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Hardness and mechanical anisotropy of hexagonal SiC single crystal polytypes

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ABSTRACT

The mechanical response of single crystal silicon carbide (SiC) of two hexagonal polytypes (six layer, 6H- and four layer, 4H) was investigated using nanoindentation. Indentations were performed on two specific crystallographic orientations of single crystals i.e., normal to the basal, (0001) and prismatic, $(10\bar{1}0)$ planes, in the load range between 25 mN and 500 mN. A significant anisotropy in the hardness is observed with the basal orientations showing a higher hardness compared to prismatic orientations. In both orientations, the 6H-SiC polytype exhibits higher hardness than the 4H-SiC polytype. It is also observed that the hardness decreases with increasing indentation load, suggesting that SiC crystals exhibit indentation size effect. However, unlike hardness, elastic modulus is independent of indentation load and the elastic anisotropy is insignificant. Severe cracking, particularly at higher indentation loads is noticed near the edges of the indentation imprints. The indentation fracture toughness, K_{IC}^{i} computed from the imprints shows slightly higher values for 6H-SiC compared to the 4H-SiC. However, for both the polytypes, a slightly higher K_{IC}^{i} is observed for basal indentations compared to the prismatic ones.

1. Introduction

Polycrystalline silicon carbide (SiC) is considered as a potential material for body armor applications because of its high compressive strength when compared to other structural ceramics [1–5]. The high strength of SiC is attributed primarily to the strong covalent bond between Si and C atoms. It is also observed that factors such as crystal structure, porosity, inclusions, fraction of polytypes, grain size and crystallographic orientation of the grains influences the mechanical properties of SiC [6–12]. SiC has a complex crystal structure and exists in several allotropic forms, but SiC used in armor plates predominantly contains hexagonal crystal structure [9,12] of which six layer (6H) and four layer (4H) hexagonal polytypes are the most commonly observed [12]. There have been few experimental investigations highlighting the role of inclusions on the dynamic performance of SiC [7,8], but the role of individual polytypes and their crystallographic orientations on the mechanical properties is not well understood. One of the reasons for this is the difficulty in producing large single crystals of SiC comprising of specific polytypes. Since, the mechanical properties of polycrystalline SiC are directly influenced [12] by the nature and fractions of each kind of polytype present in the sample, it is important to understand the role of individual polytypes and their orientations on the mechanical response of SiC. Such an understanding will help in designing new SiC based materials with improved mechanical performance. Hence, in the present study, we investigated the mechanical properties of single crystals of 6H- and 4H-SiC polytypes and the influence of orientation on the plastic deformation and fracture. We have used nanoindentation, to evaluate mechanical properties of materials because of the inherent advantages associated with this technique [13–16].

Nanoindentation is a useful technique to obtain both hardness and elastic modulus of materials even at low indentation loads (as low as few nano-newtons) which otherwise is not possible with conventional indentation tests [13–16]. It is also reported in the literature that the hardness of ceramics can be directly correlated to their ballistic performance [17]. Ceramics are generally prone to cracking during macro Vickers indentation (because of high indentation loads) and hence the true hardness of ceramics always remains questionable [17,18]. Hence, many of the successful studies on SiC till date use the Knoop hardness method, since the geometry of the indenter is less prone to cracking [19–21]. One other advantage of the Knoop indenter is that, it is possible to study the

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in-plane anisotropy of the materials and this perhaps helps in identifying the possible slip systems in the material. Adewoye and Page [19] have performed etching on the Knoop indented samples and noticed preferential removal of the material along the $11\bar{2}0$ direction suggesting that this could be the possible slip direction in 6H-SiC single crystals. Fujita et al. [20] have carried out high temperature indentation experiments using a Knoop indenter particularly on {0001} and $\{10\bar{1}0\}$ planes of 6H-SiC single crystals and noticed that the basal indentations have higher hardness than the prismatic indentations. Though Knoop indentation is a useful method to evaluate the in-plane and out-of-plane anisotropy, the geometry of the Knoop indenter do not induce high enough plastic strains in the material underneath the indenter and hence the material under the indenter many not be in a fully plastic regime to determine the true hardness. Rendtel et al. [18] have performed Vickers and Knoop indentations on polycrystalline SiC samples and determined that for the same indentation loads Vickers hardness values are found to be higher than the Knoop hardness highlighting the importance of geometry of the indenter on hardness measurements. However, severe cracking is noticed at the Vickers indentation corners, perhaps due to high indentation loads. It is possible to obtain indents without cracking at low indentation loads, but then the size of the indent makes the imaging a difficult task and sometimes leads to spurious values of the measured hardness. Instrumented nanoindentation provides a means of measuring the hardness by indirect method (typically from the *P vs. h* curve) even at low indentation loads. In the present study, we have used instrumented nanoindenation to obtain the mechanical properties of 6H- and 4H-SiC polytypes and also investigated the effect of orientation on the mechanical properties.

2. Experimental methods and materials

Single crystals of 6H- and 4H-SiC were provided by the Army Research Laboratory from Fairfield Crystal (New Milford, CT). Cuboidal samples are sectioned from the bulk samples and the crystallographic orientation of each face of cube are determined using Laue back reflection diffraction method. A comparison with the standard diffraction pattern suggests that the faces of the cuboidal specimen correspond to basal (0001), first order $(10\bar{1}0)$ and second order $(11\bar{2}0)$ prismatic planes. The specimen faces are polished to a surface roughness of 0.25 μm using standard metallographic polishing techniques. As the mechanical polishing induces considerable damage to the surface layers (which was confirmed by high nanohardness values), all the samples were chemically etched in a hydrofluoric acid for about 1 h to remove about 1 μm from the surface layers of the sample. At the same front, precautions were taken to minimize the errors in hardness measurement arising from mounting of the specimens. To achieve a firm bonding between the specimen holder and the glue, we have allowed the glue to settled for 24 h. This made sure that the samples are firmly adhered to the sample holder. Further, to minimize variation in hardness due to sample location, all the measurements were carried out at a fixed sample tray location and the indentation were carried out far away from the edge to avoid the edge effects. In addition to this, the minimum spacing between the two successive indents was about five time the penetration depth so that error in hardness due to strain field interaction of the subsequent indents is also minimized.

2.1. Nanoindentation

Nanoindentation experiments were performed using MTS nanoindenter with indentation loads varying from 25 mN to 500 mN. The hardness and elastic modulus were determined using Oliver and Pharr (O&P) method [13–16]. This method has been successfully employed to determine the hardness and elastic modulus of various single and polycrystalline ceramics. Here, we describe some of the principles and equations used in nanoindentation and a detailed description of the same are found in Refs. [13–16]. Any instrumented indentation method uses the contact area of impression, A_c, indentation load at maximum penetration depth, P_{max}, and initial unloading contact stiffness, S, to compute the hardness, H and elastic modulus of a given sample E_s.

$$Hardness, H = \frac{P_{max}}{A_c} \tag{1}$$

$$Elastic\ modulus,\ E_s = \left(1-\vartheta_s^2\right)\left[\left(\frac{2\sqrt{A_c}}{S\sqrt{\pi}}\right) - \frac{1-\vartheta_i^2}{E_i}\right]^{-1} \tag{2}$$

In the above equation, the subscripts i and s denote the indenter and sample under consideration, while E and ϑ represent the elastic modulus and Poisson's ratio respectively. It is evident from equations (1) and (2) that, H and E_s depends on A_c and the errors introduced in the measurement of A_c will lead to errors in indentation results. Unlike conventional indentation methods, where A_c is obtained by direct measurement (measuring the diagonal length in the case of Vickers method or diameter of the impression in the case of Brinell), the A_c in instrumented indentation is obtained by indirect method. It is customary to obtain A_c by performing series of calibrations on a standard sample (typically fused silica), supplied by the machine manufacturer.

In the current study, indentation experiments were carried out on (0001) and $(10\bar{1}0)$ planes using a Berkovich pyramidal diamond indenter. All the indentations were made in such a way that one of the indenter corners is oriented along easy slip direction $11\bar{2}0$. The area function of the indenter was calibrated using a standard fused silica sample with-in the depth range of 100–1000 nm. To minimize the error due to thermal drift, the machine was corrected for drift and maximum drift rate of 0.02 nm/s was allowed. The H and E of fused silica obtained using this area function are within ± 5% of the manufacturer specified values and a plot of H, E *vs.* P is presented in supplementary information as Fig. S1. All the indentations were performed under load controlled mode with a 10 s loading and unloading time and a 15 s holding time at maximum indentation load. In-order to obtain statistically significant data a maximum of 20 indentations were performed at each indentation load.

3. Results and discussion

3.1. Analysis of indentation load vs. displacement curves

Typical indentation load (P) *vs.* displacement (h) curves of single crystal 4H- and 6H- SiC specimens in basal and prismatic orientations are shown in Fig. 1. It is evident from *P vs. h* curves that, the loading portion of the curve exhibits a smooth behavior and no displacement bursts (also known as *pop-ins*) are observed. However, AFM and SEM images of the indents (shown in Figs. 4 and 6), at indentation load of 500 mN, indicate cracking at the indent corners. It is commonly observed in the literature that such cracking at the indent corners appears as displacement bursts (in the load controlled experiments) in the loading curve. *Pop-ins* in the loading curves have been previously reported during nanoindentation experiments on several materials and is generally attributed to the plastic instabilities such as pressure induced phase transitions [22], amorphization [23,24], nucleation of shear bands