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Radius and angle determination of diamond Berkovich indenter

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ABSTRACT

In this work, a new measurement method based on atomic force microscope (AFM) and confocal laser scanning microscope (CLSM) is proposed to determine the radius and angle parameters of diamond Berkovich indenter. In order to accurately describe the real tip shape, a comprehensive geometrical model that takes the effects of indenter edge radius and tip radius into account is established. Resultantly, a sophisticated area function is formulated to fit the AFM acquired topographies. Comparison reveals that the fitted data of indenter radius have a satisfactory consistency with the experimental results. For the determination of angle parameters, the CLSM relevant 3D topography acquisition, angle calculation and error correction methods are constructed. Experimental observations validate that the CLSM method is capable of accurately determining the angle parameters of diamond Berkovich indenter.

1. Introduction

The nanoindentation technique is one of the most effectively measuring methods for determining mechanical properties of materials by directly measuring the indentation load-displacement behavior on a very small scale [1–3]. In particular, the geometry of the indenter plays crucial roles in determining of the area of contact. Diamond Berkovich indenter has been widely employed in the instrumented indentation tests [2]. As compared to the indenters made from other materials, a very sharp tip apex can be polished, and its tip geometry can keep self-similarity in a small scale [3]. Because of the unmatched advantages, diamond Berkovich indenter is quite suitable for the micro/nano indentation test. Due to the restriction caused by the state-of-the-art of polishing technologies for diamond Berkovich indenter, the tip apex hardly intersects at an ideal point, which is usually rounded as a micro spherical surface. The smaller the spherical radius or the so-called tip radius is, the smaller scale the indenter geometry can keep self-similarity in, which means that the output accuracy of indentation test is heavily dependent on the tip radius [4–6]. Furthermore, the angle parameters of indenter, e.g. the included angle between the axis of diamond pyramid and each pyramid face, the included angle between any two edges of diamond pyramid, as well as the included angle between the axis of diamond pyramid and the axis of indenter holder, will evidently affect the indentation results too [7–9].

In a word, the tip shape of diamond indenter has significant influences on the instrumented assessment of the material mechanical properties. Therefore, in order to accurately evaluate the crucial parameters of diamond indenter, many interesting work have been carried out. Up to now, the methods used to measure the tip shape of diamond indenter can be divided into two types, i.e. the direct and indirect determination approaches.

According to the instrument and measuring principle, the direct determination approaches include methods based on atomic force microscope (AFM) [10–16], scanning electron microscope (SEM) [6,17–18] and scanning white light interferometer (SWLI) [19].

VanLandingham et al. [10] used AFM to determine the tip shape and area function of Berkovich and conical indenters, which are usually applied to the instrumented indentation. They found that the geometry of AFM tip, scanning velocity of AFM tip and image resolution have a negligible impact on the area function, but non-linearities in the vertical scanner of AFM yield the significant uncertainties, which in return limit the imaging size. Munoz-Paniagua et al. [11] used AFM to analyze the errors induced by instrument and nonideality of indenter tip shape. They disclosed that the hardness is more sensitive to the errors appearing in the determination of the shape area function (SAF), while the errors in estimating the instrumental compliance have a greater influence on the reduced Young's modulus. In order to accurately determine the SAF of a Berkovich indenter and optimize the SAF-compliance values, they finally proposed a hyperbolic definition to correct the SAF. Krier et al. [12] employed AFM to accurately estimate

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the size of the tip defect of diamond Berkovich indenter. Previously, the horizontal movements of the AFM system were calibrated against a 2-μm pitch silicon saw-toothed artifact. Moreover, they declared that a careful cleaning for the indenter surface before measurement is an important issue too. Monclús et al. [13] used AFM to examine the tip shape of diamond Berkovich indenter, which had been used to perform the nanoindentation tests on SiC samples and Al thin films under high temperature. They emphasized that the calibration for the indenter tip shape should be considered as a regular basis, especially when indenting the hard samples and oxidized metal films. In order to calibrate the relationship of contact area versus contact depth for a worn Berkovich indenter, Lee et al. [14] and Wheeler et al. [15,16] directly observed the 3D morphology of the indenter apex by using an AFM.

Chicot et al. [6] evaluated the size of the tip defect for different diamond indenters by means of emission field SEM analysis at different magnifications. In their observations, however, the SEM snaps are two dimensional, which will produce inconsistency with the real tip shape. By using of focused ion beam milling, Yu and Polycarpou [17] modified a worn diamond Berkovich indenter as a sharp spherical indenter, and subsequently evaluated the tip radius with SEM. Miyamoto et al. [18] fabricated the natural diamond rods as nano-tips with low and high energy ion beams, and they also analyzed the tip shapes in terms of SEM observations. Furthermore, Chen and Su [19] used SWLI to separately acquire the 2D tip topography of a Rockwell diamond indenter at different angles and positions. And subsequently, the image data were stitched by resorting to the iterative closest point algorithm. In such a way, the tip radius and cone angle were determined according to the stitched 3D tip topography.

For the AFM-based method, the AFM system should be calibrated rigorously in advance of acquiring the tip shape of diamond indenter. And subsequently, the angle parameters and SAF are determined according to the AFM sampled 3D topography. This method has high accuracy, but the measurement range is small due to the probing restriction in the vertical direction. SEM-based method requires a conductive sample, which means that the diamond indenter should be plated with gold before imaging. In such a way, the measured tip apex is more or less different from the real pyramid morphology. Moreover, only the 2D images of diamond pyramid can be captured by this means. Therefore, it has difficulties in accurately evaluating the angle parameters, tip radius and SAF. As a simple method, however, SEM method has been widely used for the qualitative assessment of diamond Berkovich indenter. In SWLI operation, the incident white light should be reflected by the pyramidal faces. However, diamond crystal is a transparent material, through which the light transmits inevitably. As a result, the SWLI imaging on the diamond pyramid is quite inconvenient, especially for the pyramid face having a large inclination angle.

For the indirect assessment method, indentation into a reference material of known Young's modulus, e.g. fused silica, should be performed firstly. And subsequently, the acquired indentation morphology is considered as the real morphology of indenter apex, in which the shape of indenter can be indirectly determined. Doerner and Nix [20] adopted a replication method to calibrate the shape of a blunt diamond indenter. Their method consists of making two-stage carbon replicas of indentations in annealed α-brass and imaging them in transmission electron microscope. Merle et al. [21] performed nanoindentations on the fused silica substrate with a diamond Berkovich indenter. They reported that plotting the Oliver-Pharr contact depth versus contact radius can yield the shape of indenter. Jha et al. [22] carried out finite element simulations of nanoindentation on the elastic and elasto-plastic solids, e.g. aluminum single crystal, steel and fused silica, which aims to gain insight into two energy based parameters, i.e. the total energy constant and elastic energy constant. The simulated load-displacement data revealed that the total and elastic energy constants can be used to characterize the indenter geometry and response of a material to indentation, respectively. Because of the nonlinear behavior of the reference material and instrument, it is evident that the indirect method has difficulties to accurately detect the tip shape of indenter.

Moreover, none of previous work considered the influence of indenter edge radius on the SAF, regardless of the bluntness appearing on the tip apex. Taking the advantages and disadvantages of different measurement methods into account, the ISO standard [23] and Takagi et al. [24] suggested that different instruments should be employed in response to different measuring ranges as required. Therefore, in this work an AFM-based method is employed to determine the tip and edge radii of diamond Berkovich indenter within a measuring range of hundreds of nanometers, in which a new algorithm of SAF considering the effect of edge radius is proposed. In the determination of angle parameters, a range of hundreds of micrometers is designed and subsequently realized by using a confocal laser scanning microscope (CLSM).

2. Determination of indenter radius

Fig. 1(a) shows the conventional assessment of radius based on an AFM acquired tip topography of diamond indenter. Tip radius

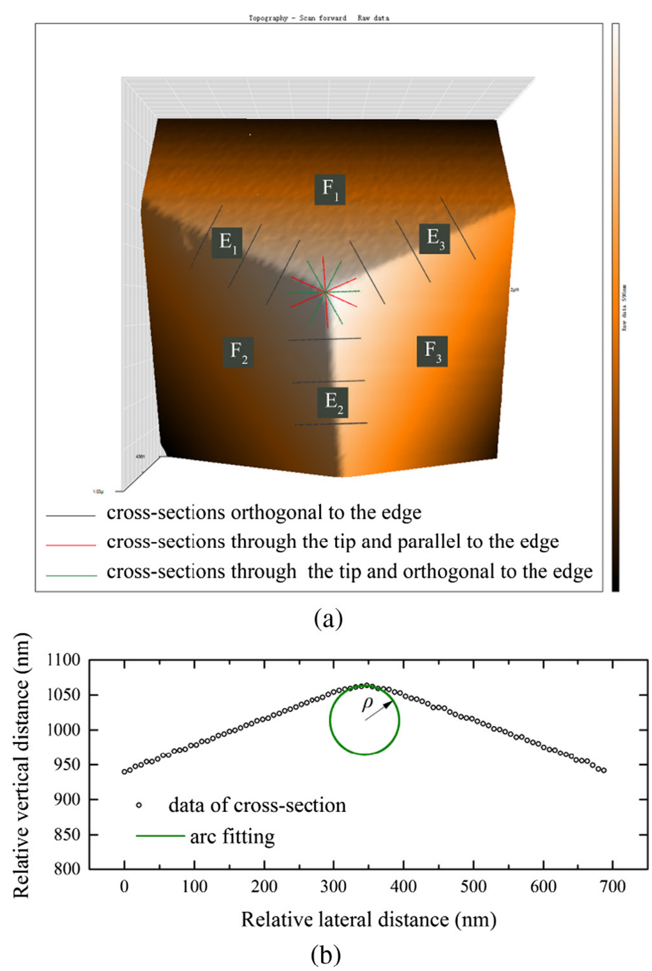

Fig. 1. Evaluation of indenter radii: (a) 3D rendering of an AFM image of a Berkovich indenter; (b) the cross-sectional data collected along the transversal lines shown in (a), which are used to read the radius.