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Calculation of Indent’t Real Contact Projection Area in Nanoindentation

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Abstract. Real contact projection area is a key parameter of the evaluation models of the materials’ mechanical properties using nanoindentation. If the geometrical parameters of the indenter are known, the real contact projection area may be determined based on the relation between the real contact depth determined according to the unloading curve and the geometrical parameters of the indenter and the real contact projection area. If the real geometrical parameters of the indenter are not known, the real contact projection area is obtained using testing method. In this paper, two kinds of testing methods, the standard block method (including the standard elastic modulus block method and the standard hardness block method) and the non-standard block method, are introduced for the determination of the real contact projection area.

1. Introduction

With the rapid development of information technology and MEMS, the application fields of thin films are wider and wider. The mechanical properties of thin films have much influence on the design, manufacturing and performances of products. Since the thickness of thin films is smaller and smaller, even the thickness of thin films is less than 10 nanometers, so the measurement of the mechanical properties of thin films will face great challenge. Even though there are many measuring methods of the properties for thin films, such as nanoindentation, micro beam bending, blister, micro tensile and so on, each measuring method has its limits in application. Because it is easy to install samples and without the special requirement for sample’s dimensions and shapes, nanoindentation has gained wide application in the measurement of the mechanical properties of thin films. Nanoindentation consists of loading and unloading processed, the principle of which is that indenter is loading on the sample based on the constant displacement or load step under the driving of the loading mechanism on the loading processes till the loading process ends when the pre-setting depth or load is realized, and then the unloading process starts on the constant displacement or load step under the driving of the loading mechanism until there is no force action between indenter and sample. On the loading and unloading processes, the synchronous measurement of the loading depth and the force of the indenter is needed by using the displacement and force sensors. Based on the unloading curve, the unloading process...
stiffness and the real contact depth may be determined, and the real contact projection area may be calculated on the basis of the real contact depth. According to the unloading stiffness, the real contact projection area, the Poisson’s ratio of the sample, the elastic modulus and the Poisson’s ration of indenter and substrate, the hardness of the sample can be evaluated by the related equation, and the elastic modulus of the sample may be gained on the basis of the evaluating models of the elastic modulus, promoted by Oliver and Pharr[1-2]. There are many influence factors on the measurement of the mechanical properties by using nanoindentation [3], and the real contact projection area is one of many important parameters in the evaluating models. For the calculation of the real contact projection area, if the parameters are known, the real contact projection area may be directly computed on the basis of the gained real contact projection area and the geometrical parameters of the indenter, and if the actual geometrical parameters of the indenter deviate from its ideal parameters and are unknown, the real contact projection area will be determined by using the polynomial of the real contact depth.

2. Calculation of the real contact projection area for the known parameters of the indenters

2.1. For the ideal indenter

In nanoindentation, Vickers, Knoop, Berkovich and Cone are the several commonly used indenters. When the used indenter is ideal and indented vertically, if the real contact depth is \( h_c \), the real contact projection area is as follows,

\[
A = \begin{cases} 
\frac{\pi h_c^2 \tan^2 \frac{\alpha}{2}}{2} & \text{for cone indenter} \\
\frac{4 h_c^2 \tan^2 \frac{\alpha}{2}}{2} & \text{for Vickers indenter} \\
\frac{4 h_c^2 \tan^2 \frac{\alpha_1}{2} \tan^2 \frac{\alpha_2}{2}}{2} & \text{for Knoop indenter} \\
\frac{9 \tan 30^\circ h_c^2 \tan^2 \alpha'}{2} & \text{for Berkovich indenter}
\end{cases}
\]

(1)

Where \( \alpha, \alpha_1 \) and \( \alpha_2 \) are the angle parameters of the indenter, \( \alpha' \) is a derived parameter of Angle \( \alpha \) of the Berkovich indenter.

2.2. For the un-ideal indenter

From the evaluating models of hardness and elastic modulus, their evaluation is directly related to the real contact projection area between indenter and sample at the maximum load. When the geometrical sizes and shape as well as installation of the indenter are ideal, after the real contact depth \( h_c \) is got, the real contact projection area \( A \) may be determined on the basis of Eq. (1). In fact, even though the manufacturing and measuring precision is very high, that the actual geometrical sizes and shape of the indenter deviate from its ideal state certainly exists. Taking example for Vickers indenter, there may exist rounding case (See Figure 1) or flattting case(See Figure 2) on its tip, and when the real contact projection area \( A \) is calculated, first, a new displacement series \( h_{i+1} \Delta h_i \) or \( h_{i+1} \Delta h_{ip} \), \( i = 1 \sim n_U \) is calculated, and then the area \( A \) may be determined on the basis of the new displacement series, where \( n_U \) is the sampling point number on the unloading process, \( \Delta h_i \) and \( \Delta h_{ip} \) can be calculated by Eq. (2).

\[ h_{i+1} \Delta h_i = h_{i-1} \Delta h_i = \frac{\Delta F_{max}}{E} 
\]

\[ h_{i+1} \Delta h_{ip} = \frac{\Delta F_{max}}{E} \]

Figure 1. Rounding of indenter tip.  
Figure 2. Flattting of indenter tip.
where the parameters are seen on Figures 1 and 2.

The half vertex angle \( \alpha/2 \) of the tip also has a half vertex angle deviation \( \Delta \alpha/2 \), its actual half vertex angle \( \alpha_a/2 = \alpha/2 - \Delta \alpha/2 \). The Vickers indenter has four half vertex angles, that is, \( \alpha_1, \alpha_2, \alpha_3 \) and \( \alpha_4 \), the theoretical values of which are \( \alpha_1 = \alpha_2 = \alpha_3 = \alpha_4 = \alpha/2 = 68^\circ \). When \( \alpha_1 = \alpha_2 = \alpha_3 = \alpha_4 = \alpha/2 = 68^\circ \), or when \( \alpha_1 \neq \alpha_2 \neq \alpha_3 \neq \alpha_4 \), \( \alpha_a/2 = (\alpha_1 + \alpha_2 + \alpha_3 + \alpha_4)/4 \). Let \( \alpha_a/2 \) substitute for \( \alpha/2 \) on Eq. (2), and the real contact projection area \( A_{\alpha_a/2} \) may be gained.

When the axis of the indenter is loading on the surface of the sample un-vertically, the real contact projection area can’t be calculated by Eq. (2). Since the axis may be inclined in the different orientations, it’ll induce the calculation of the area to be very complicated. Therefore, the calculation method of the real contact projection area \( A_1 \) is introduced only for the angle \( \beta \) between the actual axis in the axial middle section of the opposite sides at the bottom of the Vickers indenter and the ideal axis of the indenter, as shown in Figure 3. As known from Figure 3, when the indented depth is \( h_{ii} \), the real contact projection area

\[
A_1 = \frac{(a_1 + h_{ii})(a_2 + b_2)}{2}
\]

where

\[
a_1 = h_{ii} \tan \frac{\alpha}{2} \left[ \left( 1 - \tan \theta \tan \frac{\alpha}{2} \right) \cos \frac{\alpha}{2} \right]^{-1}, \quad b_1 = h_{ii} \tan \frac{\alpha}{2} \left[ \left( 1 + \tan \theta \tan \frac{\alpha}{2} \right) \cos \frac{\alpha}{2} \right]^{-1}
\]

\[
a_2 = h_{ii} \tan \frac{\alpha}{2} \left[ \left( 1 - \tan \theta \tan \frac{\alpha}{2} \right) \cos^2 \frac{\alpha}{2} \right]^{-1}, \quad b_2 = h_{ii} \tan \frac{\alpha}{2} \left[ \left( 1 + \tan \theta \tan \frac{\alpha}{2} \right) \cos^2 \frac{\alpha}{2} \right]^{-1}
\]

where the meaning of the parameters is seen in Figure 3.

\[\Delta h_{ii} = \left( \sin^{-1} \frac{\alpha}{2} - 1 \right) \]

\[\Delta h_{pi} = 0.5 \alpha \tan^{-1} \frac{\alpha}{2}\]

\[\text{(2)}\]

\[\text{Figure 3. Schematic of calculating } A_1.\]

When the geometrical sizes and shape of other types of indenters deviate from their ideal states, the calculation of their real contact projection areas may be determined according to the above principle.
3. Calculation of the real contact projection area for the unknown parameters of the indenters

Since nanoindenter is a very precise measuring instrument and the manufacturing and installing precision of its indenter is also very high, it is very difficult to measure the geometrical sizes, the form errors and the installing errors of the indenter. On the other hand, the mechanical properties of the indenter also have some influence on the real contact projection area. Therefore, the real contact area may be calculated based on the standard block method and the non-standard block method.

3.1. Standard block method

3.1.1. Standard elastic modulus block method. Suppose that a standard sample block with an elastic modulus $E_s$, which is invariable with the change of the indented depth, is indented $n$ times by using a rigid indenter or an indenter with an elastic modulus $E_i$ and a Poisson’s ratio $\nu_i$, a group of data series $h_i$, $S_i$, $i=1$~$n$ for the real contact depth and the unloading stiffness may be obtained after data processing. For the rigid indenter, the real contact projection area gained in the $i$th experiment is as follows.

$$A_{ci} = \frac{\pi}{4} \left( \frac{1 - \nu^2}{\beta E_s} \right)^2 S_i^2, \quad i = 1 \sim n \quad (4)$$

For a non-rigid indenter, the standard complex modulus $E_{sr}$ should be determined, and then the real contact projection area gained in the $i$th experiment is as follows.

$$A_{ci} = \frac{\pi}{4} \left( \frac{1}{\beta E_{sr}} \right)^2 S_i^2, \quad i = 1 \sim n \quad (5)$$

In Eqs. (4) and (5), $\beta$ is a parameter related to the indenter’s type, $\nu$ is the Poisson’s ratio of the standard sample, and the unloading stiffness and the real contact depth are calculated on the basis of Eqs. (6) and (7), respectively.

$$S = \frac{dP}{dh} |_{h=h_{max}} \quad (6)$$

$$h_c = h_{max} - \xi P_{max} S^{-1} \quad (7)$$

where $h_{max}$ and $P_{max}$ are the maximum indented depth and the maximum load, and $\xi$ is a parameter related to the indenter’s type. From Eq. (6), the unloading stiffness $S$ is a slope of the tangent of the unloading curve at the maximum indented depth.

Oliver and Pharr introduced a function of calculating the real contact projection area by using the real contact depth for Berkovich indenter on the basis of the constant elastic modulus method. The function has obtained a wide application in determining the real contact projection area according to the real contact depth. If the real contact depth $h_c$ and the real contact projection area $A_c$ satisfy the following relation [5],

$$A_c = A(h_c) + \sum_{j=1}^{N} B_j h_c^{2(j-1)} \quad (8)$$

where $A(h_c)$ is determined by using Eq. (1), the coefficients $B_j$, $j=1$~$N$ are the undecided ones, which can be determined by using $h_{ci}$, $A_{ci}$, $i=1$~$n$. Suppose that

$$F = \sum_{i=1}^{n} \left( A_{ci} - \left[ A(h_{ci}) + \sum_{j=1}^{N} B_j h_{ci}^{2(j-1)} \right] \right)^2 = \min \quad (9)$$

and

$$\frac{\partial F}{\partial B_j} = 0, \quad j = 1 \sim N \quad (10)$$
After the solution of Eq. (10) is found, the undecided coefficients \( B_j, j=1\sim N \) can be gained. Eq. (8) may also adapt to other types of indenters.

3.1.2. The standard hardness block method. Based on the same idea as the standard elastic modulus block method, Suppose that a standard sample block with a hardness \( H_s \), which is invariable with the change of the indented depth, is indented \( n \) times by using a rigid indenter or an un-rigid indenter, a group of data series \( h_{ci}, P_{\text{max}}i, i=1\sim n \) for the real contact depth and the maximum load may be obtained after data processing, and then the real contact projection area gained in the \( i \)th experiment is as follows.

\[
A_{ci} = \frac{P_{\text{max}i}}{H_S}, i = 1 \sim n
\]  

(11)

If \( h_c \) and \( A_c \) also satisfy Eq. (8), the undecided coefficients \( B_j, j=1\sim N \) in Eq. (8) may be also gained by using Eqs. (9) and (10).

3.2. The non-standard block method.

If we have no a standard hardness block or a standard elastic modulus block, an isotropy and homogeneous sample polished carefully is indented \( n \) times by using a rigid indenter or an un-rigid indenter on a nanoindenter with a super high resolution of displacement and load, and the unloading stiffness \( S_i \) and the real contact depth \( h_{ci}, i=1\sim n \) may be achieved on the basis of the unloading curve and Eq. (7). The hardness \( H_i \) and the elastic modulus \( E_i \) (or the complex modulus \( E_{ri} \)) of the \( i \)th indenting experiment can be obtained by using the related formulas. A new series of \( H_i \) and \( E_i \) (or \( E_{ri} \)), \( i=1\sim n \) is gained after they respectively queued on the basis of the real contact depth and the hardness or the real contact depth and the elastic modulus based on \( h_{ci} \) and \( H_i \), \( k=1\sim n \) or \( h_{ci} \) and \( E_i \) (or \( h_{ci} \) and \( E_{ri} \)), \( k=1\sim n \) is built as follows, that is,

\[
H = f(h_c)
\]  

(12)

and

\[
\begin{align*}
E &= g_1(h_c) \\
E_i &= g_2(h_c)
\end{align*}
\]  

(13)

After the functions were built, the \( n \) indented experiments are done with the same type of indenter as above on the other nanoindenter, and on the basis of a series of data processing, the following formulas may be founded, respectively, according to Eqs. (10), (4) and (5).

\[
A_{ci} = \frac{P_{\text{max}i}}{f(h_{ci})}, i = 1 \sim n
\]  

(14)

\[
A_{ci} = \left\{ \begin{array}{l}
\frac{\pi \left( 1 - \nu^2 \right)}{4 \bar{g}_1(h_{ci})} S_i^2 \\
\frac{\pi}{4 \bar{g}_2(h_{ci})} S_i^2
\end{array} \right. , i = 1 \sim n
\]  

(15)

where the meanings of the related parameters are seen in the Eqs. (10), (4) and (5). According to \( A_{ci} \), \( i=1\sim n \), obtained by using Eq. (14) or (15), the undecided coefficients \( B_j, j=1\sim N \), in Eq. (8) may be determined the processing method used in the standard hardness block method or the standard elastic modulus block method, respectively.

4. Conclusion

On the basis of introducing the calculation of the real contact projection area for nanoindentation and taking Vickers indenter as an example, the calculation method of the real contact projection area is given when the indenter tip is rounding or flatting, and the formulas of determining the real contact projection area is also given for the special case that the axis of the indenter is not perpendicular to the
surface, contacting initially with the indenter tip, of the sample. For the indenter with the unknown geometrical parameters, based on the constant elastic modulus method of determining the real contact projection area, which was promoted by Oliver and Pharr, the standard hardness block method and the non-standard block method are put forward.

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