Localized mechanical property assessment of SiC/SiC composite materials

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Abstract

SiC fiber-reinforced SiC matrix composites (SiC/SiC) are under consideration as a structural material for a range of nuclear applications. While these materials have been studied for decades, recently new small scale materials testing techniques have emerged which can be used to characterize SiC/SiC materials from a new perspective. In this work cross section nanoindentation was performed on SiC/SiC composites revealing that both the hardness and Young's modulus was substantially lower in the fiber compared to the matrix despite both being SiC. Using Scanning Electron Microscopy (SEM) it was observed that the grain growth of the matrix during formation was radially out from the fiber with a changing grain structure as a function of radius from the fiber center. Focused ion beam machining was used to manufacture micro-cantilever samples and evaluate the fracture toughness and fracture strength in the matrix as a function of grain orientation in the matrix. Additionally microstructural characterization techniques like Raman Spectroscopy, X-ray diffraction, and microtomography were used to evaluate differences in the matrix and fibers of the composite.

Keywords: A. Ceramic-matrix composites (CMCs), B. Fracture toughness, C. Micro-mechanics, B. Hardness

Introduction

Fission and fusion reactors are attractive options to meet the energy demands of our economy and society by providing economically competitive stable core energy generation while having a minimal detrimental impact on our environment [1,2]. Due to these attributes there have been many developmental efforts to improve and design new types of reactors [1,2]. Several advanced reactor concepts are considering significantly higher operating temperatures for process heat production and also more efficient thermodynamic cycles [3-5]. Both call for more suitable materials for high temperature applications. In addition, the recent Fukushima events have illustrated that materials with little hydrogen production in steam in the event of a Loss Of Coolant Accident (LOCA) need be evaluated in order to potentially mitigate the hydrogen issue during LOCA events. Due to the slow reaction kinetics with water and steam [6,7], as well as the high strength at elevated temperatures, and low thermal neutron cross section, SiC is being considered as a structural material for a range of advanced reactor concepts [8-12] as well as light water reactor cladding. Monolithic SiC does have the challenges of low ductility and no tolerance against brittle fracture therefore SiC matrix SiC fiber (SiC/SiC) composites are being considered to mitigate these challenges [13, 14]. The use of SiC/SiC composites in reactor applications can provide superb safety margins for high temperature stability and radiation resistance to neutrons [15]. Challenges remain for SiC/SiC composites especially in respect to understand the degradation of the material under radiation condition. It has been shown that properties like thermal conductivity degrade under neutron irradiation condition [16-18]. The SiC/SiC composites materials have excellent chemical compatibility with the gas

(He) and liquid (Li,Pb) coolants proposed for fusion and advanced fission reactors [15, 19-21-22]. While these points are important for potential reactor application it is not the main focus of this study.

However, SiC/SiC composites have challenges. While better than monolithic SiC in terms of fracture behavior [13,14,22] it does not have the ductility and fracture toughness of a metal. Design challenges related to joining and material handling remain in applying this material to nuclear reactor needs within a radiation environment [23, 24]. One of the most critical challenges is achieving consistent mechanical properties over the 14 ft long fuel rod. As for all composites the mechanical properties of a composite structure is determined by the interplay of the individual components; in this particular case by the interplay of fibers with the surrounding matrix material [11,25]. This is facilitated by an interface layer, such as pyrolytic carbon, located between the fibers and matrix. Reinforcing fibers coated with an interface layer can bridge cracks, inhibit crack propagation, and slide or pull out of the matrix, affording improved mechanical performance [13,15,19]. However, as interfacial shear strength improves the overall composite strength, it can reduce the toughness due to decreased fiber sliding and pullout [26,27]. Various composite designs attempt to balance these toughening and strengthening characteristics in order to optimize material properties for nuclear applications. While the strength and performance of the entire SiC/SiC composite structure is of interest, it can help structural modeling efforts to know more of the behavior of individual components. In addition it can be interesting to compare the properties of the deposited matrix SiC to the properties of bulk SiC as well as investigate the properties of the fibers themselves. In this research we study the separate

mechanical properties derived from the fiber and the matrix of the SiC/SiC composites. In this work, modern nanoscale and microscale mechanical testing techniques like nanoindentation and micro cantilever testing, will be combined with microstructural characterization techniques (x-ray diffraction, Raman spectroscopy, SEM and microtomography) in order to provide insight about the properties of individual composite constituents. The understanding of the individual composite constituents can lead to a better understanding about their interplay as a composite and identifying the weakest link. Small scale mechanical testing is required in order to test the matrix and fibers separately in the as manufactured composite. This work is a first step toward understanding the materials intrinsic properties rather than the extrinsic property as a composite.

Experimental part

Two different 2 directional (2D) SiC/SiC developmental composites samples, GA-1 and GA-2, were provided to the University of California Berkeley (UCB) by General Atomics (GA). These samples are not fully infiltrated at the macro-scale; however, the regions selected for analysis in this work were fully infiltrated at the micro-scale. Both specimens utilize commercially available nuclear grade stoichiometric SiC fibers (Tyranno SA3). The matrix of the SiC/SiC composite was processed using the chemical vapor infiltration (CVI) method which has the advantage of low thermal and mechanical stress during the densification process and produces a stoichiometric, crystalline β -SiC which is highly stable to neutron irradiation [10,25,28,29]. The SiC/SiC composites were polished using standard silicon carbide papers up to 4000 grit followed by diamond polish with 1 μm diamond grit.

Nanoindentation was performed on the samples, in both individual fibers and matrix, using a Micro Materials NanoTestTM (UK). The nanoindenter tip (Diamond-Berkovich) was calibrated using fused silica prior to the measurements. The calibration leads to an area function correcting for geometrical inconsistencies in the used diamond tip. The indents were placed 2 to 3 μm apart and had depths of 150nm or 200nm for higher spatial resolution. The nanoindentation was performed in both cross section and lengthwise on the fibers as well as on large pieces ($>1\text{mm}$). The nanoindentation experiment was carried out using the depth vs. load hysteresis with indents being terminated using depth control. The thermal drift correction data was collected post-indentation. The hardness (H) and reduced indentation modulus (E_r) were calculated from the indents utilizing the Oliver and Pharr method [30].

A Quanta 3D field emission gun (FEG) scanning electron microscope with focused ion beam (SEM/FIB) has been used in this research. FIB surface milling provided a clean surface for better imaging of the samples and to prepare cross-section of the fibers. Energy dispersive spectroscopy was carried out on the fibers using an Oxford EDS attached to the SEM/FIB. X-ray diffraction of the sample was complete with a Bruker D8 Advance Bruker D8 Advance Powder x-ray diffractometer in Bragg-Brentano geometry ($\theta - \theta$ setup) using $\text{Cu K}\alpha$ radiation ($1.54 \text{ \AA}$). A Renishaw Invia Laser Raman spectrometer was used for the Raman characterization. The excitation wavelength used was 488 nm line of an Ar^+ ion laser at an incident power of 10 mW. The area was analyzed using a 2 μm diameter spot through a standard $\times 50$ microscope objective. The spectra were collected with a 10 s data point acquisition time, a spectral range of $200\text{-}2000 \text{ cm}^{-1}$ and a spectral resolution of 2 cm^{-1} .

Image J [31] has been used for the image processing of the SEM images. The Microtomography beam line (8.3.2) at the advanced light source (ALS) at Lawrence Berkeley National Laboratory (LBNL) was used to perform x-ray tomography of the samples. A 10x lens was used with the system resulting in 0.6 micron pixels on the detector and 1025 images were taken in order to from the reconstructions.

Fracture toughness has been studied using a Hysitron Pi-85 Picoindenter and a Micro Materials NanoTest[™]. Micro cantilevers were manufactured parallel to the grain direction (radial to the fiber) and perpendicular to the grain direction (tangential to the fiber). There were 11 (6 parallel and 5 perpendicular) micro cantilevers manufactured by the S.G. Roberts method [32]. The FIB procedure was as follows: three trenches were cut 20 μm wide and 10 μm deep with a 15 nA beam current on three sides to shape bend bar. These trenches formed a U-shaped trench cut. The geometry was refined using a 1 nA beam current. The sample was then titled to 45 degrees along the length axis to shape cantilever and the base of the cantilever was undercut from both sides using a 3 nA beam current. Due to the anticipated fracture toughness measurements, the bend bars were notched using a 10 pA beam current. The fracture toughness K_{Ic} can be determined using the equation 1 from D. Di Maio and S.G. Roberts;

$$K_{Ic} = \sigma_c \sqrt{(\pi a)} F(a/b) \quad (1)$$

where σ_c is the fracture stress, a is the crack length (notch depth) and $F(a/b)$ is a dimensionless shape factor dependent on sample geometry [32]. The stress is calculated using a standard mechanical approach.

Results:

Mechanical properties by nanoindentation

The mechanical properties, H and E_r , were evaluated in cross-section in order to measure the hardness of the fibers. In Figure 1a, load-displacement curves for sample GA 1 of different areas have been represented (matrix and fiber). The softer part of the sample requires a lower load to make a 200 nm penetration depth indent, indicating that the fibers have lower H than the matrix. The E -modulus was also found to be lower which has been reported previously and was found to be the reason matrix often micro cracks prior to transferring the load to the fibers [33-35]. 2D dimensional hardness maps of the material were obtained and plotted in 3D with the hardness on the Z -axis in Figure 1b. The post indentation analysis by SEM is shown in Figure 1c, revealing the pyramidal imprint of the indents, and in Figure 1d, where cracks from indentation were observed.

Since the site of each individual indent was located utilizing SEM, all hardness data can be summarized in Table 1 and the words “fiber” or “matrix” have been used to indicate the location of the indents. Figure 2a shows a tilted SEM image of sample GA-1 where the indents are clearly located inside and outside the fiber. The values of the H and E_r as a function of the matrix/fiber have been plotted in Figure 2b. On average, the fibers (26-30 GPa) are significantly softer than the matrixes (38-43 GPa). Similar values for the matrix have been found in [36] for CVI produced SiC in TRISO particles.

Mechanical properties by micro cantilevers

During the SEM observation it was observed that the SiC matrix grains are elongated along the radial direction from the fiber surface which is known previous literature [17]. In

order to evaluate if there is any difference between the fracture toughness depending on the direction of grain growth, two different groups of bend bars were manufactured using the FIB instrument on sample GA-2. Five bend bars were cut parallel to the grain growth (radial direction to a fiber) while six bend bars were cut perpendicular to the grain growth (tangential direction to the fiber). Figure 3 shows a SEM image of one of the perpendicular bend bars before (a) and after testing (b). In Figure 3c, a load-displacement curve has been plotted from a bend bar oriented parallel to the grain growth direction. Ultimate failure of this cantilever could be observed at a force of $\sim 640 \mu\text{N}$. The average values of all the micro cantilever fracture toughness tests are shown in Table I and in Figure 3d. The micro testing showed that the cantilevers in parallel with the grains had slightly higher fracture toughness than the micro cantilevers perpendicular to the grain orientation.

Microstructural analysis

The micro tomography 3D reconstructions of the composites can be seen in figure 4. The aim of the microtomography was to assess the internal porosity of the composite and the overall structure of the composit(fiber arrangement). When analyzing the reconstructions it is noticed that the fibers have a different contrast than the matrix. This is particular interesting as this shows that the fibers and the matrix may have different constituents and/or densities and thus different contrasts in the X-ray microtomography imaging supporting the fact that the fibers do contain different constitence than the matrix. In the images we can see an internal porosity of the composite but as stated above this is expected due to the CVI technique. However, it does show if the pores are connected or not which is a valuable parameter when it comes to fission gas release from a cladding.

Therefore the tomography method can be a valuable tool for quality control of a fuel cladding. The images used to make the 3D reconstructions were also used to measure the porosity in the in center of the fiber tows. The measured porosity in the center of the tows was $21.5 \pm 0.6 \%$. This technique gives this unique ability with perform non-destructive testing to measure internal porosity of the composite.

Microstructural analysis using SEM has been performed in order to understand the lower mechanical behavior of the fibers. Figure 5a shows a cut of a fiber in half in the matrix for sample GA-1. The pyrolytic carbon interface layer is clearly visible while a significant number of “dark spots” that appear significantly different than the surrounding SiC material.. The cross-section SEM image shows dark spots deeper than the surface indicating that they are microstructural part of the fiber. It has been in shown in [16,17] that SA3 fibers contain excess carbon which is believed to be the dark spots. In [16,17] the amount of carbon is less than reported here bu it is known that strong batch to batch variations occur. A top view of one of the fibers is presented in Figure 5b. Based solely on SEM analysis, it was determined that the spots are excess carbon using Energy dispersive spectroscopy (EDS) and Raman spectroscopy.. Evlating the EDS line scan a gradual decrease of the Si signal is observed across the center of the fiber, however, C signal increased in the middle and at the edges of the fiber. Further XRD and Raman spectroscopy were conducted on the specimens.

SiC has a high number of compositional and structural forms and more than 250 different polytypes has been described [37]. The cubic β -SiC (3C-SiC) and the hexagonal α -SiC (6H-SiC) have been the most investigated polytypes. The XRD normalized

diffraction patterns of the as-prepared samples and the fibers are shown in Figure 6 in the region 20-80 (2θ). The diffraction patterns of both samples are very similar and show the peak with higher intensity at 35.7° , that it is associated with the (111) orientation. Also, the other typical reflections from the cubic SiC are observed. The same peaks attributed to the cubic SiC are found in the diffractogram from the fibers, although there are some differences in their relative intensities. However, the main difference of the fiber diffractogram and the samples is the presence of a small peak at in the fibers at 34.2° , associated with the (101) reflection of the α -SiC [37]. Similar results have been previously reported for similar fibers by XRD [38, 39] and Select area electron diffraction (SAED) [40].

Figure 7 shows a SEM image and the Raman spectra for the fiber and matrix from sample GA-2, which were taken from a radial distance from the center of the fiber. Through the following text 0 μm will be assigned as core-fiber (CF), 3 μm as inner-fiber (IF), 6 μm as outer-fiber (OF) and 9 μm as matrix. Peaks have been identified corresponding to the different vibrational modes of SiC and C. β -SiC phase is denoted by the presence of two narrow peaks at around 796 and 972 cm^{-1} and the graphitic carbon phase is characterized by the bands at 1370 and 1596 cm^{-1} . A decrease in the intensity of the carbon signal and an increase of the SiC vibration modes are observed moving out from the core of the fiber to the matrix.

In order to study the SiC phase, the Raman spectrum in the vicinity of the SiC peaks (Raman shift of 650-1050 cm^{-1}) was more closely examined (Figure 8a). The β -SiC (3C-SiC in Ramsdell notation) exhibits two sharp bands, corresponding to the transverse optical

phonon (TO) mode at 796 cm^{-1} and longitudinal optical phonon (LO) at 972 cm^{-1} . The different spectra measured from the fiber and matrix showed the peaks but at different intensities. A feature shown in the Raman spectra of the matrix is a shoulder band at around 766 cm^{-1} . This band could be observed for the OF but it is not so noticeable for the rest of the spectra and it is associated to another different polytype of SiC. It is believed that a small contribution of a different polytype could be present in the matrix or in the fibers, as showed previously in similar research works [41,42]. The Raman spectra of the matrix also showed a broad background assigned to a so-called “density of state” (DOS) contribution superposes between 700 and 1000 cm^{-1} . Such contribution is a common feature of disordered SiC crystals and often these disorder zones are sandwiched between different polytype layers or located near the surface [40]. An expanded Raman spectra graph for the carbon region is shown in Figure 8b. The graphitic material is confirmed by the observance of the two characteristic D- (disordered mode) and G- (graphitic) bands of carbon at 1350 - 1390 and $\sim 1580\text{ cm}^{-1}$, respectively [43,44]. It can be seen in Figure 8b that the Raman spectra of the matrix is different from that the others, in that none of the characteristic carbon peaks are observed. While the fiber always shows D- and G-bands, the spectra of the matrix shows small peaks at around 1525 , 1621 and 1705 cm^{-1} . These features are associated to second-order Raman lines from scattering by overtones of SiC [45] and they have been previously observed in SiC fibers [40, 46]. A small shoulder described in the literature like D' could be observed in the spectra taken inside the fibers [46]. An analysis of the peak intensities for the TO SiC vibration mode has been plotted in Figure 9a together with the full width at half height (FWHH). A decrease in the intensity of the TO SiC

vibration mode is observed from the matrix to the core of the fiber. It is noticeable that the FWHH does not follow the same trend than the TO intensity. FWHH of the matrix indicates that the matrix shows the smallest crystal size compared with the fiber. Focusing on the fiber, the smallest crystal size is found at the core of the fiber and it increases on the outer part of the fiber. The intensities of the D- and G- bands are plotted in Figure 9b together with the peak intensity at 1525 cm^{-1} (attributed to SiC). The Raman scattering efficiency of SiC is much lower than that of carbon [47] and it could explain why this peak is not observed in CF and IF where the amount of carbon has found higher. For the fiber, it was observed that the intensity of D- and G-bands increases closer to the core, indicating a higher amount of carbon. The I_D/I_G ratio has been plotted in the same graph and some differences could be observed. The measurement of the outer layer is influenced by the thin pyrolytic carbon film in between the fiber and the matrix and it shows the biggest ratio value. Within the fiber, a higher ratio was seen in the core-fiber. It is believed that the nature of the thin layer that surround the fiber is different from the carbon that it is in the fiber, as seen by the differences in the I_D/I_G ratio.

Additional microstructural analysis of the fibers was complete with the production of a SEM-STEM (Scanning Electron Microscopy – Scanning Transmission Electron Microscopy) foil. An overview of the foil can be seen in figure 10a while a more detail image of the fiber can be seen in figure 10b. The images of the foil were taken with a FEI Helios SEM/FIB dual beam instrument at Los Alamos National Laboratory (LANL). An analysis of the images shows that the carbon enrichments (black spots) are sitting at grain boundaries. Also in the image it is observed that there is a gradient in the crystal size with the smallest crystal size being in the center of the fiber ($\sim 140\text{ nm}$) and largest near the

perimeter ($\sim 220\text{nm}$) which was also observed in the Raman analysis that was performed. These grain size are similar to those given [17] for Tyranno fibers. There also appears to be twinning or stacking faults in some of the grains of the fibers as reported in pervious literature [17] but additional high resolution TEM would be need to verify this phenomenon

Discussion

When analyzing the results of the nanoindentation performed on the SiC/SiC composite sample there is a noticeable difference in the H and E_r between the matrix and fiber. The matrix has values of approximately 38 GPa in hardness meanwhile the fiber showed 26 GPa. The fiber is 25% softer than the matrix. This difference may just be an artifact of composite behavior on the microscale due to the possibility of: a) movement of the fiber during testing due to the pyrolytic carbon interface layer, similar to what occurs during fiber loading, b) the pyrolytic carbon interface layer being soft enough to allow the SiC to expand during the indentation. However, it did not seem likely that the fiber was sliding since they were not depressed into the matrix after testing. In addition the pieces tested were thick ($\sim 5\text{mm}$) and mounted in epoxy which would have prevented the slide of the fibers. Comparing the indentation forces as seen in figure 1 with fiber push out forces as reported in [48] it is found that the forces used for indentation is one order of magnitude less than what it takes to move a fiber in a $\sim 100\mu\text{m}$ thin slice. It also did not seem likely the fiber was expanding during the indentation process because the carbon interface was not displaced after testing. Therefore it is likely that the lower hardness is real, and is a result of inhomogeneous regions in the fiber that contain a higher amount of carbon as shown by

EDS and Raman spectroscopy. The SEM images of the samples indicate darker spots in the fibers themselves (Figure 11). Figure 11 also displays a gradient throughout the fiber with high concentration of carbon in the center and lower concentration outside while the fibers grain structure is visible. It appears that the excess carbon in the fibers is at the grain boundaries from SEM-STEM images of the fiber seen in figure 10.

From the SEM observation alone, it is difficult to determine if these dark spots are either carbon rich regions or internal porosity; however, EDS and Raman spectroscopy results verified that the dark spots are carbon enrichments areas. These carbon-rich spots can also explain the significantly weaker mechanical properties measured in the fiber compared to those of the SiC matrix. In order to quantify the amount of graphite and its effect towards mechanical properties, Image J analysis was performed and it is shown in Figure 11c. The quantification of the graphite work as follows: rings with different radius (1 μm , 2 μm , ..until the size of the fiber) were drawn around the center of the fiber. The mask function was then used to create a binary image of black and white where the black was the originally black (carbon) in the SEM image. The area of the black regions was then measured and compared to the overall area of the circle to find the % of carbon. It was observed that in the center of the fiber (the smallest circles), 30-35% of the area consists of graphite, and in some individual fibers, this can be as high as 50%. Applying the simple rule of mixtures allows an estimation of the expected mechanical property changes of the fiber due to the graphite particles. This approach is expressed in Equation 2.

$$H_{\text{estimated}} = \text{Vol\%}_{\text{graphite}} * H_{\text{graphite}} + \text{Vol\%}_{\text{SiC}} * H_{\text{SiC}} \quad (2)$$

Assuming that 70% by volume in the center of the fiber is SiC and its hardness is 38 GPa and 30% is graphite and its hardness is 4 GPa [49] it can be calculated that the hardness of the center of the fiber is approximately 28.7 GPa which is in the range that was measured with the nanoindentation (26-30 GPa). The same approach can be used for the Young's modulus (E) which results a calculated value of 327 GPa, in good agreement with the measured modulus values which ranged between 303-348 GPa. Both theoretically calculated values for H and E are in reasonable agreement with the actual measured values considering the error of the measurements. This approach ignores the fact that the graphite particles in the fiber and SiC of the fiber also have interface strengthening. The microstructure of the SiC/SiC composite reveals that when the SiC in the matrix begins to form the grains grow radially outward from the fiber. This phenomenon can be seen in Figure 12. As the grains grow outward from the fiber and meet other grains prevalent voids and cracks are produced which could be seen in Figure 1c-d. These zones could be the initiators for cracks in the matrix and have lower H and Er values. Since the pores are empty space and do not contribute to the strength of the material and the indent is placed on top of the more porous region it is obvious that the H and E-modulus must be lower in these areas. The values for the void zone are approximately 34.2 GPa for the hardness and 305.5 for the E-modulus. Further experiments were performed to see if there was a hardness gradient in the matrix due to the radial formation pattern of the grains. When the nanoindentation testing was completed the results indicated that there was no hardness gradient in the matrix.

Another structure that was observed was a helo-structure, composed of the small grains adjacent to the fiber that form a ring around the fiber. The structure is built up during an initial infiltration and ends when processing parameters are changed. Cracks appeared after indentation much more frequently near the helo-rings than in other areas. These are visible in Figure 1d under the top fiber and may suggest the outer edge of these rings are potential initiators of cracks.

The micro cantilever test gave the fracture toughness of matrix SiC of 3.37 ± 0.36 MPa m^{1/2} for the parallel and 2.29 ± 0.31 MPa m^{1/2} for the perpendicular, in good agreement with reported fracture toughness values for bulk CVD SiC [16]. This good agreement suggests that these micro-cantilever tests could be a valuable approach in cases where material availability is very limited, such as test on irradiated specimens. The slightly lower fracture toughness value measured for the perpendicular cantilevers is likely due to an effect of the grain boundaries aligned perpendicular to the cantilever orientation.

The strain of each micro cantilever test was measured, and the E-modulus of the material was calculated from the resulting stress vs. strain curves and compared with the nanoindentation performed earlier. The strain at the notched point of the sample was found using the formula below.

$$\epsilon(x) = \frac{1}{c} \left(\frac{1}{2} - \frac{x}{c} \right) \frac{6}{\sqrt{3}} \frac{w(x)}{z} \quad (3)$$

Where c is overall length of cantilever, x is the lever length (point of contact to the notch), z is half the height, w(x) is the displacement. A representative plot of the stress vs.

strain curves is shown in Figure 13, and a linear fit was applied to obtain a Young's modulus of ~472 GPa this bend bar.

The average Young modulus calculated using the micro cantilevers were 473.5 and 470.2 GPa for the parallel and perpendicular grain orientation for the matrix of the hardest sample. The value obtained by Nanoindentation was 482.3 GPa, and is in very good agreement with the value of the Young's modulus calculated with the micro cantilever method and other literature values [16].

Conclusion

The work performed here clearly shows that nanoindentation can be used to test the individual components of a composite and evaluate its mechanical properties. The ability to see constituent properties individually allows us to see how the constituents interact as a whole composite and identify potential weak spots. It is the intention to provide experimental techniques and data to evaluate the intrinsic properties of each individual constituent which then could allow a comprehensive model of the overall behavior of a composite structure. In addition microstructural analysis shows the reason for the behavior of the individual constituent (fiber and matrix) separately which is in agreement with the literature. The nanoindentation results indicated that the fiber is significant softer than the matrix and EDS and Raman confirm nanometer sized graphite particulates embedded in the fiber as seen in previous literature [16,17]. After the study of the phases by EDS and Raman spectroscopy, the rule of mixture was used to estimate the H and E_r of the fibers, in good agreement with the experimental values. This indicates that great care in evaluation and quality control of commercially-produced fibers may be needed to ensure consistent

composite properties are achieved. The results of the micro cantilever carried out on the hardest sample showed that the grain orientation in the matrix had an effect on the fracture toughness of the matrix.

These results give modelers more information about the composite to more accurate predictions of the overall composite behavior in a structural application. It could also lead to a better understanding of the mechanical property changes that happen during the lifetime of the composite because small scale testing can observe the changes in each component of the composite. These observations of the changes in each component could lead to better design of the composites.

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Table I: Description of the samples studied in this research: density and mechanical properties by means of nanoindentation and micro-cantilever analysis

	Nanoindentation						Micro-cantilevers			
	Fiber			Matrix			Grain orientation			
							parallel		perpendicular	
	H	Er	E	H	Er	E	K_{IC}	E	K_{IC}	E
	GPa						MPa $\sqrt{m}$	GPa	MPa $\sqrt{m}$	GPa
GA-1	26.44 ± 4.10	245.97 ± 29.03	303.0 ± 28.8	38.5 ± 2.6	333.5 ± 14.8	455.0 ± 14.5				
GA-2	30.00 ± 4.55	273.67 ± 23.47	347.8 ± 23.2	43.0 ± 2.4	347.4 ± 14.8	482.3 ± 14.5	3.57 ± 0.36	473.49 ± 15.23	2.39 ± 0.31	470.21 ± 19.35

Figure 1. (a) Typical load displacement curves obtained from GA-1, (b) 3D Plot of the hardness values on this particular fiber and (c) SEM image after indentation. (d) SEM image after indentation for another fiber in GA-1.

Figure 2. a) Tilted SEM image of fiber of GA-1 after nanoindentation. b) Hardness and reduced modulus of the samples as a function of matrix/fiber.

Figure 3. SEM images of a parallel oriented micro-cantilever before (a) and after (b) testing. (c) Load-displacement curve for (a) and calculated fracture toughness (d).

Figure 4: 3D reconstructions of the composite from the microtomography performed. The reconstructions allow the internal structure of the composite to be investigated.

Figure 5. FIB cross section cut of an individual fiber in GA-1 (a) and EDS line scan over a fiber in GA-2 (b).

Figure 6. X-ray diffraction patterns of the fibers and samples GA-1 and GA-2. (• and * indicates β -SiC and α -SiC respectively).

Figure 7. a) SEM image of one of the fibers and b) Raman spectra of sample GA-2.

Figure 8. Expanded Raman spectra for the SiC (a) and C-C region (b).

Figure 9. a) Intensity and FWHH of TO SiC vibration mode. b) Intensity of SiC at 1525 cm^{-1} and D and G bands and I_D/I_G .

Figure 10: a) Overview SEM-STEM image of the foil. b) A detailed SEM-STEM image of the microstructure of the fiber.

Figure 11. SEM images of a fiber from sample GA-1: (a) cross-section (a) and top-view (b). c) Image j analysis of the center of the fiber

Figure 12. Radial grain growth of the fibers in the SiC/SiC composite material in sample GA-1.

Figure 13: Representative stress vs. strain plot for a micro cantilever (parallel orientation).