

# Improvement of strength and reliability of ceramics by shot peening and crack healing

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## Abstract

In this study we propose a new method for improving the surface strength and reliability of ceramics that combines shot peening with crack-healing ability. We used Si<sub>3</sub>N<sub>4</sub>/SiC composite ceramics with high crack-healing ability and subjected the specimens to shot peening and crack healing. To evaluate the effect of our method, we investigated the residual stress after shot peening and crack healing and examined the specimens' mechanical properties, including apparent fracture resistance, contact strength and bending strength. We found that shot peening and crack healing is effective to increase apparent fracture resistance, contact strength and bending strength.

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**Keywords:** Crack healing; Shot peening; Si<sub>3</sub>N<sub>4</sub>/SiC; Residual stress; Contact strength; Ring crack

## 1. Introduction

Ceramics are candidate materials for industrial applications because of their excellent mechanical, tribological and thermal properties. These applications include bearings, turbo charger rotors, diesel engine components and cutting tools. However, because ceramics are brittle materials, fracture toughness is lower than that of metallic materials, resulting in lower reliability in mechanical properties. Thus, if the brittleness of ceramics is overcome, the reliability and product life of ceramic components will be improved. Many studies have been carried out to overcome the low fracture toughness of ceramics by (a) toughening the ceramic by fiber or whisker reinforcement, and microstructure control and (b) activating the crack-healing ability to heal surface cracks introduced during machining and service.

Significant progress has been made in the area of toughening by fiber or whiskers,<sup>1</sup> while there have been fewer studies involving crack-healing ability, though the number of works is still increasing.<sup>2–15</sup> The proposed mechanisms of crack healing have been described by many researchers. Among the several types of crack healing, that induced by the oxidation of silicon carbide

(SiC) has advantages, because the complete recovery of strength is attained by this mechanism. The SiC oxidation concept was proposed by Ando and colleagues.<sup>7,8</sup> Since then, there have been many reports on crack-healing behavior of Si<sub>3</sub>N<sub>4</sub>/SiC,<sup>8,9,12</sup> mullite/SiC,<sup>7,13</sup> Al<sub>2</sub>O<sub>3</sub>/SiC<sup>10,11</sup> and SiC/AlN.<sup>14</sup>

Shot peening is a common procedure used to increase the near-surface strength of metals.<sup>16</sup> In the shot-peening process, a stream of small, hard spheres is shot at a treated surface. Compressive residual stress is generated by the localized plastic deformation of the surface layer. Fatigue strength of metals can be increased by shot peening, because the compressive residual stress prevents fatigue crack initiation and propagation.<sup>16,17</sup> Although researchers believed that strengthening of ceramics due to shot peening would not be possible, recent studies show that the near-surface strength of ceramics can be improved by shot peening.<sup>18–21</sup>

Pfeiffer and Frey carried out shot peening on alumina and silicon nitride by using 0.61- to 0.69-mm tungsten carbide shots.<sup>18,19</sup> They reported that high compressive residual stress up to more than 1 GPa could be introduced near the surface and the compressive residual stress improved the near-surface strength of ceramics. Moon et al. reported that apparent fracture resistance at the sub-surface of alumina and silicon nitride was increased by shot peening.<sup>20</sup> Tanaka et al. carried out shot peening on silicon nitride using 0.05-mm high-strength steel

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shots and 1.1-mm tungsten carbide shots.<sup>21</sup> They reported that compressive residual stress up to 1.5 GPa was introduced near the surface, and the compressive residual stress increased the apparent fracture resistance at sub-surface up to 2.5-fold.

Considering the results obtained in previous studies, the shot peening of ceramics is a promising technique for increasing strength in the surface region. Strengthening the surface layer will increase the contact strength of ceramic components such as bearings and turbine blades in which higher contact strengths are desirable. However, if surface cracks are unintentionally induced during the machining and shot-peening process, the reliability of the ceramics components will be decreased. If crack healing is used in combination with shot peening, increasing the surface strength and reliability of ceramics will be achieved simultaneously. In this study we propose a new method for improving the strength and reliability of ceramics that combines shot peening with crack-healing ability. The effects of shot peening and crack healing on the residual stress, apparent fracture resistance at sub-surface, contact strength and bending strength were investigated.

## 2. Experimental procedures

### 2.1. Material and specimens

In this study, we used silicon nitride reinforced by silicon carbide ( $\text{Si}_3\text{N}_4/\text{SiC}$ )<sup>8,9,12</sup> which has excellent crack-healing ability. The  $\text{Si}_3\text{N}_4$  powder used in this study has a mean particle size of 0.2  $\mu\text{m}$ . The SiC powder used has a 0.27- $\mu\text{m}$  mean particle size. The samples were prepared using a mixture of  $\text{Si}_3\text{N}_4$  with 20 wt% SiC powder and 8 wt%  $\text{Y}_2\text{O}_3$  as a sintering additive powder. The  $\text{Y}_2\text{O}_3$  powder used has a 0.4- $\mu\text{m}$  mean particle size. To this mixture, alcohol was added, and the mixture was blended completely for 48 h. The mixture was placed in an evaporator to extract the solvent and then in a vacuum to produce a dry powder mixture. The mixture was subsequently hot-pressed at 1850 °C and 35 MPa for 2 h in an  $\text{N}_2$  atmosphere. The relative density of the hot-pressed material determined by the Archimedes method was 99.5% of the theoretical density (3.32 kgf/mm<sup>3</sup>). The average grain size of the matrix was  $\text{Si}_3\text{N}_4 = 0.44 \mu\text{m}$ , and the average aspect ratio was about 5.0. Most SiC particles were located in grain boundaries and distributed uniformly. This material was selected as a test material because it has an excellent crack-healing ability. The hot-pressed plate was then cut into test specimens measuring 3 mm × 4 mm × 22 mm. The specimens were polished to a mirror finish on one face.

### 2.2. Shot peening and residual stress

Shot peening was carried out on the  $\text{Si}_3\text{N}_4/\text{SiC}$  specimens with a direct pressure peening system. Three specimens were shot peened simultaneously. The zirconium oxide ( $\text{ZrO}_2$ ) beads with a diameter 0.3 mm were used. The peening pressure was 0.2 MPa. Peening time was 40 s for three specimens. The resulting Almen-intensity was 0.16 mmA. The specimens subjected to shot peening are called “SP” specimens, and the specimens

Table 1  
Conditions for X-ray diffraction.

Characteristic X-ray	Cu $K\alpha_1$
X-ray tube	Cu
Diffraction plane	$\text{Si}_3\text{N}_4$ (3 2 3)
Diffraction angle [°]	141.26
Tube voltage [kV]	40
Tube current [mA]	30

without shot peening are called “Non-SP” specimens. After shot peening, specimens were heat-treated in air for 1 or 5 h at temperatures from 500 to 1400 °C so we could investigate the effects of heat treatment on residual stress, apparent fracture resistance, contact strength and bending strength. These specimens are called “SP + heat-treated” specimens. The residual stresses were measured for Non-SP, SP and SP + heat-treated specimens by the X-ray diffraction method. The conditions for X-ray diffraction are shown in Table 1. To investigate the in-depth residual stress distribution, we removed the surface layers by polishing the specimens with diamond abrasives of 9.0, 3.0 and 0.5  $\mu\text{m}$ . We confirmed that removal of the surface layers resulted in negligible residual stress.

### 2.3. Measurement of apparent fracture resistance, contact strength and bending strength

For measurement of apparent fracture resistance at sub-surface, Non-SP specimens, SP specimens and SP + heat-treated specimens were prepared. To prepare SP + heat-treated specimens, the SP specimens were heat-treated in air for 1 h at 900–1300 °C. The apparent fracture resistance ( $K_C$ ) was evaluated by the indentation-fracture (IF) method, in which apparent fracture resistance can be estimated using the following equation in accordance with Japan Industry Standards (JIS)<sup>22</sup>:

$$K_C = 0.026E^{1/2}P^{1/2}\frac{a}{c^{3/2}} \quad (1)$$

where  $E$  is Young’s modulus,  $P$  is the indent load,  $a$  is half the diagonal length of indentation and  $c$  is half the surface crack length. Vickers indentations were introduced on the polished surface by applying an indent load of 49 N for 20 s. The Young’s modulus of the material is  $E = 300 \text{ GPa}$ . Note that the value of fracture resistance ( $K_C$ ) is affected by residual stress.<sup>23</sup> For comparison, we evaluated the intrinsic fracture toughness ( $K_{IC}$ ) of the Non-SP specimens using the single-edge V notch beam (SEVNB) method. The V notch was made on the bending test specimens where the V notch shape was as follows: depth = 1.2 mm, notch angle = 20°, and notch root radius = 0.04 mm.

For measurement of contact strength, Non-SP, SP and SP + heat-treated specimens were prepared. To prepare SP + heat-treated specimens, the SP specimens were heat-treated in air for 1 h at 900–1300 °C. The contact strength was measured by sphere indentation tests, as shown in Fig. 1. Indentations were made on the surface of the specimens using tungsten carbide (WC) spheres with a diameter of 4 mm, to prescribed loads at a crosshead speed of 0.2 kN/min. After unloading the loads, the

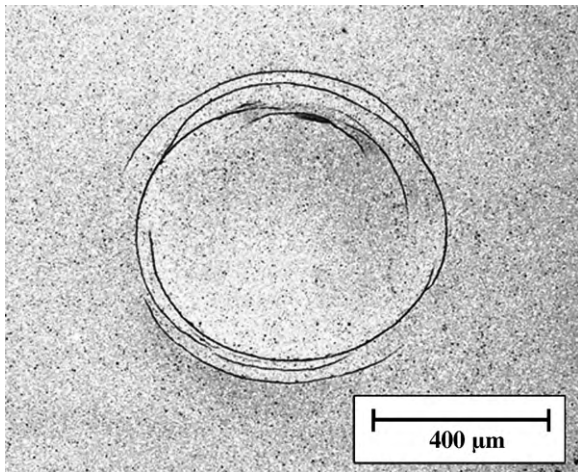


Fig. 1. Schematic illustration and photograph of ring cracks on the surface of Non-SP specimen.

indented surfaces were observed by optical microscopy to identify a crack initiation. Three specimens were used for indentation tests. We compared the critical load  $P_c$  where the crack initiation did not occur in all three specimens.

For measurement of bending strength, Non-SP, SP and SP+heat-treated specimens were prepared. A Vickers indentation was made on the center of the polished face of the Non-SP, SP and SP+heat-treated specimens at loads of 19.6 to 98.0 N to simulate the surface cracks introduced by shot peening or other contact events. These specimens are called “Non-SP+cracked”, “SP+cracked” and “SP+heat-treated+cracked” specimens, respectively. Some SP+cracked specimens and Non-SP+cracked specimens were heat-treated in air at 1100 °C for 5 h to investigate the effects of shot peening on the crack-healing process. These specimens are called “SP+cracked+heat-treated” and “Non-SP+cracked+heat-treated” specimens. The specimens were subsequently tested in three-point bending at a crosshead speed of 0.5 mm/min, using a fixture with a span of 16 mm.

### 3. Experimental results and discussion

#### 3.1. Residual stress

Fig. 2 shows the residual stress distributions induced by SP. The maximum compressive residual stress of 880 MPa was observed at the surface. The compressive residual stress decreased in-depth direction. The compressive residual stress was induced approximately up to 40 μm.

Fig. 3 shows residual stress at the surface as a function of heat-treatment temperature. Compressive residual stress decreased as the heat-treatment temperature was increased. However, the specimen heat-treated at 800–1100 °C showed compressive residual stresses of 300–400 MPa. In  $\text{Si}_3\text{N}_4/\text{SiC}$ , crack healing occurs above 800 °C. Thus, inducing compressive residual stress and crack healing were used simultaneously.

Compressive residual stress in the surface layer may reflect the volume expansion induced by micro-crack initiation within

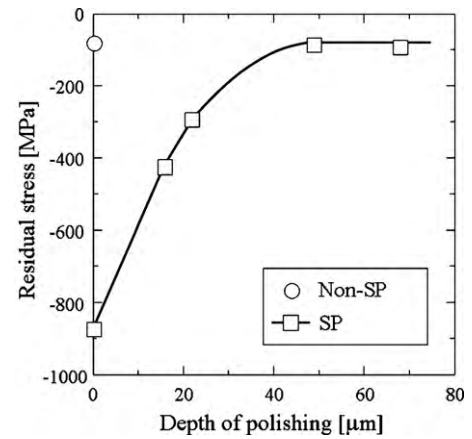


Fig. 2. Relationship between residual stress and depth from surface.

crystallites. The lattice strain was relieved due to thermal expansion as a result of increasing the heat-treatment temperature. Crack healing due to oxidation of SiC occurs above 800 °C. The oxidation of SiC includes approximately 80% volume expansion of the condensing phase.<sup>15</sup> The oxidation products fill the micro-cracks, which leads to a volume expansion at the surface. Thus, the relaxation of compressive residual stress was not pronounced at 800–1100 °C. On the other hand, the lattice strain was eliminated due to softening the grain boundary glassy phase above 1200 °C.

#### 3.2. Vickers indentation cracks and surface condition

Fig. 4 shows the optical micrograph of Vickers indentation and cracks for Non-SP and SP specimens. The Vickers indentation load was 98 N. Cracks were clearly observed in Non-SP specimens, as shown in Fig. 4(a), whereas the surface cracks were very short in SP specimens, as shown in Fig. 4(b). Table 2 shows the length of surface cracks for Non-SP+cracked, SP+cracked and SP+heat-treated+cracked specimens. The lengths of surface cracks in SP+cracked and SP+heat-treated+cracked specimens were much smaller than that of SP+cracked specimens. It is clear that the compressive residual stress increases the resistance to crack initiation at sub-surface.

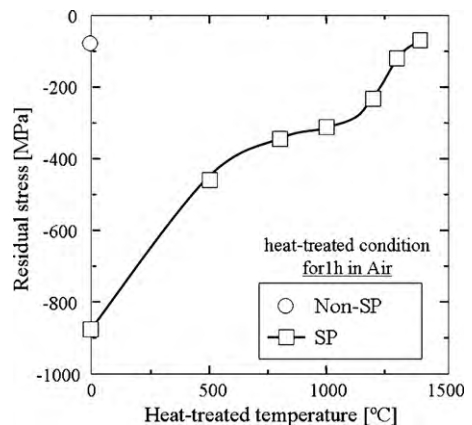


Fig. 3. Residual stress at the surface as a function of heat-treatment temperature.



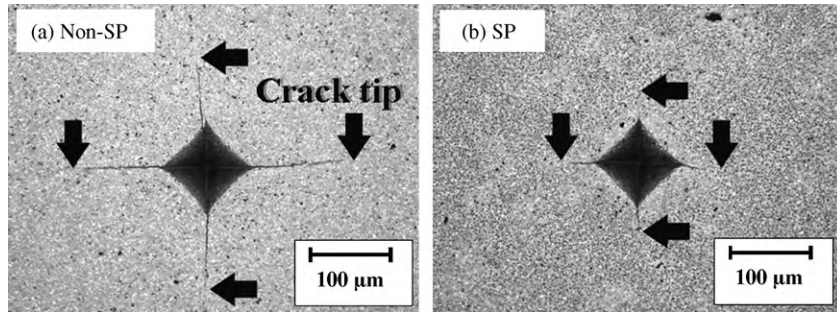


Fig. 4. Optical photograph of Vickers indentation and cracks (indented load: 98 N): (a) Non-SP specimen and (b) SP specimen.

Table 2  
Length of surface cracks for Non-SP and SP specimens.

Vickers indentation load [N]	Length of the surface cracks 2c [μm]		
	Non-SP + cracked specimens	SP + cracked specimens	SP + heat-treated + cracked specimens
19.6	107.4	63.5	87.0
49.0	188.4	106.8	117.1
98.0	300.1	156.7	179.6

The maximum height of roughness for Non-SP specimens and SP specimens was  $R_y = 0.24 \mu\text{m}$  and  $0.30 \mu\text{m}$ , respectively. Thus, the surface condition was not changed greatly by the shot peening.

3.3. Apparent fracture resistance

Fig. 5 shows the apparent fracture resistance ( $K_C$ ) at sub-surface measured by the IF method. The average value of fracture toughness ( $K_{IC}$ ) for Non-SP specimens measured by the SEVNB method was  $4.9 \text{ MPa m}^{1/2}$ . Thus, the value of  $K_C$  of the Non-SP specimens measured by the IF method ( $K_C = 4.1 \text{ MPa m}^{1/2}$ ) is close to the  $K_{IC}$  of the material. The  $K_C$  of the SP specimens increased 2.3-fold in contrast to the  $K_C$  for the Non-SP

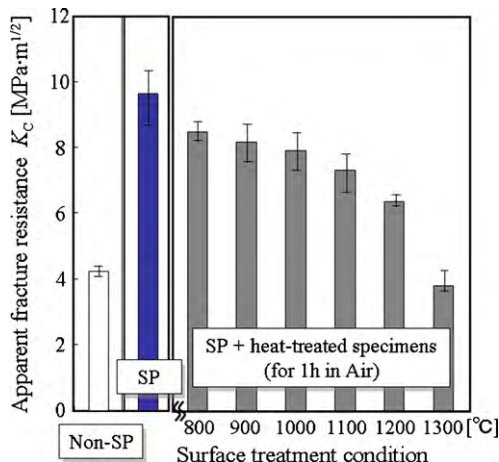


Fig. 5. Relationship between surface treatment conditions and apparent fracture resistance.

specimens. After heat treatment, the  $K_C$  for the SP specimens decreased. However, if the heat-treatment temperature was  $800\text{--}1100 \text{ }^\circ\text{C}$ , at which crack healing is possible, the  $K_C$  after heat treatment was more than 2 times as great. If the healing temperature was  $1300 \text{ }^\circ\text{C}$ , the value of  $K_C$  was the same for the SP and Non-SP specimens. Thus, increasing the  $K_C$  is closely related to compressive residual stress.

3.4. Contact strength

Fig. 6 shows the results of sphere indentation tests. The open circles indicate that no ring crack was observed on the surface of specimens, whereas the solid triangles indicate that a ring crack was observed. The critical loads  $P_C$  for crack initiation are shown by left-pointing arrows. The  $P_C$  for Non-SP specimens was  $0.5 \text{ kN}$ , whereas the  $P_C$  for SP specimens was more than  $4.5 \text{ kN}$ , which was the maximum load of the testing system. Thus, the  $P_C$  increased more than 9 times by shot peening. The  $P_C$  for the SP + heat-treated specimens decreased with increasing heat-treatment temperature. The tendency of decreasing  $P_C$  with increasing heat-treatment temperature is similar to that of  $K_C$ . This is because both  $K_C$  and  $P_C$  are closely related to the compressive residual stress. We noted that the  $P_C$  of SP + heat-treated specimens was 4 times greater than that of the Non-SP specimens if the heat-treatment temperature was  $1100 \text{ }^\circ\text{C}$ , at which crack healing is possible.

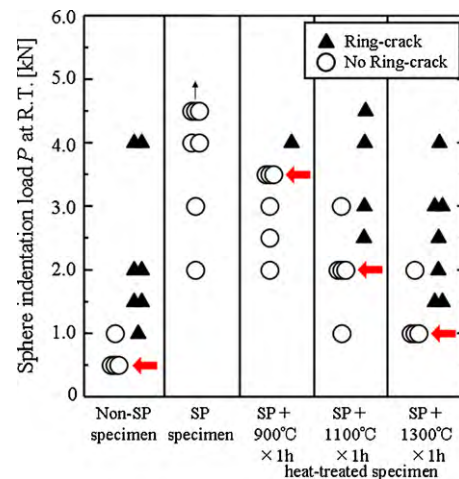


Fig. 6. Relationship between surface treatment conditions and sphere indentation load.

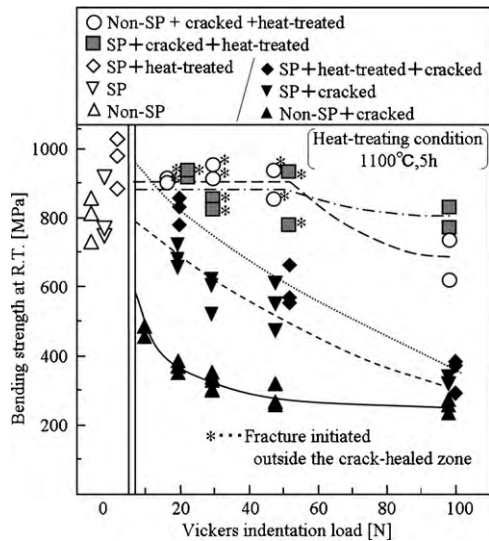


Fig. 7. Relationship between indentation load and bending strength.

### 3.5. Bending strength

Fig. 7 shows the bending strength as a function of Vickers indentation load. The bending strength of Non-SP + cracked specimens (solid triangles) decreased steeply with increasing Vickers indentation loads, although the decrease rates of the bending strength of SP + cracked specimens (solid inverse triangles) and SP + heat-treated + cracked specimens (solid diamonds) were smaller than that of Non-SP + cracked specimens. The main reason for this is that the lengths of surface cracks in SP + cracked specimens and SP + heat-treated + cracked specimens were much shorter than that of Non-SP + cracked specimens, as shown in Table 2. We note that the bending strengths of SP + heat-treated + cracked specimens were higher than those of SP + cracked specimens. Similarly, the bending strength of SP + heat-treated specimens (open triangle) was higher than that of the SP specimens (open inverse triangles). As mentioned already, the compressive residual stress decreased due to heat treatment, as shown in Fig. 3. Thus, the increase of bending strength was due to healing of small surface cracks produced by the shot-peening process. These results indicate that the shot peening in combination with crack healing increased the strength at the sub-surface of the test material.

If the indentation load was less than 49 N, the bending strength of SP + cracked + heat-treated specimens (solid squares) and Non-SP + cracked + heat-treated specimens (open circles) had recovered, and these specimens showed bending strength similar to each other and close to the strength of the specimens with no indentations. Most of the specimens fractured outside the crack-healed zone. As the indentation load was increased to 98 N, the bending strengths of SP + cracked + heat-treated specimens were higher than those of the Non-SP + cracked + heat-treated specimens. The difference of bending strength was caused by the difference of the crack length, as shown in Table 2. Thus, the effect of the shot peening on the crack-healing process increased as the indentation load increased.

## 4. Conclusion

We investigated the effects of shot peening and crack healing on residual stress, apparent fracture resistance, contact strength and bending strength of  $\text{Si}_3\text{N}_4/\text{SiC}$  composite. The results are summarized as follows:

- (1) The maximum compressive residual stress of 880 MPa was observed at the surface. The compressive residual stress was induced to approximately 40  $\mu\text{m}$  in-depth.
- (2) The apparent fracture resistance at the sub-surface was improved by a shot peening factor of 2.3.
- (3) The contact strength of  $\text{Si}_3\text{N}_4/\text{SiC}$  was increased more than 9 times by shot peening.
- (4) After heat treatment, the apparent fracture resistance and contact strength of SP specimens decreased. However, if the heat-treatment temperature was 800–1100  $^\circ\text{C}$ , at which crack healing is possible, the apparent fracture resistance and contact strength showed very high values.
- (5) The decrease rates of the bending strength against Vickers indentation loads of SP + cracked specimens and SP + heat-treated + cracked specimens were smaller than that of Non-SP + cracked specimens. The reason for this is that the surface cracks induced in SP + cracked and SP + heat-treated + cracked specimens were much shorter than those of Non-SP + cracked specimens.
- (6) The bending strengths of SP + heat-treated + cracked specimens were higher than those of SP + cracked specimens. Similarly, the bending strength of SP + heat-treated specimens was higher than that of the SP specimens. The increase of bending strength was due to healing of small surface cracks produced by the shot-peening process.
- (7) As the indentation load increased to 98 N, the bending strengths of SP + cracked + heat-treated specimens were higher than those of the Non-SP + cracked + heat-treated specimens. Thus, the effect of the shot peening on the crack-healing process increased as the indentation load increased.
- (8) Considering the experimental results of (1)–(7), we conclude that shot peening in combination with crack healing is a useful technique to improve the strength and reliability of ceramics.

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