*Research Article*

# Improvement in Contact Strength of Si$_3$N$_4$/SiC Composite by Crack Healing

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Ceramics have been used as bearing and cutting tool components, which are subjected to contact loading during their operation. The presence of surface cracks on these components decreases their contact strength. Thus, the reliability of ceramic components can be increased by improving their contact strength through crack healing. In the present study, the effects of crack healing on the contact strength of a silicon carbide-(SiC-) reinforced silicon nitride (Si$_3$N$_4$) composite subjected to various machining processes were investigated. The contact strength of this composite was evaluated using a sphere indentation test in which acoustic emission was used. The results showed that the contact strength of the composite improved when it was subjected to crack healing in combination with rapping; this was true even when the composite had cracks due to a heavy machining process.

## 1. Introduction

Structural ceramics have excellent mechanical properties, corrosion resistance, and wear resistance. However, their fracture toughness is lower than that of metals. As a result, surface cracks may develop on the surface of ceramic components during machining or operation. These cracks can considerably decrease the reliability and strength of the components. A common method for fabricating ceramic components involves the use of diamond abrasives for machining them. This process usually results in countless cracks on the surface layers of the components. Thus, the machining of ceramics is very costly, and not all minute surface cracks can be removed.

To overcome this problem, some researchers have attempted to recover the strength of ceramics by means of crack healing. Chu et al. [1] showed that a 100 $\mu$m long surface indentation crack in mullite reinforced using 20 vol% SiC particles can be completely healed by subjecting it to heat treatment at 1300°C for 1 h in air. Ando et al. [2] reported that crack healing can be achieved by the oxidation of SiC and Si$_3$N$_4$ in a Si$_3$N$_4$/SiC composite. Nakao et al. [3] stated that the oxidation of SiC generates exothermic heat and

causes a volume expansion of approximately 80% in the condensing phase. As a result, the spaces between crack walls are filled with the healing material and the walls are bonded strongly.

Crack-healing behavior in machined ceramics has also been studied by several researchers. Wu et al. [4] reported that an Al$_2$O$_3$/SiC composite showed a substantial increase in strength after grinding and heat treatment in air at 1250°C. Liu et al. [5] reported that the heating of ground ceramic components made from alumina (Al$_2$O$_3$) and silicon nitride (Si$_3$N$_4$) in air can increase their fracture strength. Lee et al. [6] reported that the numerous surface cracks resulting from the heavily machining of SiC-reinforced mullite can be completely healed by the crack-healing treatment. Osada et al. [7] investigated the high-temperature strength of machined Al$_2$O$_3$/SiC composite specimens subjected to crack healing and reported that healed specimens exhibit the same level of strength as the smooth specimens do up to 1300°C. Jung et al. [8] stated that surface cracks in a Si$_3$N$_4$/SiC composite resulting from wheel grinding are completely healed by heat treatment in air at 1200–1400°C. These results indicate that crack healing is an effective method for increasing the reliability of machined ceramic components.

Ceramic components such as bearing and cutting tools have been widely used in various engineering applications. These components are subjected to contact loading during their operation. The contact strengths of ceramics have been studied by several researchers. They have found that surface cracks on these components decrease their contact strength. Fett and Munz [9] reported that a surface crack near the contact zone decreases the Weibull exponents for strength. Strength recovery in ceramics by means of crack healing has been studied by many researchers. However, in most of these studies, the strength of ceramics after crack healing was measured by bending tests. Thus, the effects of crack healing on the contact strength of ceramics have not been studied yet.

In the present study, the effects of crack healing on the contact strength of a $Si_3N_4$/SiC composite subjected to various machining processes were investigated. The contact strength was evaluated using a sphere indentation test in which acoustic emission (AE) was used.

## 2. Experimental Procedure

*2.1. Materials and Specimens.* In this study, we used a silicon carbide-(SiC-) reinforced silicon nitride ($Si_3N_4$) composite, which has excellent crack-healing ability. $Si_3N_4$ and SiC powders used for preparing the composite had mean particle sizes of 0.2 and 0.27 $\mu$m, respectively. The samples were prepared using a mixture of $Si_3N_4$ and 20 wt% SiC powders with 8 wt% $Y_2O_3$ powder as a sintering additive. The $Y_2O_3$ powder had a 0.4 $\mu$m mean particle size. To this mixture, alcohol was added, and the mixture was blended for 48 h. The mixture was first placed in an evaporator to extract the solvent and then in vacuum to obtain a dry powder mixture. The mixture was subsequently hot-pressed at 1850°C and 35 MPa for 2 h in a $N_2$ atmosphere. This material was selected as the test material because of its excellent crack-healing ability.

The hot-pressed plate was then cut into specimens measuring $3 \times 4 \times 40$ mm. The specimens were polished to a mirror finish on one face. These specimens will hereafter be referred to as "Smooth specimens." Smooth specimens developed some minute surface defects during the polishing process. Jung et al. [8] reported that the optimum conditions for crack healing a $Si_3N_4$/SiC composite in air are 1300°C and 1 h. Thus, some Smooth specimens were heat-treated at 1300°C for 1 h in air. Such specimens are referred to as "Smooth + Healed specimens."

Smooth specimens were machined under several conditions for the specimens to develop surface cracks. Table 1 shows the machining conditions. The specimens that were ground using #200 and #400 diamond wheels are referred to as "#200 specimens" and "#400 specimens," respectively. The machined specimens were crack-healed in air at 1300°C for 1 h. These specimens are referred to as "#200 + Healed specimens" and "#400 + Healed specimens."

In order to reduce surface roughness, a 5 $\mu$m surface layer in #400 machined specimens was removed by rapping before or after crack healing. These specimens are referred to as

TABLE 1: Name of specimen and machining conditions.

| Name | Machining process |
|---|---|
| Smooth | — |
| Smooth + Healed | Smooth → heat treated |
| #200 | Smooth → #200 grinding |
| #200 + Healed | Smooth → #200 grinding → heat treated |
| #400 | Smooth → #400 grinding |
| #400 + Healed | Smooth → #400 grinding → heat treated |
| #400 + Rap | Smooth → #400 grinding → rapping |
| #400 + Rap + Healed | Smooth → #400 grinding → rapping → heat treated |
| #400 + Healed + Rap | Smooth → #400 grinding → heat treated → rapping |

Heat treated at 1300°C, 1 h in air.

"#400 + Rap + Healed specimens" and "#400 + Healed + Rap specimens," respectively. Jung et al. [8] reported that the crack depth obtained in a $Si_3N_4$/SiC composite by grinding was 20–30 $\mu$m. Thus, the rapping depth of 5 $\mu$m is less than the machining crack depth. This is because the aim of the rapping is to reduce only the surface roughness. We confirmed that the removal of surface layers by rapping resulted in negligible residual stress.

*2.2. Measurement of Contact Strength.* The contact strength of the specimens was measured using sphere indentation tests. Figure 1 shows a schematic of the testing system. Indentations were made on the surfaces of the specimens using tungsten carbide spheres with a diameter of 4 mm. These tests were carried out using a universal testing machine at a crosshead speed of 0.2 kN/min. The ring crack initiation load was determined using AE. Bouras et al. [10] stated that subcritical crack growth is one of the sources of AE in ceramic materials. The loading was interrupted at the load detected by AE. After the loads were removed, the indented surfaces were observed by optical microscopy to identify crack initiation. Figure 2 shows an example of a ring crack. The Weibull distributions of the crack initiation load were determined for each specimen on the basis of many sphere indentation tests.

*2.3. Measurement of Bending Strength.* The bending strengths of the specimens mentioned in Section 2.1 were also measured so that the effects of crack healing on the contact strength and bending strength could be compared. Three-point bending tests were carried out using a universal testing machine at a crosshead speed of 0.5 mm/min and a fixture with a span of 16 mm.

## 3. Experimental Results and Discussion

*3.1. Weibull Distribution of Crack Initiation Load.* The crack initiation loads were statistically analyzed by a two-parameter Weibull function, as shown in Figure 3. In these analyses, the failure probability $F$ was calculated using the median rank
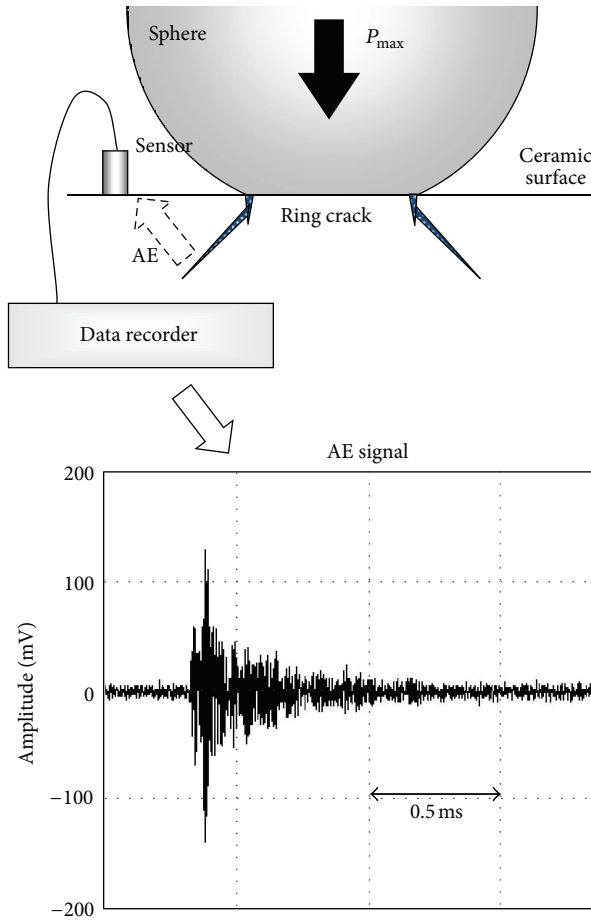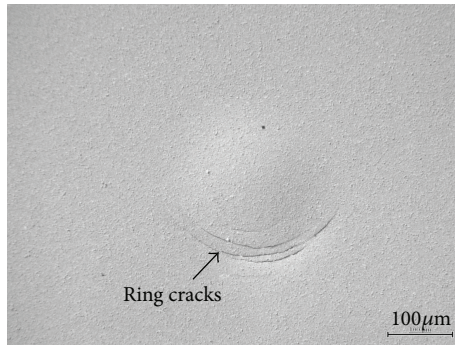
FIGURE 1: Schematic illustration of surface ring cracks and AE signal.



FIGURE 2: Ring cracks after sphere indentation test. For Smooth specimens, $P_{max} = 0.99$ kN.

estimate. These results were plotted using the two-parameter Weibull function, as expressed by the following equation:

$$F\left(P_{max}\right) = 1 - \exp\left\{-\left(\frac{P_{max}}{\beta}\right)^{\alpha}\right\}, \qquad (1)$$

where $P_{max}$ is the crack initiation load, $\beta$ is a scale parameter, and $\alpha$ is a shape parameter.

Table 2 lists the shape and scale parameters for the crack initiation load obtained from the Weibull distribution.

TABLE 2: Shape parameter $\alpha$, scale parameter $\beta$, bending strength, and roughness of specimens.

| Specimens | Shape parameter $\alpha$ (—) | Scale parameter $\beta$ (kN) | Bending strength $\sigma$ (MPa) | Surface roughness $R_z$ ($\mu$m) |
|---|---|---|---|---|
| Smooth | 1.9 | 0.65 | 861.9 | 0.10 |
| Smooth + Healed | 3.8 | 0.93 | 915.4 | 0.66 |
| #200 | 1.4 | 0.33 | 521.6 | 2.74 |
| #200 + Healed | 1.4 | 0.12 | 900.0 | 2.81 |
| #400 | 1.4 | 0.40 | 648.5 | 1.52 |
| #400 + Healed | 1.1 | 0.30 | 913.7 | 1.82 |
| #400 + Rap | 1.1 | 0.56 | 676.2 | 0.15 |
| #400 + Rap + Healed | 3.6 | 1.06 | 856.5 | 1.02 |
| #400 + Healed + Rap | 3.2 | 1.06 | — | 0.23 |

The symbols (○) and (●) in Figure 3(a) denote the crack initiation loads for the Smooth and Smooth + Healed specimens, respectively. The crack initiation load for the Smooth specimens is slightly scattered and showed 2-mode distributions. In the lower fracture probability region, the values of $P_{max}$ in the smooth specimens were affected by the surface cracks induced by machining that existed near the sphere indentation site. The $P_{max}$ depends on the size and distribution of the cracks. If a large crack existed near the sphere indentation site, the $P_{max}$ tended to be decreased.

The values of $\alpha$ exhibited by the Smooth + Healed specimens were almost twice those exhibited by the Smooth specimens. A high $\alpha$ value implies that the crack initiation load had low scatter. In addition, the $\beta$ values for the Smooth + Healed specimens were higher than those for the Smooth specimens. The Smooth specimens contained minute flaws such as surface cracks and surface pores; yet it was observed that these flaws could be healed by heat treating, resulting in an increase in the crack initiation loads in the Smooth + Healed specimens.

The symbols (□) and (■) in Figure 3(b) denote the crack initiation loads for the #200 and #200 + Healed specimens, respectively. The symbols (△) and (▲) in Figure 3(c) denote the crack initiation loads for the #400 and #400 + Healed specimens, respectively. The #200 and #400 specimens exhibited lower $\alpha$ and $\beta$ values than the Smooth specimens did. This is because the surface cracks developed in these specimens were larger in number and size than those in the Smooth specimens. Moreover, the #200 + Healed and #400 + Healed specimens exhibited lower $\alpha$ and $\beta$ values than the #200 and #400 specimens, respectively, did. The surface cracks resulting from machining were healed by crack healing. However, the surface roughness was not improved after crack healing, as shown in Table 2. Thus, the crack initiation loads for the #200 + Healed and #400 + Healed specimens were not improved.

The symbols (◇), (◆), and (▼) in Figure 3(d) denote the crack initiation loads for the #400 + Rap, #400 + Rap + Healed, and #400 + Healed + Rap specimens, respectively. As expected, the $\beta$ values for the #400 + Rap specimens
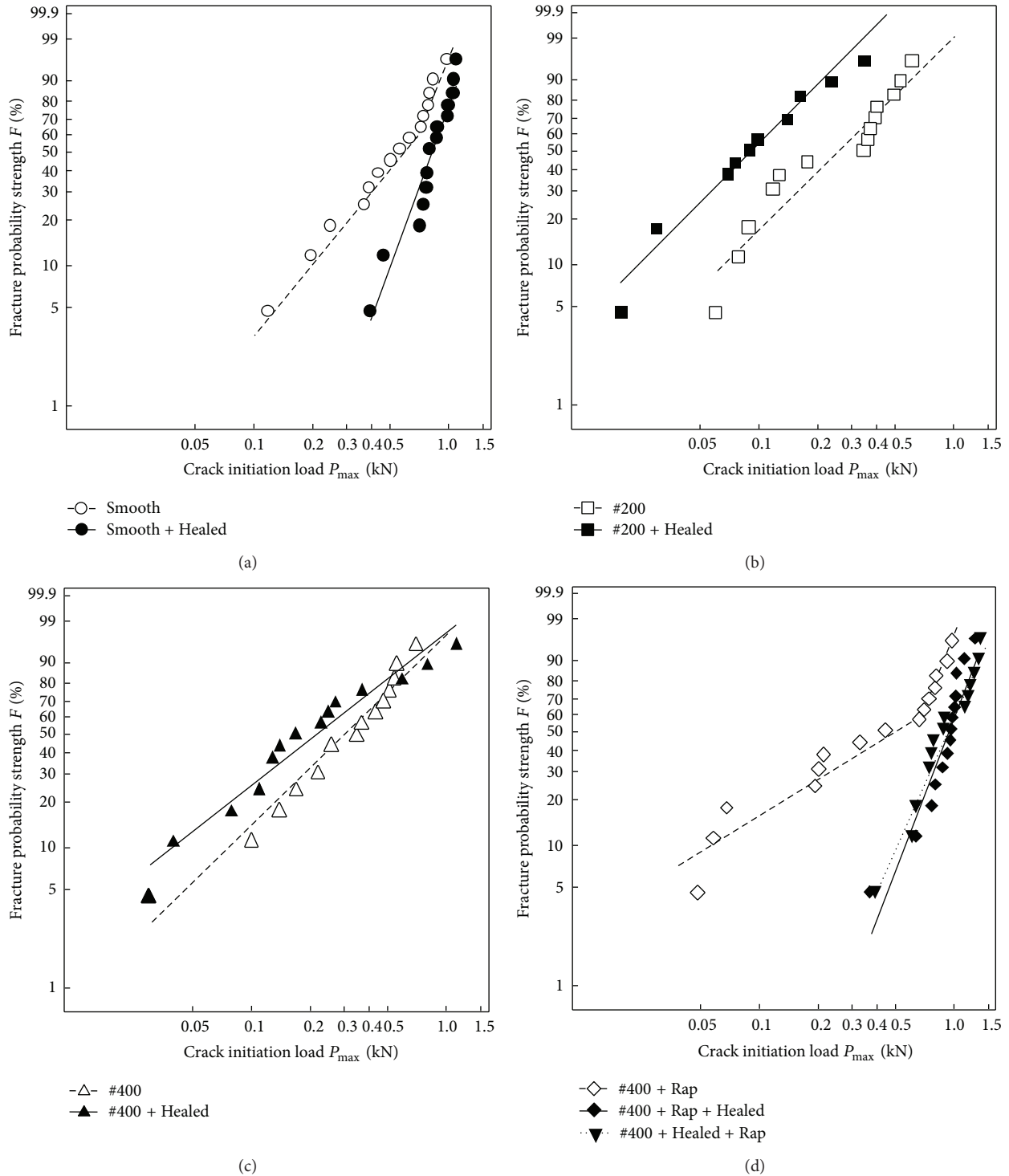
(a)



(b)



(c)



(d)

FIGURE 3: Weibull distributions of crack initiation load. (a) Smooth and Smooth + Healed specimens, (b) #200 and #200 + Healed specimens, (c) #400 and #400 + Healed specimens, and (d) #400 + Rap, #400 + Rap + Healed, and #400 + Healed + Rap specimens.

were lower than those for Smooth specimens (see Table 2). These results indicated that the surface cracks induced by #400 were not eliminated the by rapping. The #400 + Rap + Healed and #400 + Healed + Rap specimens exhibited much higher $\alpha$ and $\beta$ values than the #400 + Healed and #400 +

Rap specimens did. Moreover, the $\alpha$ and $\beta$ values for these specimens were similar to those for the Smooth + Healed specimens. Thus, reducing the surface roughness of crack-healed specimens by rapping is effective in increasing the crack initiation loads, that is, the contact strength. The effects
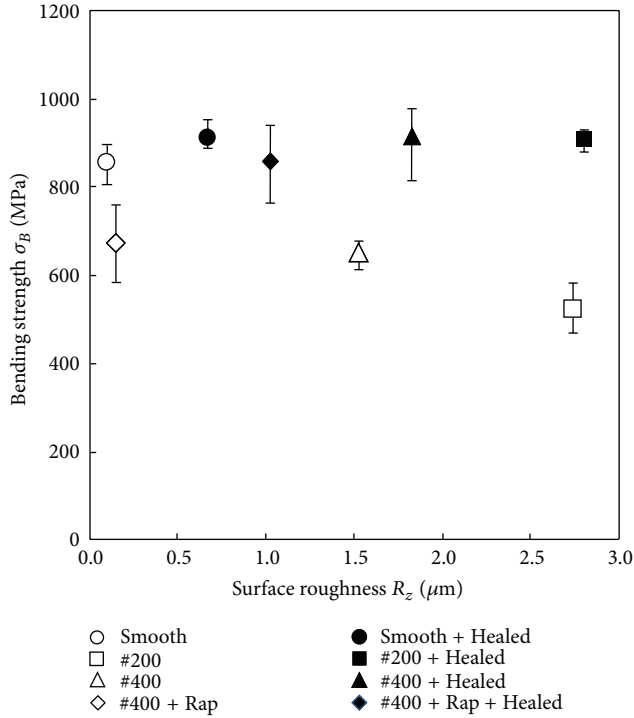
FIGURE 4: Relationship between bending strength and surface roughness of specimens.

of surface roughness on the contact strength are discussed in Section 3.3.

*3.2. Effect of Surface Roughness on Bending Strength.* Figure 4 shows the bending strength as a function of surface roughness. The values of the bending strength and surface roughness are listed in Table 2. Three specimens were used in each bending test. The error bar in Figure 4 shows the scatter of the bending strength. The open and solid marks denote the non-crack-healed and crack-healed specimens, respectively. The bending strength of the non-crack-healed specimens decreased with increasing surface roughness. However, the bending strength of the group of crack-healed specimens was similar to that of the Smooth + Healed specimens. Thus, the surface cracks resulting from machining were healed completely with respect to the bending strength. It should be noted that the bending strength of the specimens was not highly sensitive to the surface roughness at the roughness levels tested in this study. This tendency was different from that shown by the contact strength, as described in the next section.

*3.3. Effect of Surface Roughness on Contact Strength.* Figure 5 shows the relationship between the contact strength and surface roughness of the specimens. The $\beta$ values shown in Table 2 and Figure 3 are used to represent the contact strength. The surface roughness values are listed in Table 2. The open and solid marks represent the non-crack-healed and crack-healed specimens, respectively. The contact strength of the non-crack-healed specimens decreased with
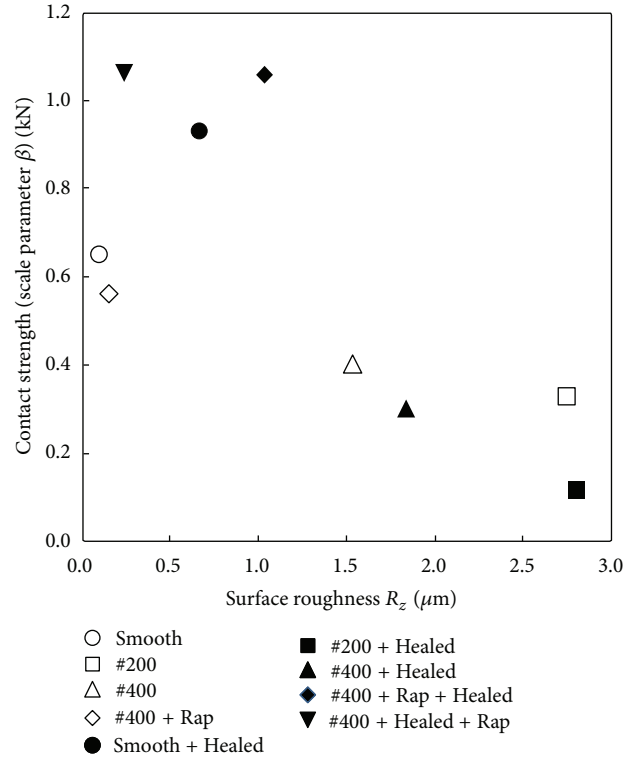


FIGURE 5: Relationship between contact strength and surface roughness of specimens.

increasing surface roughness. However, the contact strength of the Smooth + Healed specimens increased when $R_z$ was less than $1\,\mu$m because the surface cracks resulting from machining were healed completely. However, the contact strength of the #200 + Healed and #400 + Healed specimens did not increase by crack healing. Thus, the contact strength was affected by surface roughness when $R_z$ was more than $1\,\mu$m. It is possible that the surface roughness caused stress concentration at the sphere indentation site. Thus, the contact strength decreased with increasing surface roughness. On the other hand, the fracture initiation sites in bending test specimens were not necessarily on the surface. Thus, the surface roughness did not affect the bending strength significantly.

The contact strength of the #400 + Rap + Healed and #400 + Healed + Rap specimens increased more than that of the #400 + Healed specimens. On the basis of these experiments, it can be concluded that the contact strength of the $Si_3N_4/SiC$ composite was improved by subjecting it to crack healing and rapping, even when the composite had cracks resulting from heavy machining.

## 4. Conclusions

The effects of crack healing on the contact strength of a $Si_3N_4/SiC$ composite subjected to various machining processes were investigated. The contact strength was evaluated using a sphere indentation test in which acoustic emission was used. The results can be summarized as follows.

(1) The contact strength of the Smooth specimens was increased by crack healing, which eliminated surface flaws.

(2) The contact strengths of the #200 and #400 specimens were lower than that of the Smooth specimens because the surface cracks in these specimens were greater in number and size than those in the Smooth specimens.

(3) The contact strengths of the #400 + Rap + Healed and #400 + Healed + Rap specimens were quite high and were nearly equal to that of the Smooth + Healed specimens.

(4) The bending strength of the specimens with surface cracks was improved by crack healing and had almost constant values in spite of the increase in surface roughness. Thus, the bending strength of the specimens was not highly sensitive to the surface roughness.

(5) These results suggest that the contact strength of the machined $Si_3N_4$/SiC composite was improved by a combination of crack healing and rapping, even when the composite had cracks due to heavy machining.

## Acknowledgment

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