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Oxidation-induced strengthening in ground silicon carbide

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Silicon carbide has specific advantages over other structural ceramics, such as high temperature capability and excellent chemical stability. So silicon carbide is a promising candidate for high temperature structural materials as well as for corrosion resistance applications. Like brittle materials, however, strength of silicon carbide is closely related to the size and distribution of surface flaws such as machining-induced cracks, because of their inherent low toughness. Thus, a smooth polishing procedure after grinding is often needed in order to reduce the size of surface flaws, leading to an increase in cost of ceramic components. Moreover, the allowable flaws in ceramics are so small that it is almost impossible to detect the flaws and also difficult to remove them completely using machining techniques. As a result, the structural integrity of a ceramic component is seriously affected.

A method, to heal a crack, could be considered to overcome this problem. Studies on crack healing have been reported for alumina [1–3], silicon nitride [4–6], silicon nitride/silicon carbide composite [7], Mullite/silicon carbide composite [8] and silicon carbide [9–12]. Our research group recently proposed a mechanism of crack healing and strengthening by pre-oxidation procedure in silicon carbide [11, 12]. The major observation is that the residual stress produced by the thermal expansion mismatch between silica (within cracks) and surrounding silicon carbide played a significant role in the strength increase. As the crack healing technique can be adopted in practical strength applications, considerable advantages can be expected either in the reliability or in the machining and inspection costs of ceramic components. In the above perspectives, the effect of pre-oxidation on strength of silicon carbide with grinding-induced surface cracks was investigated and reported in the present communication.

SiC material (Hexoloy, Carborandom, Inc., USA) was received in a plate form. The microstructure of the material consisted of SiC grains smaller than 10 μm and average grain size is about 5 μm (see Fig. 1). The material was machined into flexure specimens of 3 mm × 4 mm × 40 mm. The specimen surface was ground using diamond wheels with girt sizes of 127 μm, 64 μm, 42 μm and 32 μm. Grinding conditions were: 3,300 rpm, 35 m/s peripheral wheel speed and 10 μm depth of cut. The specimens were mounted on a holder in such a manner that grinding was oriented along the width of the specimens (perpendicular to the tensile stresses applied during the strength measurement). The ground specimens were then heat-treated at 1,500 °C for 50 h in air. The heating rate was 10 °C/min and cooling was done by turning off the electric power of the furnace. For comparison, some heat treatment experiments were also conducted in vacuum under similar conditions. The microstructure was characterized by field-emission scanning electron microscopy (FESEM), Energy Dispersive X-ray (EDX) and X-ray Diffraction (XRD). The strength of specimens was measured using a four-point bending fixture with outer and inner spans of 30 mm and 10 mm, respectively at a crosshead speed of 0.5 mm/min, as per the standard procedure [13]. Specimens were placed in the fixture such that the surface containing grinding-induced cracks was in

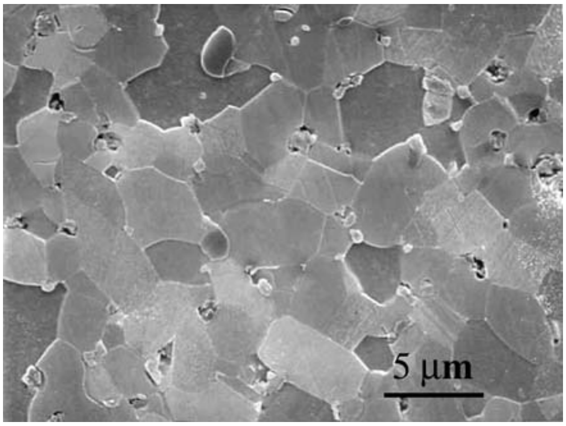

Fig. 1 Microstructure of silicon carbide

tension. High temperature strength test was also conducted according to the above-mentioned method up to 1,400 °C in air. At least five specimens were tested at each condition to obtain the average strength and standard deviation.

The influence of heat treatment on flexural strength of silicon carbide, ground with various grit sizes is shown in Fig. 2. Mecholsky et al. [14] have reported that grinding perpendicular to the tensile axis generates flaws which are 60% deeper with slightly greater stress intensity factor than flaws resulting from parallel grinding. In the present set of experiments, it was ensured that grinding orientation was perpendicular to the tensile axis because a great strength reduction is expected. The plotted data in Fig. 2 reveal that strength of as-machined specimen is decreased with increasing the grit size of diamond wheel. The strength of specimen that was ground with 32-µm grit diamond wheel is 358 ± 17 MPa. In contrast, a 47% reduction in strength is recorded 167 ± 4 MPa, when 127-µm grit wheel is used as compared to 32-µm grit. On the other hand, the strength of the heat-treated specimens is not only greater than that of their peers without heat treatment, but also that of as-polished ones. The strength showed a higher value of about 460 MPa, which was 25% higher in comparison with that of ground and polished specimen (~360 MPa) and was independent of diamond grit size used. It is interesting to note here that no change in strength occurred when the ground specimen was heat-treated in vacuum.

Detailed microstructural investigation revealed the presence of a new phase on the heat-treated specimens, presumably formed during oxidation. XRD and EDX analyses confirmed that the new phase is silica (amorphous and crystalline). The previous studies [11, 12] on crack-healing in silicon carbide have shown that heat treatment above 1,000 °C in air could heal a crack of about 2 mm length, which was filled with silica, and thereby increased the strength. The present results now indicate that this improvement resulted in healing of both grinding-induced cracks and pre-existing flaws on surface of specimens, due to oxidation by heat treatment in air.

Silicon carbide is a promising structural ceramic for high temperature application, because of its excellent strength retention at elevated temperature (above 1,000 °C). Hence, it is important to measure high temperature strength of the heat-treated specimen and the test results are shown in Fig. 3. The strength of heat-treated sample did not change up to 1,400 °C when compared to that of at room temperature. In contrast, the strength of as-machined specimens, which was ground using 127-µm grit diamond wheel is increased above 600 °C and then reached to

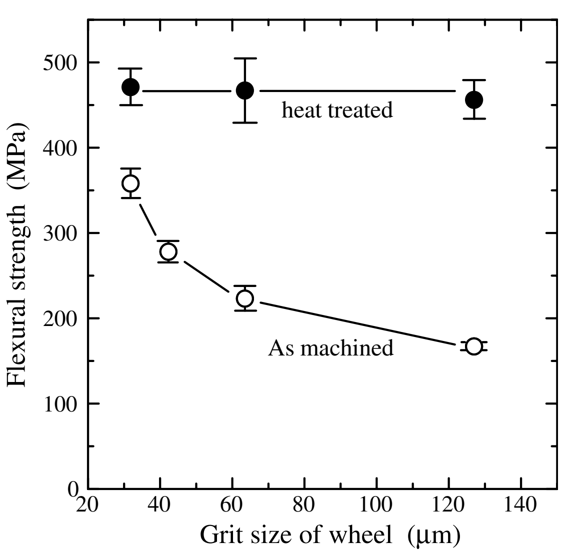

Fig. 2 Influence of heat treatment on flexural strength of silicon carbide ground with various grit sizes of wheel

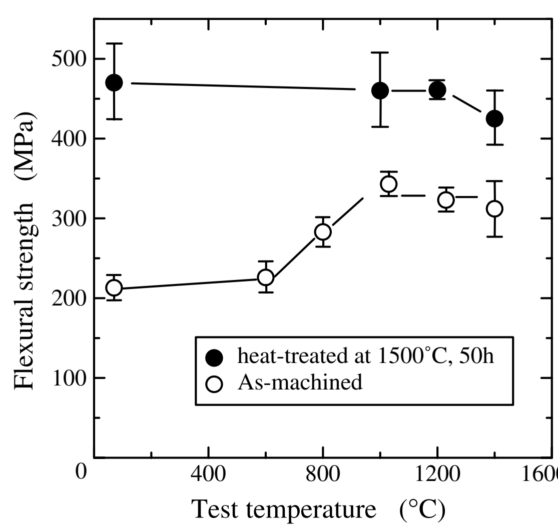

Fig. 3 High temperature strength of silicon carbide ground with 127-µm diamond grit wheel before and after heat treatment in air

320 MPa at 1,000 °C. Above 1,000 °C, there is no change in strength of machined samples up to 1,400 °C. It is considered that this improvement in strength tested at elevated temperature resulted in healing of grinding-induced cracks due to oxidation, when the machined specimens were exposed to the atmosphere for 30 min during test measurements.

These results clearly implicate that pre-oxidation procedure is a useful technique not only to improve strength, but also to reduce machining costs. Furthermore, this technique is expected to improve reliability of silicon carbide, because most of the surface flaws can be healed using this technique.

The main results of this study are as follows:

(a) The room temperature strength of ground and heat-treated specimens was improved because both grinding-induced cracks and pre-existing surface flaws were healed due to oxidation by heat treatment in air. After heat-treating, the strength indicated a constant value of about 470 MPa independent of their surface condition.
(b) The strength of as-machined specimen increased with increasing testing temperature. This indicates that the surface cracks could be healed by oxidation during testing at elevated temperature.
(c) The strength of heat-treated SiC remains almost constant (~470 MPa) up to 1,400 °C, when the ground specimen was heat-treated at 1,500 °C for 50 h in air.

From the present study, it should be clear that the pre-oxidation technique is a useful way to strengthen the silicon carbide after machining.

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References

1. Lange JJ, Radford KC (1970) J Am Ceram Soc 53:420
2. Gupta TK (1976) J Am Ceram Soc 59:259
3. Moffatt JE, Plumbridge WJ, Hermann R (1996) Br Ceram Trans 95:23
4. Choi SR, Tikara V (1992) Scripta Metall Mater 26:1263
5. Chu MC, Ando K, Sato S, Yao F, Kobayashi Y (1998) High Pressure Inst Jpn 36:82
6. Zhang YZ, Edwards L, Plumbridge WJ (1998) J Am Ceram Soc 81:1861
7. Yao F, Ando K, Chu MC, Sato S (2000) J Mater Sci Lett 12:1081
8. Chu MC, Sato S, Kobayashi Y, Ando K (1995) Fatigue Fract Eng Mater Struct 18:1019
9. Petrovic JJ, Jacobson LA (1976) J Am Ceram Soc 59:34
10. Korous Y, Chu MC, Nakatani M, Ando K (2000) J Am Cerm Soc 83:2788
11. Chu MC, Cho SJ, Park HM, Yoon KJ, Ryu H (2004) Mater Lett 58:1313
12. Chu MC, Cho SJ, Lee YC, Park HM, Yoon DY (2004) J Am Ceram Soc 87:490
13. ISO 14740 (2000) Fine ceramics – Test method for flexural strength of monolithic ceramics at room temperature
14. Mecholsky JJ, Freiman SW, Rice RW (1977) J Am Ceram Soc 60:114