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Strength recovery behavior of machined Al_2O_3/SiC nano-composite ceramics by crack-healing

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Abstract

The machined alumina/15 vol.% SiC nano-composites having a semicircular groove were conducted to various heat treatments. The change of the local fracture stress at the bottom of semicircular groove has been investigated as a function of crack-healing temperature and time. By heating at 1400 °C at 1 h, the machined specimen was fractured not from the cracks introduced by the hard machining but from the other region. Thus, it was concluded that the machining cracks were completely healed by the above heat treatment. Moreover, The strength at room temperature and elevated temperature (<1300 °C) of the machined sample healed exhibited the same levels as that of polished sample healed. It can be demonstrated that the crack-healing was useful for an increase in the strength of machined ceramics economically.

Keywords: Crack-healing; Composites; Al_2O_3; SiC; Strength

1. Introduction

An alumina is a very popular ceramics, exhibits excellent mechanical properties and excellent oxidation resistance than metals at high temperature. Thus, it is expected to apply to structural component operating at high temperature in various fields. However, an alumina has following three weakness: (1) low strength (≈400 MPa), (2) low fracture toughness (≈3.0 MPa $m^{1/2}$) and (3) low heat-resistance temperature for mechanical service (∼900 °C). To overcome these weakness, nano-composite alumina (Al_2O_3/SiC) proposed by Niihara et al.[1,2] maybe useful. However, Ando and co-workers revised Niihara's nano-composite concept a little. The sintering policies of Ando's mullite/SiC, Si_3N_4/SiC and Al_2O_3/SiC composite are[3–5,15,18]: (a) by adding 15–20 vol.% nanosize SiC particles, prevent the grain growth during sintering and increase strength, (b) by adding 15–20 vol.% nanosize SiC particles, induce the excellent crack-healing ability and (c) by distributing nanosize SiC particles not only on grain boundary but also in Al_2O_3 grain, increase the heat-resistance temperature for mechanical service (bending strength at 1300 °C ≈400 MPa).

It is well known that the flawed ceramics recover strength by heat treatment.[6–13] The recovery is caused by following three reasons: (A) by relieving the tensile residual stress, (B) by re-sintering the flaws and (C) by crack-healing. In the case of Al_2O_3 and Al_2O_3/SiC composite, tensile stress relieving and re-sintering can be achieved even in vacuum, nitrogen, argon and air atmosphere. However, crack-healing cannot be achieved in vacuum, nitrogen and argon atmosphere[3,4] because crack-healing can be achieved by the following chemical reaction:

$$SiC + \frac{3}{2}O_2 \rightarrow SiO_2 + CO + 943\ \text{kJ/mol} \quad (1)$$

So, for the crack-healing, O_2 is necessary, thus embedded crack can not be healed.[14–16] For the complete strength recovery, following three conditions are necessary[14–16]: (1) healing material (SiO_2) have to exhibit same level or higher strength than that of base material, (2) SiO_2 have to be bonded to base material strongly and (3) SiO_2 have to fill the crack completely. To achieve the above (1) and (2) conditions, large heat generation of 943 kJ/mol is very important and to achieve above (3) condition, nanosize SiC have to be added more than 10 vol.%. If above three conditions were filled completely, most samples fractured outside the crack-healed zone.

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Recently, Ando et al. proposed through-life reliability management method of ceramics components using crack-healing and proof-testing.[17,18] The methodology is composed of following three phases: (a) after machining, heal the surface cracks and recovery the strength, (b) by the proof-testing, omit the components which have non-acceptable embedded flaws and (c) if crack initiate during service, heal the crack during service and continue the service. As to above (b), Ando et al. proposed proof-testing theory already and showed that temperature dependence of calculated minimum fracture stress showed good agreement with experimental one of Si_3N_4/SiC and Al_2O_3/SiC. As to above (c), Ando et al. succeed to sinter the SiC, Si_3N_4/SiC, Al_2O_3/SiC and mullite/SiC with excellent crack-healing ability. It was shown that these ceramics were able to heal crack even under static or cyclic stress and exhibited the same strength and fatigue limit at the temperature of healing. They also investigated the threshold stress condition for crack-healing and found if applied stress intensity factor is lower than $0.6K_{1C}$, crack could be healed completely.[19–21]

As to a strength recovery behavior of machined ceramics related to above (a), many useful studies have been made using Al_2O_3 and Al_2O_3/SiC composite. However, studies related to Al_2O_3/SiC composite which was added more than 10 vol.% SiC and exhibit excellent crack-healing ability are few yet.

In this study, the nano-composite Al_2O_3/SiC was sintered and small bending specimen was made. A semicircular groove was machined heavily at the center of the specimen's surface. The machined specimens were crack-healed under various conditions. The fracture stress of these specimens after crack-healing were evaluated systematically at RT and high temperatures. It was found that the machined specimen recovered the strength almost completely by healing. Therefore, it was concluded that the crack-healing was the excellent technique to improve the structural integrity of machined Al_2O_3/SiC composite, economically.

2. Experimental

2.1. Materials

The alumina powder (AKP-20, Sumitomo Chemical Co. Ltd., Japan) used in the present experiment has an average particle size of 0.5 μm. The SiC powder (Ultrafine grade, Ibiden Co. Ltd., Japan) used has a mean particle size of 0.27 μm. The mixture of alumina powders and 15 vol.% SiC powders were blended well in ethanol for 48 h using alumina balls and an alumina mill pot. Rectangular plates (90 mm × 90 mm × 6 mm) were hot pressed in N_2 at 1600 °C under 35 MPa for 4 h. The sample nano-composite alumina was sintered at a relatively low temperature (1600 °C). The sintered plates were cut into 3 mm × 4 mm × 22 mm rectangular bar specimens as shown in Fig. 1. The specimens were polished to a mirror finish according to the JIS standards[22] on a tensile surface. The edges of the specimens were beveled 45° to prevent fractures due to edge-cracks. In this paper, these specimens are called "smooth specimens". A semicircular groove was made at the center of the smooth specimens by using a diamond-coated ball-drill as shown in Fig. 1. The grinding direction was perpendicular to the long side of the specimens. The other grinding conditions are listed in Table 1. A photograph of the as-machined specimen is shown in Fig. 2.

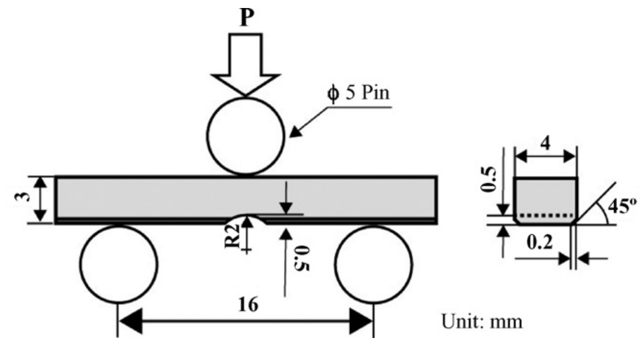

Fig. 1. Three-point loading system, test specimen size and semicircular groove.

Table 1
Grinding conditions

Figuration of machined specimens	Semicircular groove
Grindstone	Ø4 #140 Diamond electrocoated
Number of rotations (rpm)	5000
Table feed speed (mm/min)	100
Depth of cut by one pass (μm)	10

2.2. Crack-healing conditions and test method

The machined specimens were crack-healed in air at 1300–1400 °C for 1 or 10 h. The healed specimens are here called "machined specimens healed". As well, the smooth specimens were crack-healed at 1300 °C for 1 h in air. These specimens are defined as "healed smooth specimens". All fracture tests were performed on a three-point bending system with a span of 16 mm as shown in Fig. 1. The crosshead speed was 0.5 mm/min. The tests took place at room temperature and the high temperatures ranged from 500 to 1300 °C. From the maximum bending moment as the specimens fractured, M_F, the following equation was used to evaluate the nominal fracture

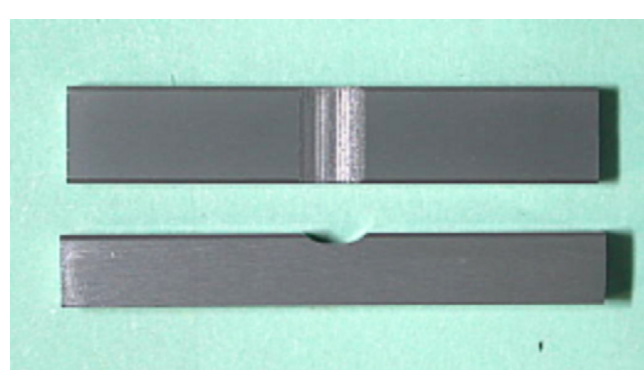

Fig. 2. Photograph of the machined specimen.

stress (σ_{NF}) of the machined specimens.

$$\sigma_{NF} = \frac{M_F}{Z}, \quad (1)$$

where Z was the section modulus of the machined specimens.

Using stress concentration factor, α, and the following equation, one can evaluate the local fracture stress at the bottom of the semicircular groove as follows:

$$\sigma_{LF} = \alpha\sigma_{NF}, \quad (2)$$

where the value of α was calculated to be 1.4.[23]

3. Results and discussion

3.1. Crack-healing behavior

Fig. 3 shows the effect of the crack-healing conditions on the strength recovery behavior of the machined specimen. The symbol (◇) shows the local fracture stress (σ_{LF}) of the as-machined specimen. The σ_{LF} shows large scatter from 200 to 400 MPa. The symbol (●) shows the fracture stress of the healed smooth specimen, with the average fracture stress being 845 MPa. Even the smooth specimen contains minute flaws such as surface cracks and surface pores, yet it was seen that these flaws can be healed by heat-treating, resulting in an increase in strength of approximately 38%.[14,15] By machining, σ_{LF} was drastically reduced from approximately 850 to 200–400 MPa. Of course this decrease in σ_{LF} was attained by the cracks introduced by machining.

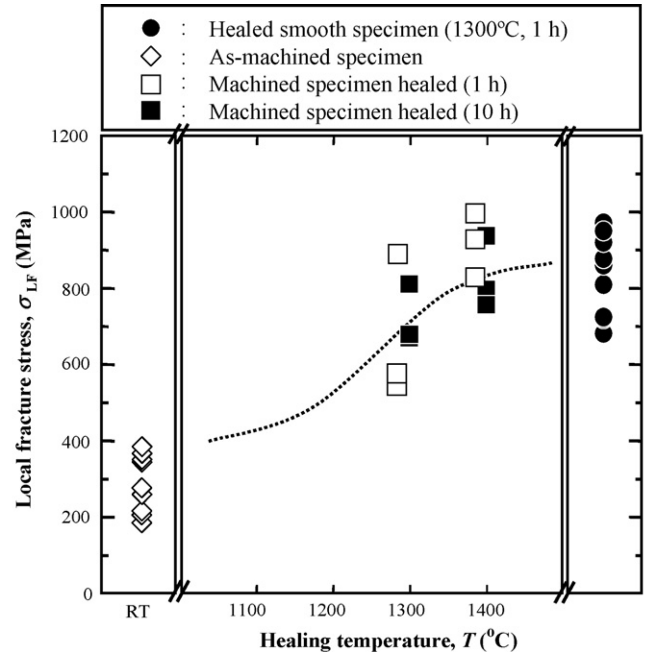

Fig. 3. Effect of crack-healing temperature and time on the strength recovery behavior of machined specimen.

Fig. 4(a) shows SEM image of the fracture surface of as-machined specimens. Also, Fig. 4(b and c) show the detail SEM images of points A and B in the Fig. 4(a), respectively. The fracture origins were confirmed to be cracks introduced by machining as shown in Fig. 4(a). This fact suggested that

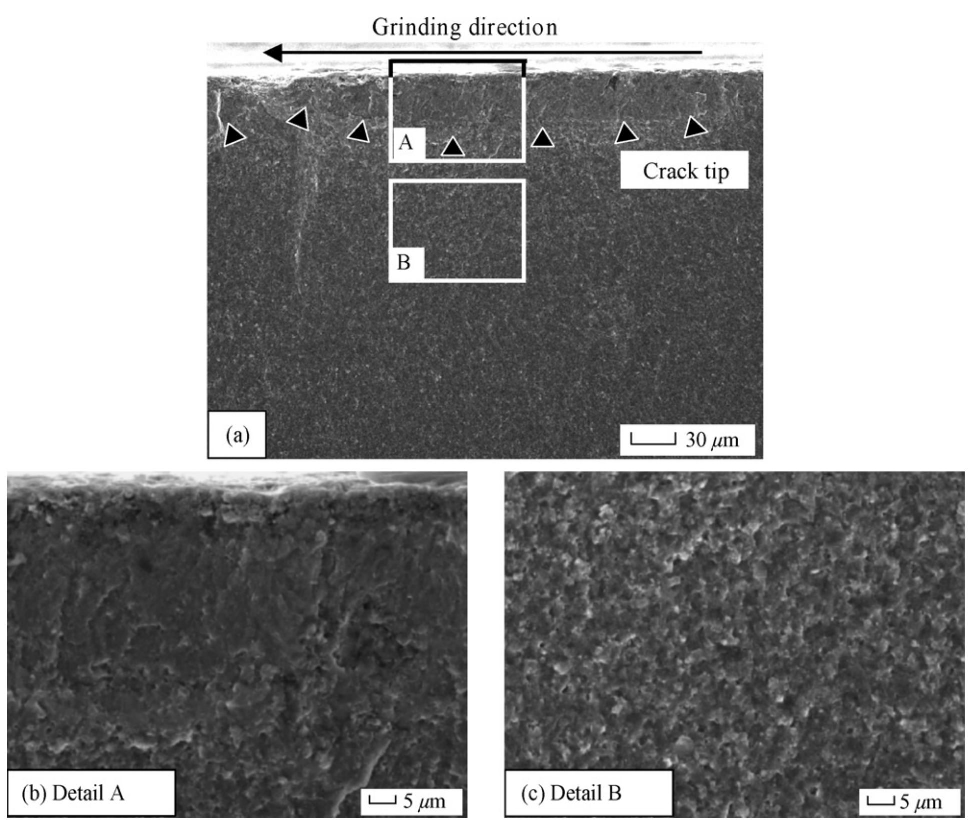

Fig. 4. SEM images of surface crack introduced by machining. (a) σ_{LF} = 205 MPa, (b) detail A and (c) detail B.

the machining crack should be the severest flaws of all the machining damages and the embedded flaws, and then its size determined the strength of as-machined specimen. As shown in Fig. 4(b and c), fracture mode was changed by this boundary: i.e., the fracture mode at point A was intragranular and that at point B was the mode mixed intergranular and intragranular. Therefore, the boundaries shown as closed triangles in the figures were assumed to be the crack tip. This crack was likely formed by connecting the semi-elliptical cracks, thereby having wavy tip. Moreover, the maximum depth of this crack was found to be about 30 μm. By using the same method, the depth of crack of the other as-machined specimens was determined. As a result, it was found that there is a large scatter of the machining crack depth, and the σ_{LF} decreased as the crack depth increased.

Again in Fig. 3, the symbols (□) and (■) show the σ_{LF} of the machined specimen healed for 1 and 10 h, respectively. As the healing temperature increases, the σ_{LF} increases significantly. The average fracture stress of the specimens crack-healed at 1400 °C for 1 or 10 h were found to reach a maximum value and the σ_{LF} was almost equal to the fracture stress of the healed smooth specimen (●). Thus, the standard crack-healing condition for the machined specimen is defined as heating at 1400 °C for 1 h. This condition is quite different from the optimized crack-healing condition for the indented crack heated at 1300 °C for 1 h.[14,15]

The optimized crack-healing temperatures of the machining crack and indented crack are 1400 and 1300 °C, respectively. The machining crack required 100 °C higher temperature than that of indented crack. This reason has not been clarified yet. However, the following two reasons can be pointed out: (1) the difference in the state of the residual stress acted at the subsurface between those cracks and (2) oxidation of SiC by the heat generation during machining. The machining crack was closed by the action of the compressive residual stress. Which would result in reduction of supply of the oxygen in air. Moreover, before the crack-healing treatment, if SiC particles were already covered with a thin oxidation layer by the grinding heat, this would lead to the decrease in the oxidation rate of SiC particles.

3.2. Statistical properties of local fracture stress and SEM image

Fig. 5 shows the Weibull plots of the local fracture stresses of the as-machined specimen (◇), the machined specimen healed (□) and the healed smooth specimen (●), where all the fractures of the healed smooth specimen initiated from the largest embedded flaw such as a pore.[18] A two parameter Weibull function is given by the following equation:

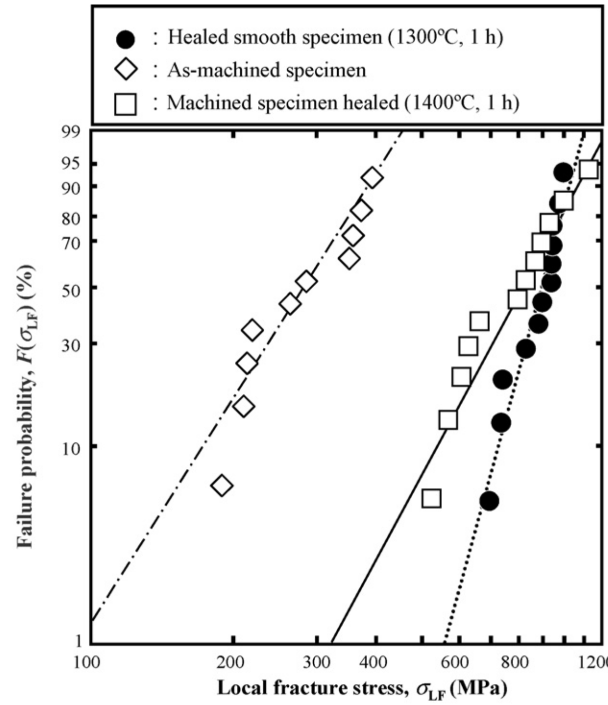

Fig. 5. Weibull plot of local fracture stress.

$$F(\sigma_{LF}) = 1 - \exp\left\{-\left(\frac{\sigma_{LF}}{c}\right)\right\}^{b},$$

where b is a shape parameter and c is a scale parameter.

Table 2 shows the shape parameter, b, the scale parameter, c and the average of the σ_{LF} of the three kinds of samples shown in Fig. 5. The shape parameter of the machined specimen healed is a slightly larger value than that of the as-machined specimens. However, the scale parameter of the machined specimen healed increased considerably from 307 to 846 MPa, with this value being almost equal to that of the healed smooth specimen. This means that machined specimen healed recovered its strength almost completely due to crack-healing.

However, a comparison of the b and σ_{LF} of both specimens reveals that the healed smooth specimen exhibited a slightly larger value than that of the machined specimen healed. Thus, the goodness of fit test (χ^2 test) was used to evaluate whether the σ_{LF} distributions of the machined specimens healed and the healed smooth specimens were independent or not. The statistical hypothesis, H_0, was defined as "distributions were not independent" and the alternative hypothesis, H_1, was "distributions were independent". The value of χ^2 with the degree of freedom of 2 was obtained to be 1.51. This value was lower than

Table 2
Shape parameter (b), scale parameter (c) and average of local fracture stress (σ_{LF})

	Shape parameter	Scale parameter (MPa)	Average of local fracture stress (MPa)
Healed smooth specimen (1300 °C, 1 h)	8.95	891	845
As-machined specimen	3.85	307	278
Machined specimen healed (1400 °C, 1 h)	4.53	846	773

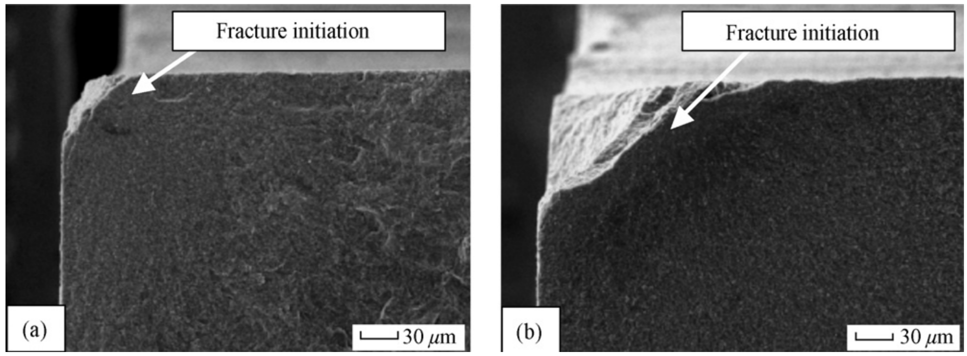

Fig. 6. SEM images showing the fracture initiation from the chipping. (a) $\sigma_{LF} = 981$ MPa and (b) $\sigma_{LF} = 559$ MPa.

that of the type-I error rate (significance level) of 5%. Thus, the H_0 cannot be rejected, and then H_0 was acceptable. From this statistical analysis, it was concluded that the machined specimen healed at 1400 °C for 1 h exhibits almost the same level strength to the healed smooth specimens, though the machined specimen has smaller effective volume than that of smooth specimen. Thus, it can be concluded that strength recovery of machined specimen by crack-healing is not complete. In order to investigate this reason, all fracture surfaces were investigated systematically as shown in Figs. 6 and 7.

Fig. 6 shows a SEM image of the fracture initiating from the chipping zone at the edge. Ten of the 12 machined specimens healed fractured from the chipping zone. Fig. 6(a) shows the smallest chip from which fracture initiated, the specimen having $\sigma_{LF} = 981$ MPa. Fig. 6(b) shows the largest chip from which a fracture initiated, the specimen having $\sigma_{LF} = 559$ MPa. The other two specimens fractured from outside the chipping zone. These specimens fractured from surface at center of the bottom of the semicircular groove, as shown in Fig. 7. In the specimen which exhibited the highest value of $\sigma_{LF} = 1106$ MPa, crack initiated from the surface of machined groove, as shown in Fig. 7(a and b). On the other hand, the specimen shown in Fig. 7(c) had the sharp chip at the center of the bottom of the semicircular groove (with a length of approximately 90 μm and a depth of approximately 30 μm), thereby it fractured from the chip and exhibiting the lowest $\sigma_{LF} = 516$ MPa. From these observations, the reason that machined specimen healed was not able to recover the strength completely was attributed the chipping occurred at the end of machined groove or the bottom of the machined groove.

From these test results, it can be concluded that: (1) crack-healing at 1400 °C for 1 h led to an almost complete strength recovery of the machined specimens, (2) chipping cannot be healed and the specimen with chipping exhibits a slightly lower σ_{LF}. Thus, it was demonstrated that almost all of the cracks of the alumina/SiC particle composites could be healed by crack-healing at 1400 °C for 1 or 10 h.

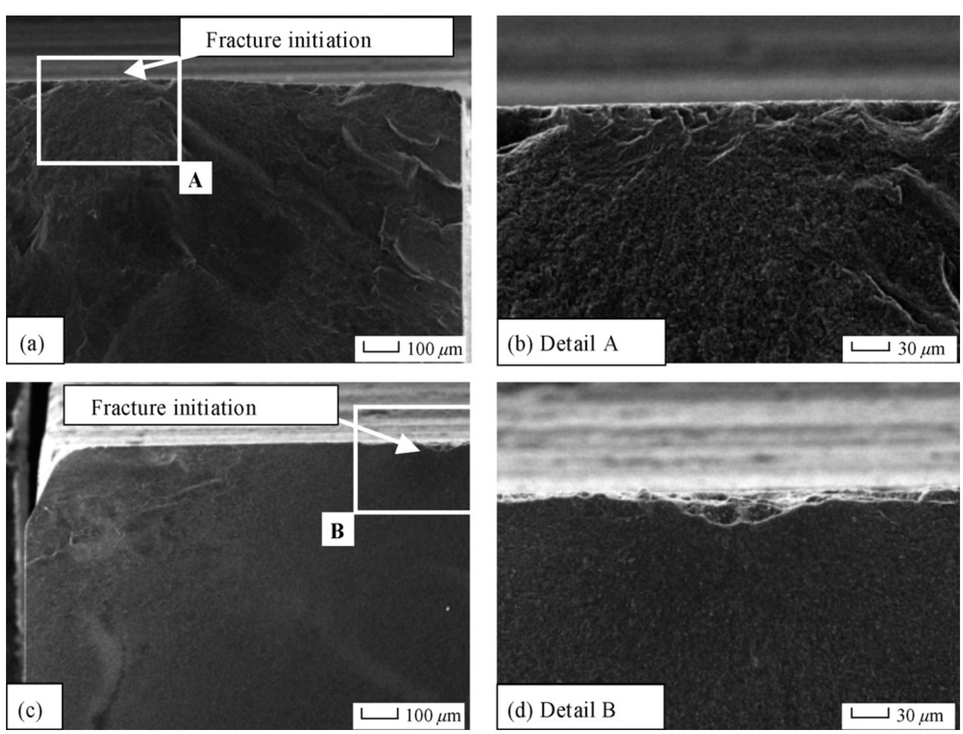

Fig. 7. SEM image showing the fracture initiation from the bottom of the semicircular groove. (a) $\sigma_{LF} = 1106$ MPa, (b) detail A, (c) $\sigma_{LF} = 516$ MPa and (d) detail B.

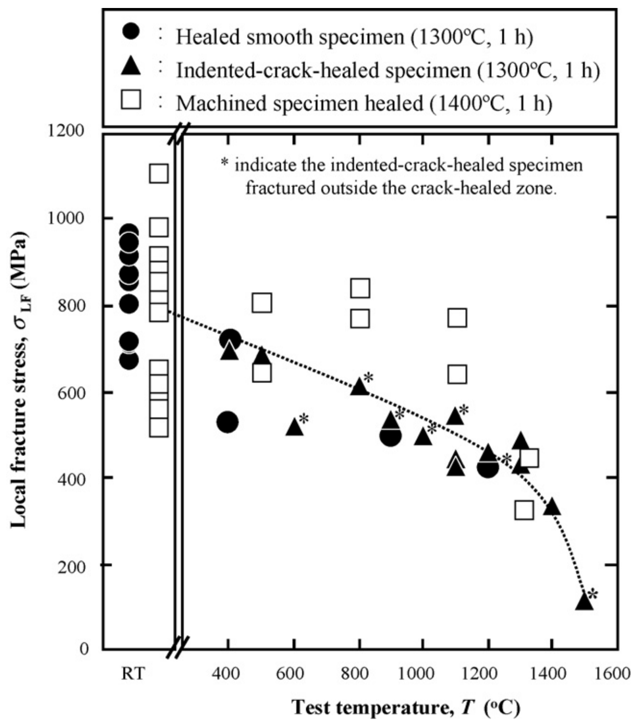

Fig. 8. Temperature dependence of local fracture stress.

3.3. *Effect of test temperature on the local fracture stress of machined specimens healed*

Fig. 8 shows the temperature dependence of the local fracture stress of the machined specimen healed (□). For comparison, the temperature dependences of the fracture stresses of the indented-crack-healed specimen (▲) and the healed smooth specimen (●) are also shown in this figure. The symbols with asterisks (*) indicate the crack-healed specimens fractured outside the crack-healed zone. These specimens indicate that the pre-crack was completely healed and the healed zone exhibited the same level strength to the base material even up to 1500 °C.[14,15]

The local fracture stresses of the machined specimens healed were almost constant in the temperature range from room temperature to 1100 °C. The constant value of σ_{LF} was found to be about 700 MPa, and was a little higher than that of the crack-healed specimens and the healed smooth specimen at 800–1100 °C. This would result from the local plasticity at high temperature. At the high temperature range, alumina is able to exhibit the small plasticity. In this study, the fracture stress was obtained from the elastic solution. Thus, the fracture stress obtained using the elastic solution was overestimated. Above 1100 °C, the σ_{LF} decreases markedly with the increase of the test temperature. However, it exhibited the same σ_{LF} of the healed smooth specimen at 1300 °C. Moreover, it was found that all the machined specimen did not fractured from the crack-healed zone from SEM observations. Thus, it could be concluded that machined specimen healed exhibited the same levels strength and the high reliability to the based material (Al_2O_3/SiC composite) up to 1300 °C.

4. Conclusions

A nano-composite Al_2O_3/SiC was sintered and small bending specimens (22 mm × 4 mm × 3 mm) were made. A semicircular groove was machined hardly using a diamond ball-drill at the center of the specimen's surface. The as-machined specimens were crack-healed under various conditions. The fracture stresses of these specimens after crack-healing were evaluated systematically at RT and high temperatures. The main conclusions are as follows:

(1) Hard machining introduced many cracks and chips in the specimens. All as-machined specimen were fractured by the cracks introduced by machining. The cracking reduced the local fracture stress at RT from approximately 845 to 250 MPa.
(2) The cracks introduced by machining were healed almost completely, and the local fracture stress of the machined specimen recovered from approximately 250 to 773 MPa by healing at 1400 °C for 1 h. However, the chips were not healed and affected the strength of the specimen; thus most machined specimens healed (10 of 12) fractured from the chip. As a result, the average local fracture stress (773 MPa) exhibited a slightly lower value than that (845 MPa) of the healed smooth specimen.
(3) The machined specimen healed exhibited a little higher or the same level strength to the healed smooth specimen up to 1300 °C.
(4) These results demonstrated crack-healing to be a very effective technique to reduce machining costs and to improve the structural integrity of machined alumina.

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