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Weibull distribution for Vickers hardness of peened ZrO_2 ceramics according to shot sizes

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The effects of shot peening (SP) on the Vickers hardness of ZrO_2 were studied. The size of the shot was pinned using φ180 μm and φ300 μm. Vickers hardness was measured for indentation loads of 98 N and 294 N. The effects of Vickers hardness was evaluated according to the ball size and the indentation load by performing a Weibull statistical analysis. The scale parameters were significantly affected with an indentation load of 98 N and a shot size of φ180 μm. Generally, the shape parameters were also significantly affected in the small short ball (180sp). Thus, the introduction of a compressive residual stress by SP is an effective technique for increasing the mechanical properties of ZrO_2.

Key words: ZrO_2 ceramic, Weibull statistical analysis, Vickers hardness, Shot peening, Ball size.

Introduction

Ceramics with excellent mechanical properties and high crack-healing ability possess potential applicability for the next generation of machine structural member and cutting tools. Many researchers have studied the crack healing phenomenon [1-7] with ceramics such as SiC, Si_3N_4/SiC, and Al_2O_3/SiC. ZrO_2 is widely used in implants and artificial bones, but is somewhat lacking for applications as structural member. In order to apply ZrO_2 as a structural member, Weibull statistical analysis was performed to evaluate the crack healing characteristics, mechanical properties [8] and Vickers hardness [9-14]. ZrO_2 shows a slight reduction in strength with the addition of SiC, however, the crack healing ability was improved by the addition of TiO_2. In addition, the Vickers hardness followed the Weibull probability distribution and researchers have evaluated the fracture toughness and mechanical properties using surface modified ceramics by shot peening. [15-17]

This study used two types of shot balls for surface modification in a monolithic specimen and a composite specimen of ZrO_2. The Vickers hardness for the as-received specimen and the shot-peened specimen were measured with indentation loads. Weibull statistical analysis was performed to evaluate the effect of the shot ball size and the indentation load.

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Materials and Experimental Method

A powder composed of 0.026 μm ZrO_2 (TZ-3Y-E, Tosoh), 0.27 μm SiC (Wako pure chemical industries), and a sintering additive (commercially purchased anatase 0.3 μm TiO_2) was used for the experiments. SiC was added to give crack healing characteristics. 3 wt.% TiO_2 was added for superior strength recovery by crack healing. Circular plates of φ60 × 5 mm were sintered in a vacuum atmosphere for 1 hr 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 ZrO_2 balls (Nikkato Ltd, YTZ® ball) of φ180 and φ300 μm with a Vickers hardness of 1,250 Hv. The shot projection nozzle diameter was φ5 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 seconds. Table 2 shows the conditions of the SP treatment. The shot peened specimens using shots of φ180 μm and φ300 μm were 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 was 160 μm (cut off 0.80 mm) and the measurement speed was set at 0.30 mm/sec.

The Vickers hardness was measured for 10 seconds under a load of 98 N and 294 N (HV-114, Mitutoyo). The specimens used were the as-received specimen,

Table 1. Batch composition and processing conditions.

Specimen	Batch composition (wt.%)	Conditions	
		Sintering	Heat treatment
Z	100 wt.% ZrO_2	30 MPa, 1723 K, 1 hr in vaccum	1073 K, 5 hr in air
ZS	90 wt.% ZrO_2 10 wt.% SiC		1173 K, 1 hr in air
ZST	88.8 wt.% ZrO_2 10 wt.% SiC 1.2 wt.% TiO_2		1073 K, 1 hr in air

Table 2. Shot peening conditions.

Shot system	Direct pressure system
Shot material	ZrO_2 (1250 Hv)
Shot diameter	φ180 μm, φ300 μm
Shot pressure	0.2 MPa
Projector distance	100 mm
Time	30 sec
Coverage	300%

heat treated specimen, 180sp specimen and 300sp specimen. Weibull statistical analysis was performed with a hardness of 20 for each specimen.

Results and Discussion

Weibull statistical analysis of as-received specimen and heat treated specimen

Fig. 1 shows the Vickers hardness of the Z, ZS and ZST specimens. Z, ZS and ZST indicate the as-received specimens, while Zh, ZSh and ZSTh indicate the heat-treated specimen. Vickers hardness was measured with an indentation load of 98 N. Each specimen was exhibited at 1200 ~ 1450 Hv in the Z specimen, 1290 ~ 1350 Hv in the ZS specimen and 1270 ~ 1310 Hv in the ZST specimen. The hardness distribution of the Zh specimen was less than that of the Z specimens. The hardness of the ZSh and ZSTh specimens was slightly higher than that of the ZS and ZST specimens. Especially, the hardness of the ZSTh specimen was increased by approximately 6%. In addition, it can be seen that Vickers hardness is not a determined value, and changes statistically. Accordingly, considering the ease of analysis and the weakest link assumptions, Weibull statistical analysis had to be performed with a two-parameter distribution as shown below.

$$F(x) = 1 - exp\left[-\left(-\frac{x}{\beta}\right)^{\alpha}\right] \quad (1)$$

Here, α is the shape parameter, which refers to the variability of the probability parameter, and β is the scale parameter indicating the characteristic lifetime, which is the failure probability of 63.2%.

Fig. 2 shows the Weibull probability of the Vickers hardness from the as-received and heat-treated specimens of Z, ZS and ZST. Table 4 shows the shape parameter and scale parameters of the Weibull distribution function estimated from the Vickers hardness of the as-received and heat-treated specimens of Z, ZS and ZST. The table also shows the average, standard deviation

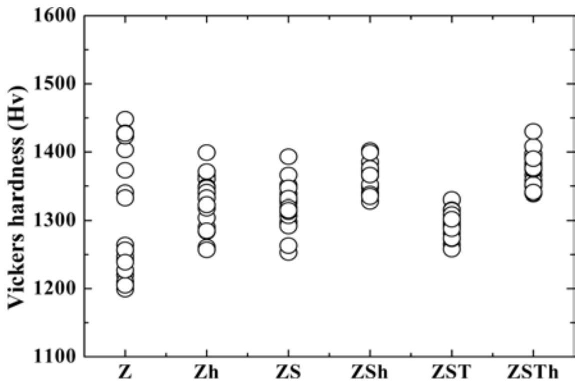


Fig. 1. Comparison of Vickers hardness between as-received specimen (Z, ZS and ZST) and heat treatment specimen (Zh, ZSh and ZSTh).

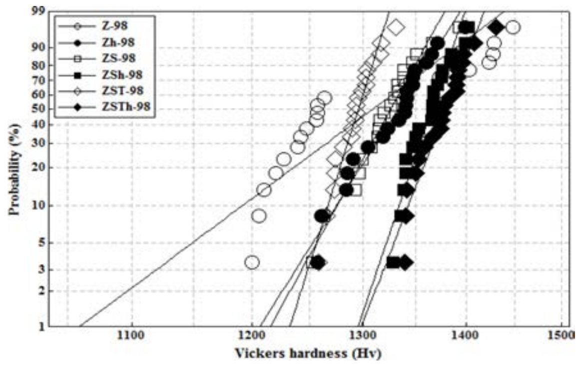


Fig. 2. Weibull plot of Vickers hardness of as-received specimen (Z, ZS, ZST) and heat treatment specimen (Zh, ZSh, ZSTh) under indentation load of 98 N.

Table 3. Condition for X-ray diffraction.

Characteristic X-ray	Cu-Kα
X-ray tube	Cu
Diffraction plane	Si_3N_4(323)
Diffraction angle [deg]	141.26
Tube voltage [kV]	40
Tube current [mA]	30

Table 4. The estimated Weibull parameters of 3 kinds of specimen under 98 N.

Parameter / specimen	Shape parameter	Scale parameter	Mean/STD/COV
Z	19.9994	1332.99	1305/98.19/0.075
Zh	42.2839	1344.40	1328/37.37/0.028
ZS	48.7790	1335.54	1321/32.47/0.025
ZSh	79.2620	1371.89	1363/21.04/0.015
ZST	85.5526	1300.63	1293/18.12/0.014
ZSTh	69.1720	1386.97	1376/24.36/0.018

(Std), and coefficient of variation (COV) according to mathematical statistics. The scale parameters of the Z and ZS specimens were similar, while that of the ZST specimen was about 2.5% smaller. The shape parameter of the Z specimen was about 20, while those of the ZS and ZST specimens were about 49 and 86, respectively. That is, the shape parameter was increased with the increase of synthetic elements in ZrO_2. However, the Zh specimen was approximately 111% larger than the Z specimen at about 42, the ZSh specimen was approximately 62% greater than the ZS specimen, and the ZSh specimen was about 20% smaller than the ZST specimen. COV also showed a similar pattern with the shape parameter. That is, the Z specimen was the largest by 0.075. The ZSh, ZS, ZSh, ZST and ZSTh specimens were similar.

Weibull statistical analysis by indentation load

Figs. 3(a-c) shows the Weibull probability of the Vickers hardness for the as-received specimen and the heat-treated specimens of Z, ZS and ZST. These were obtained from the indentation load of 98 N and 294 N. Tables 5-7 show the shape parameters and the scale parameters of the Weibull distribution function estimated from the Vickers hardness. The table also shows the average, standard deviation (Std), and coefficient of variation (COV) according to mathematical statistics. The hardness distribution for the Z, ZS and ZST specimens was higher by 98 N, and the dispersion was small. The hardness distribution of the ZST specimen with the addition of TiO_2 differed significantly according to the load. The hardness distribution of the Zh specimen was almost similar to that of the Z specimen and the dispersion was small. The hardness distributions of ZSh and ZSTh were slightly smaller than that of the as-received specimen, but the dispersion was also small. Especially, the Zh and ZSh specimens were smaller at 294 N, but the ZSTh specimen was higher at 294 N with the addition of TiO_2. It was determined that TiO_2 has a reinforcing effect following heat treatment. In addition, the hardness of the heat-treated specimens was smaller than that of the the as-received specimen. This is due to

Table 5. The estimated Weibull parameters of Z specimen by indentation load.

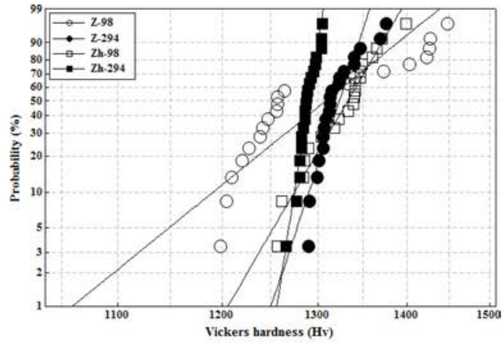
Parameter / specimen	Shape parameter	Scale parameter	Mean/STD/COV
Z-98	19.9994	1332.99	1305/98.19/0.075
Z-294	74.2877	1329.96	1320/23.58/0.018
Zh-98	42.2839	1344.40	1328/37.37/0.028
Zh-294	157.5700	1293.22	1289/9.876/0.008

Table 6. The estimated Weibull parameters of ZS specimen by indentation load.

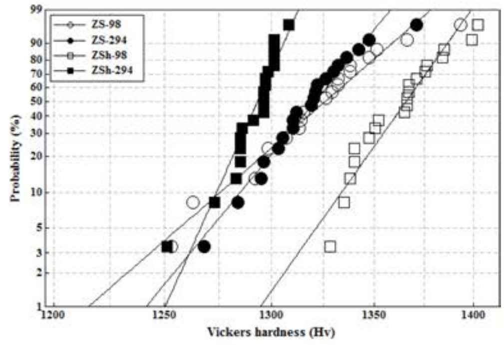
Parameter / specimen	Shape parameter	Scale parameter	Mean/STD/COV
ZS-98	48.7790	1335.54	1321/32.47/0.025
ZS-294	68.3922	1327.78	1317/23.18/0.018
ZSh-98	79.2620	1371.89	1363/21.04/0.015
ZSh-294	125.2040	1296.92	1291/12.63/0.010

Table 7. The estimated Weibull parameters of ZST specimen by indentation load.

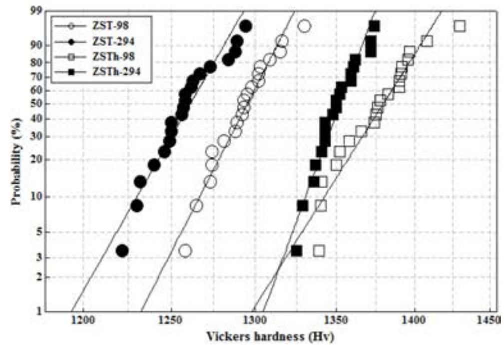
Parameter / specimen	Shape parameter	Scale parameter	Mean/STD/COV
ZST-98	85.5526	1300.63	1293/18.12/0.014
ZST-294	76.3153	1267.26	1258/20.02/0.016
ZSTh-98	69.1720	1386.97	1376/24.36/0.018
ZSTh-294	117.6830	1356.64	1350/13.85/0.010



(a) Z specimen

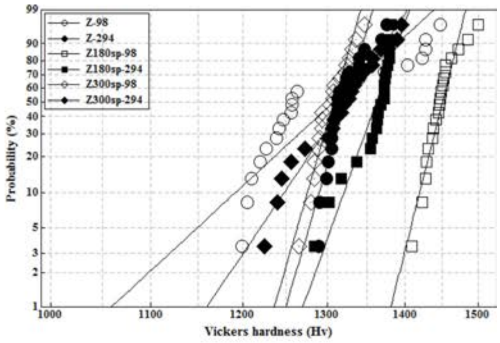


(b) ZS specimen

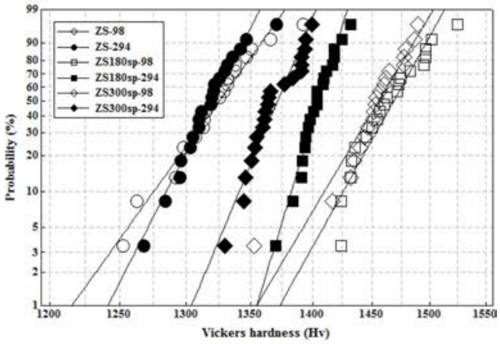


(c) ZST specimen

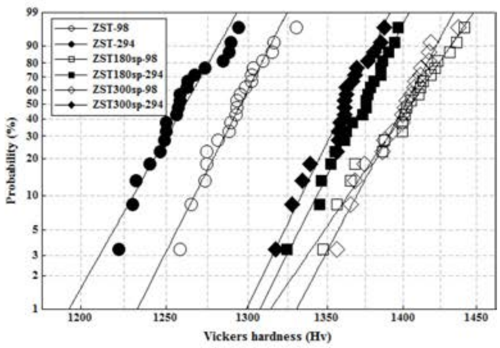
Fig. 3. Weibull plot of Vickers hardness according to indentation load.



(a) Z specimen



(b) ZS specimen



(c) ZST specimen

Fig. 4. Weibull plot of Vickers hardness according to indentation load.

the oxide formed on the surface by heat treatment.

Figs. 4(a-c) show the Weibull probability of the Vickers hardness from the as-received specimens of Z, ZS and ZST, the 180sp specimen and the 300sp specimen. Table 8-10 show the shape parameter and the scale parameters of the Weibull distribution function estimated from the Vickers hardness. The table also shows the average, standard deviation (Std), and coefficient of variation (COV) according to mathematical statistics. Each of the SP specimens had a higher probability distribution than the as-received specimen regardless of the indentation load. (a) The probability distribution of the Z180sp-98 specimen was higher than those of the Z-98 specimen and Z300sp-98 specimen. The indentation load at 98 N had a smaller dispersion than at 294 N.

Table 8. Weibull parameters of Z specimens.

Parameter / specimen	Shape parameter	Scale parameter	Mean/STD/COV
Z-98	19.9994	1332.99	1305/98.19/0.075
Z-294	74.2877	1329.96	1320/23.58/0.018
Z180sp-98	86.1561	1456.83	1448/21.20/0.015
Z180sp-294	60.4441	1370.82	1359/27.60/0.020
Z300sp-98	74.6613	1314.99	1306/21.30/0.016
Z300sp-294	32.1173	1338.44	1317/48.46/0.037

Table 9. Weibull parameters of ZS specimens.

Parameter / specimen	Shape parameter	Scale parameter	Mean/STD/COV
ZS-98	48.7790	1335.54	1321/32.47/0.025
ZS-294	68.3922	1327.78	1317/23.18/0.018
ZS180sp-98	63.2353	1477.35	1465/28.63/0.020
ZS180sp-294	114.6060	1410.36	1404/14.61/0.010
ZS300sp-98	58.8670	1464.94	1452/29.98/0.021
ZS300sp-294	83.2629	1377.94	1369/20.02/0.015

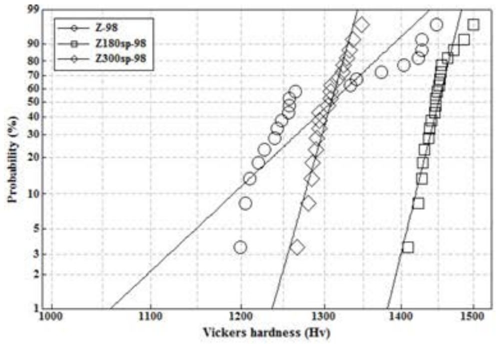
Table 10. Weibull parameters of ZST specimens.

Parameter / specimen	Shape parameter	Scale parameter	Mean/STD/COV
ZST-98	85.5526	1300.63	1293/18.12/0.014
ZST-294	76.3153	1267.26	1258/20.02/0.016
ZST180sp-98	63.1090	1412.85	1401/26.15/0.019
ZST180sp-294	85.9629	1378.78	1370/18.70/0.014
ZST300sp-98	81.4170	1407.34	1398/20.37/0.015
ZST300sp-294	102.4820	1368.06	1361/15.97/0.012

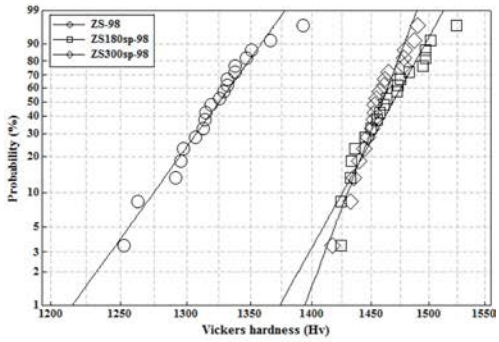
The probability distribution of the Z180sp-294 specimen was smaller than that of the Z180sp-98 specimen, but higher than those of the Z-294 and Z300sp-294 specimens. (b) The probability distribution of the ZS180sp-98 specimen was higher than those of the ZS300sp-98 specimen and the ZS-98 specimen. The ZS180sp-294 specimen was also higher than the ZS300sp-294 and ZS-294 specimens. (c) The probability distribution of the ZST specimen was high in the order of ZST-98 > ZST300sp-98 > ZST180sp-98. The probability distribution at 294 N was smaller than at 98 N, but the trends were the same. The probability distribution with an indentation load of 294 N in sp specimen was higher than that at 98 N.

The shape and scale parameters of 98 N were described above. The corresponding results for 294 N are as follows. (a) The shape parameters of the Z180sp and Z300sp specimens were smaller by about 20% and 57% compared to that of the Z specimen. The scale parameters of the Z180sp and Z300sp specimens were higher by about 3% and 6% compared to that of the Z specimen. (b) Shape parameters for ZS180sp and ZS300sp specimens were higher by 68% and 22% compared to that of the ZS specimen. Scale parameters for ZS180sp and ZS300sp specimens were about 6% and 4% higher than that of the ZS specimen. (c) Shape parameters for ZS180sp and ZS300sp specimens were 13% and 34% higher than that of the ZST specimen. Scale parameters for ZS180sp and ZS300sp specimens were about 8% higher than that of the ZST specimen. The hardness distribution with the indentation load of 98 N was higher than that at 294 N.

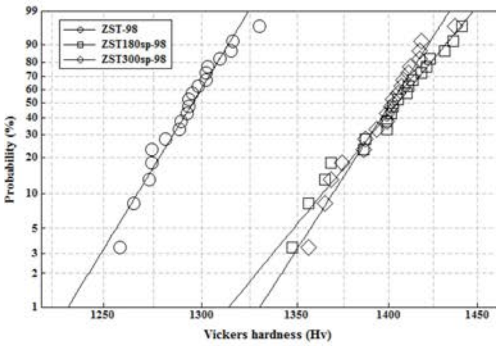
The mean Vickers hardness of the as-received specimen at 98 N was similar, but the sp specimens, except for the Z300sp specimen, were about 10% higher than the as-received specimen. That is, the shape and scale parameters of the 180sp specimen were higher than those of the as-received specimen and the 300sp specimen. The mean Vickers hardness for the as-received specimen at 294 N was similar, but the ZST specimen was smaller by about 5%. Z180sp and ZS180sp specimens were 3% and 6% higher than each of the Z and ZS of the as-received specimens. The 300sp specimen was 0%, 4% and 8% higher than the Z, ZS and ZST of the as-received specimens. This was affected by the surface modification according to the size of the shot ball.



(a) Z specimen



(b) ZS specimen



(c) ZST specimen

Fig. 5. Weibull plot of Vickers hardness according to shot ball sizes.

Weibull statistical analysis by size of shot ball

Figs. 5(a-c) show the Weibull probability of the Vickers hardness from the as-received specimens of Z, ZS and ZST, the 180sp specimen and the 300sp specimen. The images were obtained with an indentation load of 98 N. Table 11-13 shows the shape parameter and the scale parameters of the Weibull distribution function estimated from the Vickers hardness. The table also shows the average, standard deviation (Std), and coefficient of variation (COV) according to mathematical statistics. (a) The shape parameter of the Z-98 specimen was about 20, but those of the Z180sp-98 and Z300sp-98 specimens were 86 and 75, respectively. The dispersions of Z180sp-98 and Z300sp-98 specimens were smaller than that of the Z-98 specimen. The scale parameter of the Z180sp-98 specimen was higher than that of the Z specimen, and exhibited a high probability distribution. However, the Z300sp-98 specimen was similar to the Z-98 specimen. (b) The shape parameters of the ZS specimens were in the order of ZS180sp-98 > ZS300sp-98 > ZS-98. Dispersion was reduced by shot peening (sp). Scale parameters also exhibited the same tendency

Table 11. The Estimated Weibull parameters of Z specimens by shot ball size.

Parameter / Speci.	Shape parameter	Scale parameter	Mean/STD/COV
Z-98	19.9994	1332.99	1305/98.19/0.075
Z180sp-98	86.1561	1456.83	1448/21.20/0.015
Z300sp-98	74.6613	1314.99	1306/21.30/0.016

Table 12. The Estimated Weibull parameters of ZS specimens by shot ball size.

Parameter / Speci.	Shape parameter	Scale parameter	Mean/STD/COV
ZS-98	48.7790	1335.54	1321/32.47/0.025
ZS180sp-98	63.2353	1477.35	1465/28.63/0.020
ZS300sp-98	58.8670	1464.94	1452/29.98/0.021

Table 13. The Estimated Weibull parameters of ZST specimens by shot ball size.

Parameter / Speci.	Shape parameter	Scale parameter	Mean/STD/COV
ZST-98	85.5526	1300.63	1293/18.12/0.014
ZST180sp-98	63.1090	1412.85	1401/26.15/0.019
ZST300sp-98	81.4170	1407.34	1398/20.37/0.015

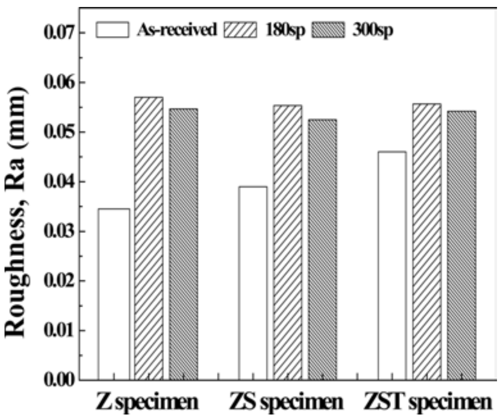


Fig. 6. Surface roughness of Z, ZS and ZST specimens according to shot peening.

as the shape parameter. (c) The shape parameter of the ZST specimen was higher than that of the Z and ZS of the as-received specimen. Dispersion of the hardness was minimal. The scale parameters of the ZST specimen were smaller than that of each Z and ZS specimen. That is, the 180sp specimen showed probability distributions higher than the as-received specimen and the 300sp specimen.

Roughness before and after shot peening

Fig. 6 shows the surface roughness before and after shot peening. The surface roughness of the as-received specimen showed a sequence of ZST> ZS> Z specimens. However surface roughness after shot peening was similar in the three types of specimens, and the surface roughness of the 180sp and 300sp specimens was higher than that of the as-received specimen. Also, the surface roughness of the 180sp specimen was slightly larger than that of the 300sp specimen.

Conclusions

The Vickers hardness of a monolithic specimen (Z specimen) and a composite specimen (ZS and ZST specimen) of ZrO_2 was measured with indentation loads of 98 N and 294 N. The reliability of the Vickers hardness was analyzed with Weibull statistics. The following results were obtained.

(1) The shape parameters of the as-received specimens for Z, ZS and ZST with an indentation load of 98 N were about 20, 49 and 86. The hardness of ZrO_2 was less dispersed with an increase of the synthesis element. The shape parameter of the heat treated Z specimen was about 111% higher than that of the as-received Z specimen, while the heat treated ZS specimen was about 42% higher than the as-received ZS specimen. However, the heat-treated ZS specimen was about 20% lower than the as-received ZST specimen. The shape parameter at 294N was higher than that at 98N. Therefore, dispersion was a small.

(2) The scale parameters of the as-received Z and ZS specimens were close to 1330, and that of the ZST specimen was 1300. The scale parameters of the heat treated specimens increased by 0.6, 6 and 7% compared to the as-received specimen, respectively.

(3) The scale parameters of the as-received and heat treated specimens with an indentation load of 98 N were increased by 0.2% (Z specimen), 4% (Zh specimen), 0.5% (ZS specimen), 6% (ZSh specimen), 3% (ZST specimen), and 2.2% (ZSTh specimen) compared to that of the indentation load of 294 N.

(4) The scale parameters of the 180sp and 300sp specimens with an indentation load of 98N were generally higher than with an indentation load of 294N. The scale parameters of the 180sp-98N specimen were 1457 (Z specimen), 1477 (ZS specimen) and 1413 (ZST specimen). The scale parameters for the 180sp-98 N specimen were higher than for the 180sp-294N, 300sp-98N and 300sp-294N specimens.

(5) From the above results, surface modification of ceramics is believed to be superior to with small short balls.

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