Improvement of the Contact Strength of $\text{Si}_3\text{N}_4/\text{SiC}$ by a Combination of Shot Peening and Crack-Healing*

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Abstract
The combination effects of shot peening (SP) and crack-healing on the contact strength of ceramics were investigated. $\text{Si}_3\text{N}_4/\text{SiC}$ composite ceramics with high crack-healing ability were subjected to SP using zirconium oxide shots with several peening pressures and shot diameters. Then, some of the specimens subjected to SP were heat-treated in air to heal the surface cracks induced by SP. The residual stress, the apparent fracture toughness and the Weibull distribution of the contact strength were investigated. As a result, it was found that the combination of SP and crack-healing is effective for increasing the contact strength and decreasing the scatter of the contact strength.

Key words: Ceramics, Fracture Toughness, Residual Stress, Shot Peening, Crack-Healing, Contact Strength

1. Introduction
Structural ceramics have excellent wear resistance and high temperature strength. Thus, structural ceramics are expected to be a good candidate material for bearings, gas turbines and cutting tools. However, the fracture toughness of a ceramic is fairly low compared with that of a metallic material, and thus the mechanical properties of ceramics tend to be less reliable. Therefore, if the brittleness of ceramics could be overcome, the reliability and product life of ceramic components would be improved.

Shot peening (SP) is a common procedure used to increase the strength of metals. The compressive residual stress generated by SP prevents fatigue crack propagation and stress corrosion cracking. Recent studies have shown that the near-surface strength of ceramics can be improved by SP. Pfeiffer and Frey carried out SP on alumina and silicon nitride ($\text{Si}_3\text{N}_4$) by using 0.61 to 0.69 mm tungsten carbide shots and reported that high compressive residual stress of up to more than 1 GPa could be introduced near the surface. Tanaka et al. carried out SP on $\text{Si}_3\text{N}_4$ using 0.05 mm high-strength steel shots and 1.1 mm tungsten carbide shots. They reported that compressive residual stress of up to 1.5 GPa was introduced near the surface, and the compressive residual stress increased the apparent fracture toughness at the sub-surface up to 2.5 fold. Considering the results obtained in previous studies, the SP 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 SP process, the
reliability of the ceramics components will be decreased.

Some structural ceramics have a crack-healing ability.\(^{(3)-(6)}\) If crack-healing is used in combination with SP, the surface strength and reliability of ceramics can be increased. Takahashi et al.\(^{(7)}\) investigated the effects of SP and crack-healing on the contact strength of an Si\(_3\)N\(_4\)/SiC composite. They carried out SP on the Si\(_3\)N\(_4\)/SiC using 300 \(\mu\)m zirconium oxide (ZrO\(_2\)) shots and reported that the contact strength of the Si\(_3\)N\(_4\)/SiC was increased by SP.\(^{(7)}\) However, the contact strength of the SP specimens decreased after crack-healing because the compressive residual stress decreased after crack-healing. It is presumed that the contact strength of ceramics subjected to SP has considerable scatter depending on the size of the cracks induced by SP. Thus, it is necessary to investigate the distribution of the contact strength of ceramics subjected to SP. However, no such analysis has been performed. Also, the combination effects of SP and crack-healing on the distribution of the contact strengths have not been investigated. In this study, the effects of shot peening (SP) and crack-healing on the residual stress, the apparent fracture toughness and the Weibull distribution of contact strength were investigated on Si\(_3\)N\(_4\)/SiC composite.

2. Test procedure

2.1 Materials and specimens

In this study, silicon nitride reinforced by silicon carbide (Si\(_3\)N\(_4\)/SiC) was used. This material was selected as a test material because it has excellent crack-healing ability.\(^{(5)}\) The Si\(_3\)N\(_4\) powder used in this study has a mean particle size of 0.2 \(\mu\)m, while the SiC powder used has a mean particle size of 0.27 \(\mu\)m. The samples were prepared using a mixture of Si\(_3\)N\(_4\) with 20 wt% SiC powder and 8 wt% Y\(_2\)O\(_3\) as a sintering additive powder. Alcohol was then added to the solution, 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. The mixture was subsequently hot-pressed at 1850°C and 35 MPa for 2 h in an N\(_2\) atmosphere. The hot-pressed plate was then cut into test specimens measuring 3×4×40 mm. The specimens were polished to a mirror finish on one face.

2.2 Shot peening, residual stress and surface roughness

Shot peening (SP) was carried out on the Si\(_3\)N\(_4\)/SiC specimens with a direct pressure peening system. Commercial zirconium oxide (ZrO\(_2\)) shots with a diameter of 300 and 500 \(\mu\)m were used. The Vickers hardness of the ZrO\(_2\) balls was 1250HV. The peening pressures selected were 0.1, 0.2 and 0.3 MPa. The three specimens were shot peened simultaneously for 40 s. The peening coverage was 300%. The specimens subjected to SP are referred to here as “SP” specimens, and the specimens without SP are referred to as “non-SP” specimens. After SP, some specimens were heat-treated in air at 1100°C for 5 h. This heat-treatment condition was selected based on the results of a previous study.\(^{(7)}\) Surface cracks introduced by SP can be healed while maintaining the compressive residual stress introduced by SP. These specimens are called “SP + crack-healed” specimens. The residual stresses on the surface were measured for non-SP, SP and SP + crack-healed specimens by the X-ray diffraction method. The conditions for X-ray diffraction are shown in Table 1. The surface roughness was measured at five points on each specimen using a stylus-type surface roughness tester.

<table>
<thead>
<tr>
<th>Characteristic X ray</th>
<th>Cu-Ka</th>
</tr>
</thead>
<tbody>
<tr>
<td>X ray tube</td>
<td>Cu</td>
</tr>
<tr>
<td>Diffraction plane</td>
<td>Si(_3)N(_4) (323)</td>
</tr>
<tr>
<td>Diffraction angle [deg]</td>
<td>141.26</td>
</tr>
<tr>
<td>Tube voltage [kV]</td>
<td>40</td>
</tr>
<tr>
<td>Tube current [mA]</td>
<td>30</td>
</tr>
</tbody>
</table>
2.3 Measurement of apparent fracture toughness

For the measurement of apparent fracture toughness \( (K_c) \) at the sub-surface, non-SP specimens, SP specimens and SP + crack-healed specimens were prepared. The \( K_c \) was evaluated by the indentation-fracture (IF) method, in which \( K_c \) can be estimated using the following equation in accordance with Japan Industry Standards (JIS) \(^{(8)}\):

\[
K_c = 0.026E^{1/2}P^{1/2}\frac{a}{c^{3/2}},
\]

where \( E \) is Young’s modulus, \( P \) is the indent load, \( a \) is half the diagonal length of the 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 \( E=300 \) GPa, as determined by the strain measurements in the bending test.

2.4 Measurement of contact strength

For the measurement of contact strength, "non-SP," "SP," "non-SP + crack-healed" and "SP + crack-healed" specimens were prepared. The contact strength was measured by sphere indentation tests. Figure 1 shows the testing system. \(^{(9)}\) Indentations were made on the surfaces of the specimens using tungsten carbide (WC) spheres with a diameter of 4 mm. The sphere indentation tests were carried out at a crosshead speed of 0.2 kN/min using a universal testing machine. The ring crack initiation load \( (P_{\text{max}}) \) was determined using acoustic emission (AE). At the load at which AE detected the crack initiation, the loading was interrupted. After removing the loads, the indented surfaces were observed using optical microscopy to identify crack initiation.

In this study, the ring crack initiation loads \( (P_{\text{max}}) \) were statistically analyzed by a two-parameter Weibull function. It was assumed that the \( P_{\text{max}} \) for each specimen follow the two-parameter Weibull distribution to evaluate the \( P_{\text{max}} \) with the same measure. In these analyses, the failure probability, \( F \), was calculated using the median rank estimate. These results were plotted using the two-parameter Weibull function, expressed by the following equation:

\[
F(P_{\text{max}}) = 1 - \exp \left\{- \left( \frac{P_{\text{max}}}{\beta} \right)^{\alpha} \right\},
\]

where \( P_{\text{max}} \) is the ring crack initiation load, \( \beta \) is the scale parameter, and \( \alpha \) is the shape parameter. The scale parameter \( \beta \) describes the crack initiation load when \( F(P_{\text{max}}) = 63.2\% \). The shape parameter \( \alpha \) describes the width of the distribution of the crack initiation load.
3. Results and discussion

3.1 Residual stress

Figure 3 shows the residual stress at the surfaces of the specimens. The diameter of the shot did not have a major impact on the compressive residual stress on the surfaces. However, the compressive residual stress increased as the peening pressure increased. Compressive residual stress on the surface layer may reflect the volume expansion induced by micro-crack initiation within crystallites. It is assumed that the number of micro cracks increased with increasing peening pressure. Thus, the compressive residual stress may also increase as the peening pressure is increased.

Figure 4 shows the residual stress distributions for the SP and SP + crack-healed specimens. The shot diameter was 300 µm and the peening pressure was 0.2 MPa. To investigate the in-depth residual stress distribution, the surface layers were removed by polishing the specimens with diamond abrasives of 9.0, 3.0 and 0.5 µm. The residual stress induced by polishing was also investigated by measuring the residual stress after polishing. A maximum compressive residual stress of 500 MPa was observed on the surface in this SP specimen. The compressive residual stress decreased in the depth direction. Compressive residual stress of up to approximately 40 µm was generated. In the SP + crack-healed specimen, the compressive residual stress decreased. However, the SP + crack-healed specimen showed compressive residual stresses of 330 MPa. Thus, the induction of compressive residual stress and crack-healing occurred simultaneously. The compressive residual stresses at about 40 µm were slightly larger than that of the non-SP specimens. This is because the compressive residual stress induced by polishing remained on the surface.

The lattice strain was relieved due to thermal expansion as a result of heat treatment. However, crack-healing due to oxidation of SiC occurs above 800°C. The oxidation of SiC includes approximately 80% volume expansion of the condensing phase. 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 after crack-healing.

Fig. 2. A ring crack after the sphere indentation test.

Fig. 3. Residual stress at the surface of each specimen.
3.2 Surface roughness

Table 2 shows the average value of the surface roughness for each specimen. It can be seen that the surface roughness after SP increased as the shot diameter increased. However, the rate of increase was quite small. Thus, it can be said that SP can be used in ceramic components. The surface roughness after heat-treatment increased. This is because the surface layer was oxidized by heat-treatment in air.

<table>
<thead>
<tr>
<th>Specimens</th>
<th>Maximum height roughness Rz [µm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Non-SP</td>
<td>0.287</td>
</tr>
<tr>
<td>300µm,0.1MPa-SP</td>
<td>0.296</td>
</tr>
<tr>
<td>300µm,0.2MPa-SP</td>
<td>0.324</td>
</tr>
<tr>
<td>300µm,0.3MPa-SP</td>
<td>0.336</td>
</tr>
<tr>
<td>500µm,0.1MPa-SP</td>
<td>0.307</td>
</tr>
<tr>
<td>500µm,0.2MPa-SP</td>
<td>0.489</td>
</tr>
<tr>
<td>500µm,0.3MPa-SP</td>
<td>0.432</td>
</tr>
<tr>
<td>Non-SP + crack-healed</td>
<td>0.607</td>
</tr>
<tr>
<td>300µm,0.2MPa-SP + crack-healed</td>
<td>0.587</td>
</tr>
</tbody>
</table>

3.3 Apparent fracture toughness

Figure 5 shows the apparent fracture toughness ($K_C$) at the sub-surface measured by the IF method. The $K_C$ of the SP specimens increased 33-93% for 300 µm-SP specimens and 19-79% for 500 µm-SP specimens in contrast with the $K_C$ for the non-SP specimens. The diameter of shot did not have a pronounced effect on the $K_C$. The $K_C$ increased as the peening pressure increased. As mentioned in Section 3.1, the compressive residual stress also increased as the peening pressure increased. However, the values of $K_C$ for the 0.3MPa-SP specimens were not as large as expected based on the values of compressive residual stresses shown in Fig. 3. Tanaka et al. indicated that the value of $K_C$ increased almost linearly with the magnitude of compressive residual stress at the surface when the compressive residual stress was smaller than about 900 MPa. However, the value of $K_C$ became saturated when the compressive residual stress reached 1000 MPa. Similar experimental results were observed in this study.
After heat treatment, the $K_C$ for the SP specimens decreased from 8.1 MPa$m^{-1/2}$ to 6.6 MPa$m^{-1/2}$. However, the $K_C$ after heat treatment was 57% higher than that of the non-SP specimen. Thus, the combination of SP and crack-healing is effective for increasing the $K_c$.

Fig. 5 Apparent fracture toughness of each surface-treated specimen.

### 3.4 Effects of peening pressure on contact strength

Figure 6 shows the effect of peening pressure on the Weibull distribution of the $P_{\text{max}}$. The shot diameter was 300 µm, and the peening pressures were 0.1, 0.2 and 0.3 MPa. The test results of non-SP specimens are also shown for comparison. The shape parameter and scale parameter of the $P_{\text{max}}$ obtained from the Weibull distribution are summarized in Table 3. In the non-SP specimens, the Weibull distribution of $P_{\text{max}}$ showed good linearity. However, in the SP specimens, the Weibull distribution of $P_{\text{max}}$ did not show good linearity. The possible reasons for the non-linearity of the Weibull distribution in the SP specimens are considered as follows.

In the lower fracture probability region, the values of $P_{\text{max}}$ in the SP specimens were smaller than those of the non-SP specimens. This is because the surface cracks induced by SP existed near the sphere indentation site. The $P_{\text{max}}$ depends on the size and distribution of the cracks induced by SP. If a large crack existed near the sphere indentation site, the $P_{\text{max}}$ tended to be decreased.

Fig. 6 Effects of peening pressure on the Weibull distribution of crack initiation load.
In higher fracture probability regions, the values of $P_{\text{max}}$ in the SP specimens were larger than those in the non-SP specimens. It was presumed that the size of the surface cracks near the sphere indentation site was small. Thus, the crack initiation load increased because of the effect of the compressive residual stress.

As mentioned in Section 3.3, the $K_C$ increased as increasing peening pressure from 0.1 to 0.3 MPa. However, the specimen with a peening pressure of 0.2 MPa (0.2 MPa-SP specimens) showed the highest $P_{\text{max}}$ among the SP specimens. Figure 7 shows the surface of the 300 µm, 0.2 MPa-SP and 300 µm, 0.3 MPa-SP specimens observed by optical microscope. It is clear that the size of cracks introduced in 0.3 MPa-SP specimens were larger than that in the 0.2 MPa-SP specimens. Thus, the 0.2 MPa-SP specimens showed the highest $P_{\text{max}}$. In the following examination, a peening pressure of 0.2 MPa was used.

![Image](a) 300 µm, 0.2 MPa - SP specimen  (b) 300 µm, 0.3 MPa - SP specimen

Fig. 7 Surface cracks induced by shot peening.

<table>
<thead>
<tr>
<th>Specimens</th>
<th>Shape parameter $\alpha$</th>
<th>Scale parameter $\beta$ [kN]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Non-SP</td>
<td>7.99</td>
<td>0.90</td>
</tr>
<tr>
<td>300µm,0.1MPa-SP</td>
<td>1.29</td>
<td>0.92</td>
</tr>
<tr>
<td>300µm,0.2MPa-SP</td>
<td>0.97</td>
<td>2.42</td>
</tr>
<tr>
<td>300µm,0.3MPa-SP</td>
<td>1.22</td>
<td>1.30</td>
</tr>
<tr>
<td>500µm,0.2MPa-SP</td>
<td>0.92</td>
<td>2.01</td>
</tr>
<tr>
<td>Non-SP + crack-healed</td>
<td>6.98</td>
<td>1.48</td>
</tr>
</tbody>
</table>
| 300µm,0.2MPa-SP + crack-healed | 3.19 | 2.26

3.5 Effects of shot diameter on contact strength

Figure 8 shows the effect of shot diameter on the Weibull distribution of the crack initiation load. The shot diameters were 300 and 500 µm and the peening pressure was 0.2 MPa. The test results for the non-SP specimens are also shown for comparison. It can be seen that the Weibull distributions of the $P_{\text{max}}$ in both SP specimens were similar. Thus, the shot diameter did not affect the $P_{\text{max}}$ very much. This is because the diameter of the shot did not notably affect the $K_C$, as mentioned in Section 3.2.

The surface roughness after SP increased as the shot diameter increased (see Table 2). From the viewpoint of surface roughness, a shot diameter of 300 µm was preferable. In the following examination, a shot diameter of 300 µm was used.

3.6 Effects of shot peening and crack-healing on contact strength

Figure 9 shows the effects of shot peening and crack-healing on the Weibull distribution of the $P_{\text{max}}$. The shot diameter was 300 µm and the peening pressure was 0.2 MPa. The test results of the non-SP and the non-SP + crack-healed specimens are also shown for comparison. It is clear that the $P_{\text{max}}$ of the non-SP + crack-healed specimens increased in contrast with the non-SP specimens. This is because the minute surface cracks introduced
by machining of the test specimens were healed. (9)

In the lower fracture probability region, the values of $P_{\text{max}}$ in the SP + crack-healed specimens were higher than those in the SP specimens. This is because the cracks induced by SP were healed by crack-healing.

In the higher fracture probability region, the values of $P_{\text{max}}$ in the SP + crack-healed specimens were larger than those in the non-SP and non-SP + crack-healed specimens. This is because the minute surface cracks introduced by machining of the test specimens were healed and compressive residual stress of 330 MPa remained on the specimen surface. However, the $P_{\text{max}}$ in the SP + crack-healed specimens was slightly lower than that in the SP specimens. This is because the compressive residual stress decreased due to the heat treatment, as discussed in Section 3.1.

It is clear that the SP + crack-healed specimens showed higher $P_{\text{max}}$ than did the non-SP and non-SP + crack-healed specimens. It is also clear that the SP + crack-healed specimens showed a smaller scatter of $P_{\text{max}}$ than the SP specimens. Thus, SP in combination with crack-healing is a useful technique to improve the contact strength of ceramics.

Fig. 8 Effects of shot diameter on the Weibull distribution of crack initiation load.

Fig. 9 Effects of the shot peening and crack-healing on the Weibull distribution of crack initiation load.
4. Conclusions

The effects of shot peening (SP) and crack-healing on the residual stress, the apparent fracture toughness and the Weibull distribution of contact strength were investigated on an Si$_3$N$_4$/SiC composite. The results are summarized as follows:

1. Compressive residual stresses from 292 MPa to 942 MPa were observed at the surface of SP specimens. The compressive residual stress increased as the peening pressure increased. The diameter of the shot did not notably affect the compressive residual stress at the surfaces.

2. The apparent fracture toughness ($K_C$) at the sub-surface of the SP specimens increased 33 - 93% for 300 $\mu$m-SP specimens and 19 - 79% for 500 $\mu$m-SP specimens in comparison with the $K_C$ for the non-SP specimens. The values of $K_C$ were affected by the peening pressures rather than the diameter of the shot.

3. After crack-healing of the SP specimens, the compressive residual stress and the $K_C$ decreased in contrast with the SP specimens. However, the $K_C$ after the heat treatment was 57% higher than that of the non-SP specimen. Thus, the induction of compressive residual stress and crack-healing occurred simultaneously.

4. The specimens subjected to SP with a peening pressure of 0.2 MPa and a shot diameter of 300 $\mu$m (300 $\mu$m, 0.2MPa-SP specimens) showed the highest crack initiation load ($P_{max}$) among the SP specimens. However, the scatters of the $P_{max}$ in SP specimens were larger than those of the non-SP specimen. This is because cracks induced during the SP process existed near the specimen surface.

5. The scatter of the $P_{max}$ in the SP specimens could be improved by crack-healing in air at 1100°C for 5 h. This is because the surface cracks induced by SP were healed by the crack-healing.

6. Considering experimental results (1) to (5), it is concluded that SP in combination with crack-healing is a useful technique to improve the contact strength of ceramics.

Acknowledgement

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References


(8) Japan Industrial Standards R1607, Testing Method for Fracture Toughness of Fne