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Weibull statistical analysis on Vickers hardness of corroded ZrO_2 composites ceramics

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This study measured the Vickers hardness of ceramics, and Weibull statistical analysis was used to evaluate the reliability of the measured data. The specimens were heat-treated for 1, 5 and 10 hrs at temperatures of 1073 K and 1173 K, and were corroded for 400 hours in acidic and alkaline solutions. The specimens were as follows: yttria-stabilized ZrO_2 monolithic ceramics, ZrO_2/SiC composite ceramics with SiC added to improve crack healing ability and ZrO_2/SiC/TiO_2 composite ceramics with TiO_2 added for the increase of strength. The 2-parameter Weibull probability distribution can be applied to the Vickers hardness. In the Weibull statistical analysis of the corroded ZrO_2 composite ceramics, the shape parameters and scale parameters can be used to determine the dispersion and to predict the strength / hardness.

Key words: ZrO_2 ceramics, Corrosion, Acidic solution, Alkaline solution, Vickers hardness, Weibull statistical analysis.

Introduction

Ceramics has excellent properties, such as heat resistance, corrosion resistance, and wear resistance. However, ceramics has a high cracking susceptibility because the fracture toughness is much lower than metal materials. Therefore, it is difficult to use for machining, due to the higher processing costs and the lack of reliability. This problem has become an important research topic, with several approaches aimed at providing crack-healing ability to ceramics. Similarly, granting self-crack-healing ability to ZrO_2 ceramics is expected to reduce the cost of processing complex shaped implants and artificial bones, which require reliability.

Many researchers are actively studying the crack healing of ceramics (heat treatment) using oxidation. The healing conditions require a temperature of 1273 ~ 1573 K.[1-7] ZrO_2/SiC (20 wt.%) composite ceramics has a self-healing capability that can be activated within 30 ~ 100 hours at a low temperature of 873 ~ 1073 K [8]. Previous studies also confirmed the ability of crack healing with the addition of SiC (10 wt.%) and TiO_2 to ZrO_2 ceramics [9], but the bending strength of ZrO_2/SiC is shown to be approximately 50% that of ZrO_2. If the phase transformation (ie, tetragonal $\rightarrow$ monoclinic transformation) of ZrO_2/SiC composite ceramics can be harnessed for crack-healing, this is a new crack healing mechanism can be developed and is expected to improve the crack-healing ability and strength of ceramics with the addition of other ingredients and materials. However, research on the corrosion of ceramics is necessary because of potential exposure to a variety of environments. Studies on the corrosion resistance and chemical resistance of ceramics have been performed, and the corrosion resistance of crack-healed SiC ceramics has been evaluated. [10-12] In addition, ceramics have diverse mechanical properties. Researchers reported the statistical characteristics and quantitative probability characteristics to the average value or dispersion on mechanical properties of ZrO_2/SiC composite ceramics. [13, 14] Mechanical properties such as tensile strength and hardness are very important for the design, manufacture and development of materials.

This study measured the Vickers hardness of ceramics, and Weibull statistical analysis was used to evaluate the reliability of the measured data. The specimens were heat-treated for 1, 5 and 10 h at temperatures of 1073 K and 1173 K, and were corroded in acidic and alkaline solutions. The specimens were as follows: Yttria-stabilized ZrO_2 monolithic ceramics, ZrO_2/SiC composite ceramics with SiC added to improve crack healing ability and ZrO_2/SiC/TiO_2 composite ceramics with TiO_2 added for the increase of strength.

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. Silicon carbide was added for crack healing. TiO_2 was

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Table 1. Batch composition and processing conditions.

Speci.	Batch composition (wt.%)	Conditions		Relative density (%)
		Hot Pressing	Heat treatment	
Z	ZrO_2 (100)	30 MPa, 1723 K, 1 hr in vaccum	1073 K and 1173 K from 1 to 10 hrs in air	100.17
ZS	ZrO_2 (90) SiC (10)			100.90
ZST	ZrO_2 (88.8) SiC (10.0) TiO_2 (1.2)			98.45

added to 3 wt.% to achieve the most strength recovery by crack healing. Sintered materials were sintered in a vacuum atmosphere for 1 hr via a hot press under 30 MPa at 1,723 K. Afterward, the ZrO_2, ZrO_2/SiC and ZrO_2/SiC/TiO_2 specimens were named Z, ZS, and ZST, respectively. The batch compositions of the specimens are given in Table 1.

Specimens for measuring Vickers hardness were heat treated. The heat treatment was carried out for 1, 5 and 10 hrs at 1073 K and 1173 K. The surfaces of the specimens were polished prior to hardness measurement and corrosion testing at room temperature for 400 hours in solution. Sintering and heat treatment conditions for each specimen are shown in Table 1.

Corrosion testing was conducted using the acidic and alkaline corrosion test method for fine ceramics under the KS standard, KSL1607. Solutions of H_2SO_4 3 mol/L and NaOH 5 mol/L were used.

Hardness was measured using a Vickers hardness tester (HV-114, Mitutoyo). The specimen was measured for 10 seconds with indentation loads of 9.8 N. Weibull statistical analysis was used with the hardness data of 10 measured for each specimen.

Results and Discussion

Figs. 1-6 show the Vickers hardness of the corroded specimens (Z, ZS and ZST) in an acidic solution

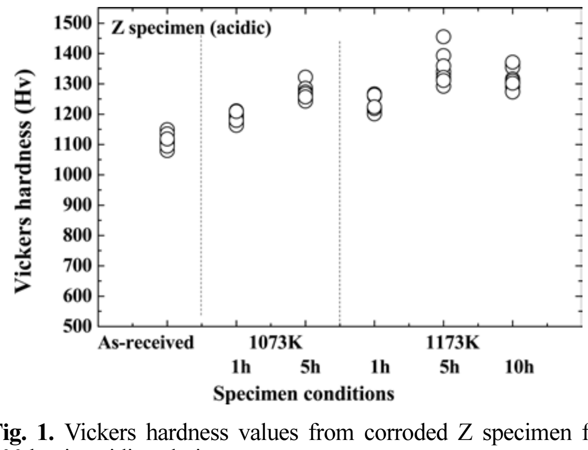

Fig. 1. Vickers hardness values from corroded Z specimen for 400 hrs in acidic solution.

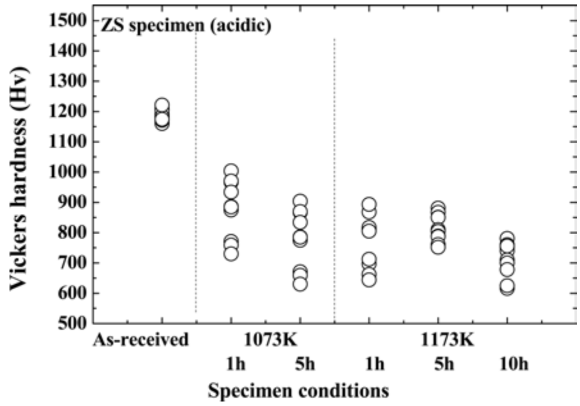

Fig. 2. Vickers hardness values from corroded ZS specimen for 400 hrs in acidic solution.

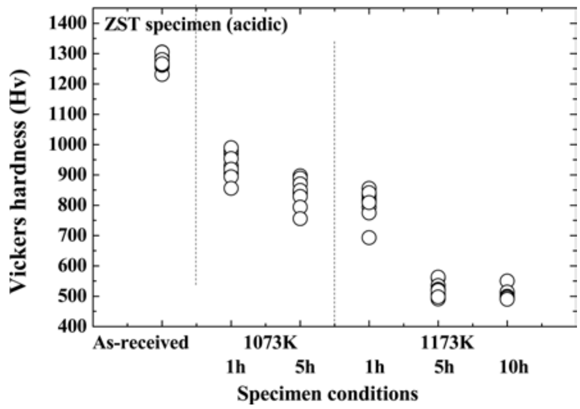

Fig. 3. Vickers hardness values from corroded ZST specimen for 400 hrs in acidic solution.

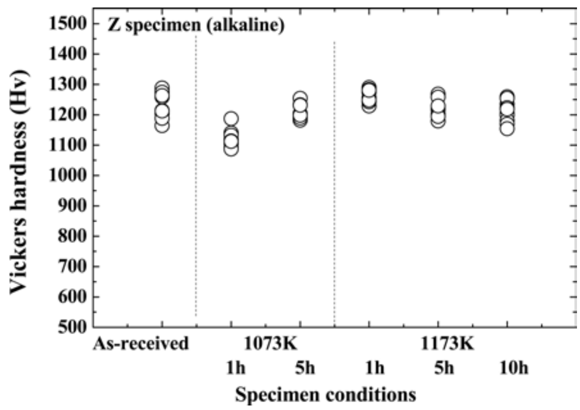

Fig. 4. Vickers hardness values from corroded Z specimen for 400 hrs in alkaline solution.

(H_2SO_4) and an alkaline solution (NaOH). The Vickers hardness differs according to the kinds of specimen, but a variation can be clearly observed. For the strength evaluation of the ceramics, as a brittle material, a probabilistic evaluation considering the variation distribution is important in order to improve the accuracy of the assessment. 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, the

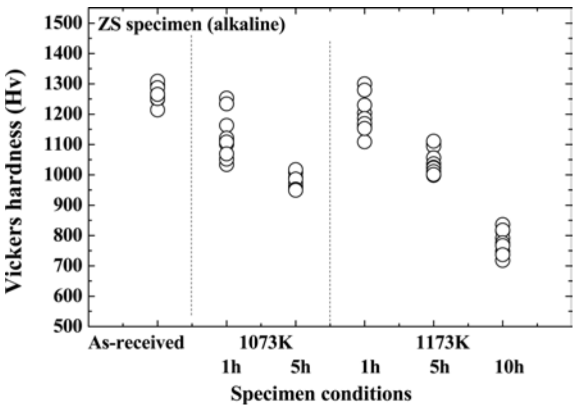

Fig. 5. Vickers hardness values from corroded ZS specimen for 400 hrs in alkaline solution.

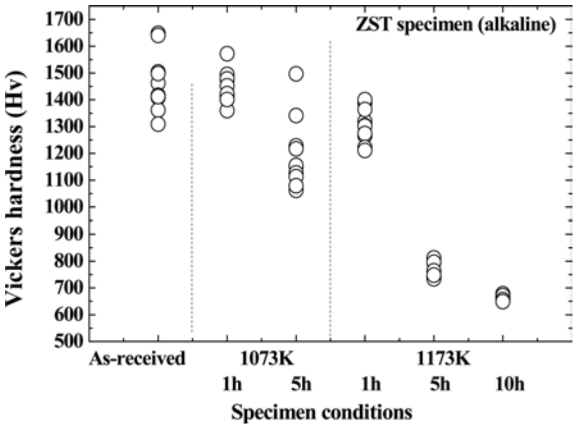

Fig. 6. Vickers hardness values from corroded ZST specimen for 400 hrs in alkaline solution.

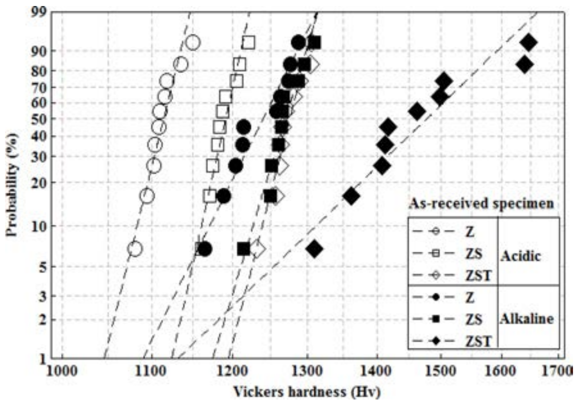

Fig. 7. Weibull plot of Vickers hardness from as-received specimen.

Weibull statistical analysis needs to be applied as a two-parameter Weibull distribution as shown below.

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

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. 7 shows the Vickers hardness according to the

Table 2. The estimated Weibull parameters for as-received specimen.

Condition		Shape parameter	Scale parameter	Mean/SD/COV
Acidic	Z	66.5	1120	1112/19.6/0.018
	ZS	73.5	1196	1188/18.8/0.016
	ZST	64.5	1282	1272/22.7/0.018
Alkaline	Z	32.9	1253	1234/42.9/0.035
	ZS	54.3	1278	1266/26.8/0.021
	ZST	15.9	1511	1466/110.6/0.075

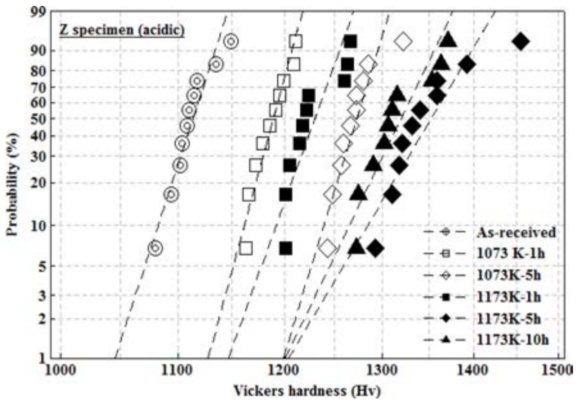

Fig. 8. Weibull plot of Vickers hardness from corroded Z specimen in acidic solution.

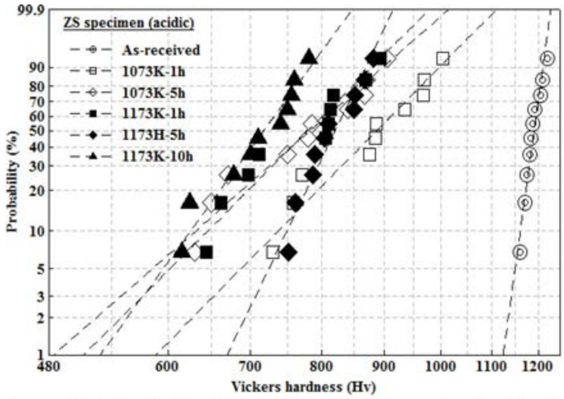

Fig. 9. Weibull plot of Vickers hardness from corroded ZS specimen in acidic solution.

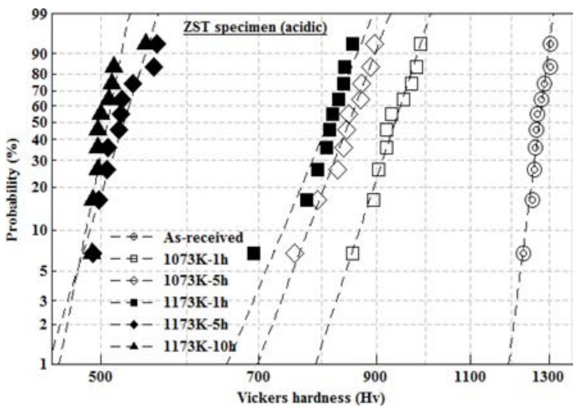

Fig. 10. Weibull plot of Vickers hardness from corroded ZST specimen in acidic solution.

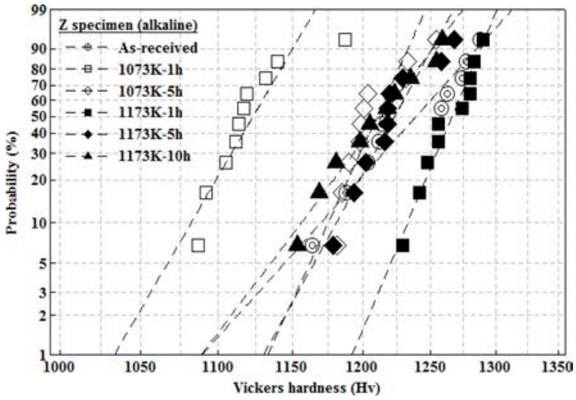

Fig. 11. Weibull plot of Vickers hardness from corroded Z specimen in alkaline solution.

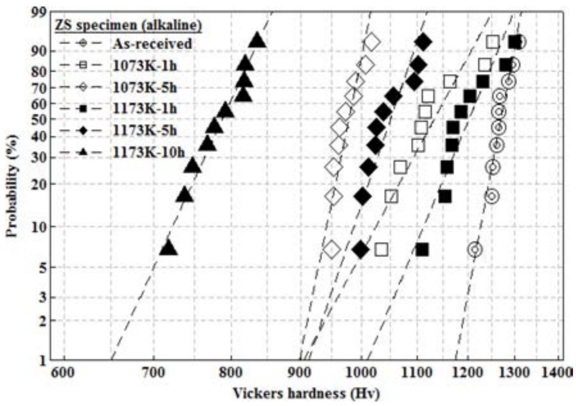

Fig. 12. Weibull plot of Vickers hardness from corroded ZS specimen in alkaline solution.

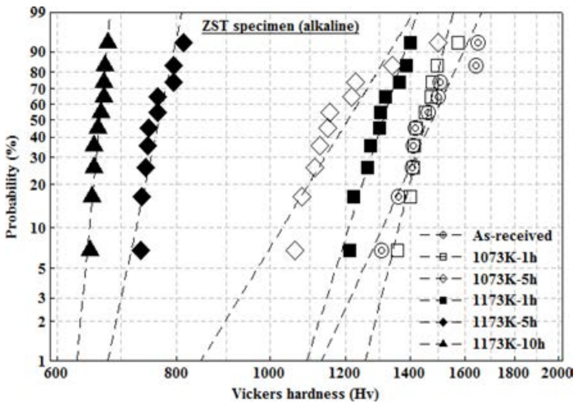

Fig. 13. Weibull plot of Vickers hardness from corroded ZST specimen in alkaline solution.

Weibull probability. Since hardness is expressed as a straight line, it can be seen as applicable to the Weibull probability distribution.

The Vickers hardness of the as-received corroded ZS specimen in both solutions was lower than those of the as-received corroded Z and ZST specimens. The Vickers hardness of the as-received corroded Z, ZS and ZST specimens in alkaline solution was higher than those of the corroded specimens in acidic solution, but the hardness of the acidic solution was lower than that of the alkaline solution and the distribution was small.

Table 2 shows the shape parameter and the scale parameters of the Weibull distribution function estimated from the Vickers hardness of the corroded Z, ZS and ZST specimens in both solutions. The table also shows the average, standard deviation (STD), and coefficient of variation (COV) according to mathematical statistics.

Figs. 8-13 show the Vickers hardness according to the Weibull probability. Vickers hardness was obtained from the corroded Z, ZS and ZST in both an acidic and alkaline solution. Vickers hardness can be seen as applicable to the Weibull probability distribution.

In Fig. 8, the Vickers hardness of all corroded Z specimens was higher than that of the corroded Z as-received specimens. The value for the corroded Z specimen at 1173 K was higher than that at 1073 K.

In Fig. 9, the Vickers hardness of all corroded ZS specimen was lower than that of the corroded ZS as-received specimen. The value for the corroded ZS specimen at 1073 K was a little higher than that at 1173 K.

In Fig. 10, the Vickers hardness of all corroded ZST specimen was lower than that of the corroded ZST as-received specimen. The value for the corroded ZST specimen at 1073 K was significantly higher than that at 1173 K. This is similar to the non-corroded specimens. Especially, the corroded 1173 K-5 hrs and 10 hrs ZST specimens exhibited almost the same probability distribution, while the 1173 K-1 hr and 1073 K-5 hrs corroded ZST specimen exhibited almost the same distribution.

In Fig. 11, the probability distribution for the 1173 K-1hr corroded Z specimen was higher than that of the as-received Z specimen. The distribution for the corroded Z specimen at 1073 K and 1173 K was lower than that of the Z as-received specimen. The corroded 1073 K-1 h Z specimen exhibited the lowest probability distribution while the 1173 K-1 hr specimen exhibited the highest. The other specimens exhibited similar probability distributions.

In Fig. 12, all corroded ZS specimens had lower probability distributions than the as-received corroded ZS specimens. The corroded ZS specimens at 1073 K and 1173 K exhibited lower probability distributions with increased heat treatment time. The corroded ZS specimen at 1173 K exhibited slightly higher probability distribution than that at 1073 K.

In Fig. 13, the Vickers hardness for all corroded ZST specimens was lower than that of the corroded ZST as-received specimens. The Vickers hardness for the corroded ZST specimen at 1073 K was significantly higher than that of 1173 K with each heat treatment time. This is similar to the non-corroded specimens. Especially, the 1173 K-5 hrs and 10 hrs corroded ZST specimens exhibited almost the same probability distribution, but the 1173 K-1 hr and 1073 K-5 hrs ZST corroded specimens exhibited almost the same distribution. Especially, the 1173 K-10 hs corroded ZST specimen exhibited a much lower probability distribution than the others.

Table 3. The estimated Weibull parameters for Z specimen (acidic).

Condition	Shape parameter	Scale parameter	Mean/SD/COV
AS-received	66.5	1120	1112/19.6/0.018
1073K-1hr	79.7	1195	1187/17.1/0.015
1073K-5hrs	70.4	1280	1271/22.5/0.018
1173K-1hrs	59.9	1238	1228/26.0/0.020
1173K-5hrs	36.4	1367	1348/47.3/0.035
1173K-10hrs	44.8	1331	1316/35.5/0.027

Table 4. The estimated Weibull parameters for ZS specimen (acidic).

Condition	Shape parameter	Scale parameter	Mean/SD/COV
AS-received	73.5	1196	1188/18.8/0.016
1073K-1 hr	10.3	919	878/96.0/0.109
1073K-5 hrs	8.8	816	775/98.1/0.127
1173K-1 hrs	10.1	808	772/82.3/0.107
1173K-5 hrs	21.0	835	815/44.8/0.055
1173K-10 hrs	13.9	737	712/57.1/0.080

Table 5. The estimated Weibull parameters for ZST specimen (acidic).

Condition	Shape parameter	Scale parameter	Mean/SD/COV
AS-received	64.5	1282	1272/22.7/0.018
1073K- 1hr	24.9	951	932/42.0/0.077
1073K-5 hrs	21.6	864	844/43.2/0.051
1173K-1 hrs	19.5	828	807/46.6/0.058
1173K-5 hrs	26.0	532	522/24.3/0.047
1173K-10 hrs	40.7	511	505/17.8/0.035

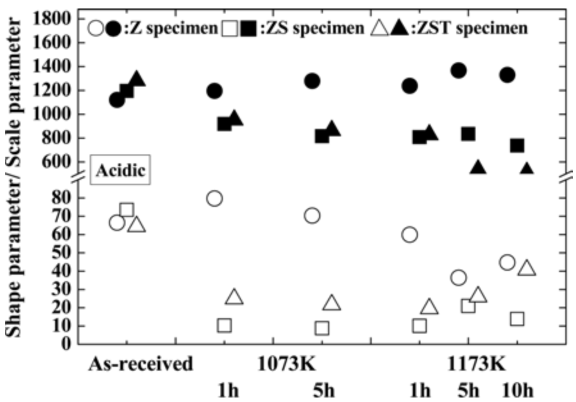

Fig. 14. Shape parameter and scale parameter from Weibull probability of Z, ZS and ZST specimens immersing in acidic solution.

Tables 3-5 show the shape and scale parameters of the Weibull distribution function estimated from the Vickers hardness of the corroded specimens. These were obtained from an acidic solution. The tables also show the average, standard deviation (STD), and coefficient of variation (COV) according to mathematical statistics.

Fig. 14 shows the shape parameter and the scale from an acidic solution. The symbols (○, ●) were obtained from the corroded Z specimen. The shape parameters at 1073 K were about 20% and 6% larger than those of the corroded as-received specimen, but those at 1173K were smaller by about 10%, 45% and 33%, respectively. The scale parameters at 1073 K were about 7% and 14% larger than those of the corroded as-received specimen, while those at 1173 K were larger by about 10%, 22% and 19%, respectively. The symbols (□, ■) were obtained from the corroded ZS specimen. The shape parameters at 1073 K were about 86% and 88% smaller than those of the corroded as-received specimen, and those at 1173 K were smaller by about 86%, 71% and 81%, respectively. The scale parameters at 1073 K were about 23% and 32% smaller than those of the corroded as-received specimen, while those at 1173 K were smaller by about 32%, 30% and 38%, respectively. The symbols (△, ▲) were obtained from the corroded ZST specimen. The shape parameters at 1073 K were about 61% and 66% smaller than those of the corroded as-received specimen, and those at 1173 K were smaller by about

Table 6. The estimated Weibull parameters for Z specimen (alkaline).

Condition	Shape parameter	Scale parameter	Mean/SD/COV
AS-received	32.9	1253	1234/43.0/0.035
1073K-1 hr	50.6	1132	1121/28.4/0.025
1073K-5 hrs	64.4	1217	1208/23.7/0.020
1173K-1 hrs	70.5	1273	1264/20.4/0.016
1173K-5 hrs	53.7	1232	1220/26.9/0.022
1173K-10 hrs	39.0	1225	1210/35.0/0.029

Table 7. The estimated Weibull parameters for ZS specimen (alkaline).

Condition	Shape parameter	Scale parameter	Mean/SD/COV
AS-received	54.3	1278	1266/26.7/0.021
1073K-1 hr	18.9	1154	1125/72.6/0.065
1073K-5 hrs	49.9	985	975/24.0/0.025
1173K-1 hrs	24.3	1220	1195/59.2/0.050
1173K-5 hrs	30.1	1063	1046/42.7/0.041
1173K-10 hrs	22.2	800	782/39.6/0.051

Table 8. The estimated Weibull parameters for ZST specimen (alkaline).

Condition	Shape parameter	Scale parameter	Mean/SD/COV
AS-received	15.9	1511	1466/110.6/0.075
1073K-1 hr	29.0	1473	1448/59.9/0.041
1073K-5 hrs	12.9	1243	1197/133.4/0.111
1173K-1 hrs	23.1	1333	1305/64.9/0.050
1173K-5 hrs	34.4	775	763/27.7/0.036
1173K-10 hrs	76.2	668	663/10.1/0.015

70%, 60% and 37%, respectively. The scale parameters at 1073 K were about 26% and 33% smaller than those of the corroded as-received specimen, and those at 1173 K were smaller by about 35%, 59% and 60%, respectively.

From the results obtained in the acidic solution, the shape parameters of the corroded Z specimen at 1073 K were larger than those of the as-received specimen, but all parameters at 1173K were smaller and all the scale parameters were slightly larger. Both the shape and scale parameters of the corroded ZS and ZST specimen were smaller.

Tables 6-8 show the shape and scale parameters for the Weibull distribution function estimated from the Vickers hardness of the corroded specimen. These were obtained from an alkaline solution. The tables also show the average, standard deviation (STD), and coefficient of variation (COV) according to mathematical statistics.

Fig. 15 shows the shape parameter and the scale from an alkaline solution. The symbols (○ , ●) were obtained from the corroded Z specimen. The shape parameters at 1073K were about 54% and 96% larger than those of the corroded as-received specimen, and those at 1173K were larger by about 114%, 63% and 19%, respectively. The scale parameters at 1073 K were about 10% and 3% smaller than those of the corroded as-received specimen whereas those at 1173 K were larger by about 2% after 1 hr, and smaller by about 2% after 5 and 10 hrs. The symbols (□ , ■) were obtained from the corroded ZS specimen. The shape parameters at 1073 K were about 65% and 8% smaller than those of the corroded as-received specimen, and those at 1173 K were smaller by about 55%, 46% and 59%, respectively. The scale parameters at 1073 K were about 10% and 23% smaller than those of the corroded as-received specimen, and those at 1173 K were smaller by about 5%, 17% and 37%, respectively. The symbols (△ , ▲) were obtained from the corroded ZST specimen. The shape parameters for 1073 K-1 hr were about 82% larger than those of the corroded as-received specimen, but those for 1073 K-5 hrs were smaller by about 19% and those at 1173 K were larger by about 45%, 116% and 380%, respectively. The scale parameters at 1073K were about 3% and 18% smaller than those of the corroded as-received specimen, and those at 1173 K were smaller by about 12%, 49% and 100%, respectively.

From the results obtained in the alkaline solution, the shape parameters of the corroded Z specimen at 1073 K and 1173 K were larger than those of the as-received specimen, but the scale parameters were similar or smaller. Both the shape and scale parameters of the corroded ZS and ZST specimen were smaller.

From the results obtained in the alkaline solution, all the shape parameters of corroded Z specimen at 1073 K and 1173 K were larger than those of the as-received specimen, but the scale parameters were similar or smaller. Both the shape and scale parameters of the corroded ZS specimen were smaller. The shape parameters of the corroded ZST specimen were generally larger, but all the scale parameters were smaller.

Figs. 16-17 show the mean Vickers hardness of the corroded Z, ZS and ZST specimens. Fig. 16 and Fig. 17 were obtained from immersion in acidic and alkaline solutions, respectively. The standard deviation is shown by the solid line, and the solid symbols represent the average hardness of the as-received specimen.

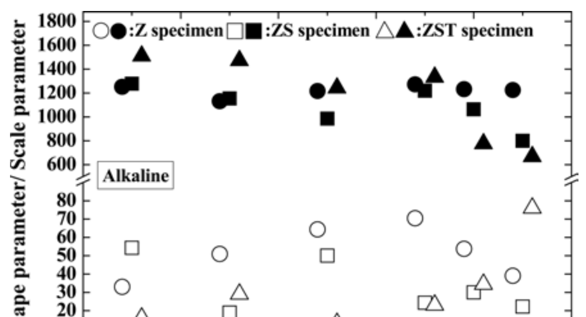

Fig. 15. Shape parameter and scale parameter from Weibull probability of Z, ZS and ZST specimens immersing in alkaline solution.

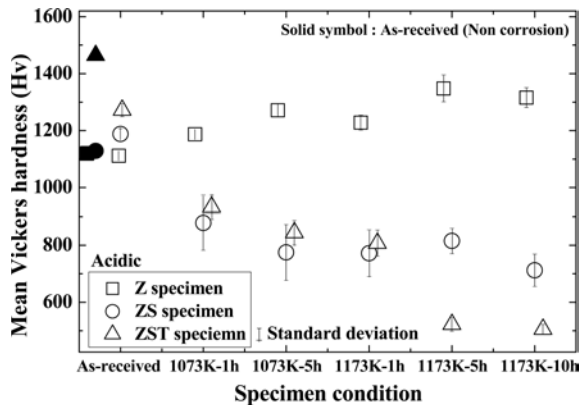

Fig. 16. Mean Vickers hardness according to specimen conditions of Z, ZS and ZST specimens immersing in acidic solution.

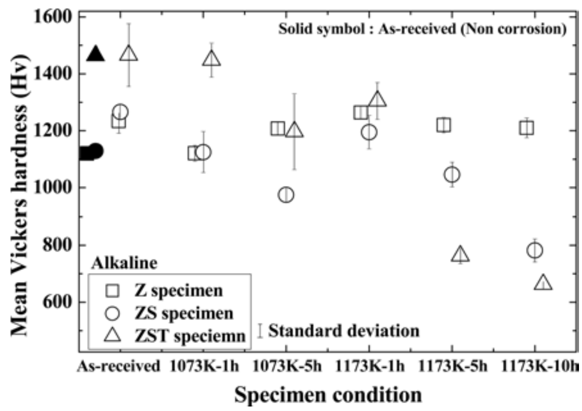

Fig. 17. Mean Vickers hardness according to specimen conditions of Z, ZS and ZST specimens immersing in alkaline solution.

Fig. 16 was obtained from immersion in acidic solution. The mean Vickers hardness of the as-received Z specimen is 1120 Hv, and that of the corroded as-received specimen is 1110 Hv. Both were similar. However, the heat-treated specimens exhibited increased mean hardness. In particular, the hardness of the specimens that received heat treatment for a long time was further increased. The strength of monolithic zirconia decreases when used for a long time at high temperatures over 1273 K. This is because it was partially changed to Monoclinic. However, the strength of monolithic zirconia is enhanced when it is heat-treated at temperatures lower than 1073 and 1173 K. The mean Vickers hardness of the ZS and ZST as-received specimens were 1130 and 1464 Hv, and that of the corroded as-received specimen was 1188 and 1272 Hv, respectively. Additionally, the mean hardness of the corroded specimen decreased with increasing temperature and heat treatment time. This trend was further reduced by the addition of TiO_2 to the 1173 K-5 hrs and 10 hrs ZST specimens. That is, ZrO_2 facilitates tetragonal $\rightarrow$ monoclinic transformation and the thermal expansion coefficient changes with the addition of TiO_2, resulting in a large crack or tear. These spots are more susceptible to corrosion.

Fig. 17 was obtained from immersion in an alkaline solution. The mean Vickers hardness of the Z as-received specimen was 1120 Hv, and that of the corroded as-received specimen was 1230 Hv. The mean Vickers hardness of the corroded as-received specimen was slightly higher than that of the corroded as-received specimen. The mean Vickers hardness of the heat-treated specimen was nearly similar to or slightly higher than that of the corroded as-received specimen.

The reason for this is explained above. The mean Vickers hardness of the ZS and ZST as-received specimens was 1130 and 1464 Hv, and that of the corroded as-received specimen was 1266 and 1466 Hv, respectively. Additionally, the mean hardness of the corroded specimen decreased with increasing temperature and heat treatment time. This trend was similar to the results with an acidic solution.

Conclusions

ZrO_2 monolithic ceramics and ZrO_2 composites ceramics were heat-treated for 1, 5, and 10 hours at 1073 K and 1173 K. These specimens were corroded for 400 hrs by acidic and alkaline solutions. Vickers hardness testing was performed with Weibull statistical analysis in order to evaluate the reliability of the measurement data.

1. The 2-parameter Weibull probability distribution can be applied to the Vickers hardness.

2. For the Z, ZS and ZST as-received specimens, the Vickers hardness of the corroded specimen in acidic solution was smaller than that in alkaline solution, but the probability distribution of the acidic solution was smaller than that of the alkaline solution.

3. With the corroded heat-treated specimens in acidic solution, the hardness distributions for all corroded Z specimens were larger than those of the corroded Z as-received specimens. The hardness distributions of all corroded ZS and ZST specimens were smaller than those of the corroded ZS and ZST as-received specimens.

4. For the corroded heat-treated specimens in alkaline solution, the hardness distribution of the 1173 K-1hr corroded Z specimen was larger than that of the corroded Z as-received specimen, but the hardness distributions of the corroded Z specimen at 1073 K and 1173 K were smaller than that of the corroded Z as-received specimen. However, the hardness distributions of all corroded ZS and ZST specimens were smaller than those of the corroded ZS and ZST as-received specimens.

5. The shape parameters of the corroded Z specimen at 1073 K were larger than those of the Z as-received specimen, but all shapes parameters at 1173 K were smaller. All scale parameters were slightly larger. All the shape and scale parameters of the corroded ZS and ZST specimens were smaller than those of the ZS and ZST as-received specimens.

6. In the Weibull statistical analysis of the corroded ZrO_2 composite ceramics, the shape parameters and scale parameters can be used to determine the dispersion and to predict the strength / hardness.

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