O U R N A L O F

Ceramic Processing Research

Characteristics of crack growth of zirconia composites by shot peening

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The effect of shot peening on the crack propagation and bending strength of zirconia (ZrO_2) composites was investigated. Shot peening was carried out on ZrO_2 composites using Yttrium stabilized ZrO_2 beads. It was found that shot peening could introduce compressive residual stress to the surface of ZrO_2 composites. In Z, ZS, and ZST specimens, the shot peening treatment suppressed crack growth caused by the micro-crack introduced to the surface and by the residual stress, while the bending strength of the cracked specimen was improved.

Key words: ZrO₂ composites, Shot peening, Compressive residual stress, Crack growth.

Introduction

Ceramic has excellent properties, including heat resistance, corrosion resistance, and wear resistance. However, ceramics have much lower fracture toughness than metal materials, and have high crack-sensitivity. Therefore, machining of ceramics is difficult, while machining costs are high and reliability decreases. Research for developing ceramic material with crack healing ability is being conducted to overcome this problem. If ZrO₂ ceramics have a crack-healing ability, the implant and the artificial bone can be expected reduction of machining cost. Many researchers who study crack-healing have used the method of the oxidation of the material, in which the crack-healing temperature of $1273 \sim 1573$ K in the atmosphere is required. [1-3] ZrO₂/SiC composites developed by various researchers was found to have a crack-healing ability at a low temperature of about 1073 K.[4] If this low temperature healing material was to be commercialized, the cost of facilities for machining would be reduced. In addition, since low temperature healing is expected to suppress the dimensional change caused by oxidation, as well as the changes of surface shape and the high-temperature corrosion, etc., it is considered to be an extremely advantageous industrial technology.

On the other hand, because the use of shot peening for ceramics causes compressive residual stress, shot peening is considered to be an effective method to suppress crack propagation under a constant stress. Shot peening is a surface modification method used to introduce compressive residual stress and is widely used in the fabrication of metallic materials. Recently, the possibility of introducing a compressive residual stress to the surface of ceramic material was presented. [5,7] In addition, the effect of improving the fracture toughness and introducing compressive residual stress by shot peening for Si_3N_4 /SiC composites was reported [8].

This study discusses observations of the crack growth and the shot peening effect by loading a constant stress on ZrO_2 composites material.

Materials and Experimental Method

A powder composed of $0.026 \,\mu\text{m}$ ZrO₂(TZ-3Y-E, Tosoh), $0.27 \,\mu\text{m}$ SiC (Wako pure chemical industries), and a sintering additive (commercially purchased anatase $0.3 \,\mu\text{m}$ TiO₂) was used for the experiments. SiC was added to give crack healing characteristics. 3 wt.% TiO₂ was added for superior strength recovery by crack healing. [4] Circular plates of $\phi 60 \times 5 \,\text{mm}$ were sintered in a vacuum atmosphere for 1 h via hot pressing under 30 MPa at 1,723 K. The batch compositions of specimens are given in Table 1.

For shot peening (SP), a shot peening apparatus of a direct pressure system was used (FDQ type, Fuji Seisakusho, Japan). The shots were ZrO2 balls (Nikkato Ltd, YTZ® ball) of φ 180 and φ 300 µm with a Vickers hardness of 1,250 Hv. The shot projection nozzle diameter was φ 5 mm and the projection distance was 100 mm. The pressure of the projected shot was set to 0.2 MPa and the projection time was 30 s. Table 2 shows the conditions of the SP treatment. The shot peened specimens using shots of φ 180 µm and φ 300 µm are named the SP180 specimen and SP300 specimen, respectively.

The surface roughness was measured using a contact stylus roughness measuring instrument (Kosaka Laboratory Ltd, SE1200), with a measurement method based on JIS B0601. The measurement length is 160 μ m (cut off 0.80 mm) and the measurement speed was set at 0.30 mm/sec. The hardness and crack growth

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Table 1. Batch composition and processing conditions.

Specimen	Database approximation	Conditions			
	(wt.%)	Hot	Crack		
		pressing	healing		
Z	100 wt.% ZrO2				
ZS	90 wt.% ZrO_2	30 MPa,	1073 K and		
2.5	10 wt.% SiC	1723 K, 1 hour	1173 K from 1 to 10 hour		
787	$8/ \text{ wt.} \% \text{ ZrO}_2$	in vacuum	in air		
231	$3 \text{ wt.}\% \text{ TiO}_2$				
Table 2. Shot	peening condition.				
Sho	t system	Direct pressure system			
Shot	t material	ZrO ₂ (1250 Hv)			
Shot	diameter	φ180 μm, φ300 μm			
Shot	pressure	0.2 MPa			
Projec	tor distance	100 mm			
	Time	30 sec			
Co	overage	300%			
0	P Φ 5 pi 16		4 45°		

Fig. 1. Three point bending specimen and rough scheme of test system.

of the SP treated specimen (as-SP) surface was used for the Vickers hardness test. Vickers hardness was measured under a load of 490 N and the crack growth was observed under a load of $9.8 \sim 490$ N. Then, the SPtreated specimens in which crack growth was observed with loads of 98 N and 196 N using ZrO₂ shots of φ 180 and φ 300 µm were named the SP180 (98 N) specimen, SP180 (196 N) specimen, SP300 (98 N) specimen, and SP300 (196 N) specimen, respectively.

Bending tests were performed on a three-point loading system with a span of 16 mm and a cross head speed of 0.5 mm/minute. Fig. 1 shows the three-point bending specimen and a rough scheme of the test system.

Results and Discussion

Roughness of the SP treated surface

The roughness of the SP treated surface is shown in figures 2 and 3. Figs. 2(a), 2(c), and 2(e) show the Z, ZS, and ZST specimens by SP180, respectively, and Figs. 2(b), 2(d), and 2(f) show the Z, ZS, and ZST specimens by SP300, respectively. Using an optical microscope, the surface was observed to be uneven.



Fig. 2. Appearance of the surface by shot peening. (a) Z specimen by SP180, (b) Z specimen by SP300, (c) ZS specimen by SP180, (d) ZS specimen by SP300, (e) ZST specimen by SP180, (f) ZST specimen by SP300.



Fig. 3. Surface roughness of Z, ZS and ZST specimen according to shot peening.

Fig. 3 shows the roughness of the Z, ZS, and ZST specimens. The roughness of the SP180 and SP300 specimens was greater than that of the as-received specimen. The roughness of the SP180 specimen was also slightly greater than that of the SP300 specimen.

Compressive residual stress before and after SP treatment

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In order to determine the cause of the crack growth reduction after the SP treatment described in the previous section, compressive residual stress was measured. Fig. 4 shows the results. The compressive residual stress of the Z specimens was about -178 MPa for the as-received specimen, about -1940 MPa for the SP180 specimen, and about -494 MPa for the SP300 specimen. Compared to the as-received specimen, the compressive residual stress of the SP180 and SP300 specimens increased up to about 1000% and 176%, respectively. Therefore, the compressive residual stress of the SP treatment, and the compressive residual stress by SP180 treatment improved more than that by SP300 treatment.

The compressive residual stress of the ZS specimens was about -298 MPa for the as-received specimen, about -1988 MPa for the SP180 specimen, and about -232 MPa for the SP300 specimen. Compared to the asreceived specimen, the compressive residual stress of the SP180 specimen was increased by about 566%, but that for the SP300 specimen was decreased by about -22%. Therefore, for the ZS specimen, the compressive residual stress was improved by the SP180 treatment, but the SP300 treatment showed no effect on the reduction of compressive residual stress.

The compressive residual stress of the ZST specimens was about -28 MPa for the as-received specimen, about -1525 MPa for the SP180 specimen, and about -25 MPa for the SP300 specimen. Compared to the as-received specimen, the compressive residual stress of the SP180 specimen was increased by about 5340%, but that of the SP300 specimen was decreased by about -11%. Therefore, for the ZST specimen, the compressive residual stress was improved more by the SP180 treatment than that for the Z and ZS specimens.

As such, for the zirconia composite ceramic, it was found that the compressive residual stress by SP treatment had a greater effect on the SP180 specimen than on the SP300 specimen. Therefore, the crack growth reduction is determined to be affected by the compressive residual stress introduced to the surface by SP treatment.



Fig. 4. Residual stress by shot peening.



Fig. 5. Vickers hardness of Z, ZS and ZST specimen according to shot peening.

Vickers hardness of the SP treated specimen

Vickers hardness was measured for 20 seconds under the indentation load of 490 kN. Fig. 5 shows the results of the Z, ZS, and ZST specimens. A white square symbol refers to the as-received specimen, a white circle symbol refers to the SP180 specimen, and a white triangle symbol refers to the SP300 specimen. The measured reasons by high indentation load are as following. As the sizes of the ZrO₂ shots used in the SP treatment are φ 180 and 300 μ m, the sizes of the surface traces of the shot are assumed to be a maximum diameter of 180 and 300 µm, respectively. When the surface was treated with a small indentation load, the Vickers indenter value was small. Therefore, the interior of the boundary of the shot traces was measured. In this case, the Vickers hardness due to dispersion will reduce the reliability. Therefore, the high indentation load will improve the reliability of the hardness. The mean hardness of the as-received Z, ZS, and ZST specimens (white square symbol) was close to 1289, 1334, and 1332 Hv, respectively. In the Z and ZS specimens, the mean hardness was 1344 and 1375 Hv, respectively, by SP180 (white open symbol), which was slightly higher than that of the as-received specimens. Meanwhile, the mean hardness of the Z and ZS specimens of 1210 and 1251 Hv, respectively, by SP300 (white triangle symbol) were reduced, respectively. However, the mean hardness of the ZST specimen by SP180 (white circle symbol) and SP300 (white triangle symbol) are 1240 and 1229 Hv, respectively. These were lower than the mean hardness (1332 Hv) of the as-received specimen (white square symbol).

Bending strength of as-received specimen after SP treatment

Fig. 6 shows the bending strength of the Z, ZS, and ZST specimens after the SP treatment. The shot peening was carried out on the as-received specimen. The white square symbol refers to the as-received



Fig. 6. Bending strength of Z, ZS and ZST specimens according to shot peening.



specimen, the white circle symbol refers to the SP180 specimen, and the white triangle symbol refers to the SP300 specimen. The asterisk symbol (*) shows fracture caused by porosity or inclusions in the specimens, which were excluded from the mean strength measurement.

The mean strengths of the Z specimen were 1659, 1384, and 1592 MPa, for the as-received Z, SP180, and SP300 specimens, respectively. The mean strengths of the SP180 and SP300 specimens decreased by about 17% and 4% compared to that of the as-received Z specimen, respectively. The mean strengths of the ZS specimen were 724, 1404, and 1243 MPa, for the as-received Z, SP180, and SP300 specimens, respectively. The mean strengths of the SP180 and SP300 specimens increased by about 94% and 72% compared to that of

Specimen Load(N)	Z			ZS			ZST		
	As- received	SP180	SP300	As- received	SP180	SP300	As- received	SP180	SP300
9.8	Х	Х	Х	0	Х	Х	0	Х	Х
19.6	0	х	х	О	х	х	0	х	0
29.4	Ο	х	х	О	х	х	0	х	Ο
49.0	Ο	х	Ο	О	х	0	0	х	Ο
98.0	Ο	х	О	О	х	О	Ο	Ο	О
196.0	Ο	0	О	О	Ο	О	Ο	Ο	Ο
294.0	Ο	Ο	О	О	О	О	Ο	Ο	Ο
490.0	0	0	0	0	0	0	0	0	0

x: Non crack propagation, O : Crack propagation.



Fig. 7. SEM photographs of indentation and crack according to Vickers load. (a) As-received Z specimen, (b) Z specimen by SP180, (c) Z specimen by SP300, (d) As-received ZS specimen, (e) ZS specimen by SP180, (f) ZS specimen by SP300, (g) As-received ZST specimen, (h) ZST specimen by SP180, (i) ZST specimen by SP300.

the as-received ZS specimen, respectively. The mean strength of the ZS specimen with a crack-healing ability by the addition of SiC differs from that of the Z specimens. That is, the mean strength of the SP180 specimen increased more than that of the SP300 specimen. On the other hand, the mean strengths of the ZST specimen were 1347, 1354, and 1091 MPa for the as-received Z, SP180, and SP300 specimens, respectively. The mean strength of the as-received ZST specimen with SiC and TiO₂ added was increased by about 86% compared to that of the as-received ZS specimen with added SiC. Meanwhile, the mean strength of the SP180 specimen of ZST was similar to that of the as-received specimen, but that of the SP300 specimen was reduced by approximately 19% compared to that of the as-received specimen.

For providing crack healing ability, the mean strength of the ZS specimen with added SiC increased two times more than that of the as-received specimen by the SP treatment. However, the mean strength of the as-received ZST specimen with added SiC and TiO₂ was increased more than that of the ZS specimen, but the mean strength of the SP180 and SP300 specimens was decreased.

Crack growth behavior

After the SP treatment, the crack growth and crack length were evaluated according to the Vickers indentation. The results are shown in Table 3 and Fig. 7.

When the crack propagation was introduced five times by the Vickers indentation of each load, Even if a crack was occurred once or even a little propagation, it has been judged that the crack was propagated. This is because the fracture of the ceramics is significantly affected, even by fine cracks.

In Table 3, x indicates that the crack did not propagate and O shows that the crack was propagated. Cracks propagated in the three types of as-received specimens, except for that of the Z specimen with a load of 9.8 N. The SP180 and SP300 specimens of the Z and ZS specimens showed better crack growth resistance than the ZST specimen. It was confirmed that, with the SP treatment, cracks do not occur with the Vickers indentation.

Fig. 7 shows the Vickers indentations and cracks according to the Vickers load using the Z, ZS, and ZST specimens. The crack lengths of the Z, ZS, and ZST specimens were as-received specimen > SP300 specimen > SP180 specimen.

Fig. 8 shows the crack length. The crack length of the as-received Z, ZS, and ZST specimens increased sharply compared to that of the SP180 and SP300 specimens. Meanwhile, the crack propagation length of the SP300 specimen was slightly greater than that of the SP180 specimen. It is judged that the micro-cracks and residual stress introduced to the surface by the SP treatment caused interference of the crack growth.



Fig. 8. Crack length according to Vickers load. (a) Z specimen, (b) ZS specimen, (c) ZST specimen.

Bending strength of as-cracked specimen after SP treatment

In the previous section, it was confirmed that, even with the Vickers indentation of high load after SP treatment, the crack does not propagate well. Fig. 9 shows the bending strength of the as-cracked specimen after SP treatment. In the figure, the asterisk symbol (*)



Fig. 9. Bending strength of cracked specimen according to Shot peening (a) Z specimen, (b) ZS specimen, (c) ZST specimen.

indicates that a fracture occurred on the outside of the cracks.

Fig. 9(a) shows the results of the Z specimen. The bending strength of the SP180 (98N) specimen is almost similar to that of the as-received Z specimen and the as-received SP180 specimen because the crack did not propagate in spite of the Vickers indentation. Meanwhile, the strength of the SP180 (196 N) specimen was reduced.

This is a cause of the large indentation, although the crack did not propagate. The bending strength of the as-SP300 specimen is slightly greater than that of the as-received specimen. The bending strength of both the SP300 (98 N) and SP300 (196 N) specimens decreased due to crack propagation. The SP300 (98 N) specimen showed a higher bending strength than that of the as-cracked specimen, but that of the SP300 (196 N) specimen was reduced to a similar strength to that of the as-cracked specimen. Therefore, it was determined that the Z specimen by SP180 has a toughness without a decrease in the strength in spite of the Vickers indentation load of 98 N.

Fig. 9(b) shows the results of the ZS specimen. The bending strength of the SP180 (98 N) specimen was almost similar to that of the as-received SP180 specimen because the crack did not propagate in spite of the Vickers indentation. The mean strength of the SP180 (98 N) specimen and as-received SP180 specimen was increased by about two times that of the as-received ZS specimen. Meanwhile, the mean strength of the SP180 (196 N) specimen was reduced. This is due to the large indentation, even though the crack did not propagate. The bending strength of the SP180 (196 N) specimen is slightly higher than that of the as-received specimen. The bending strength of both the SP300 (98 N) and SP300 (196 N) specimens decreased due to the crack propagation. The SP300 (98 N) specimen showed a higher bending strength than that of the as-received ZS specimen, but the SP300 (196 N) specimen showed a strength similar to or lower than that of the as-received ZS specimen. Therefore, the as-SP180 and as-SP300 specimens exhibited a higher strength than the asreceived ZS specimen, while the SP180 specimens exhibited a higher strength than the as-received ZS specimen in spite of the Vickers indentation load of 98 and 196 N, respectively. The SP300 specimen also showed a higher strength than that of the as-received ZS specimen in spite of the Vickers indentation load of 98 N. It was determined that the SP300 specimen also had excellent toughness.

Fig. 9(c) shows the results of the ZST specimen. The SP180 (98 N) specimen was almost similar to the bending strength of the as-received SP180 specimen and the as-received specimen because the crack did not propagate in spite of the Vickers indentation. Meanwhile, the strength of the SP180 (196 N) specimen was reduced. This is due to the large indentation, although the crack did not propagate. The bending strength of the SP180 (196 N) specimen. Although the crack propagated with the SP300 (98 N) and SP300(196 N) specimens, the SP300(98 N) specimen showed a similar strength to that of the as-SP300 specimen. The bending strength of the SP300 (196 N) specimen was slightly reduced, but was higher than that of the as-cracked specimen.

Conclusions

Zirconia (ZrO_2) composites can introduce a compressive residual stress to the surface by shot peening. The effect of the SP180 appears to be more significant than that on the SP300. By the SP treatment, the crack growth was reduced due to the micro-cracks and the residual stress introduced to the surface, with the bending strength of the ascracked specimen treated by shot peening was improved.

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