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PLEASE CITE THE PUBLISHED VERSION

<https://doi.org/10.1080/02670836.2020.1755139>

PUBLISHER

Taylor & Francis

VERSION

AM (Accepted Manuscript)

PUBLISHER STATEMENT

This is an Accepted Manuscript of an article published by Taylor & Francis in Materials Science and Technology on 24 April 2020, available online: <http://www.tandfonline.com/10.1080/02670836.2020.1755139>.

LICENCE

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REPOSITORY RECORD

Zhang, Dezhou, Ligu Zhao, and Anish Roy. 2020. "A Multiscale-indentation Study of Deformation and Fracture in 6H Polycrystalline Silicon Carbide". Loughborough University.
<https://hdl.handle.net/2134/12097584.v1>.

A Multiscale-Indentation Study of Deformation and Fracture in 6H Polycrystalline Silicon Carbide

D Zhang, LG Zhao*, A Roy

Wolfson School of Mechanical, Electrical and Manufacturing Engineering, Loughborough
University, Loughborough, LE11 3TU, UK

*Corresponding author; Email: L.Zhao@Lboro.ac.uk; Tel.: 0044-1509-227799

Abstract

The mechanical behaviour of a polycrystalline silicon carbide across length scales was studied using Vickers indentation, focusing on the hardness, fracture toughness and failure mechanism of the material. For macroscopic and microscopic indentations, the hardness decreased with an increase in load, which was associated with the well-known indentation size effect as well as the internal flaws. For nanoindentation, severe plastic deformation was discovered beneath the imprints on the basal plane (0001) which is the most favourable crystallographic plane for dislocation movement. Alternative sources of plastic deformation, including deformation twinning and stacking faults, were found for nanoindentations with an increased load. Also, cracking was observed for indents made at 100 mN and above, which was used to study the fracture toughness.

Keywords: Silicon carbide; Vickers indentation; Length scale; Modulus; Hardness; Fracture toughness

1. Introduction

Due to its superior properties, silicon carbide (SiC) has a wide range of industrial applications and used for manufacturing of micro-electro-mechanical systems [10], turbine engine

components, automobile brakes [34], bulletproof armour [35], cutting tools and abrasives [37]. Numerous experimental studies were carried out to determine the mechanical properties of SiC. For instance, Sarva and Nemat-Nasser [30] performed quasi-static compression tests on hot-pressed sintered silicon carbide at ambient temperature. The sample failed at a compressive stress of 4.2 GPa by axial splitting which was believed to be caused by the tensile cracks propagated in the direction of the maximum compressive load. Using quasi-static compression, Shin et al. [32] studied the fracture mechanisms for two types of α -SiC (6H-SiC). Specifically, SiC-I was hot pressed with boron and carbon as sintering additives, while SiC-II was sintered with Al-based compound. For SiC-I, transgranular fracture was observed; whereas intergranular fracture was the dominating failure mode for SiC-II. In addition, it was found that intergranular cracking leads to a higher fracture toughness than that for transgranular cracking due to a greater area involved in fracture for intergranular cracking [28]. Additionally, Zhang et al. [42] carried out both quasi-static and dynamic compression on polycrystalline 6H-SiC, it was found that brittle fracture was the major failure mode under quasi-static compression whereas, evident plastic deformation (dislocations, kinks etc.) was the dominating deformation mechanism under dynamic compression.

Despite the efforts, there are still research gaps in understanding the properties of SiC which require further investigation. For instance, brittleness of SiC is an undesirable weakness commonly seen for ceramics, and not ideal for load-bearing structures due to the risk of catastrophic failure. On the other hand, SiC is also used in manufacturing microchips for applications at high temperature in order to increase the performance. The mechanical behaviour of SiC at a small length scale is as important as its electrical properties in order to maintain the structural integrity of the components. Consequently, nanoindentation tests were carried out to study further the mechanical behaviour of SiC. Based on nanoindentation of single crystal 6H-SiC, Shim et al. [33] found that the geometry of the indenter tip had a great impact on the

measured mechanical properties. For example, the displacement at peak load, as well as the plastic deformation and hardness, increased with the decrease of indenter tip angle (centreline to face). In their study, the yield strength was found to be 7.2 GPa, close to the theoretical shear strength of 6.8 GPa. Yan et al. [39] also conducted nanoindentation on single-crystal 6H-SiC using Berkovich indenter tip to investigate the substrate deformation and damage under indentation. From the transmission electron microscopy (TEM) analyses, it was found that the depth ($\sim 1 \mu\text{m}$) of substrate deformation was 2.5 times and 5 times the total and residual depths of the indent, respectively. Below the deformed zone, there was a long micro-crack propagating straight downwards to a depth of $4 \mu\text{m}$. Substrate cracks were also observed by other researchers when investigating the plastic deformation in 4H-SiC [13, 14]. However, the cracks in 4H-SiC were either lateral or radial [9, 40], as opposed to median cracks found in 6H-SiC [6, 39], due to the crystalline structure difference between 6H and 4H SiC.

In addition, many experiments have shown that the plastic flow for crystalline materials is dependent on the length scale. In the work of De Guzman et al. [5], it was found that the hardness was independent of the indentation depth for amorphous materials; while the hardness increased with the decreasing indentation depth for crystalline materials. It was suggested that for crystalline materials, high dislocation density was expected beneath the shallow indentation and caused the increase of hardness. When the depth increased, the geometrical necessary dislocation (GND) density became too low to have a significant effect on hardness. Such theory was known as strain gradient theory and was one of the most accepted explanations for indentation size effect (ISE). ISE was not only observed in metals, but it has also been reported for ceramics [2, 21, 45]. For instance, Balog et al. [2] performed macroscopic scale and nanoscale indentations on SiC which was sintered with oxides and aluminium nitride (AlN) as additives. The hardness measured from macroscopic indentation was 5~7 GPa lower than that measured from nanoscale indentation, largely due to indentation size effect. In the work of Zhu et al. [45], nanoindentation

experiments were performed on various materials including ceramics and metals using spherical indenter, and the yield stress was found proportional to the cubic root of indenter radius due to the ISE. While Machaka et al. [21] performed a series of microindentation tests on B_6O , and the ISE was attributed to the bulk material's indentation elastic recovery, the indenter/specimen friction resistance coupled with elastic resistance of the specimen.

On the other hand, fracture toughness is also an important parameter that describes the resistance of materials to fracture. The initial approach to determine the fracture toughness of ceramics using Vickers indentation was proposed by Palmqvist [26] based on the proportionality of the crack length to the applied load [15, 26]. There are normally two types of cracks observed in Vickers indentation, namely, the radial-median cracks (half-penny cracks) and Palmqvist cracks [20, 22, 29]. According to Matsumoto's work [22], materials with high toughness would exhibit Palmqvist cracks while radial-median cracks would appear for materials with low toughness. However, the type of cracks does not only depend on the material itself but also on the applied load. In the work of Coric et al. [4], it was found that for yttria-stabilized tetragonal zirconia (Y-TZP) ceramics, when the applied load was low (29.42 N and 49.03 N), Palmqvist cracks occurred; whereas radial cracks appeared when the applied load was high (196.13 N and 294.20 N). In literature, different methods were proposed to calculate the fracture toughness using indentation techniques. For silicon carbide, Niihara et al. [24] and Kaur et al. [12] proposed formulae for calculation of fracture toughness based on Palmqvist cracking; while Lawn and Fuller [17] and Anstis [1] introduced equations for calculation of fracture toughness based on radial-median cracking.

Despite the considerable effort in characterising silicon carbide, there are still areas that require significant research effort. In particular, the size effect and the failure mechanisms of SiC at different length scales and loading conditions require an improved understanding, since the

application of polycrystalline SiC involves mechanical loading and contact at different length scale. In this study, a series of indentation tests were carried out in order to provide a better understanding of mechanical behaviour of polycrystalline SiC at different length scales. The focus of this research is on the size effect as well as the response of SiC to different load levels. Deformation and failure mechanisms were also studied according to microscopic examinations of indents, including the crack mode at macroscopic scale and plastic deformation due to dislocation activities at nanoscale.

2. Experimental details

2.1 Material and Specimen preparation

The material used in this study is commercially sold as Hexoloy SA silicon carbide produced by SAINT-GOBAIN using pressureless sintering of submicron silicon carbide powder at a temperature above 2000°C. The sintered sample has fine grains, and the grain size ranges from 4 to 10 microns with a density of 3.10 g/cm³. The as-received silicon carbide tiles were of hexagonal shape with a side length of 27 mm and a thickness of 4 mm. Smaller samples were cut from these tiles using a diamond blade fitted on the Accutom 5 machine, and then hot mounted using polyfast resin for scanning electron microscopy (SEM) examination. The mounted samples were then ground using Struers Piano plates (Struers Ltd, UK, grit 220 to 1200) followed by polishing procedure using polishing plate/cloths with diamond slurries of 9 µm, 3 µm and 1 µm, consecutively. The grinding took a few minutes and finished when the surface was flat with a consistent scratch pattern. Polishing was then carried out to remove all surface damages caused by previous steps including the cutting and grinding. Between each polishing step, the sample was washed in an ultrasonic bath to remove any potential contaminations, i.e., large diamond particles. Each polishing step took more than 1 hour and as a result, a smooth mirror finishing surface was accomplished with a surface roughness (Ra) of 9-18 nm.

2.2 Macroscopic scale indentation

According to the ISO (International Organization for Standardization) standard [11], indentation tests fall in the macroscopic scale when the force applied is in a range between 2 N and 30 kN. In this study, indentation tests were carried out by using a Mitutoyo bench-top hardness testing machine (HV-100 Series, AVK-CO) installed with Vickers indenter. Several factors were considered before the selection of load levels for the macroscopic scale indentation. This includes the suggestion from ISO standard, the load range of the machine and the thickness of the specimen (should have sufficient substrate material). In addition, a coherent transition to microscopic scale indentation was also taken into account. Consequently, the indentation loads were chosen to be 0.1, 0.2, 0.5 and 1 kgf, corresponding to 980, 1960, 4900 and 9800 mN, respectively. Based on the work of Wade et al. [36] and machine capability, the dwell time and loading rate were chosen as 10 s and 1 $\mu\text{m/s}$, respectively. For each load, 10 indentations in a row were performed and the gap between each indent was 10 times the diagonal length of the imprint in order to eliminate their interactions [36].

2.3 Microscopic scale and nanoscale indentations

Both micro and nanoindentation were carried out using the NanoTest machine (Micro materials Ltd Wrexham, UK). For microscopic scale indentation, loads of 100 mN, 500 mN and 1000 mN were imposed; whereas 30 mN, 60 mN and 100 mN were imposed for nanoscale indentation. The length scale was distinguished based on the specified parameter (load and penetration depth) on ISO standard, and the load level was chosen based on the standard as well as the machine's capability. For example, the ISO 14577-1 standard [11] suggested that nanoscale indentations should have penetration depths of less than 200 nm. On the other hand, microscopic scale indentations should have penetration depths greater than 200 nm and the load applied should be less than 2 N. The lowest load at which the machine can perform with reliable accuracy was 30 mN which correspond to a penetration depth of ~ 180 nm. In order to gather sufficient data for

comparison, indentation experiments were also carried out at 60 mN and 100 mN using the nanoscale indentation system. To have a coherent transition across length scale, the lowest load for microscopic scale indentation was chosen to be 100 mN which was the same as the maximum load used in nanoscale indentation.

Here, the loading rate and the dwell time were kept the same as those used for macroscopic scale indentation. All tests were performed in a temperature-controlled lab at 25°C. Total 20 indentations were made under 30 mN load with a 10 μm gap between individual indents. The 20 indents were made in a matrix area of 40 μm $\times$ 30 μm . Similar patterns were made for all other load levels except that the distance between indents increased with increasing load. Here, the gap between each indent was 10 times the depth of the penetration which is sufficient to eliminate the possible interaction of stress fields generated from previous indentation [36].

2.3 Focused Ion Beam (FIB) lift out

In order to verify the plastic deformation, FIB lift-out (FEI nanolab 600 dual beam machine) was carried out for TEM scans. The imprint selected for FIB lift-out was made under 60 mN and the overall imprint pattern can be seen in Fig. 1 (a) with imprint locations marked using circles (a red circle for the FIB lift-out, and yellow circles for the rest). The locations were selected as there were no obvious defects near the imprint, and the force-displacement curve shows a reasonable response. In the enlarged image (Fig. 1 (b)), the imprint of interest was marked with a thin platinum (Pt) layer for protection from damage potentially caused by the impinging electron beam. Figure 1 (c) shows a thick Pt layer was deployed above the imprint for protection from damage caused by the FIB. Large current coarse milling and small current fine milling were shown in Figs. 1 (d) and (e) respectively. Once the lamella was cut free from the substrate material, it was mounted on a copper grid using Pt for further thinning (Fig. 1 (f)). The finished lamella is shown in Fig. 2, where a notch (cross-sectional view of the imprint) can be

seen. The thickness of the lamella was about 180 nm which can be seen in Fig. 2 (b). These images were taken under electron beam mode within the FIB machine. Lines were drawn on the figure to distinguish the boundary of the Pt layer and SiC lamella due to bad contrast. The nanometer-thickness lamella was then scanned for dislocation activities using a JOEL 2000FX TEM (operating at 200 kV, point-to-point resolution of 0.28 nm, lattice fringes at 0.20 nm resolution).

Fig. 1, (a) The overall imprint pattern for 60 mN load, (b) the enlarged image showing the imprint of interest and neighbour imprints with thin Pt layer covered, (c) thick Pt layer for protection from (d) coarse and (e) fine ion beam milling, and (f) mounted lamella

Fig. 2, (a) Top and (b) front views of the finished lamella.

3 Results and discussion

3.1 Macroscopic scale indentation

Figures 3(a-d) show SEM images (JSM-7100F thermal field emission electron microscope) of representative imprints on the sample surface after indentation tests at 980, 1960, 4900 and 9800 mN, respectively. The imprints were inevitably influenced by the existing voids and secondary phases in the material. The diagonal length of the imprint was measured based on the SEM imaging. The length of the crack and residual imprint increased with an increase in load as expected. For the indentation corresponding to 980 mN load, the crack length was shorter than the corresponding diagonal length of the imprint, whereas the imprints produced under higher load had a longer diagonal length than the crack size. The hardness values were calculated to be in a range between 25.5 GPa and 29 GPa, as summarised in Table 1 together with other parameters according to equation [7],

$$H = 2 \frac{P}{d^2}, \quad (1)$$

where H is the hardness, P is the load and d is the diagonal length of the residual imprint measured from centre to corner. For the indentation experiment with the lowest load (980 mN), the hardness was the largest (29 GPa). For higher loads, the hardness value range from 25.5 to 27 GPa. This was believed to be associated with the deformed volume (i.e., imprint size) and the flaws presented in the material. Specifically, the deformed volume for the lower load level was less than that for the higher load, therefore, the probability of finding flaws was smaller. These effects are negligible in macroscopic indentation.

Fig. 3, SEM images of the imprints at different load level: (a) 980 mN, (b) 1960 mN, (c) 4900 mN and (d) 9800 mN.

Classification of induced cracks was based on the ratio of the crack length (from the centre of the imprint) to the half diagonal length of the imprint (c/a) (Fig. 4). According to literature [3, 20, 24], the crack is regarded as a Palmqvist crack if c/a is less than 2.5 and becomes a radial-median crack if c/a is greater than 2.5. Table 1 shows the measured hardness and the values of a and c for different load level. For the indentation made under 980 mN the crack is classified as a Palmqvist crack. On the other hand, when the applied load was larger than 980 mN, the cracks are radial-median cracks. This is consistent with the work of Yang et al. [41], where Vickers indentation was performed on solid-phase-sintered SiC. It was found that when the applied load was greater than 10 N, clear radial-median cracks were observed. Similar results were also reported in [8, 21], where Palmqvist cracks were observed at low load level while median cracks were shown at high load level.

Fig. 4, Schematics of (A) radial-median and (B) Palmqvist cracks.

Table 1, Macroscopic scale hardness and measured crack length and diagonal length of the imprint.

Load (mN)	Hardness (GPa)	a (μm)	c (μm)	c/a
980	28.80 ± 1.62	4.22 ± 0.23	10.34 ± 1.11	2.45 ± 0.08
1960	28.04 ± 2.03	5.3 ± 0.53	16 ± 1.37	3.01 ± 0.14
4900	27.73 ± 1.45	8.7 ± 0.72	28.7 ± 1.66	3.30 ± 0.16
9800	25.86 ± 2.59	12.45 ± 1.03	43.05 ± 2.32	3.46 ± 0.11

For a Palmqvist crack, the fracture toughness can be calculated using following two equations (2) and (3) which were suggested by Niihara et al. [24] and Kaur et al. [12], respectively,

$$K_{IC} = \frac{0.0114E^{0.4}P^{0.6}a^{0.8}}{c^{3/2}} \quad (2)$$

$$K_{IC} = 0.0294\left(\frac{HP}{l}\right)^{1/2} \quad (3)$$

where E is the elastic modulus, P is the applied load and H is the measured hardness and a , c , l are dimensions measured from the imprint as shown in Fig. 4.

For a radial-median crack, Lawn and Fuller [17] and Anstis et al. [1] proposed Eq. 4 and 5, respectively, for the calculation of the fracture toughness,

$$K_{IC} = 0.0726\left(\frac{P}{c^{3/2}}\right) \quad (4)$$

$$K_{IC} = 0.016\left(\frac{E}{H}\right)^{1/2}\left(\frac{P}{c^{3/2}}\right) \quad (5)$$

Table 2 compares the fracture toughness calculated from the equations proposed above. The values obtained based on the equations for a Palmqvist crack seems to be lower than that calculated using the equations for a radial-median crack. However, they are all within the reported range (i.e., $0.7\text{-}3.2 \text{ MPa}\sqrt{\text{m}}$) in literature [23]. Fracture toughness is an essential property of engineering materials, especially for ceramics which are often regarded as brittle materials. In addition, in most load-bearing applications, the limiting criteria for the selection of ceramics is the fracture toughness. Therefore, it is very important to quantify the fracture toughness of the SiC studied in the present work. Materials fracture toughness can be enhanced through intrinsic and extrinsic mechanisms. Intrinsic toughening can be realised by a change in the distribution of interface properties of second-phase particles to suppress damage caused by micro-cracking or micro-void ahead of the crack (increase the cracking resistance). However, such toughening mechanism normally works for metallic materials. For ceramics, toughness can be improved by extrinsic toughening which takes place behind the crack tip including crack bridging and phase transformation. As opposed to intrinsic toughening which increases the cracking resistance, extrinsic toughening reduces the crack driving force through different mechanisms which act behind the crack tip. It is also known as crack-tip shielding which can occur in form of crack bridging, crack deflection, constrained micro-cracking and phase transformation [16]. Extrinsic toughening leads to a resistance-curve (R-curve) behaviour where the crack driving force to sustain cracking increases with crack extension, i.e., the fracture toughness increases with increasing crack length. Fracture toughness calculated using equations developed for radial-median crack showed a smooth increase with increasing load level as shown in Table 2. However, if the cracks observed for 980 mN load were considered to be Palmqvist cracks, there would be a sudden increase of fracture toughness when the load increased from 980 mN to 1960 mN. This indicates that the cracks caused by 980 mN load may not be Palmqvist cracks after all since the value of $c/a=2.45$ is very close to the threshold value 2.5. Figure 5 shows the SEM image of the imprint produced by 980 mN load. The diagonal length of the

imprint is around 6.19 μm , and the radial crack at the imprint corner has a length of 5.69 μm . The yellow lines represent grain boundaries, and a transgranular crack can be seen within the grain. The cracks initiated from the imprint corner are primary cracks, and those from the imprint edge are considered as secondary cracks as indicated in Fig. 5. The presence of voids and secondary phases affect crack propagation. For example, the overall crack length ' l ' is 5.69 μm as indicated in the figure where no voids or other defects were observed along the path of the crack. In contrast, crack lengths are 7.14 and 7.28 μm for cracks propagated from other corners where voids and secondary phase promoted the crack propagation.

Table 2, Fracture toughness calculated using different equations.

Load (mN)	K_{IC} MPa $\sqrt{m}$ (Niihara et al. [24]) (Palmqvist)	K_{IC} MPa $\sqrt{m}$ (Kaur et al. [12]) (Palmqvist)	K_{IC} MPa $\sqrt{m}$ (Anstis et al. [1]) (Radial-median)	K_{IC} MPa $\sqrt{m}$ (Lawn & Fuller [17]) (Radial-median)
980	0.77 ± 0.23	1.46 ± 0.17	1.90 ± 0.13	2.14 ± 0.21
1960	/	/	2.00 ± 0.16	2.23 ± 0.19
4900	/	/	2.16 ± 0.18	2.31 ± 0.26
9800	/	/	2.27 ± 0.22	2.52 ± 0.31

Fig. 5, SEM image of an imprint showing the crack caused by macroscopic scale indentation.

3.2 Microscopic scale indentation

Figure 6 shows the overall force-displacement curves for microscopic scale indentation. The load-displacement curve showed a good match for the three loading states in the elastic region (initial part). A small variation the curves presented was probably due to the underlying grain orientation during the indentation process. As an example, the imprint size was $\sim 2.5 \mu\text{m}$ for the 100 mN load, and the probability of indenting a single grain was high given that the grain size is in the range of 4 to $10 \mu\text{m}$. On the other hand, the largest load used for micro-scale indentation was 1000 mN and the corresponding imprint size was about $8.7 \mu\text{m}$. This imprint size was larger than most grains of the material ($4\text{-}10 \mu\text{m}$) which leads to a low probability of indenting only a single grain. Therefore, for indentations made under larger load levels, the shifts in the force-displacement curve were mostly due to the voids and secondary phase. In addition, for

indentation at 500 mN and 1000 mN loads, there was a ‘kink’ in the unloading part of load-displacement curves, i.e., when the load was approaching zero (marked in red circles). This was caused by the correction of thermal drift, and such effect was negligible at smaller load (100 mN).

Fig. 6, The overall F-D curves for micro-scale indentation.

The Oliver-Pharr’s method [25] was used to calculate the elastic modulus for each load. Table 3 summarizes the results. With an increasing load, the elastic modulus reduced significantly. This phenomenon is different from that reported in Zhao et al. [44] who showed almost constant elastic modulus (527 GPa) with increasing indentation depth (200 to 1000 nm) for a polycrystalline 3C-SiC. The decreased elastic modulus in this study was due to the increasing size of imprint. As the imprint size gets bigger, the probability of interacting with voids increased.

Therefore, a ‘softening’ response was observed in terms of decreasing elastic moduli. Such effect was first proposed by Leonardo da Vinci according to Parsons [27], who found that the fracture strength decreased with increasing sample size and it was attributed to the higher probability of finding flaws within the larger sample volume. Similarly, samples tested in tension normally exhibit lower strength than that tested in bending. This is because, for bending tests, the volume of the sample subjected to peak stress was lower than that in a tensile test [16].

Table 3, Elastic modulus and hardness obtained from micro-scale indentation.

Max load (mN)	Min E (GPa)	Max E (GPa)	Average E (GPa)	Hardness (GPa)
100mN	579.74	726.94	659.72±38.90	30.91±0.88
500mN	240.95	403.87	324.26±15.59	28.45±0.93
1000mN	208.47	244.98	227.42±6.54	26.23±1.76

A total of 60 indents were made at three different load levels (i.e., 20 indents for each load level). Figure 7 (a) shows the SEM image of all indents at 100 mN load (marked in red circles). Representative SEM images of indents are presented in Fig. 7 (b-d) for 100 mN, 500 mN and 1000 mN loads, respectively. It was observed that for imprints made under microscopic scale load, the crack lengths were smaller than the diagonal lengths of imprints, which was similar to macroscopic scale indentation at 980 mN load. The c/a ratio and the fracture toughness for each load are summarised in Table 4. The fracture toughness was calculated using Lawn and Fuller’s method [17]. Although the c/a ratio for the imprint produced at 100 mN was 2.44 which was less than the threshold (2.5), it was assumed to be a radial-median crack for the reasons discussed earlier. The fracture toughness showed an increasing trend with increasing loads which was due to extrinsic toughening, known as R-curve behaviour. This is similar to the situation observed in macroscopic scale indentation.

Table 4, Imprint crack length and fracture toughness at microscopic scale indentation.

Load (mN)	a (μm)	c (μm)	c/a	Fracture toughness ($\text{MPa}\sqrt{m}$) (Lawn & Fuller [17])
100	1.25 ± 0.21	3.05 ± 0.43	2.44 ± 0.12	1.36 ± 0.14
500	3.125 ± 0.45	8.125 ± 0.69	2.60 ± 0.08	1.56 ± 0.17
1000	4.0 ± 0.53	10.6 ± 1.24	2.65 ± 0.17	2.10 ± 0.24

Fig. 7, The SEM image of (a) all indents made under 100 mN load and imprints at different load levels: (b) 100 mN, (c) 500 mN and (d) 1000 mN.

3.3 Nanoscale indentation

The force-displacement curves for nanoindentation tests are plotted in Fig. 8, showing a good consistency for the loading part of the force-displacement curves at all load levels. The elastic modulus was calculated using the same method as that used for microscopic scale indentation. It can be seen in Table 5, both nanoscale and microscopic scale indentations showed a decrease of the elastic modulus with increasing load. For nanoscale indentation, the average elastic modulus was 847 GPa for 30 mN, which was higher than those reported in the literature (300-550 GPa; [23, 31, 33]). The modulus reduced to 521 GPa at 100 mN load. The modulus calculated from nanoscale and microscopic scale indentations was different for the same load level. For 100 mN load, the modulus was calculated as 521 GPa and 659 GPa for nanoscale and microscopic scale indentations, respectively. As the load applied was the same, the major difference between nanoindentation and micro-indentation tests was the radius of the Vickers indenter tip. In this study, the tip radius was measured as ~ 500 nm for microscopic scale indenter, almost twice the tip radius for nanoscale indenter. Therefore, the modulus difference was mainly caused by the tip radius. In fact, the tip radius was proved to have a significant effect on the properties obtained through indentation [38], where the hardness of a copper increased from 0.9 GPa to 1.9 GPa when the indenter tip radius was reduced from 20 μm to 1 μm .

Fig. 8, The force-displacement curves for nano-scale indentation.

Table 5, Comparison of elastic modulus and hardness obtained from nanoscale and microscopic scale indentations.

Max load (mN)	Min E (GPa)	Max E (GPa)	Average E (GPa)	Average H (GPa)
(Nanoscale indentation)				
30 mN	772.04	936.29	847.23 ± 58.57	47.07 ± 1.54
60 mN	465.64	741.69	620.79 ± 38.75	41.10 ± 1.05
100 mN	427.95	573.60	521.49 ± 25.89	39.56 ± 1.45
(Microscopic scale indentation)				
100 mN	579.75	726.94	659.72 ± 38.90	30.91 ± 0.88
500 mN	240.96	403.87	324.26 ± 15.59	28.45 ± 0.93
1000 mN	208.48	244.98	227.42 ± 6.54	26.23 ± 1.76

Crack initiated at a load level ~ 100 mN. Figure 9 shows the SEM images of the residual imprint for (a-c) Vickers indentation with increasing load (30, 60 and 100 mN, respectively) and (d) Berkovich indentation (100 mN). There were no cracks observed on the surface of the sample for 30 mN and 60 mN. However, for the imprint at 100 mN load, the crack length was measured to be $1.05 \mu\text{m}$ for Vickers indentation compared to $0.88 \mu\text{m}$ observed for Berkovich indentation. The fracture toughness was calculated to be $2.22 \text{ MPa}\sqrt{\text{m}}$ using Eq. 4 (Vickers indentation), which was lower than $2.57 \text{ MPa}\sqrt{\text{m}}$ obtained from Berkovich indentation.

Fig. 9, SEM images of (a-c) residual imprint caused by Vickers indentation under 30, 60 and 100 mN load, respectively, and (d) residual imprint caused by Berkovich indentation under 100 mN load.

Figure 10 shows the hardness values of 6H-SiC measured across the length scale (log scale used for x-axis). The hardness values calculated from nanoscale indentation were larger than those obtained from the microscopic scale and macroscopic scale indentations. The magnitude varied between 48 GPa and 37 GPa in the nanoscale indentation regime, whereas the values were around 30 GPa for microscopic scale and macroscopic scale indentations. This was due to the indentation size effect, as previously explained. The presence of voids also influenced the results, especially with an increase in length scale. A similar explanation was reported in micro-pillar compression and Berkovich indentation tests carried out by Shin et al. [31] and Zhang et al. [43], respectively. Another potential factor was the grain orientation effect, as the indentation for a lower load could be confined to a single grain whereas the indentation for a larger load would involve a group of randomly oriented grains. The grain orientation effects were studied by Zhang et al. [43], showing a significant influence on the properties obtained from nanoindentation. Specifically, a “softer” response in terms of lower elastic modulus and hardness were observed if the grain orientation was more favourable for dislocations to slip. While for orientations which inhibit dislocation movement, cracks were formed as a result of excessive stresses.

Fig. 10, The hardness of 6H SiC measured across length scale (log scale for x-axis).

3.4 TEM analysis

For TEM analysis, lift-out lamellas were prepared. Figure 11 shows some representative TEM images of the overall lamella, the zoomed-in area beneath the imprint and the diffraction pattern beneath the imprint. From the overall lamella (Fig. 11 (a)), it can be seen that there were no voids or noticeable defects around the imprint despite the fact that the material was sintered with a large volume fraction of voids. This lamella also contains multiple grains, with grain size that ranged from 2 to 5 μm . From Fig. 11 (b), shear bands can be observed, with dislocation pile-ups. The spacing of shear bands decreases at locations near the imprint. Similar features were reported for experiments in 3C-SiC in Zhao et al.'s work [44]. Since the region close to the imprint was heavily deformed, more shear bands were naturally generated to accommodate this deformation. Selected area diffraction (SAD) was carried out in the JOEL 2000FX TEM to determine the grain orientation beneath the imprint, with locations marked using red circles. The

diffraction patterns were identical for both locations which imply the grain beneath the imprint was a single grain, i.e., the indentation was not made on grain boundaries or multiple grains. The typical diffraction pattern shown in Figure 11 (d) was indexed as a basal plane, suggesting that the FIB lift-out was on the basal plane (0001) which was the most favourable crystallographic plane for dislocation movement in SiC. Figure 11 (c) shows an enlarged area with no cracks observed, indicating the plastic deformation was purely due to dislocation activity.

Fig. 11, TEM images of (a) the overall lamella, (b-c) the enlarged area underneath imprint and (d) the diffraction pattern of the grain beneath the imprint for the imprint caused by 60 mN load.

FIB lift-out was also performed on the indent corresponding to 100 mN indentation, with observed cracking. Figure 12 shows the lamella with a clear view of the residual imprint, the

evidence for inelastic deformation such as dislocations, deformation twinning, microcracks, stacking fault, as well as large radial/lateral cracks. According to Fischer-Cripps [7], radial-median cracks are cracks extending into the specimen, whereas, lateral cracks are horizontal cracks caused by residual tensile stresses that formed underneath the surface. The enlarged TEM images in Fig. 13 provide such details. Dislocation pile-ups and deformation twinning were observed beneath the imprint. Deformation twinning was also seen in bioceramic armour which was believed to be an efficient energy dissipation mechanism [19]. In addition, discrete microcracks were nucleated at the twinning site (Fig. 13 (a-b)). According to Shin et al. [32], tensile stresses initiate dilatant crack at the twinning position thereby forming a transgranular crack. Radial-median cracks were observed beneath the imprint (Fig. 13 (c)) and according to Lawn and Swain [18], such cracks usually initiated from the flaws induced by plastic deformation. TEM image showed the occurrence of severe plastic deformation, manifested as stacking faults and the intense diffraction contrast (Fig. 13 (c)). The initiation of cracks for SiC was caused by stress concentrations beneath the indenter tip as a result of stacking fault and deformation twinning, leading to the development of a visible microcrack. In addition, shear bands were seen beneath the imprint and the interaction between shear bands and stacking fault lead to the initiation of microcracks (Figs. 13 (c) and (d)). Similarly, in the work of Zhao et al. [44], microcracks were formed by the interaction of shear bands for polycrystalline 3C-SiC. According to the TEM observations for both 60 mN and 100 mN indentation, it was concluded that prior to crack formation, dislocation activities were the major sources for inelastic deformation. With increasing load, stacking faults, deformation twinning and shear bands started to occur, contributing further to the plastic deformation. Eventually, the formation of microcracks was observed, facilitating the dissipation of strain energy.

Fig. 12, TEM image for the FIB lift-out lamella, showing stacking fault, dislocation pile-ups, microcracks and radial-median cracks.

Fig. 13, TEM images showing (a-b) deformation twinning and microcracks and (c-d) stacking fault, shear bands and microcracks.

4. Conclusion

Indentation experiments across length scales were carried out using Vickers indenter to analyze the mechanical response of polycrystalline 6H-SiC at different load levels. Major conclusions are highlight below.

- For a load less than 30 mN, the imprint size was found smaller than that of a single grain, and therefore, grain orientation had an impact on the force-displacement responses and

the measured material properties.

- For microscopic and nanoscale indentations, the elastic modulus was found to be dependent on the load level. The higher the load the lower the elastic modulus, due to the size effect.
- For nanoscale indentation, the hardness dropped with increasing load. While, for indentations made at the microscopic and macroscopic scale, the hardness remained relatively constant.
- Cracking was observed for indentation carried out at microscopic and macroscopic scales, and the fracture toughness increased with increasing loads which ranged from 1.36 to 2.52 MPa m^{1/2}.
- Dislocation movement was the major source for the plastic deformation at low loads. With increasing load levels, twinning and stacking faults were observed, contributing further to the plastic deformation.

5. Acknowledgement

The authors acknowledge the use of facilities within the Loughborough Materials Characterisation Centre.

6. References

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