methods, a method for determining the area function at very small indentation depth based on the indenter geometry is proposed. It is realized by combining the conventional measurement method of area function and the indenter geometry. Experiment results have shown that this method can improve the measurement accuracy of the indentation hardness and the reduced modulus of material at small indentation depth.

2. MEASUREMENT METHODS OF AREA FUNCTION

The methods for determining the area function of indenter, which are most commonly used in nanoindentation test, can be divided into three types. The first is the method based on the measurement of standard sample and the iteration process [3, 4]. The second is the direct measurement method based on the three-dimensional topography measurement technique [5, 6]. The third is the method based on the measurement of indenter geometry [7, 8]. The first two methods are recommended as the area function calibration methods in ISO 14577 [10, 11].

2.1 The method based on measurement of standard sample

When Oliver and Pharr [3] studied the technique for determining hardness and elastic modulus using load and displacement sensing indentation, they proposed a method for determining the indenter area function by means of an iteration process based on the indentation test data of standard samples. They used aluminium and fused quartz as standard materials to determine the load frame compliance and the diamond area function. First, they performed a series indentation on the aluminium sample and processed the data using an iteration process to determine the load frame compliance and indenter area function. Then they performed indentation test on the fused quartz sample to determine the area function of indenter at small indentation depths. The area function of a Berkovich indenter obtained by this method can be expressed as

$$A = 24.5h_c^2 + C_1h_c^1 + C_2h_c^{1/2} + C_3h_c^{1/4} + \ldots\ldots + C_8h_c^{1/128} \qquad (3)$$

where C_1 through C_8 are constants. The lead term in Eq. 3 describes a perfect Berkovich indenter, the following terms represent the shape deviation of the indenter. Similar methods of area function measurement have been widely used in nanoindentation tests. For the instrument that the load frame compliance has been calibrated, the area function can be obtained directly by the measurement of a standard sample with known reduced modulus.

2.2 The method based on three-dimensional topography measurement technique

The area function measurement method based on the three-dimensional topography measurement technique is to measure the residual indentation or the indenter directly to determine the projected contact area between the indenter and material using the scanning probe microscope or similar instruments. Barone et al. [5] studied the direct measurement method of area function using atomic force microscopy (AFM). They used an MFP3D instrument to perform a measurement of the three-dimensional topography of a Berkovich indenter. Fig. 2 shows an image they obtained with a sweep range of 10 μm. The data were processed and the areas of the sections orthogonal to the pyramid axis at different heights were determined with an accuracy step of one nanometer up to a depth of 25 nm, and then at increasing steps up to 1000 nm. Herrmann et al. [6, 12] measured a Berkovich indenter by using the scanning force microscopy (SFM). They used the measurement data to calculate the regression curves, and obtained an area function as shown in Eq. 4.

$$\sqrt{A} = ah_c + b \qquad (4)$$

where a and b are regression coefficients.

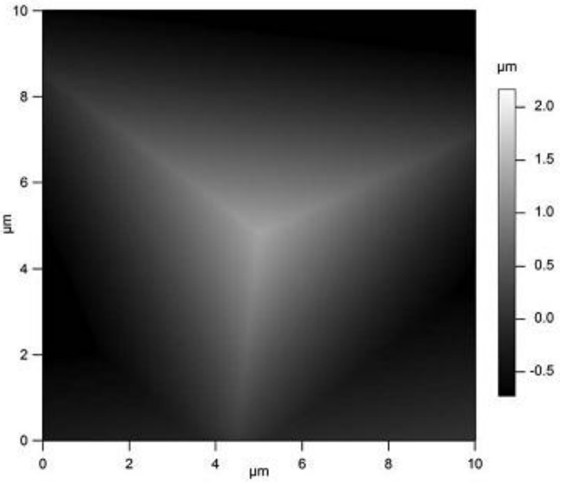

Fig. 2 AFM image of a Berkovich indenter [5]

2.3 The method based on indenter geometry

This area function measurement method determines the indenter area function directly from the geometry of the indenter tip. Thurn and Cook [7] took the indenter profile as a harmonic average of a conical tip and a spherical tip, and derived an area function as shown in Eq. 5.

$$A = \frac{\pi h_c^2}{\cot^2\alpha} + 4R\pi h_c + 4R^2\pi\cot^2\alpha \qquad (5)$$

where α is the effective included half-angle and R is the radius of curvature of the indenter tip. The first term in Eq. 5 describes a perfect indenter, and the latter two describe the effects of tip rounding. Liu and Zhang [8] also proposed a method based on the indenter geometry to create the area function. They took the indenter as a geometry shown in Fig. 3, where the behaviour of indenter is modelled by that of the cone with an included half angle that gives the same area-to-depth relationship. A similar area function was obtained, as shown in Eq. 6.

$$A = \frac{\pi h_c^2}{\cot^2\alpha} + \frac{2\pi RBh_c}{\cot\alpha} + \pi R^2B^2 \qquad (6)$$

where α is the equivalent cone half angle, R is the radius of curvature of indenter tip, and B=(1-sinα)/cosα.

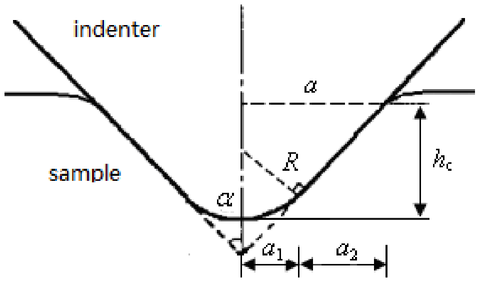

Fig. 3 Schematic diagram of indenter geometry with tip-rounding [8]

2.4 Comparison of the methods

Three types of commonly used measurement methods of indenter function have been reviewed briefly above. Each of them has advantage and limitation. The first method can obtain the area function by performing the indentation test on the standard materials and analyzing the test data. It can be implemented by using the nanoindentation instrument itself without requirement for other equipment. However, it needs to determine the load frame compliance of the instrument accurately, and the polynomial area function may have limited accuracy for the very small contact depth. The second method can realize the traceable measurement of area function by using a calibrated AFM. However, it needs to use atomic force microscope or other complex micro-scale imaging equipment in a good laboratory environment. The third method establishes a direct relationship between the projected contact area and the radius of curvature of the indenter tip. The parameters in the area function have clear physical meanings. However, this method also requires accurate measurement of the radius of curvature of indenter, which is not easy to realize, and the tip radius may change with the wear in application.

3. A NEW MEASUREMENT METHOD OF AREA FUNCTION FOR SMALL DEPTH INDENTATION

In order to improve the measurement accuracy of the mechanical property of material by nanoindentation test, the area function of indenter needs to be calibrated regularly. Thus, it is important to look for a simple and fast method of area function measurement. As is shown from the analysis in 2.4, the measurement method of area function based on the measurement of standard sample is easy to realize. However, it is found in practical application that the accuracy of projected contact area for the very shallow indentation is limited. In contrast, the measurement method based on indenter geometry can give a better description of the effect of tip rounding on the projected contact area at very small indentation depth, but it is difficult to measure the radius of curvature of indenter. Based on these analyses, a measurement method of area function, which integrates the indenter geometry with the measurement of standard sample, is proposed in this paper.

3.1 The procedures for determining area function

The new measurement method of area function can be realized using the following procedures. First, an area function including the geometrical information of indenter, such as the radius of curvature of indenter tip and the characteristic angle of indenter, and the contact depth between the indenter and material is established. Next, the area function of the indenter is calibrated using the conventional method by measuring a standard sample using the nanoindentation instrument to get an area function expressed by a polynomial. Third, the area function based on indenter geometry is fitted to the polynomial area function using the least squares method to determine the radius of curvature and the angle of the indenter. Finally, the new area function based on indenter geometry is used to replace the polynomial one at the very small indentation depth.

The new area function established in this way consists of two parts. For most of the measurement range, the area function is a polynomial obtained by measuring the standard sample. For the very small contact depth, it is a function of indenter geometrical parameters, which is obtained by fitting to the polynomial. This measurement method of area function combines the advantages of both the method based on indenter geometry and that based on the measurement of standard sample.

3.2 Establishment of area function based on indenter geometry

In this paper, a Berkovich indenter is selected to study the measurement method of area function. A perfect Berkovich indenter has a shape of regular triangular pyramid with a face angle of 65.3°. Due to the machining error and wearing in application, the tip geometry may deviate from the design shape to have a radius of curvature.

In the establishment of area function, the shape of indenter tip is supposed to be a sphere that is tangent with the indenter, as shown in Fig. 4. The indenter can be described by two parameters, the radius of curvature of indenter tip, *R*, and the angle between the edge and axis of the probe, α. In this figure, T is the tangent point between the indenter edge and the sphere, and J is the intersection of the indenter face and the sphere.

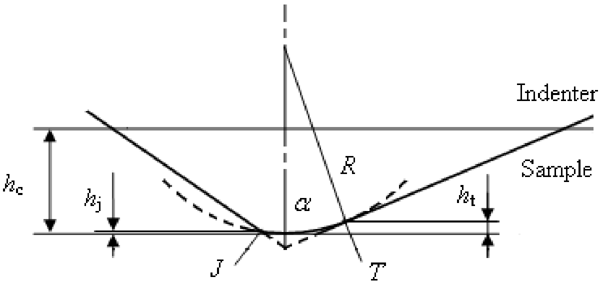

Fig. 4 Schematic representation of cross section of Berkovich indenter

According to the indenter geometry, the area function of Berkovich indenter can be expressed as

$$A = \begin{cases} \frac{3\sqrt{3}}{4}(\tan\alpha)^2\left(h_c + \frac{1-\sin\alpha}{\sin\alpha}R\right)^2 & h_c \ge h_t \\ \left[\pi - 3\arccos\left(\frac{b}{a}\right)\right]a^2 + 3b\sqrt{a^2-b^2} & h_j < h_c < h_t \\ \pi(2Rh_c - h_c^2) & h_c \le h_j \end{cases} \tag{7}$$

where

$$a = \sqrt{R^2 - (R-h_c)^2} \tag{8}$$

$$b = \frac{h_c\sin\alpha + (1-\sin\alpha)R}{2\cos\alpha} \tag{9}$$

$$h_t = (1-\sin\alpha)R \tag{10}$$

$$h_j = \frac{(3-2\sqrt{3})\sin\alpha + 4 - 2\sqrt{3}}{(\tan^2\alpha + 4)(\sin\alpha + 1)}R \tag{11}$$

In Eq. 7, *A* is the projected contact area, *R* is the radius of curvature of indenter, α is the angle between the edge and the axis of indenter, and h_c is the contact depth between the indenter and the sample.

4. EXPERIMENT AND RESULTS

An experiment was carried out to establish the area function based on indenter geometry for small contact depth and examine the effectiveness and practicability of the method.

4.1 Calibration of indenter area function

In this experiment, the area function of a Berkovich indenter was calibrated firstly on a nanoindentation instrument, the load frame compliance of which was calibrated previously. To determine the area function, a series of indents at various contact depths were performed in a fused quartz sample of known elastic modulus. During the indentation test, a load control load function with a 5 s loading time, 2 s holding time and a 5 s unloading time is used. The peak load changes from 10 mN down to 100 μN in the test to create indents of different contact depths. A load function with peak load of 10 mN is shown in Fig. 5 as an example. Then an automated testing method of the instrument was run to make a 10 by 10 array of indentation on the fused quartz sample. The contact depths of indents obtained in the test were in the range of around 8.0 nm to 176.2 nm. After the indentation process had been completed, the measurement data were analyzed to calculate the projected contact area at different contact depths.

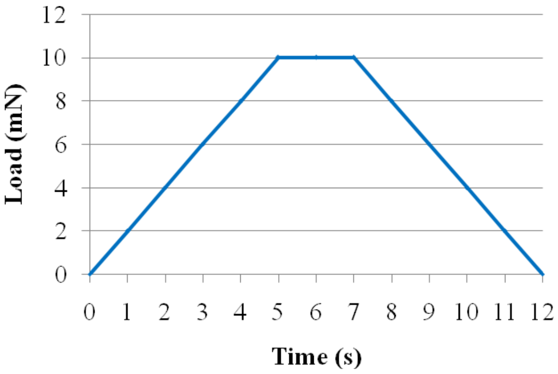

Fig. 5 Load function

An area function expressed as a six order polynomial was obtained as below by fitting the curve of projected contact area, A, versus contact depth, h_c.

$$A = 24.5h_c^2 + \sum_{i=1}^{5} C_i h_c^{1/2^{i-1}} \qquad (12)$$

where, C_1=25588.57, C_2=-2075867.17, C_3=22979610.46, C_4=-60465349.35 and C_5=39907986.34. As in Eq. 3, the first term in this equation describes a perfect Berkovich indenter and the following terms represent the shape deviation of the indenter. The measurement result of area function is shown in Fig. 6.

4.2 Determination of geometrical parameters of indenter

In order to determine the radius of curvature and the angle of the indenter, the area function based on indenter geometry is fitted to the area function obtained by measurement of standard sample. Since the polynomial area function is generally not accurate enough for the very small contact depths, the data in such area can be removed in the fitting.

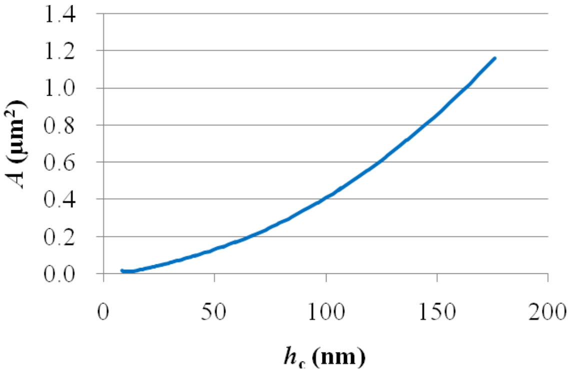

Fig. 6 Measurement result of area function

In this experiment, only the data in the range of h_c >40 nm were used in the fitting process. By fitting the equation in the first line of Eq. 7 to the area function given in Eq. 12 using the least squares method, the radius of curvature and the angle of indenter are obtained to be 631 nm and 78.68°, respectively. Substituting the results to Eq. 7, the new area function was established. The new area function is shown in Fig. 7 along with the area function given by Eq. 12 for the contact depth below 40 nm. It can be seen by comparing the two area functions that the projected contact area given by Eq. 7 is obviously larger than that given by Eq. 12 for the contact depth between 8 nm and 25 nm.

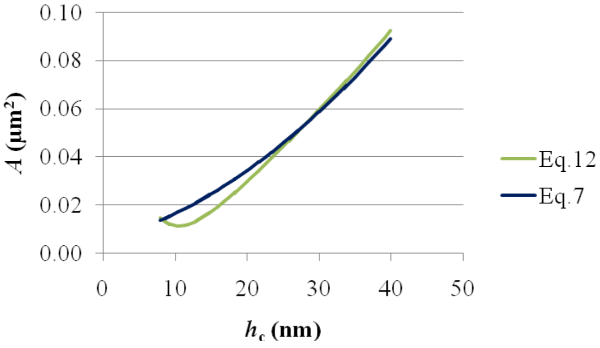

Fig. 7 Difference between two area functions for contact depth below 40 nm

4.3 Measurement of indentation hardness and reduced modulus

The indentation hardness and reduced modulus of the fused quartz sample were measured using the area function shown in Eq. 12. The plots of the hardness and reduced modulus versus contact depth are shown in Fig. 8 and Fig. 9, respectively, by the data series 1. It can be seen from the two figures that the measurement values are stable when the contact depths are larger than 40 nm. The average values in this area are 8.85 GPa for hardness and 69.64 GPa for reduced modulus, which conforms to the given values of the sample. However, when the contact depth is below around 40 nm, especially when below 30 nm, the measured values of the indentation hardness and reduced modulus are significantly different from that at larger contact depths. The maximum deviation of indentation hardness is about 56.3%

and the maximum deviation of reduced modulus is about 40.6%, which occur at the contact depth of 12.9 nm. It can be seen that the measurement results of indentation hardness and reduced modulus are unreliable for the very small contact depths. The measurement error is mainly resulted from that the values of projected contact area are not accurate enough at these very small contact depths due to the tip rounding of indenter.

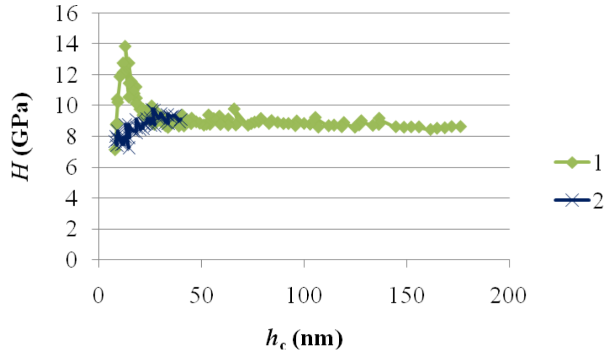

Fig. 8 Measurement result of indentation hardness

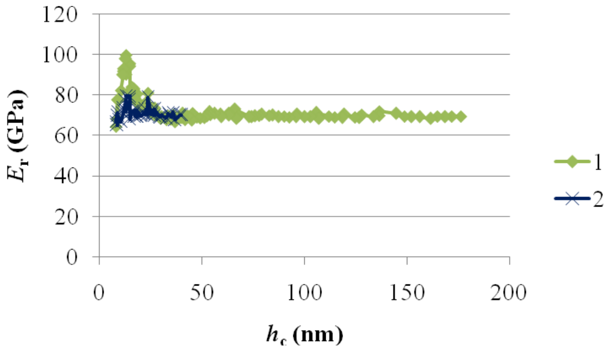

Fig. 9 Measurement result of reduced modulus

By using the area function shown in Eq. 7 to replace the area function shown in Eq. 12 for the contact depth smaller than 40 nm, the indentation hardness and reduced modulus were recalculated from the test data. The new measurement results for the contact depth below 40 nm are also shown in Fig. 8 and Fig. 9 by the data series 2 for comparison. It can be seen from the two figures that the indentation hardness and the reduced modulus obtained by this new area function becomes much closer to the stable values compared to the previous one. The maximum deviation of indentation hardness from the stable value is reduced from 56.3% to 18.0%, and the maximum deviation of reduced modulus from the stable value is reduced from 40.6% to 14.0%. Thus, the measurement accuracy at small indentation depth can be improved by using the new area function.

It is indicated from the experiment results that the new area function is helpful to improve the accuracy of measurement results of the indentation hardness and reduced modulus for the very small contact depths.

5. CONCLUSIONS

Based on the summarization of the existing measurement methods of area function in nanoindentation tests and the analysis of their characteristics, a method for determination of the area function for very small depth indentation is presented in this paper. The new area function relates the projected contact area to the radius of curvature and the angle of indenter. It is established by assuming that the indenter tip has a shape of sphere that is tangent with the indenter, and determining the parameters by fitting to the area function obtained by traditional method. It combines the advantages of both the commonly used area function calibration method and the method based on indenter geometry. Experiment results have shown that this measurement method of area function can improve the accuracy of area function, and thus improve the measurement accuracy of indentation hardness and reduced modular, for very small depth indentation. The method is valuable for high-accuracy measurements of mechanical properties of material at nano-scale.

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