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Influence of SiC whisker morphology and nature of SiC/Al_2O_3 interface on thermomechanical properties of SiC reinforced Al_2O_3 composites

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Abstract

Thermomechanical properties of a 35 vol.% SiC whiskers/Al_2O_3 matrix composite were investigated as a function of whisker surface quality. Two batches of SiC whiskers (Tateho-SCW-1-S) were studied. Whisker surface chemistry, as determined by X-ray photoelectron spectroscopy and whisker morphology, as determined by SEM or TEM, was correlated to the thermomechanical properties of the composites. The surface oxygen content of the whiskers was shown to strongly affect the composite thermomechanical properties. High oxygen surface content appears to affect the whisker/matrix interfacial bonding thus decreasing the amount of crack deflection, whisker pullout and whisker bridging which are required to reach high fracture toughness values.

Keywords: SiC; Whiskers; Interfaces; Composites; Thermal properties; Mechanical properties; Al_2O_3/SiC

1. Introduction

In ceramic matrix composites, whisker reinforcements are primarily used to enhance the fracture toughness and the flexural strength of the composite at temperatures to 1000 °C. Essentially, the whisker reinforcement prevents catastrophic brittle failure by providing processes that dissipate energy during the fracture process. Toughening mechanisms, such as crack deflection,[1–2] whisker pullout,[3–7] and whisker bridging,[8–10] depends to a large extent on the nature of the whisker/matrix interface. Several factors affect the whisker/matrix interface, including matrix chemistry, whisker surface chemistry, whisker morphology and thermal expansion mismatches. The internal stresses are also expected to affect the toughening behaviour of SiC-whiskers-reinforced alumina matrix composite as shown by Predecki et al.[11] and Li and Bradt.[12]

Recent works on ceramic matrix composites have demonstrated that fracture toughness and flexural strength of polycrystalline Al_2O_3 can be significantly improved by addition of SiC whiskers. Becher and Wei,[13] Wei and Becher,[14] Becher et al.,[15] and Homeny et al.[16] have achieved fracture toughness values approaching 10 MPa $m^{0.5}$ and flexural strength values approaching 800 MPa. Furthermore, Homeny and Vaughn[17] have demonstrated that the fracture toughness could vary with whisker type from 4 to 9 MPa $m^{0.5}$ when utilising whiskers that were similar in all aspects, except for surface chemistry. They have associated the high fracture toughness with the presence of carbon and silicon oxycarbide phases on the whiskers surface. Tiegs et al.[18] have also performed a detailed study on whiskers from numerous sources and have correlated the oxygen and carbon concentrations of the whisker surfaces with the fracture toughness. According to them as well, the high fracture toughness is associated with the presence of carbon excess on the surfaces, while the low fracture toughness is attributed to oxygen excess.

The present work deals specifically with the effect of whiskers quality on the thermomechanical properties of SiC

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whiskers/Al_2O_3 matrix composites. Two whiskers batches (Tateho-SCW-1-S) have been used to obtain different morphologies and surface oxidation states.

2. Experimental procedures

2.1. Material preparation

Two batches of SCW-1 grade SiC whiskers (Tateho, Japan) have been used as reinforcement material: one batch with a low surface oxygen content of 6 at.% oxygen as received and another batch with a high surface oxygen content of 39 at.% oxygen as received, respectively, labelled 'L' and 'H'. TEM analysis of the 'H' batch sample (Fig. 1) reveals the occurrence of an oxygen layer on the whisker surface. SEM observations allow making comparisons between the different morphologies of the batches. The whiskers 'L' have mainly small cross section (Fig. 2a) whereas the whiskers 'H' show mainly large cross section with undulated surfaces (Fig. 2b). The polycrystalline alumina powder utilized for the matrix is SM8 (Baïkowski Chimie, France, 99.9% alumina, <50 ppm Na, Mg and Ca). The mean particle size diameter of the alumina powder is about 0.25 μm and the specific surface area is 10.4 m^2/g.

The details of processing technique are described elsewhere.[23–24] Briefly, the SiC whiskers (35 vol.%) and Al_2O_3 powder are mixed using a water-based slurry method. First, a slurry of alumina powder is prepared in distilled water, the pH of the slurry is adjusted to 4, and the suspension

Fig. 1. TEM micrograph of Tateho SiC whiskers 'H'.

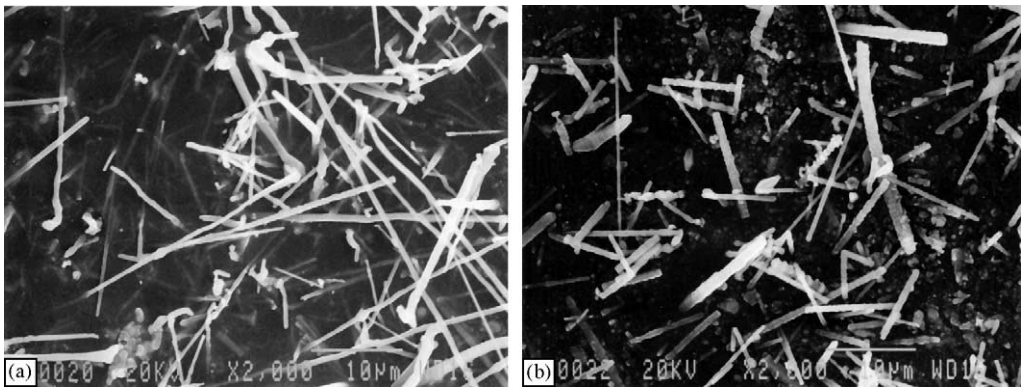

Fig. 2. SEM micrographs of Tateho SiC whiskers: (a) 'L' whisker and (b) 'H' whisker.

is dispersed ultrasonically. Concerning the SiC whiskers, the slurry is prepared using a basic solution and is subjected to ultrasonic dispersion for 10 min. The final composite is obtained by adding these slurries, each of them having a pH value corresponding to a maximum zeta potential. The mixture is subsequently dried through evaporation of water and filtered successively through 60 and 250 mesh sieves.

Hot pressed discs of alumina and Al_2O_3/SiC_w have been obtained under a pressure of 45 MPa in an argon atmosphere at 1850 °C/1 h.

2.2. Experimental techniques

Final densities of the sintered samples have been measured using the Archimede's principle. Vickers hardness, load of 100 N, has been determined on polished surfaces.

Flexural strength and fracture toughness have been determined in temperature range from 25 to 1300 °C in air atmosphere, using the 4-point bending technique with a cross-head speed of 0.1 mm/min. The outer and inner spans were, respectively, 35 and 10 mm. The dimensions of flexural strength bars were 3 mm × 4 mm × 40 mm and their tensile surfaces were polished with a 3 μm diamond-grinding wheel in the direction of tensile axis to avoid the effect of machining defects on the intrinsic characteristic material. The edges on the tensile surface were rounded. Thereafter, Young's modulus has been measured by the Grindo-Sonic technique.

The fracture toughness measurement has been performed using centre notched bars (6 mm × 4 mm × 40 mm) to less one half of the thickness with a 0.3 mm thick diamond blade.

Creep tests have been conducted in air under 100 MPa stress level at several temperatures (1000, 1200 and 1300 °C). Specimens have been deformed in a 4-point bending device whose inner and outer spans were, respectively, 18 and 36 mm. The applied stress and resulting strain have been calculated from the load and displacement data using the procedure described by Hollenberg et al.,[19] the secondary creep rates were determined from the variation of the displacement versus time when the values are stabilized.

Fracture resistance curves (R-curves) have been determined following single edge notched beam (SENB) technique in 4-point bending at a cross-head speed of 4 μm/min. The samples are machining with a 300 μm diamond saw continued by a thin notch made with a 70 μm saw. The initial ratio of the precrack depth (a_0) to sample width (w), a_0/w, was chosen as 0.6.

Slow crack growth behaviour was determined by a double torsion method. The specimens, 40 mm × 20 mm × 2 mm, were centre notched using a diamond saw. The notch length was about 10 mm and subsequently was precracked at a low cross-head speed of 4 μm/min. The relaxation tests have been made on these samples to obtain the variation of the load as a function of time and finally to allow the determination of the V–K_I curves.[20–21]

3. Results and discussion

Table 1 shows mechanical properties at room temperature of monolithic alumina and Al_2O_3/SiC_w composites prepared with whiskers 'L' or 'H'. After hot pressing, all samples show relative density close to the theoretical value. The microstructure of Al_2O_3/SiC_w composites was observed by an optical micrograph on polished surface and has shown a homogeneous dispersion of the whiskers into the alumina matrix either perpendicular or parallel to the hot pressing axis.

No significant variation of Young's modulus is observed between alumina and composites samples. On the opposite, Vickers hardness, flexural strength and fracture toughness of the Al_2O_3/SiC_w composites are higher than for monolithic alumina. For the composites containing 'L' SiC whiskers, the fracture toughness is twice that of monolithic alumina (4 MPa $m^{0.5}$). These results are quite comparable with the mechanical properties values reported by Becher and coworkers[13–15,22–25] for a similar material.

Fracture surfaces of the two composites were also observed, micrographs are shown in Fig. 3a and b. The fracture surfaces generally exhibit both intergranular and intragranular mode of failure, with some appearance of whiskers pullout. Many observations performed on polished surfaces have been made on fracture surface and indentation crack. It was pointed out that several toughening mechanisms occur in the material such as: crack deflection, debonding, bridging and pullout. Nevertheless, the main contribution of the alumina matrix reinforcement is due to cracks deflection as it can be seen in Fig. 4.

As previously noted, flexural strength and fracture toughness of polycrystalline alumina are improved by addition of SiC whiskers. However, this improvement closely depends on the SiC whiskers surface oxygen content. For the composite 'H', flexural strength and fracture toughness, are observed to be higher than for alumina but lower than for composite containing 'L' SiC whiskers (Table 1).

Table 1
Mechanical properties at room temperature of monolithic material and Al_2O_3–35 vol.% SiC whiskers 'L' and 'H'

Mechanical properties	Al_2O_3	Al_2O_3 + 35 vol.% SiC_w 'L'	Al_2O_3 + 35 vol.% SiC_w 'L'
Relative density (dth %)	99.1	100	99.6
Young's modulus (GPa)	406 ± 10	421 ± 10	407 ± 9
Hardness Vickers (10 kg)	1854 ± 38	2107 ± 32	2032 ± 62
Flexural strength (MPa)	488 ± 151	639 ± 21	549 ± 41
Fracture toughness (MPa $m^{0.5}$)	5.4 ± 0.4	7.9 ± 0.3	6.9 ± 0.2

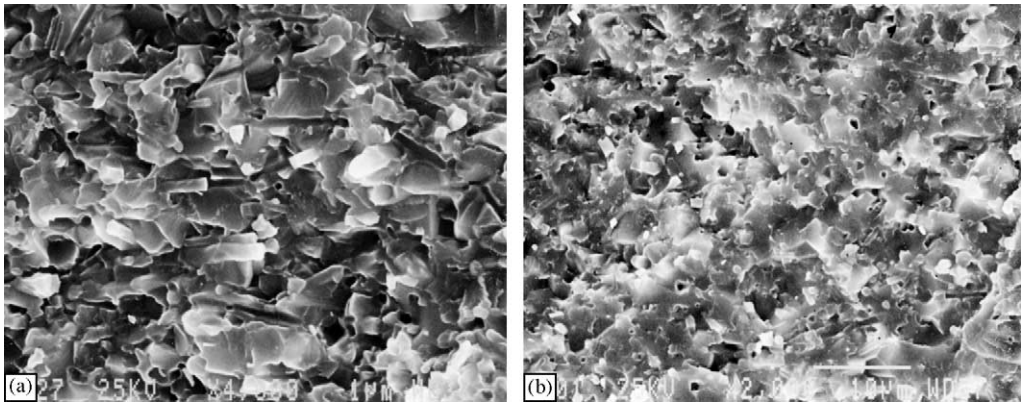

Fig. 3. SEM micrographs of a fracture surface of Al_2O_3/35 vol.% SiC whiskers, showing inter and intragranular mode failure and whisker pullout, (a) 'L' whisker and (b) 'H' whisker.

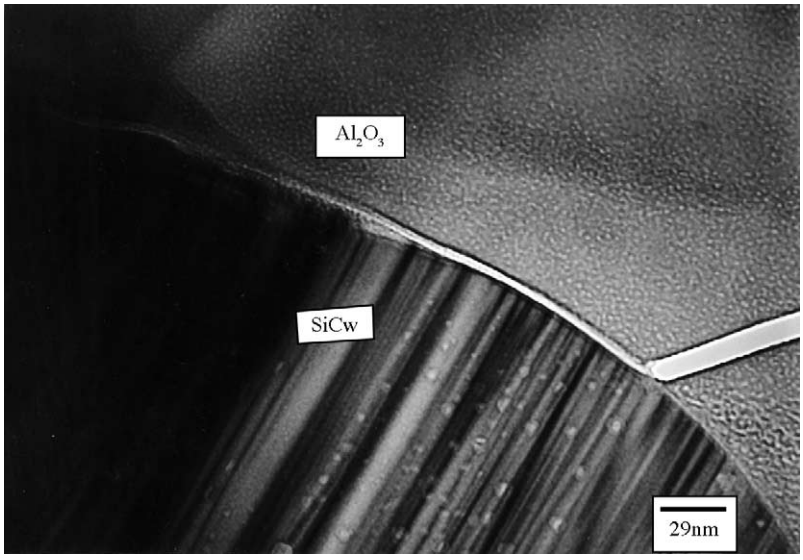

Fig. 4. TEM micrograph of Al_2O_3/35 vol.% SiC whisker 'L' composite, showing crack deflection along whisker/matrix interface.

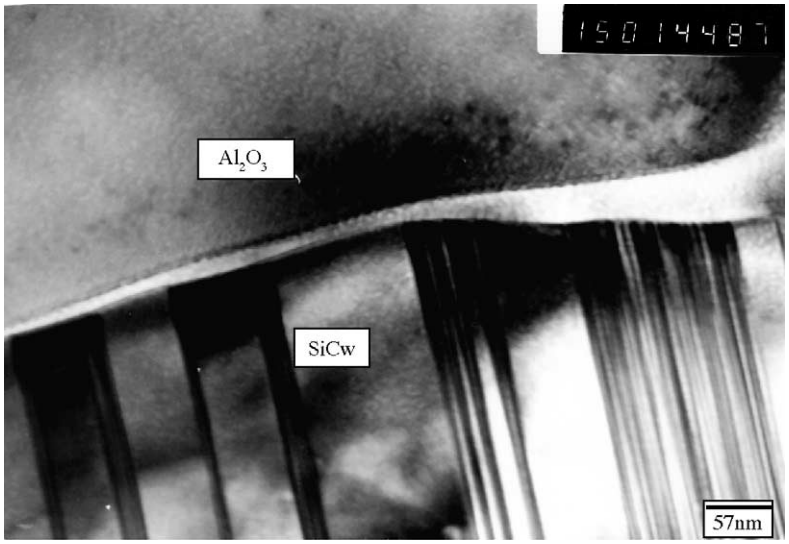

Fig. 5. TEM micrograph of Al_2O_3/35 vol.% SiC whiskers 'L' composite, showing the appearance of a glass layer along the alumina/whisker interface.

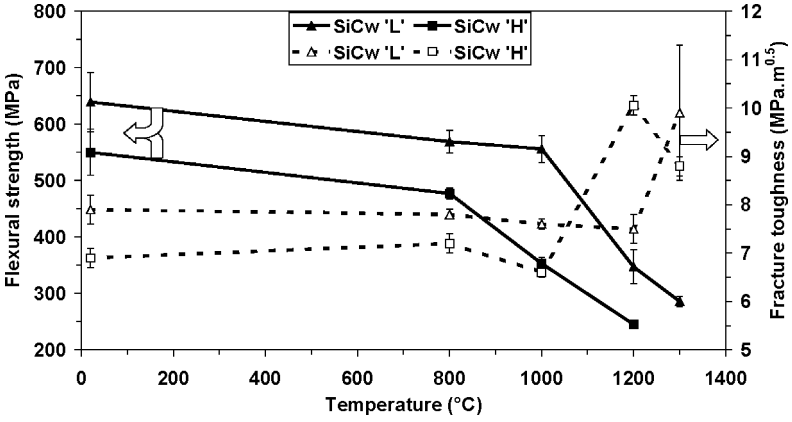

Fig. 6. Flexural strength and fracture toughness as a function of temperature.

The lower σ_f and K_{IC} values obtained when increasing SiC whiskers surface oxygen content can be explained by the degradation of silicon carbide in presence of oxygen and/or by the surface chemical reaction between SiO_2 and Al_2O_3. As a consequence, a strong interface whisker-matrix is created (see Fig. 5), which minimizes the amount of cracks deflection along the interface, whisker bridging and pullout.[25–26]

Flexural strength and fracture toughness were also measured at higher temperatures in air atmosphere (from 800 to 1300 °C). The variation of flexural strength and fracture toughness for the two composites are shown in Fig. 6 as a function of temperature. For the 'L' composite, K_{IC} and σ_f decrease slowly with increasing temperature up to 1000 °C. At temperatures above 1000 °C, σ_f significantly decreases suggesting that fracture is governed by a different mechanism. Then, the fracture toughness remains constant up to 1200 °C, and at higher temperatures, above 1200 °C, the fracture toughness increases rapidly up to 10 MPa $m^{0.5}$. During high-temperature air annealing of alumina silicon carbide composites, silicon carbide is oxidizing. This oxidation produces an amorphous phase that softens above 1200 °C and is responsible for the composite behaviour at 1300 °C. Results of flexural strength and fracture toughness obtained for 'H' composites are similar with those obtained for 'L' composites and agree with the above assumption. However, the decrease of the flexural strength as well as the increase of the fracture toughness started about 200 °C earlier than the former. These observations confirm an effect of the surface oxygen content of the original SiC whiskers on the mechanical properties of the final composites at high temperatures.

According to Becher and Tiegs,[27] the marked strength degradation, which occurs above 1000 °C in air, is associated with creep. At this temperature, the viscosity of the glassy phase must be sufficiently low to allow the liquid phase to penetrate along the matrix grain boundaries and enhanced creep and associated crack generation. Observations of the fracture surface sample tested at 1200 °C support this conclusion (see Fig. 7).

Fig. 8 shows the creep deformation for composites at 1200 °C under 100 MPa. For the 'L' composites, the behaviour indicates that the creep deformation involves a short primary stage of creep, during which the strain rate decreases, and then a long steady-state region follows this stage. Tertiary creep is not observed at all and the specimen is not broken after a testing period of 80 h.

The creep resistance of polycrystalline alumina, >3% without failure limited only by test fixture, can be significantly improved through the addition of SiC whiskers. The

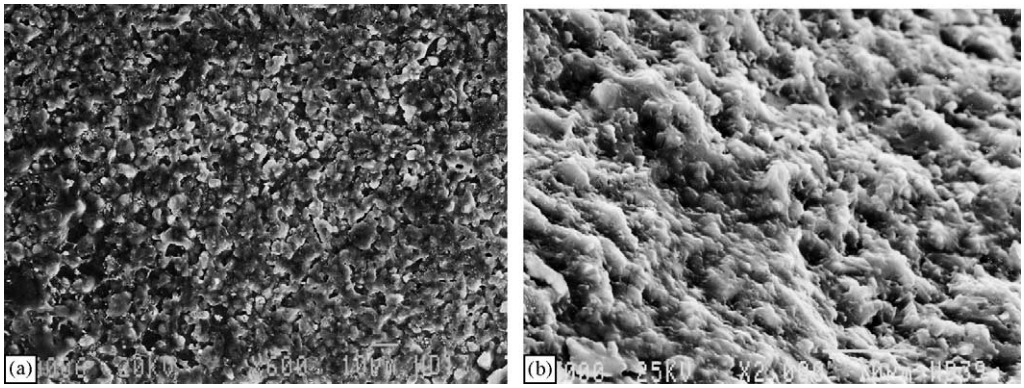

Fig. 7. SEM micrographs of (a) the tensile surface and (b) room temperature fracture surface of the Al_2O_3/35 vol.% SiC whisker 'L' composite after creep testing at 1200 °C in air, showing the SiC oxidation and the liquid phase, respectively.

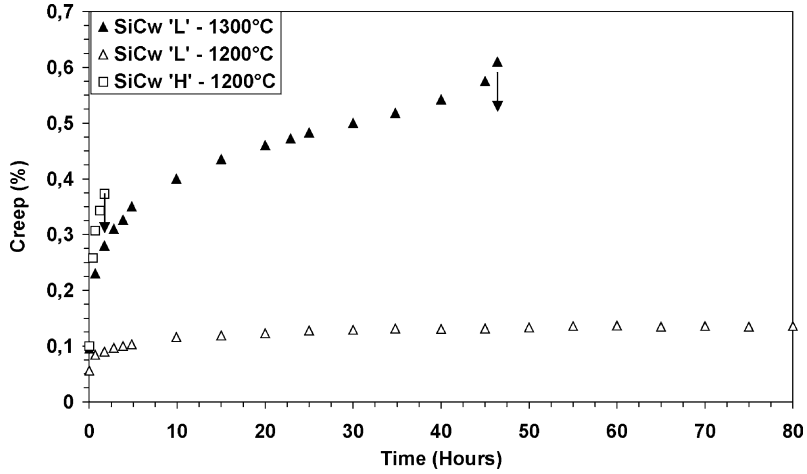

Fig. 8. Creep curves, at 1200 and 1300 °C under 100 MPa, for alumina–SiC whisker composites.

creep rate is $7 \times 10^{-10}\,s^{-1}$ for the 'L' composite after 60 h of exposure under load 100 MPa and the corresponding final strain is 0.15%, same results have been observed earlier by several authors.[28–30]

Observations of Al_2O_3/SiC_w composites after creep testing have revealed that the SiC whiskers on the surface as well as in the composite core have been oxidized. Precise examination of a sample fracture surface, broken at room temperature after creep testing at 1200 °C in air, has been performed. The SEM micrographs (Fig. 7) show the occurrence of the glassy phase on the surface fracture. The liquid phase formed on exposed surfaces could migrate near the surface grain boundaries of alumina composite and accelerate creep deformation process. At higher temperatures (>1200 °C), the volume fraction of liquid phase increases and the viscosity of liquid phase decreases. Consequently, grain boundaries sliding are more important and induce the formation of grain boundary cavities, the creep behaviour may be deeply modified. The observed creep deformation for 'L' composite at a stress of 100 MPa and 1300 °C confirms the precedent hypothesis (Fig. 8). At 1300 °C, the creep strength of the composite with 'L' SiC whiskers has been significantly reduced by 2 orders of magnitude ($=1.5 \times 10^{-8}\,s^{-1}$). On the other hand, a short stage of tertiary creep is observed before breaking at a strain of about 0.6% and after 40 h.

Concerning the composites with 'H' whiskers (Fig. 8) the specimen has been broken after only 2 h of testing. This creep behaviour degradation and the increase of the fracture toughness under air, at high temperatures, may be explained by the SiC oxidation and the amount of glassy phase in the grain boundaries.

As it has been discussed earlier, toughening by deviation and crack bridging occur in whisker reinforced materials. Consequently, the process zone size is not negligible and non-linear macroscopic fracture behaviour must be observed, producing R-curve effect in which toughness (K_R) varies with crack growth. So, the existence of a R-curve behaviour has been investigated by SENB method. At room temperature and 1000 °C, no stable crack propagation could be obtained for the composites prepared using 'L' SiC_w, suggesting that there are no extended R-curve behaviour. But hereafter 1200 °C stable crack propagation was observed showing a significant

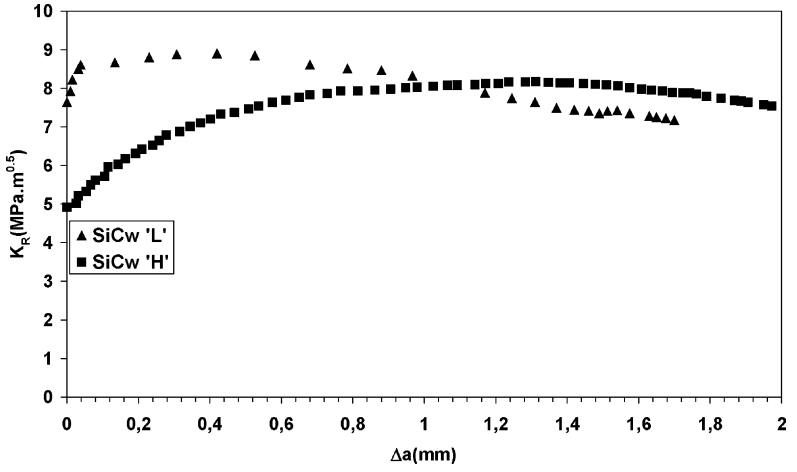

Fig. 9. Rising crack-growth resistance (R-curve) of alumina–SiC whisker composites at 1200 °C.

improvement of the R-curve behaviour with temperature. At high temperatures the viscous liquid phase fastens cracks and leads to the formation of well-developed wake zone, associated with whisker bridging.

The Fig. 9 shows R-curve behaviour of two composites prepared using 'L' and 'H' SiC_w. For all composites, R increases with crack extension. In the 'L' type composite, a rapid increase of K_R ($K_R = 8.8\,MPa\,m^{0.5}$) is observed followed by a plateau value and a slow decrease after the crack extension Δa^* has reached the value of 600 μm. This K_R value corresponds to the steady state of whiskers and may be referred as fracture toughness for crack propagation. In this state, the bridge generation is balanced by its extension and the length of the well-developed bridging zone is given by Δa^*.

The R-curve behaviour concerning the 'H' composite is different (Fig. 9), the initial K value is much lower and the rising domain is longer than for the 'L' composite. This difference in the behaviour may be attributed to the presence of high volume fraction of liquid phase in the 'H' composite, thus reinforcement mechanisms such as crack deflection along the interface, whisker bridging and pullout are disabled.

It has been shown that Al_2O_3/SiC whisker composites have higher toughness than monolithic alumina, it is believed that these composites also have a higher resistance to slow crack growth. A plot of the V–K_I curves (crack growth rate versus stress intensity factor) obtained from the relaxation test at room temperature is showed in Fig. 10 for alumina and two Al_2O_3/35 vol.% SiC_w composite materials. The crack velocity measured in the composites also shows a single stage, corresponding to the first stage, but the slope is much higher than alumina ($n = 432$ for Al_2O_3–35% SiC_w and $n = 35$ for alumina). This suggests that the whisker composite is less sensitive to slow crack growth than alumina. This difference in the behaviour can be attributed to the difference in their microstructures. For the whisker composite, the crack has greater difficulty to move by or through a whisker than only to propagate through the polycrystalline Al_2O_3,[31] in addition the differences may become more pronounced during slow crack growth.

4. Discussion

Thermomechanical properties of alumina were significantly improved by the addition of SiC whiskers. However, this improvement depends on the whisker quality, particularly the morphology and the surface oxygen content.

Thermomechanical properties of the composites prepared with high surface oxygen content whiskers were lower than those obtained with the composites prepared with low surface oxygen content whiskers. This result suggests two possible explanations. First, the superficial silica whiskers and alumina matrix may react to form mullite, producing a strong interface. Consequently, this strong interface minimizes the usual reinforcement mechanisms: crack deflection along the interface, whisker pullout, and resistance to crack opening. In this case reducing the amount of surface oxygen contamination on the SiC whiskers should increased the composite toughness.

If it is not the case a second explanation must be found. The SiC whiskers could be degraded through the following reactions:

$$SiC(s) \rightarrow Si(g) + C(g) \quad (1)$$

$$SiC(s) + 2SiO_2(s) \rightarrow 3SiO(g) + CO(g) \quad (2)$$

$$SiC(s) + O_2(g) \rightarrow SiO(g) + CO(g) \quad (3)$$

Singhal[32] lists these reactions as the most thermodynamically significant reactions above 1727 °C, especially under dynamic vacuum conditions where the gaseous products are continuously removed from the system. Reaction (1) represents the dissociation of SiC into its constituents and is strongly dependent on carbon potential in the system. The extent of reaction (2) in degrading the SiC whiskers is significant, especially when the products are constantly removed in

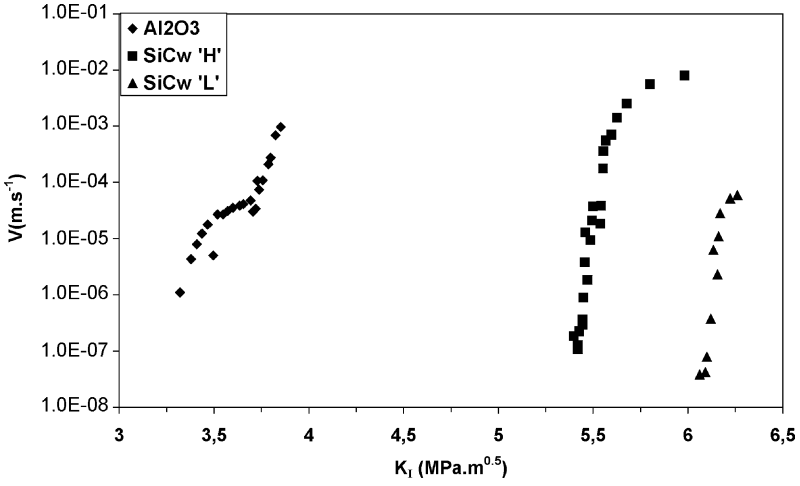

Fig. 10. The slow crack growth resistance of alumina in region I is significantly increase at room temperature with the addition of the SiC whiskers.

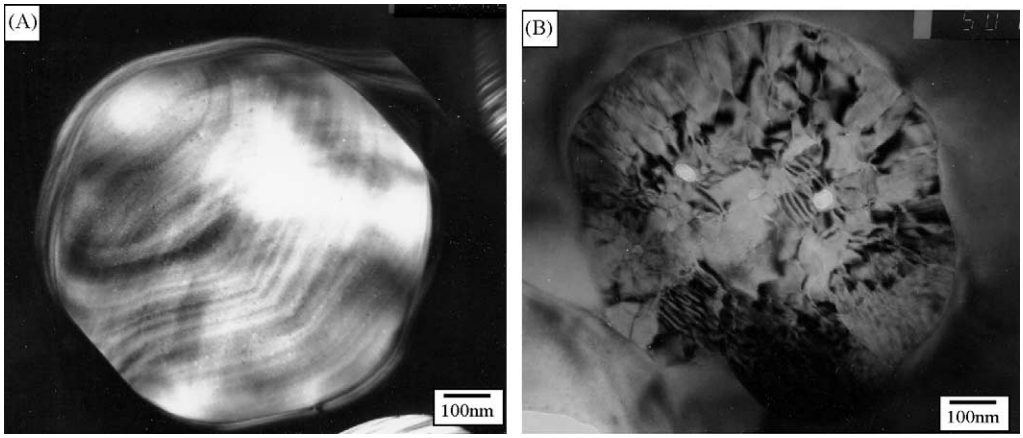

Fig. 11. TEM micrographs of SiC whiskers after hot-pressing: (a) 'L' whisker and (b) 'H' whisker.

a dynamic vacuum. Reaction (3), which represents active oxidation of the SiC whiskers, is severely inhibited by the Al_2O_3 matrix, which protects the whiskers, and by the reaction of ambient oxygen with the graphite die which significantly reduces the oxygen partial pressure in the hot-pressing chamber. Considering the above discussion, degradation of the SiC whiskers by reaction (2) is the most plausible explanation for the thermomechanical properties decrease of composites prepared with high surface oxygen content whiskers.

TEM observations of the two SiC whiskers after hot pressing are in agreement with this hypothesis. Whiskers with high surface oxygen content have a polycrystalline structure, while whiskers with a low surface oxygen content have a monocrystalline structure (Fig. 11). In addition, for composite with low oxygen content, the presence of a thin glass layer along alumina/whiskers interface is observed (Fig. 5). This is not the case for the composite with high surface oxygen content whiskers.

5. Conclusion

SiC whisker reinforced alumina composites containing up to 35 vol.% SiC whiskers were hot-pressed to more than 99% of the theoretical density and were shown to have substantial improved fracture toughness and strength compared to monolithic alumina. In addition these mechanical properties remain relatively constant to 1000 °C. Above 1000 °C in air, the low viscosity of the liquid phase, resulting from the oxidation of the SiC whiskers, leads to crack-shielding, thus enhancing mechanical properties and R-curve behaviour. Observations have shown that crack deflection by the whiskers and whisker bridging and pullout are the significant toughening mechanisms.

The surface oxygen content of SiC whiskers has a major impact on the mechanical properties of Al_2O_3/SiC_w matrix composites. The oxygen surface content appeared to affect the whisker/matrix interfacial bonding thus decreasing the amount of deflection, whisker pullout and whisker bridging which are required to achieve high fracture toughness values.

Mechanical behaviour of whisker-reinforced material was also found to be strongly dependent on the whisker content. The creep rate of alumina at 1200 °C could be reduced by one or two orders of magnitude with the addition of 35 vol.% whiskers. However, an optimum exists where the deformation rate starts to decrease with increasing whiskers content.

Acknowledgments

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