Energy Analysis of Spherical and Berkovich Indentation contact damage in commercial polycrystalline Silicon Carbide.

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ABSTRACT

This research aims at enhancing the understanding of the role of processing and microstructure on the deformation of and fracture of polycrystalline silicon carbide based ceramics when subjected to blunt (spherical) and sharp (pyramidal) indenters. The role of microstructure and material properties on the energy absorption capability of SiC is studied. By examining the behavior of several SiC materials during nanoindentation experiments using spherical and pyramidal indenters, it is possible to make predictions about methods to improve the ductility and fracture toughness of SiC to optimize its energy absorption. The applicability of the area under the irreversible part of the indentation load displacement curve (energy dissipated during loading) to predict the performance of SiC under contact loading is examined. Results from experimental data show that the polycrystalline SiC material properties like hardness and modulus are relatively insensitive to the amount of indentation damage when the indent size is more than the average grain size. It can also be concluded that during pyramidal indentation for the polycrystalline samples analyzed, grain-size plays a much more important role in energy dissipation than the grain boundary phases or any other indentation damage relate effects.

1. Introduction

1.1 Silicon carbide

SiC is the only compound of silicon (Si) and carbon (C) which exists in nature in the solid state at room temperature and pressure. SiC crystallizes in over 200 polytypes [1, 2] with different Si-C stacking sequences. The most common polytypes are the 3C (Cubic) or β and 6H (Hexagonal) or α Silicon carbide [4]. Most polytypes have similar mechanical and thermal properties but different electrical and optical properties. Silicon carbide has high hardness, good flexural strength, good high temperature strength, low density, and is relatively less expensive compared to many other high hardness materials. SiC, due to covalent bonding, has among the highest known elastic modulus (~450 GPa) and has shear yield strength of about one tenth of the shear modulus [5]. SiC has superior creep behavior and wear resistance [6, 7] which make it an ideal material for use in wear resistant coatings in a variety of automotive components like pistons, valve heads, bearings etc and in micro-electromechanical systems. The physical (wide bandgap, high thermal conductivity and high electric field strength) and chemical properties of SiC-6H polytype make it an ideal material for high temperature, high frequency, high power devices, including radar and microwave applications under extreme conditions [8]. SiC is used in coatings in military jet engines, heat exchangers in gas turbines for tanks and ships, composite armor and as the ceramic plates in bulletproof vests.

1.2 Nanoindentation

Nanoindentation, i.e probing mechanical properties at the nanometer scale has emerged as an important technique during the last decade for characterizing mechanical properties and behavior at small length scales. Although initial application of nanoindentation was for thin films

[9-13] and small length scale properties [14, 15], recently, nanoindentation has been used as a characterization tool for high and low temperature testing [16], high strain rate testing [17], dynamic testing for storage and loss moduli [18], creep testing [19, 20] and testing properties in a variety of environments (liquids etc.) [21]. Nanoindentation systems, which represent the current state of the art for hardness testing machines, are effectively non-destructive. Due to the ability to analyze small specimens, considerable research is being done into further extending the capabilities of nanoindentation systems. Nanoindentation systems are now being used to investigate materials under dynamic conditions (scratch, creep, in-situ, etc.) which opens up the possibility to obtain new and unique information about materials. Nanoindentation is also being used to analyze new materials like nanocomposites to obtain more information about interface bonding. This is difficult to do with traditional testing methods.

1.3 Pyramidal Indentation

The indenter proposed by E.S Berkovich in 1951 [22] is the most commonly used three sided pyramidal tip for nanoindentation experiments and is preferred because of its well defined geometry. A typical indentation load displacement curve obtained with a Berkovich indenter is shown in Figure 1. In Figure 1, W_e (the elastic work done) is the area under the unloading curve, W_T (total work done) is the area under the loading curve W_p is the Irreversible or plastic work ($W_T - W_e$). Cheng and Cheng [23, 24] developed a method to calculate the modulus and hardness of a material based on the area under the loading and unloading curves. They developed a relation between the indentation hardness and the reduced modulus using scaling analysis and finite element simulations in terms of work which is given by:

$$\frac{W_e}{W_T} \approx \lambda \frac{H}{E_r} \quad (1)$$

where, λ depends on the indenter geometry and has values were found to be in the range from 5.33 to 4.678 [23-25]. A comparison of the values of hardness and modulus obtained using the method outlined by Cheng-Cheng with those obtained using the Oliver-Pharr approach should give an indication of the pile-up/sink in behavior of the material during nanoindentation experiments. The Cheng-Cheng model was developed using continuum mechanics and Mises effective stress which applies to metal plasticity and also agrees with results on ceramics and silica [23, 24].

1.4 Indentation testing of Silicon carbide (SiC)

Most commercial polycrystalline SiC is composed of the SiC-6H polytype. Elastic modulus measurements of polycrystalline SiC have been made using various methods, including sonic resonance, tensile, flexural, and indentation techniques with both Vickers indenters and nanoindentation [6, 7]. SiC has a high resistance to yielding, but unlike metals, cracks and pre-existing flaws easily propagate because there is very limited dislocation mediated plastic deformation. Qian et al. [36] measured the Knoop and Vickers hardness and fracture toughness of SiC-6H single crystals and observed that the values at the asymptotic-hardness region are significantly smaller (~20%) than those at low loads indicating an indentation size-effect. Previous research on commercial polycrystalline SiC materials by Wereszczak et al.[37] (Figure 2), using spherical indenters showed that the cracking features and the load displacement curves for different forms of SiC are different. This indicates a difference in the impact response of the different SiC polycrystalline samples which can be studied using indentation experiments. Various other researchers have also used nanoindentation using Berkovich indenters to evaluate the modulus and hardness of polycrystalline ceramics [38, 39].

1.5 Objectives of this research

Ceramics have an inherent brittleness and are very sensitive to flaws. Since structural applications of SiC involve mechanical loading and contact, an understanding of the mechanical anisotropy of SiC is needed. The performance of polycrystalline SiC in applications that involve high loading and contact depend on mechanisms that redistribute loads, reduce stress concentrations and dissipate elastic strain energy. Understanding the response of polycrystalline SiC to blunt and sharp indenters, and the role of microstructural variables like grain size, grain boundary fracture strength, grain aspect ratio, etc. on deformation and fracture mechanisms in polycrystalline SiC ceramics. The role of material properties in energy absorption ability of the ceramic is useful since these microstructural parameters can be varied during processing to enhance the impact performance of the material.

2. Materials and Methods

2.1 Nanoindentation

A Nanoindenter XP® (Nano Instruments Innovation Center, MTS Corporation, Knoxville, TN) was used to perform nanoindentation experiments at ambient temperature using Berkovich, pyramidal and spherical tips of varying radii on both single and polycrystalline SiC samples used in this research. All nanoindentation experiments were done using the CSM (Continuous Stiffness Measurement) technique [40] which gives the load on the sample and the contact stiffness as a function of the displacement of the indenter into the samples. The commercial polycrystalline SiC samples were obtained from BAE Systems Advanced Ceramics (formerly Cercom, Inc.), and are manufactured using a pressure assisted densification technique (PAD). The materials, manufactured in slightly different ways involving variations in the powder

composition, grain size, and sintering parameters and are designated SiC-N, SiC-HPN and SiC-RN. Basic physical properties of the polycrystalline SiC samples are listed in Table 3 [37]. The commercial polycrystalline samples were cut and then mounted in resin. Polishing of the mounted samples was done by mechanical polishing with SiC grit paper followed by polishing with Buehler MasterMet 2 non-crystallizing colloidal silica (SiO_2) polishing suspension ($0.02\ \mu\text{m}$) using a vibratory polishing machine. Scanning electron microscopy was used to image the post indentation residual impressions made by different indenters on various SiC samples. A Zeiss Auriga dual beam FIB/SEM instrument was used for scanning electron. This area function for a Berkovich indenter is basically a polynomial which, when used with fused silica gives a modulus of 74.1 GPa in our case. The tip radii for the spherical indenters used in this research were determined using tungsten single crystal specimens of known modulus ($E=411\ \text{GPa}$) and Poisson's ratio ($\nu=0.28$). Assuming the P - h curve prior to the pop-in events to be completely elastic, the data was fit to the Hertz spherical contact solution to estimate the tip radius of the spherical indenters.

3. RESULTS AND DISCUSSION

3.1 Berkovich Indentation

Nanoindentation experiments using a Berkovich indenter angles and spherical indenters with different tip radii were carried out on several polycrystalline SiC ceramics primarily to analyze the variation of the elastic energy to total energy ratios, and modulus and hardness with respect to microstructure. Berkovich indentation tests were carried out at peak loads ranging from 25 mN to 500 mN. The modulus and hardness values obtained in an indentation experiment is compared to values obtained using ultrasonic measurements and other traditional testing methods available in literature. Table 2 below shows the properties of single crystal Cree SiC-6H

H_{OP} , E_{OP} represent the hardness and modulus measured using Oliver-Pharr method and H_{Corr} , E_{Corr} represent the hardness and modulus after correcting for pile-up during indentation. Table 3 summarizes the results for the Berkovich indentation experiments on different polycrystalline SiC samples. Figure 3(a) shows the $P-h$ curves for Berkovich indentation of SiC-N at various peak loads. Figure 3(b) shows the average load-displacement curves for Berkovich indentation of polycrystalline SiC samples at a peak load of 100 mN. The load displacement response for a polycrystalline material during a nanoindentation experiment is due to the contributions of the main phase and the intergranular phase. This is partially due to the fact that at a 100 mN peak load the size of the indent is about 2-3 μm which is more than the average grain size in SiC-RN but not the other samples. Figure 3(c) shows average load-displacement curves for Berkovich indentation at a 500 mN peak load. At this load, the size of the indent of about 7 μm is greater than the average grain size of all the materials and therefore the load-displacement response for a 500 mN peak load Berkovich indentation experiment is similar for all the materials. The response of SiC-RN at 100 mN peak load is different than the other two polycrystalline samples, due to the different grain size 1.5-3 μm compared to 3-5 μm . Figure 3(e) and Figure 3(f) show the modulus and hardness vs displacement into the surface for different polycrystalline SiC samples during a Berkovich indentation experiment. It can be seen that all the polycrystalline SiC samples show very similar hardness and modulus values the difference between the highest modulus (SiC-N) and the lowest modulus (SiC-RN) being approximately 10%. It can be seen from Figure 3, that all the three polycrystalline samples show an indentation size effect. The effect is more pronounced for SiC-N than the other two. The SiC-RN seems to show an indentation size effect up to a penetration depth of 400 nm after which it is relatively constant. The indent size at 400 nm is about 2.5 - 3 μm while the average grain size for SiC-RN is about

1.5 – 3 μm . It is conceivable that the indentation size effect at low loads and low penetration depths in SiC-RN is due to orientation effects. As soon as the indent size is equal to or greater than the average grain size of the material the indentation size effect is no longer seen. Similar arguments can be made for SiC-N and SiC-HPN. Figure 3(d) shows representative SEM images of different polycrystalline ceramics (SiC-N, SiC-RN and SiC-HPN) along with Cree SiC-6H at 500 mN peak load. At 500 mN peak load, the indent size in these SiC polycrystalline ceramic samples is about 7 μm . It can be seen from the SEM images that the SiC-RN sample with the smallest average grain size (1.5 - 3 μm), has more grain push out than the SiC-N and SiC-HPN with a larger average grain size (3 – 5 μm). Figure 4(a) shows representative SEM images of maximum and minimum damage in SiC-RN during Berkovich indentation at various peak loads. The indentation response of a material is primarily due to the contributions of the main phase (SiC), the intergranular phase and cracking effects. The secondary phase in the polycrystalline SiC samples used in this research is primarily inorganic oxides which are softer and more compliant (stiffness). The local volumetric ratio between the main phase and the intergranular phase can affect the response at low indentation loads (low penetration depths). Figure 4(b) shows the modulus and hardness during a Berkovich indentation experiment at various indentation depths in SiC-RN. The modulus and hardness are relatively constant over the entire range of indentation depths. At peak loads of less than 25 mN, when the indentation depths are approximately 200 nm, which corresponds to an indent size of approximately 1.5 μm , there is an increased scatter in the modulus and hardness values. Since the indent size is less than or equal to the average grain size, the scatter may be due to local volumetric ratio between the main and secondary phase and cracking in addition to the anisotropy. However, for peak loads greater than 100 mN which corresponds to a penetration depth of approximately 450 nm there is hardly any

scatter in the modulus and hardness values. The modulus and hardness values at peak load greater than 200 mN are relatively constant. It can be seen from Figure 3 and Figure 4, that the grain boundary phase, the secondary phase or cracking effects does not play a significant role in the hardness and the modulus material response when the indent size is greater than the average grain size.

3.2 Energy analysis of Berkovich Indentation in polycrystalline ceramics

Figure 5(a) and Figure 5(b) show the PE/TE ratios at various peak loads and peak displacements, respectively. The parameter S shown in the Figure 5 is defined as the length of the side of the indent at a particular depth, which is equal to approximately 7.5 times the depth for a Berkovich indenter. The two main differences in the different polycrystalline ceramic samples are the grain size and the secondary phase. Previous research has shown that the variation between the modulus and hardness of the three grades is very small (<10%).

The PE/TE ratios for all the three polycrystalline materials increase with an increase in the peak load or peak displacement during Berkovich indentation experiments. It can be seen from the Figure 5(a) and Figure 5(b) that, in SiC-N for peak loads of 50 mN, when the size of the indent is less than the average grain size of the material, the average values of PE/TE is about ~ 0.39 and it increases to ~ 0.46 at higher loads indicating that there is possibly a grain size effect. Interestingly, in Figure 5 the values of the PE/TE ratios become relatively constant at higher loads (> 300 mN) for all polycrystalline SiC samples. At peak loads greater than 300 mN, when the indent size is large enough that the stressed volume of the material beneath the indenter incorporates more than one grain, the material response is the same. The PE/TE ratios for SiC-RN at 50 mN are the highest of all three polycrystalline materials. SiC-RN has the smallest grain size and at 50 mN peak loads the indent size is equal to the average grain size indicating that the

deformation response is from just a few grains. SiC-N and SiC-HPN have a larger grain size, and at the 50 mN peak load the indent size is small enough that the response is only from one grain or a few grains. SiC-N and SiC-HPN show different PE/TE ratios at low peak loads, even though the average grain size is the same due to different grain boundary phases. Berkovich indentation experiments at 25 mN peak load were carried out on SiC-N samples. Figure 6 shows the PE/TE ratios for various indents in SiC-N. SEM images of a few representative indents are shown in Figure 7 and Figure 8. The results in Figure 6 show that when an indent is near the center of the grain, the PE/TE ratios are similar to the ratios of single crystal SiC-6H. The PE/TE ratios for indents close to the grain boundary or on the grain boundary are either higher or lower than single crystal SiC-6H. This indicates that the grain boundary phase might play a major role in the energy dissipation mechanism at low loads and small contacts. At low loads, when the indent size is less than grain size, if the indent is at or near the grain boundary it is conceivable that a lot of energy is dissipated in grain boundary motion, which would indicate higher PE/TE ratios compared to when the indent is at or near the center of the grain. Since the material response for an indent at 25 mN peak load, at the center of the grain is from the main phase SiC, it is expected that the PE/TE ratio is similar to single crystal SiC-6H.

3.3 Spherical Indentation

Figure 9 and Figure 10 shows load-displacement curves for spherical indentation experiments using a 7.5 μm diameter and a 50 μm diameter tip respectively. It can be seen that there is hardly any difference in the response of the three polycrystalline SiC materials examined in this research during spherical indentation experiments. The indentation response for the 15 μm diameter spherical tip is elastic plastic for all materials while the response for 50 μm diameter tip is completely elastic for all materials. In previous work, Wereszczak et al. [37]

showed that there is a difference in the fracture and cracking modes in different polycrystalline ceramics. However, those loads were in excess of 100 N which is outside the testing limits of the current nanoindentation equipment.

4. Summary Conclusions

Nanoindentation experiments using Berkovich and spherical indenters were used in to analyze the material response of polycrystalline SiC samples. Analysis of indentation experiments on polycrystalline SiC at various peak loads show the following observations:

- The material response especially hardness and modulus is relatively insensitive to the amount of indentation damage (cracking, grain push out etc,) when the indent size is larger than the average grain size.
- At low peak loads when the indent size is smaller or equal to the average grain size the variance in the hardness and modulus values is primarily due to the location of the indent and the material anisotropy. At low peak loads up to 100 mN the difference in the load displacement curves between the three polycrystalline SiC samples is primarily due to the difference in grain size. At larger peak loads > 300 mN when the indent size is greater than the average grain size the load displacement response for all three SiC polycrystalline materials is almost the same.
- The response to spherical indentation using a 15 μm and 50 μm diameter spheres is similar for all three polycrystalline SiC samples contrary to the results shown by Wereszczak et al. [37]. This is due to the load limitations on the equipment. It is found that at maximum testing loads of 500 mN, the response is elastic-plastic for the 15 μm and completely elastic for the 50 μm diameter sphere for all polycrystalline SiC samples.

- Energy methods used to analyze the load displacement curves obtained during Berkovich indentation experiments on polycrystalline SiC samples show that, for a given indent size a smaller grain size material SiC-RN will have more grains push out than a larger grain size material (SiC-N and SiC-HPN), which will affect the material response especially the PE/TE ratios. The PE/TE ratios for polycrystalline material remain relatively constant after a load at which the indent size is large enough that the stressed volume of material beneath the indenter incorporates more than one grain. The increase in the PE/TE ratio for polycrystalline ceramics from low to high loads follows a similar trend to SiC-6H when indented in the $\langle 0001 \rangle$ direction but the change is much bigger. Indentation at 25 mN peak load in SiC-N showed that if the indent size is smaller than the grain size and the indent is near or at the center of the grain then the PE/TE ratio is similar to the SiC-6H $\langle 0001 \rangle$ single crystal ratio. PE/TE ratios for SiC-N go from ~ 0.39 @ 100 mN indentation load to ~ 0.46 @ 500 mN indentation load. This indicates that the change in the ratios is due to the grain size and possibly grain boundary effects. It can be concluded that a material with smaller grain size will have greater energy dissipation at lower peak loads i.e for smaller contact.

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LIST OF TABLES

Table 1: Basic properties of commercial polycrystalline SiC.

Table 2: Modulus and hardness of Cree SiC-6H measured by Berkovich indentation.

Table 3: Experimental hardness and modulus values of different polycrystalline SiC.

Table 1: Basic properties of commercial polycrystalline SiC.

Material	Source	Av. Grain Size (μm)	Density g/cm^3	E (GPa)	Poisson's Ratio	K_{IC} ($\text{MPa} \cdot \text{m}^{1/2}$)*
SiC-N	BAE Systems	3-5	3.212	453.7	0.171	2.75
SiC-HPN	BAE Systems	3-5	3.192	443.1	0.155	3.17
SiC-RN	BAE Systems	1.5-3	3.185	431.5	0.154	2.84
* K_{IC} data listed is from various sources measured by different methods and should not be used for comparison in absolute magnitude ** Data is from various sources that include ultrasonic and mechanical testing [37].						

Table 2: Modulus and hardness of Cree SiC-6H measured by Berkovich indentation.

Sample	Berkovich Indentation			
	E_{OP}	H_{OP}	E_{Corr}	H_{Corr}
	(GPa)	(GPa)	(GPa)	(GPa)
SiC-6H <0001>	549.66 $\pm$ 20.63	40.97 $\pm$ 1.21	503.13 $\pm$ 28.19	32.26 $\pm$ 5.01
SiC-6H <01-10>	513.71 $\pm$ 19.84	39.13 $\pm$ 0.79	461.94 $\pm$ 22.49	30.81 $\pm$ 2.1

Table 3: Experimental hardness and modulus values of different polycrystalline SiC.

	E_{OP} (GPa)	H_{OP} (GPa)	$E_{Literature}$ (GPa)	$H_{Literature}$ (GPa)
SiC-N	530.13 $\pm$ 31.12	40.04 $\pm$ 4.09	453.7	27.2
SiC-HPN	527.99 $\pm$ 36.67	38.16 $\pm$ 4.34	443.1	25.3
SiC-RN	483.74 $\pm$ 34.14	36.96 $\pm$ 3.10	431.5	28.9

LIST OF FIGURES

Figure 1: Schematic P - h curve for Berkovich indentation of a homogeneous elastic-plastic solid.

Figure 2: Hertzian Indentation tests with a 300 μm diameter diamond sphere [37].

Figure 3: (a) Average P - h curves for Berkovich indentation of SiC-N. (b) Average P - h curves for Berkovich indentation of polycrystalline SiC at 100 mN peak load. (c) P - h curves for Berkovich indentation of polycrystalline SiC at 500 mN peak load. (d) Representative SEM images of Berkovich indentation in polycrystalline SiC at 500mN peak load (e) Hardness vs displacement into surface curves for different polycrystalline ceramics using Berkovich indentation. (f) Modulus vs displacement into surface curves for different polycrystalline ceramics using Berkovich indentation.

Figure 4: (a) Representative images showing maximum damage and minimum damage during Berkovich indentation in SiC-RN at different peak loads. (b) H - h and (c) E - h curves for SiC-RN in a Berkovich indentation experiment.

Figure 5: (a) PE/TE vs Peak load for Berkovich indentation of polycrystalline SiC. (b) PE/TE vs Peak displacement for Berkovich indentation of polycrystalline SiC.

Figure 6: PE/TE ratio for various 25mN Berkovich indents in SiC-N.

Figure 7: SEM images of Berkovich indentations at 25mN peak load in SiC-N indents (1-6).

Figure 8: SEM images of Berkovich indentations at 25mN peak load in SiC-N indents (10-15).

Figure 9: Load displacement response of different polycrystalline SiC materials during spherical indentation experiments with a 15 μm diameter tip.

Figure 10: Load displacement response of different polycrystalline SiC materials during spherical indentation experiments with a 50 μm diameter tip.

Figure 1: Schematic P - h curve for Berkovich indentation of a homogeneous elastic-plastic solid.

Figure 2: Hertzian Indentation tests with a 300 μm diameter diamond sphere [37].

Figure 3: (a) Average P - h curves for Berkovich indentation of SiC-N. (b) Average P - h curves for Berkovich indentation of polycrystalline SiC at 100 mN peak load. (c) P - h curves for Berkovich indentation of polycrystalline SiC at 500 mN peak load. (d) Representative SEM images of Berkovich indentation in polycrystalline SiC at 500mN peak load (e) Hardness vs displacement into surface curves for different polycrystalline ceramics using Berkovich indentation. (f) Modulus vs displacement into surface curves for different polycrystalline ceramics using Berkovich indentation.

Figure 4: (a) Representative images showing maximum damage and minimum damage during Berkovich indentation in SiC-RN at different peak loads. (b) $H-h$ and (c) $E-h$ curves for SiC-RN in a Berkovich indentation experiment.

Figure 5: (a) PE/TE vs Peak load for Berkovich indentation of polycrystalline SiC. (b) PE/TE vs Peak displacement for Berkovich indentation of polycrystalline SiC.

Figure 6: PE/TE ratio for various 25mN Berkovich indents in SiC-N.

Figure 7: SEM images of Berkovich indentations at 25mN peak load in SiC-N indents (1-6).

Figure 9: Load displacement response of different polycrystalline SiC materials during spherical indentation experiments with a 15 μm diameter tip.

Figure 10: Load displacement response of different polycrystalline SiC materials during spherical indentation experiments with a 50 μm diameter tip.