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Influence of loading rate on nanohardness of sapphire

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ABSTRACT

This work reports the loading rate effect on nanohardness of sapphire. The intrinsic nanoscale contact deformation resistance of sapphire increased with the loading rates following empirical power law dependence with a positive exponent. The results showed a significant enhancement (e.g., ~66%) of the nanohardness of sapphire with the increase in loading rates from 10 to 10,000 μN s^{-1}. These results were explained mainly in terms of the maximum shear stress generated underneath the nanoindenter, dislocation density and critical resolved shear stress of the sapphire.

1. Introduction

Sapphire is a leading candidate material for many extreme environments e.g., ground and air vehicle windows [1], optical lenses exposed to harsh environments [2] and transparent armors [3,4]. All such applications involve contact with air borne hard dust particles and moving projectiles e.g., bullets, splinters etc. from regular weapons as well as improvised explosive devices. It is true that alumina is one of the most well known armour materials that has been used as a protective material against regular weapons. But in view of global insurgency and consequent attacks made by improvised explosive devices on public lives and society at large it is important to understand the deformation behaviour of alumina at microstructural length scale; to make them more usable for protective applications which were not thought of in earlier times when it was used only to protect against regular weapons. Indentation technique offers one unique way to study the contact induced deformation and controlled fracture events over a large length scale. Particularly in this context the still evolving nanoindentation technique [5,6] comes handy as it can help us to study the micro- and nano-scale evolution of contact induced deformation and/or damage initiation over a length scale that can be as small as the microstructural length scale itself e.g., sub-μm to a μm to as big as a length scale that covers a number of microstructural length scale units, e.g., few μm to tens of μm. Therefore, it is not at all surprising to note that huge amount of literature exists [5–22] on the nanoindentation behaviour of sapphire. These reports typically encompassed dedicated experimental work [5,7–12,15,18–22], theoretical work [9,13,14,16,17] and few attempts which combined both experimental investigation and theoretical interpretation [19–21].

However, dedicated study on the effect of loading rate ($\dot{P} = \frac{dP}{dt}$) on nanoindentation behaviour of sapphire was far from significant [19–21]. For instance, the magnitudes of ($\dot{P}$) were varied in the range of as small as 35 μN s^{-1} to as large as 0.4 mN s^{-1} [19–21]. One interesting characteristic feature of these investigations [5,7–22] was the presence of *"pop-in"s* that signify the transition from classically elastic to elasto-plastic deformation of sapphire under nanoindentation. It is true that particularly in ceramic thin films and especially in ceramic coatings pop-ins can occur due to crack generation beneath the nanoindentation and not due to plasticity. But, in the case of structural bulk ceramics like the polycrystalline alumina ceramics and the single crystal alumina of the present work, the corresponding loads at which pop-ins were reported were of the order of mN and μN [5,7–22] and hence, such a small load is highly unlikely to cause crack generation beneath the nanoindentations in them as they require much higher loads for crack initiation [3,4]. However, a definite unambiguous mechanism of the genesis of pop-ins is yet to be unequivocally established. The ultralow load at which they initiate is called the critical load (P_c) corresponding to a critical depth of penetration (h_c) [5].

Based on pertinent literature data [5,7,8,10,11,16,18–21] of alumina single crystals, a typical illustrative scenario for

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Table 1
Literature survey on nanomechanical properties of sapphire.

P (μN)	$\dot{P}$ (μN s^{-1})	P_c (μN)	H (GPa)	E (GPa)	Remarks	Ref
45,000	–	10,000	15–35	–	Presence of ISE	[5]
3000	–	2200	–	–		[5]
2500		2200	–	–		[7]
5000	–	–	24	–		[8]
500–10^5	–	–	25	–		[10]
300–5×10^5	–	–	–	420		[11]
1000–9000	–	1300	30	475		[16]
18,000	–	2600	29	482		[18]
800–8000	40–400	350–580	27.5	420.6	(a) P_c decreases with the increase in $\dot{P}$ (b) Presence of ISE	[19]
1000–8000	35–400	400–620	27.5	420.6	(a) P_c decreases with the increase in $\dot{P}$ (b) Presence of ISE	[20]
1000–8000	20–400	380–540	27	423	(a) With increasing $\dot{P}$, P_c change with irregular amplitude within $0 < h < 60$ nm. (b) Presence of ISE	[21]

P: Load, $\dot{P}$: Loading rate, P_c: Critical load, H: Hardness, E: Young's modulus, –: Not mentioned, ISE: Indentation size effect.

nanoindentation load (P), loading rate (dP/dt), critical load (P_c), nanohardness (H) and Young's modulus (E) are presented in Table 1. The data from literature survey (Table 1) showed that there was wide variation in the values of P_c. For instance, it was reported [5] that P_c of sapphire was as high as e.g., ~10 mN [5]. On the other hand as reflected in data of Table 1, other researchers reported that P_c of sapphire was low as e.g., 420 μN [19,20]. It was interesting to note that the loading rates were not always reported in literature data [5,7,8,10–16]. It could have been possible to calculate the loading rates from the experimental details provided the loading times were made available in the aforesaid references. However, in absence of the loading times, the loading rates could not be exactly calculated for the works reported in [5,7,8,10,11,16], as reflected in literature survey provided in Table 1.

The magnitudes of P_c were reported to be dependent on the choice of crystallographic orientations of the sapphire crystal e.g., C-, M- or R-plane [5,19–21]. But an equivocal picture was yet to emerge whether P_c was dependent on loading rate ($\dot{P}$) or independent of it [19–21]. For instance, work reported in [19,20] suggested that the magnitudes of P_c were sensitive to variations in ($\dot{P}$). On the contrary, work reported in [21] indicated that the magnitudes of P_c were insensitive to variations in ($\dot{P}$). It also appeared that it was a complex function of the particular plane concerned and the range of loading rates. For instance, with increase in ($\dot{P}$) from 35 to 400 μN s^{-1} on the (0001) plane e.g., the C-plane of sapphire the magnitudes of P_c gradually decreased from ~620 to 520 μN [19–21]. But, as far as the $(10\bar{1}2)$ plane of sapphire was concerned P_c did not show any kind of systematic variation with variations in $\dot{P}$ [21]. On the contrary, when loading rate ($\dot{P}$) was enhanced from 20 μN s^{-1} to 100 μN s^{-1} the magnitudes of P_c had increased from ~380 to 540 μN [21] on the (1012) plane of sapphire. For the same plane, with further increase in loading rate ($\dot{P}$) from 100 μN s^{-1} to 200 μN s^{-1} the magnitudes of P_c had decreased from ~540 to 360 μN [21]. However, the magnitudes of P_c had again regained back e.g., from ~360 to 540 μN when an even higher loading rate e.g., 100 μN s^{-1} to 200 μN s^{-1} was used during nanoindentation on the same plane [21]. These results clearly pointed out that the nature of variations of P_c with respect to variations in ($\dot{P}$) was yet to be unambiguously established.

For a C-plane sapphire the magnitude of yield point load (e.g., P_c) varied in the range of ~700–850 μN for $\dot{P}$ varying between 100 and 1500 μN s^{-1} [22]. Nanoindentation conducted on the R-plane gave a comparatively lower P_c of ~400–500 μN [22]. These facts established that the present knowledgebase about loading rate dependence of critical load was far from comprehensive and sufficient. It acted as the motivational backdrop behind the present work.

Thus, the objective of the present work was to study the loading rate effect on nanohardness of sapphire. The loading rate was varied over a wide range e.g., 10–10,000 μN s^{-1}. The other objectives were to examine if nanoscale plasticity events were really occurring in sapphire during nanoindentation. Further, it was decided to check out that if the nanoscale plasticity events happened in sapphire whether their occurrence was affected at all by the variations in loading rate. Finally, it was planned to examine the roles of maximum shear stress generated underneath the nanoindenter, dislocation density and critical resolved shear stress in influencing the physics of deformation in the case of the sapphire.

2. Materials and methods

The sapphire (0001) sample grown by the Verneiul process was obtained commercially from the Union Carbide Corporation. A nanoindentation machine (Tribo Indenter Ubi 700, Hysitron Inc., Minneapolis, MN) was used to evaluate the nanomechanical properties of sapphire. The depth sensing resolution of the machine was 0.04 nm. The machine was capable to resolve load variations of even as small as 1 nN. The machine provided a surface topography of constant contact force in scanning probe microscopy (SPM) mode. In addition, it provided a load versus depth of penetration ((P-h)) plot in nanoindentation mode. A diamond indenter was used for this purpose. The indenter had a tip radius of ~150 nm and a semi-apex angle of 65.3°. The area function of the indenter tip was calibrated prior to each experiment to ensure the reproducibility and reliability of the experimental data using a calibrated, standard fused quartz sample provided by the supplier of the machine. The fused quartz sample had a certified nanohardness (H) of 9.25 GPa and Young's modulus (E) of 69.6 GPa. The dedicated software available in the control system of the machine corrected the experimentally obtained (P-h) data for tip blunting effect. Next, the Oliver-Pharr model [10] was used to evaluate the H and E data of the sapphire from the experimentally measured (P-h) data plots.

A square array of at least 5×5 indents was utilized for this purpose. No particular bias was associated with the location selection for the positions of the nanoindentation arrays. Thus, at least 25 individual measurements of nanohardness values were used for each reported average data. The error bars indicated ± 1 standard deviation. The present nanoindentation experiments were conducted in the load controlled mode. The loading axis of the present sapphire was parallel to the c-axis. In all the nanoindentation experiments, for a given loading rate the unloading