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Fracture and flexural characterization of SiC_w/SiC composites at room and elevated temperatures

HASSAN MAHFUZ, DURGA P. ZADOO, F. WILKS, MD. MANIRUZZAMAN, SHAIK JEELANI
Materials Research Laboratory, Tuskegee University, Tuskegee, AL, USA

The fracture and flexural behaviour of monolithic SiC and SiC–whisker reinforced SiC composites (SiC_w/SiC) has been investigated at room and elevated temperatures. Flexure and fracture tests were conducted in a four-point beam configuration at 23 °C, 800 °C and 1200 °C to study the effects of whisker reinforcements especially in respect of mechanical and thermal stability at high energy environments. Flexural strengths and fracture toughness data within the test temperature range are presented in graphical as well as in Weibull form, and experimental observations are analysed and discussed. Increase in flexural strength as well as in fracture toughness has been observed with the whisker reinforcement. However, it was found that the trend discontinues after a certain range of temperature. Post-failure analyses have been performed with the scanning electron microscope (SEM). Formation of glass phase has been observed at the whisker/matrix interface and the crack growth was found to be shifting from intergranular to transgranular with the rise in temperature. Effects of whisker reinforcement and the degradation of flexural and fracture properties at elevated temperature are investigated. Ultrasonic velocity measurements have been performed through the thickness of the untested as well as fractured specimen, and the variation in the sonic wave velocity is discussed in this paper.

1. Introduction

Ceramic matrix composites (CMC) have emerged in recent years as potentially excellent engineering materials because of their superior mechanical properties such as specific strength and stiffness at elevated temperature. These attractive thermomechanical properties of CMCs accompanied by their low moisture susceptibility and long-term thermal stability at high energy environments have generated considerable interest among researchers. Significant improvements have been observed in the 1990s in the manufacture of these composites. However, the very brittle nature of ceramics imparts one of their most undesirable properties – fracture toughness. Recent studies have shown that fracture toughness of monolithic ceramics can be moderately improved by the incorporation of whiskers into ceramic matrices [1–4]. There are several fracture toughening mechanisms for whisker-reinforced composite materials: crack bridging, whisker pullout, crack deflection, microcracking and change of fracture mode from inter- to transgranular [5, 6]. These mechanisms predict a different dependence of toughness upon microstructure. Although the failure of the ceramic is still catastrophic, the whisker reinforcements have generated considerable interest because of the relatively simple manufacturing process, namely, the powder metallurgy method. Among various ceramic matrices, SiC is considered to be the most suitable for high-temperature applications because of its mechanical integrity and resistance to oxidation and corrosion at elevated temperature. Due to this thermomechanical stability SiC is being extensively investigated as an oxidation protection coating for carbon/carbon composites in aerospace applications [7, 8].

Knowledge of the critical intensity factor, K_{Ic}, along with the elastic modulus, the defect size and the relative geometries of the defect and the structure, it is possible to predict the failure stress of that structure. The fracture toughness approach, therefore, constitutes a useful failure criterion for ceramics [3]. With brittle matrices such as ceramics, catastrophic failures are common once the fracture stress has been reached. Therefore, a measure of fracture toughness of the composite is essentially a measure of success of its fabrication process. It has also been observed that the surface chemistry of whiskers (in whisker-reinforced composites) plays a major role in forming the interface which in turn significantly controls the fracture toughness of the composites. It has been found that at elevated temperatures, presence of oxygen in some fibres triggers oxidation, and the subsequent degradation of fibres causes considerable concern for high-temperature applications. In the current research, whisker toughening of SiC matrix with SiC whiskers was used to investigate the effects of temperature in the flexural strength and fracture toughness. Monolithic SiC was also tested at corresponding temperatures to study and compare the improvements in the mechanical properties.

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2. Experimental details

2.1. Processing and specimen preparation

The processing and hot pressing of monolithic SiC and SiC_w/SiC composite was carried out at the facilities of Cercom*. The raw materials used were SiC powder supplied by Cercom, and SiC whiskers by Tokai Carbon†. SiC whiskers used in this study were TWS-100. SiC billet was supplied as "PAD" SiC, type B (Billet # 2–638–2f). The billet had fine-grained microstructure, characteristic of pressure assisted densified "PAD" SiC. The hot pressing was carried out in an inert atmosphere at 2080 °C and 2500 psi pressure. The bulk density of the billet (Alpha SiC) was 3.18 g cm^{-3} and the average grain size was 1.9–2.2 μm. The hot pressing of the composite billet was carried out in an inert atmosphere at 1750 °C and 3500 psi. Hot pressing temperature was limited to 1750 °C as per the recommendation of the manufacturer of SiC whiskers. The density of the composite billet was 3.225 g cm^{-3}. Billets of both monolithic SiC and SiC_w/SiC composites were prepared to study the improvements in flexural strength and fracture toughness values of the composite. All the specimens for the above tests were cut and machined at Bomas Machine Specialities‡. It was observed that whiskers tended to align in a direction perpendicular to the hot pressing direction. Accordingly, proper care was taken during specimen machining so that the whisker orientation was perpendicular to the direction of applied load.

2.2. Measurement of flexural strength

The flexural strength was measured for both monolithic SiC and SiC_w/SiC composites by using a 4-point bend test. An Instron 8502 machine with data acquisition system was used. The 4-point bend fixture was fabricated from cast SiC and the push rods were made out of alumina. The outer and inner spans of the fixture were 30 mm and 15 mm, respectively. The size of the test specimen used was 3 mm wide, 4 mm deep and 48 mm long, and is shown in Fig. 1. The cross-head speed for all tests was 0.508 mm min^{-1}. Equations derived from the Euler–Bernoulli beam theory [9] were used to calculate the flexural strength and stiffness.

2.3. Measurement of fracture toughness

Fracture toughness tests were carried out on chevron notched specimens. The 4-point fixture as mentioned earlier was used. The size of the specimen was same as that of the flexure specimen. The width of the chevron notch was 0.2 mm as shown in Fig. 2. The cross-head speed was same as it was with the flexural tests. The equation used for evaluating the fracture toughness of chevron notched specimen by 4-point bend test is as follows [10, 11]

$$K_{Ic} = \frac{P}{BW^{1/2}}Y \tag{1}$$

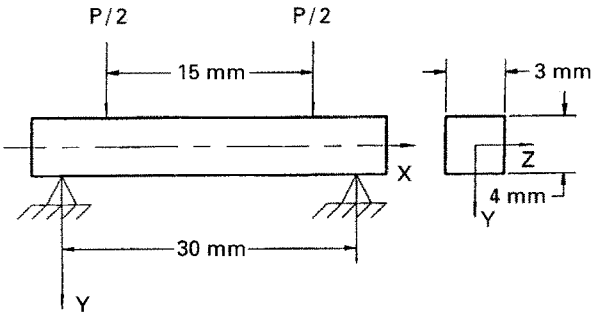

Figure 1 Four-point beam specimen.

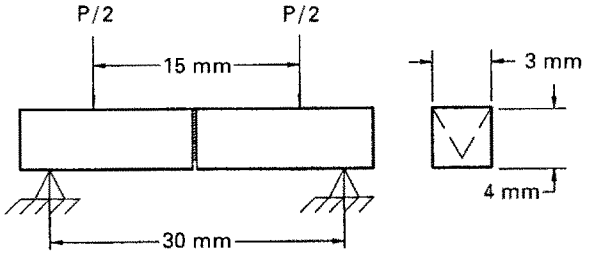

Figure 2 Chevron-notched specimen.

where

$$Y = (2.92 + 4.52\alpha_0 + 10.14\alpha_0^2)\frac{S_1 - S_2}{W} \times \left(\frac{\alpha_1 - \alpha_0}{1 - \alpha_0}\right)^{0.5} \tag{2}$$

$$\alpha_0 = \frac{a_0}{W} \tag{3}$$

$$\alpha_1 = \frac{a_1}{W} \tag{4}$$

where P = applied load, W = depth of the specimen, B = width of the specimen, S_1 = outer span for loading the bar specimen, S_2 = inner span for loading the 4-point bar specimen, a_0 = initial notch length (distance from crack mouth to chevron vertex), and a_1 = distance from crack mouth to intersection of chevron notch and specimen edge.

2.4. Non-destructive evaluation

The non-destructive evaluation of the flexure test specimens as well as untested specimens were performed in a Test Tech Ultrasonic Scanning machine equipped with a DAS-100 data acquisition system. Sound waves in the 2.25 MHz frequency range were produced by pulsing a lithium sulphite transducer which also functioned as the receiver element. The resulting waveform was digitized to eight bits and processed via a digital oscilloscope. The calculation of the wave velocity was performed from the time of flight using the following equation

$$t = 2(t_1 + t_2) \tag{5}$$

or

$$t = 2\left(\frac{d_1}{V_1} + \frac{d_2}{V_2}\right) \tag{6}$$

*Cercom Incorporated, 1960 Watson Way, Vista, CA 92083 USA
†Tokai Carbon, American Inc., 375 Park Avenue, Suite 3803, NY 10152 USA
‡Bomass Machine Specialities, 334, Washington Street, Somerville, MA. 02143 USA.

TABLE I Maximum deflection, flexural strain, flexural strength, fracture toughness and modulus of elasticity at various temperatures

Parameter	At 23 °C		At 800 °C		At 1200 °C	
	SiC_w/SiC	SiC	SiC_w/SiC	SiC	SiC_w/SiC	SiC
Deflection (mm)	0.05469	0.06472	0.04361	0.03635	0.03767	0.02954
Flexural strain ($mm\,mm^{-1}$)	0.001062	0.001256	0.000847	0.000705	0.000731	0.00057
Flexural strength (MPa)	500.9	468.1	436.9	412.0	270.7	324.5
Fracture Toughness ($MPa\,m^{-1/2}$)	5.96	3.43	5.83	3.10	–	–
Modulus of elasticity (GPa)	491.2	337.98	509.39	574.15	363.22	553.89

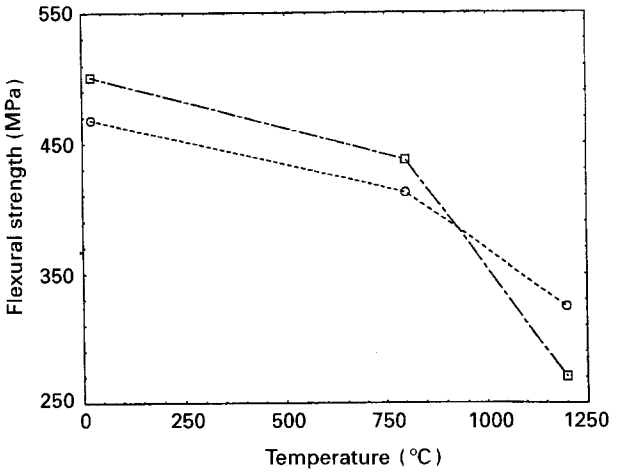

Figure 3 Flexural strength variation with temperatures. ⊡–⊡ SiC_w/SiC; ○–○ SiC.

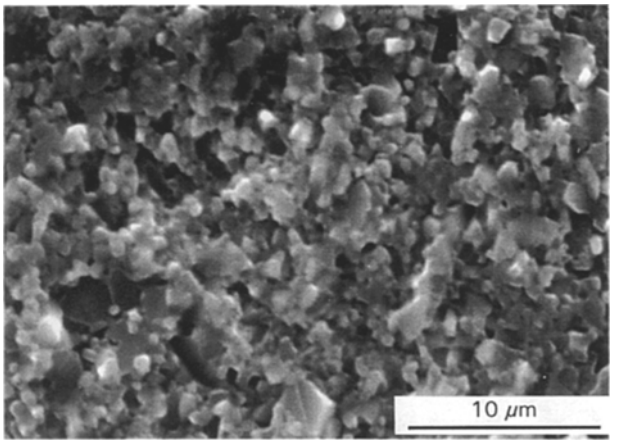

Figure 4 SEM micrograph of flexural specimen of SiC_w/SiC composites at room temperature

where t = total time of flight (s), t_1 = time of flight through the specimen (s), t_2 = time of flight through the water between the back surface of the specimen and the reflector plate (s), d_1 = specimen thickness (in), d_2 = distance between the back surface of the specimen and the reflector plate (in), V_1 = sonic velocity through the specimen ($in\,s^{-1}$), and V_2 = sonic velocity through the water ($in\,s^{-1}$).

3. Results and discussion

Both monolithic SiC and SiC_w/SiC composites were tested at room and elevated temperatures for flexural strength and fracture toughness. Maximum deflection (v_{max}), maximum flexural strain, flexural strengths, fracture toughness (K_{Ic}), and elastic modulii for both SiC_w/SiC and monolithic SiC, obtained from the flexure tests at various temperatures are shown in Table I. Five specimens of SiC_w/SiC and four specimens of monolithic SiC were tested at each temperature level for each type of test. Mean values of these tests are shown in the table. It is interesting to note from the maximum deflection and flexural strain data that the absolute values of both deflection and strain are smaller at elevated temperatures than those at room temperature indicating that the material becomes stiffer with the rise in temperature. A comparison of the flexural strengths of SiC_w/SiC with monolithic SiC, and the variation of strength with temperature is shown in Fig. 3. It is observed from the flexural strength data that the strength of SiC_w/SiC composite at room temperature increased approximately by 23% from that of the monolithic SiC. The improvement in strength is due to the whisker reinforcement. However, Fig. 3 shows that this improvement does not continue throughout the range of the test temperatures. Beyond 900 °C, SiC_w/SiC appears to show lesser strength than that of SiC. The figure also reveals that the monolithic SiC remains stable up to 800 °C and then loses strength rapidly. On the other hand, SiC_w/SiC composite loses strength more or less uniformly throughout the test temperature range, and at 1200 °C the strength values were found to be less than that of SiC. However, the decrease in flexural strength was gradual at elevated temperatures and there was no drastic loss in strength, suggesting a moderately stable system to sustain temporary overheating without any catastrophic failure [12]. It is believed that the uncoated SiC whiskers underwent partial degradation at elevated temperature which led to early failures.

SEM micrographs of flexural specimen of SiC–whisker reinforced SiC composites (SiC_w/SiC) are shown in Figs 4, 5 and 6. At room temperature, very limited whisker pullout is visible and fracture behaviour is mainly intergranular. But at 800 °C, it is mainly transgranular. Due to the change in fracture mode from inter- to transgranular, strength decreases with increase in temperature. At higher temperatures SiC

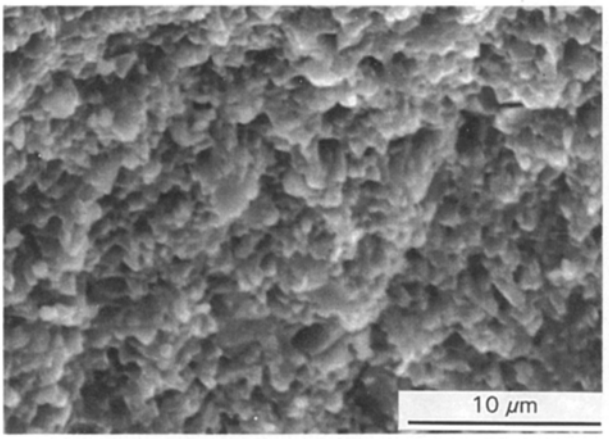

Figure 5 SEM micrograph of flexural specimen of SiC_w/SiC composites at 800 °C.

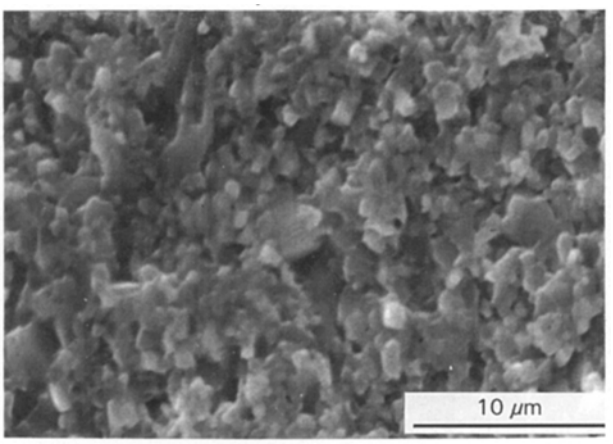

Figure 7 SEM micrograph of chevron-notched fracture specimen of SiC_w/SiC composites at room temperature.

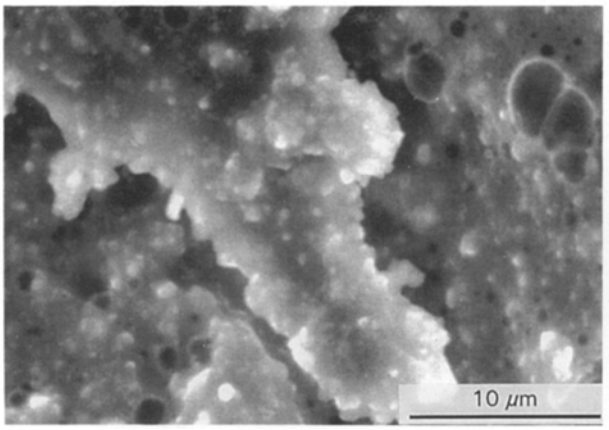

Figure 6 SEM micrograph of flexural specimen of SiC_w/SiC composites at 1200 °C.

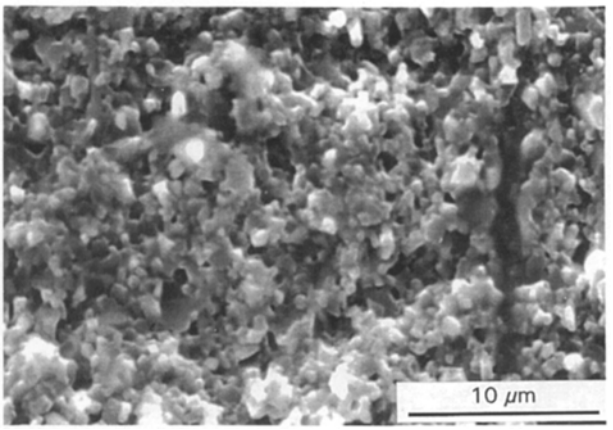

Figure 8 SEM micrograph of chevron-notched fracture specimen of SiC_w/SiC composites at 800 °C.

reacts with oxygen at the crack tip region, thus forming a glass phase [13] which is visible in SEM of the specimen shown in Fig. 6 at 1200 °C. This glass forms at the whisker/matrix interface and penetrates the matrix grain boundaries; that is, it fills the sites of potential microcracks and may even fill microcracks as they develop. At the same time the glass pockets and films provide weak paths for easy crack extension.

From the fracture toughness data it is observed that the value of K_{Ic} for SiC_w/SiC has increased from 3.43 to 5.96 by whisker reinforcement. This improvement in fracture toughness becomes more prominent at 800 °C. SiC_w/SiC composite maintains K_{Ic} value at 5.83, whereas, for monolithic SiC, the value drops to 3.01. The improvement in K_{Ic} both at room and elevated temperatures is attributed to the whisker pullout, crack deflection and microcracking due to the presence of whiskers. To determine the extent of whisker pullout and crack deflection, SEM of the fracture surface was carried out. The micrographs (Figs 7 and 8) show a limited whisker pullout both at room and elevated temperatures. Whiskers perpendicular to the crack plane have fractured close to the crack plane. The fracture behaviour at room temperature is intergranular but at elevated temperature it is transgranular. Nevertheless, intergranular fracture still occurred and more whisker pullout and porosity in matrix is visible at elevated temperature. Fracture toughness data presented in the Table I show that both SiC_w/SiC and SiC have become more brittle (reduction in K_{Ic} value) at higher temperatures. It is believed that this increase in brittleness is the cause of lesser strain at the failure condition. It is also observed that in the case of SiC_w/SiC composite, the reduction in fracture toughness value between 23 and 800 °C is 2.18%, and the corresponding reduction in flexural strain is 6.8%. In the case of monolithic SiC, the reduction in K_{Ic} value and the flexural strain value are, respectively, 12.24 and 26.65%. Therefore, it is clear that the reduction in failure strain is proportional to the reduction in the fracture toughness.

The drop in fracture toughness as well as in flexural strength with the rise in temperature suggests that the formation of glass phase plays an important role in controlling these mechanical properties at higher temperatures. The increasing amount of glass and its falling viscosity would enhance faster crack growth and the formation of more extensive microcrack zones. Eventually, the increasing interaction effects

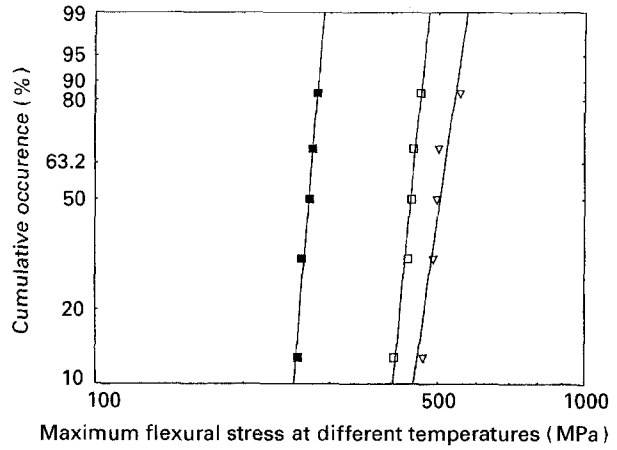

Figure 9 Weibull analysis for flexural strengths of SiC_w/SiC composites. ▽ 23 °C, CL (η) = 523.8439, SF (β) = 18.01542, r^2 = 0.8389611; □ 800 °C, CL (η) = 442.5486, SF (β) = 27.10411, r^2 = 0.9074559; ■ 1200 °C, CL (η) = 274.9708, SF (β) = 29.07775, r^2 = 0.9623235.

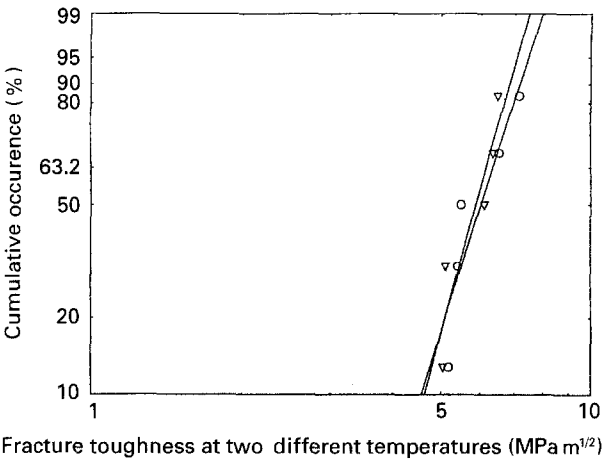

Figure 11 Weibull analysis for fracture toughness of SiC_w/SiC composites. ○ 23 °C, CL (η) = 6.273177; SF (β) = 8.274299, r^2 = 0.8572845; ▽ 800 °C, CL (η) = 6.109434, SF (β) = 9.199717, r^2 = 0.8572845.

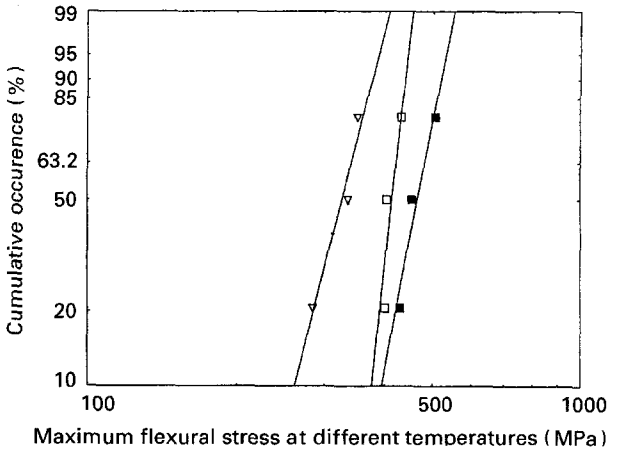

Figure 10 Weibull analysis for flexural strengths of monolithic SiC. ■ 23 °C, CL (η) = 481.2029, SF (β) = 15.22765, r^2 = 0.8394343; □ 800 °C, CL (η) = 419.023, SF (β) = 26.10605, r^2 = 0.7505386; ▽ 1200 °C, CL (η) = 339.9612, SF (β) = 9.031252, r^2 = 0.9436725.

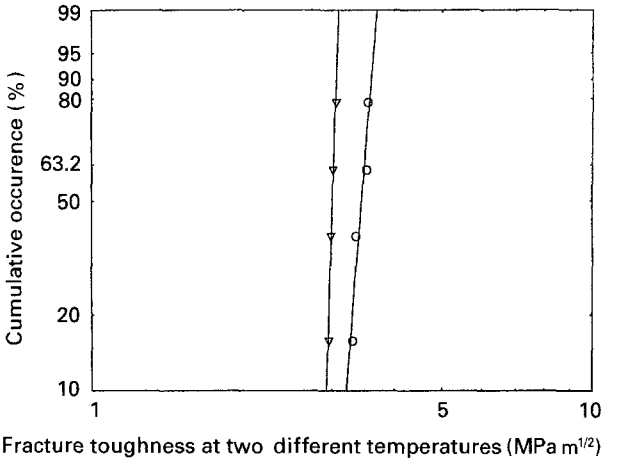

Figure 12 Weibull analysis for fracture toughness of monolithic SiC. ○ 23 °C, CL (η) = 3.480214, SF (β) = 30.37986, r^2 = 0.8893443; ▽ 800 °C, CL (η) = 3.027275, SF (β) = 72.06953, r^2 = 0.9709086.

between the main crack and microcrack zone leads to a decrease in toughness and strength [13].

Weibull analyses of flexural strength data for various temperatures are shown in Figs 9 and 10. Fracture toughness tests were performed at 23 and 800 °C only. Weibull analyses for fracture toughness data are shown in Figs 11 and 12. It is observed from the Weibull analysis of SiC_w/SiC at various temperatures that the Weibull distribution, β, lies between 17.275 and 28.81. These Weibull distributions are the slopes of the straight lines, and are shown in Figs 9 and 10 as shape factors (SF). These values of β suggest a moderate scatter of flexural data at room and elevated temperatures. The corresponding Weibull analyses for the monolithic SiC show close similarity except that the characteristic lives (shown as CL in the Weibull diagrams) are different. These characteristic life values which are indicative of 63.2% of the distribution, are independent of the Weibull modulus, and are found to be close to the tabulated average values. In the case of fracture toughness analyses (Figs 11 and 12) different situations are observed between the composite and the monolithic SiC, especially in respect of β values. For SiC_w/SiC, the Weibull distribution is seen to be very low compared to that of SiC. Besides high scatter in the fracture data, this low Weibull distribution indicates more random behaviour of SiC_w/SiC to chevron notch sensitivity both at room and elevated temperatures.

Degradation in flexural strength at elevated temperature is obvious from the ultrasonic measurements. Ultrasonic velocities through the specimens of flexural tests at room and elevated temperature are listed in Table II. These velocity measurements indicate quantitative measures of processing defects and localized inhomogeneities in the material. Localized damage incurred in the specimen during the static failure tests reduces the velocity of the sound wave as observed in Table II. The reduction in wave velocity at 1200 °C from that of room temperature suggests degradation of the material leading to lesser strength at elevated temperature as shown earlier. It is observed from Table II that wave velocity reduces by about 8.5% from room temperature (RT) to 1200 °C, whereas the

TABLE II Ultrasonic velocities through the specimens of flexural tests at room and elevated temperature

Specimen	t(s)	V_1	
		in s^{-1}	ms^{-1}
Untested	18.8972×10^6	114015	2896
Room temperature	20.996×10^6	55972	1421
800 °C	21.372×10^6	51390	1305
1200 °C	21.3852×10^6	51242	1301

corresponding reduction between untested and RT is about 51%. From the SEM micrographs it has been shown that the fracture mode change from inter- to transgranular at elevated temperature. Therefore, velocity measurements at RT and at 1200 °C suggest that shift in granularity has insignificant effect on the wave velocity. On the other hand, the difference between the untested and the RT fractured specimens is mainly the matrix microcracks, whisker debonding and whisker pullout in the neighbourhood of the failure site. These observations indicate that the ultrasonic parameter, like sonic velocity is more affected by the damage in the composite than by the change in the granularity of the microstructure.

4. Conclusions

From the results of the investigation the following conclusions may be drawn:

1. Reinforcement of SiC with SiC whiskers increases the flexural strengths of the composite at room temperature, and up to 800 °C. However, no improvement in strength is observed at 1200 °C.

2. It has been observed that SiC_w/SiC composite loses strength uniformly throughout the test temperature range, while the strength of the monolithic SiC drops rapidly only after 800 °C. This indicates a trend for SiC more towards catastrophic failure beyond 800 °C.

3. At elevated temperatures a slight reduction in fracture toughness values of SiC_w/SiC composite is found, but a higher reduction is observed in the case of monolithic SiC. The observed fracture behaviour suggests that microstructural modification, incorporated via whisker reinforcements, imparted resistance to crack growth both at room and elevated temperatures.

4. The SiC_w/SiC composite as well as the monolithic SiC show similarity in Weibull distribution both at room and elevated temperatures in respect of flexural strengths. However, in the case of fracture toughness, there is a large reduction in Weibull shape factor with SiC_w/SiC composite.

5. Ultrasonic measurements indicate that the sonic wave velocity is inversely proportional to the extent of damage in the SiC_w/SiC composite, and this growth of damage rather than the microstructure, has more influence on the velocity.

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