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Nanoscale elastic–plastic deformation and stress distributions of the C plane of sapphire single crystal during nanoindentation

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Abstract

The nanoscale elastic–plastic characteristics of the C plane of sapphire single crystal were studied by ultra-low nanoindentation loads with a Berkovich indenter within the indentation depth less than 60 nm. The smaller the loading rate is, the greater the corresponding critical pop-in loads and the width of pop-in extension become. It is shown that hardness obviously exhibits the indentation size effect (ISE), which is 46.7 ± 15 GPa at the ISE region and is equal to 27.5 ± 2 GPa at the non-ISE region. The indentation modulus of the C plane decreases with increasing the indentation depth and equals 420.6 ± 20 GPa at the steady-state when the indentation depth exceeds 60 nm. Based on the Schmidt law, Hertzian contact theory and crystallography, the possibilities of activation of primary slip systems indented on the C surface and the distributions of critical resolved shear stresses on the slip plane were analyzed.

Keywords: Single crystal sapphire; Nanoindentation; Critical resolved shear stress; Multiple pop-in events; Mechanical properties

1. Introduction

Sapphire (α-Al_2O_3) is an important crystal material due to its high hardness, chemical inertness, superior mechanical performance, and thermodynamic stability.[1–3] Generally, sapphire is brittle and its brittle-to-ductile transition temperature is ~1373 K. However, the plastic deformation of sapphire crystals may occur under low loads at room temperature.[4] Recently, the evaluation of the initial stages of plasticity and elastic–plastic deformation properties of sapphire with different surface orientations at room temperature have been extensively investigated by nanoindentation tests.[4–12] These studies show that single and/or multiple displacement discontinuity (pop-in) events during loading are found to distinguish between the fully elastic and the elastic-plastic regimes, associated with the nucleation of dislocations. As we know, the C plane (0 0 0 1) of sapphire is generally selected as a calibration medium during nanoindentation[13] and substrate in the preparation of many kinds of functional thin film/substrate systems.[14,15] It is necessary to study the mechanical properties of the C plane under different size scales and measurement conditions. Although many studies have been done on elastic-plastic behaviors of the C plane by micro-/nano-indentation with relatively large Berkovich or spherical indenters, the interpretation of indentation results obtained at room temperature is not straightforward because these indentation measurements are strongly affected by factors such as the anisotropic elasticity of sapphire, surface roughness, the radius and shape of indenter tip, loading rate and indentation depth.[4–8] In this paper, the nanoscale elastic/plastic deformation and mechanical properties of the C plane have been systematically studied by depth-sensing nanoindentation experiments at room temperature. The contact area function and radius of the Berkovich tip were carefully calibrated under very small indentation depth by nanoindentation and atomic force microscopy (AFM) instruments. The mechanical properties and surface deformation mechanism of sapphire crystal indented on the C plane were analyzed under the anisotropic elastic characteristic and small indentation depth. The critical resolved shear stresses (CRSS) at slip planes were evaluated with the aid of the Hertzian contact theory, compared with the corresponding the-

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oretical shear strength. The experimental and analytical results would greatly shed light on the understanding of the nanoscale deformation features of sapphire crystal indented on the C plane.

2. Experimental

The samples of sapphire crystal were obtained from Semiconductor Wafer, Inc in Taiwan. As specified by the manufacturer, these single crystals were *C*-axis oriented, double-side polished, and free from defects and residual stress with a root-mean-square roughness < 0.2 nm. Our X-ray diffraction θ–2θ measurements confirmed that the samples were high quality single crystals. All nanoindentation experiments were conducted using an indenter (Triboscope, Hysitron Inc.) equipped with a three-sided pyramidal Berkovich diamond tip and an in situ scanning probe microscopy (SPM) at room temperature. The force and displacement sensitivities of the instrument are 100 nN and 0.2 nm, respectively. Indentations were performed under a variety of peak loads in the range of 0.7–8 mN. For all nanoindentation tests, the loading and unloading times were 20 s and holding time was set as 10 s at the peak indent loads, which resulted in a series of different loading rates ranging from 0.035 to 0.4 mN/s. To obtain reliable data, each cycle was repeated 8 times. The thermal drift was kept below ± 0.05 nm/s for all indentations.

3. Results and discussion

3.1. Calibration of indenter tip radius

Prior to nanoindentation tests, the contact area function and effective indenter tip radius should be carefully calibrated by using a standard sample such as fused quartz.[16] As shown in Fig. 1, the experimental data are fitted by the area function[16]

$$A = C_0h_c^2 + C_1h_c + C_2h_c^{1/2} + C_3h_c^{1/4} + \cdots + C_8h_c^{1/128} \quad (1)$$

where C_O, C_1, ..., C_8 are constants, and h_c is the contact depth. The lead term describes a perfect Berkovich indenter;

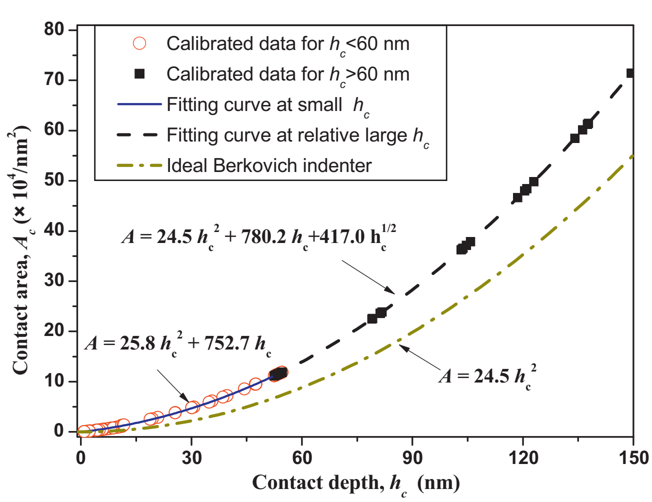

Fig. 1. The calibrated function of indenter contact area and contact depth with standard fused quartz sample.

others represent deviations from the Berkovich geometry due to blunting at the tip. These coefficients should be fitted by the *A* and h_c data based on the relevant indentation depth range.[16] In this work, we focus on the two different ranges of 10–100 nm and 100–500 nm indentation depths. Under the small range of 10–100 nm, the Berkovich tip is generally regarded as spherical or parabola revolution.[13,16,17] The relationship between *A* and h_c can be well described by a simple two-parameter relationship, $A = C_0h_c^2 + C_1h_c$. In the limit of $h_c \ll R$ (here *R* is the radius of indenter tip), the relationship can approximately reduce to $A = 2\pi Rh_c$.[17,18] As shown in Fig. 1, for $h_c < 60$ nm, the relationship was found to be $A = 25.8h_c^2 + 752.7h_c$ for our Berkovich indenter, where the units of *A* and h_c are nm^2 and nm, respectively. Consequently, the tip radius *R* obtained from the second term, $C_1 = 2\pi R$, is equal to 120 ± 5 nm. However, for a relative large indentation range of 100–500 nm, we must re-calibrate the tip area function,[16] $A = 24.5h_c^2 + 780.2h_c + 417h_c^{1/2}$. Therefore, according to the indentation depth of sapphire single crystal, two different tip area functions are chosen to analyze the indentation data.

To verify the tip radius, the three-dimensional geometry of an instrumented indenter was carefully measured by atomic force microscopy (AFM). The AFM image data can be obtained by scanning the Berkovich tip apex. For each AFM image, scanning is along the orthogonal directions of cross-sections through the apex of indenter, which are parallel to the *x*- and *y*-directions, respectively, as shown in Fig. 2(a). A parabolic curve was fitted to the near-apex region of the cross-sectional data, as shown in Fig. 2(b) and *R* was determined from the parabolic equation at the apex location. To discern the difference,[19] four curves are dissociated by adjusting the values of relative vertical distance in Fig. 2(b). It is shown that the average value of the tip radius, *R* = 130 ± 10 nm obtained by AFM, is slightly larger than that from nanoindentation measurements. In the subsequent analysis, *R* is regarded as about 125 nm.

3.2. Characteristics of load–displacement curves

Fig. 3 shows the total of the 8 representative load–displacement (*P*–*h*) curves of the indented C plane during nanoindentation. It is obvious that multiple displacement discontinuities or pop-in events occur during loading. The load corresponding to the first pop-in event is defined as the critical indentation load P_{cr}, which varies in the range of 0.40–0.62 mN (see inset in Fig. 3). The width of pop-in extension is denoted with Δh_{cr} due to the slip of activated dislocation. The pop-in phenomena are consistent with the earliest observations reported by Page[4,5,7] However, P_{cr} in their studies were much higher than P_{cr} in our work, which may ascribe to the radius magnitude of indenter tip. It is also found the fluctuation phenomena of the P_{cr} among these pop-in events, which is similar to other experimental results.[20,21] The reason may be due to the effects of thermal drift and surface roughness under the condition of ultra-low loads.

In nanoindentation tests, the loading rate $\dot{P}$ ranges from 0.035–0.4 mN/s. The relationships of the average P_{cr}, average pop-in extension width Δh_{cr} and $\dot{P}$ for the C plane are shown