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Weibull statistical analysis on mechanical properties of immersed silicon carbide in acidic and alkaline solution

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Weibull statistical analysis on mechanical properties was performed on silicon carbide by immersion in acidic and alkaline solution. The heat treatment was carried out on 1373 K. The corrosion of silicon carbide was conducted in acidic and alkaline solution under KSL1607. Results showed that heat treatment in air could significantly increase the strength of the silicon carbide. The strength of the corroded and cracked specimen was similar to that of the cracked specimen. The bending strength of the corroded, crack healed specimen decreased 47% and 75% more than that of the crack healed specimen in acidic and alkaline solution, respectively. The corrosion of silicon carbide is faster in alkaline solution than in acidic solution. The probability distributions of Vickers hardness closely followed the Weibull distribution. The scale parameter and shape parameter are evaluated in as-received silicon carbide and corroded silicon carbide, respectively.

Key words: Weibull analysis, Silicon carbide, Acidic solution, Alkaline solution, Bending strength, Vickers hardness.

Introduction

Silicon carbide has excellent characteristics for radiation resistance and high temperature. Silicon carbide ceramics have been studied as high temperature gas furnace reactor core components and nuclear fuel cladding for fourth-generation nuclear reactors, the structural material and insulation of the fusion reactor blanket, applications in the energy industry, and as high temperature component materials for aerospace industrial applications [1-3]. Silicon carbide composites show excellent performance as a structural material. Therefore, a great deal of research has been carried out on silicon carbide in the nuclear industry as well as in other industries [4]. However, ceramics have a limited application due to the problem of low toughness; ceramics with self-crack-healing ability have greatly improved reliability [5].

In such a perspective, research on imparting the self-healing ability of the structural ceramics has been actively carried out. Structural ceramics with self-crack healing have superior mechanical properties than the as-received material [7-10]. Especially, silicon carbide have been considered in application to the blanket of a fusion reactor [11, 12]. Plasma cannot be applied as a metal material because characteristics of corrosion are strong. As such, while many ceramics studies have been performed on corrosion resistance and chemical resistance [13, 14], in this current study, we carried out a probability analysis of the crack healing material [15, 16]. The evaluation of the quantitative probability distribution characteristics of ceramics and their statistical characteristics such as average or dispersion of mechanical properties is very important as the basis of the data of the design, development, and manufacture of the material [17].

In this study, an as-received specimen and a crack healed specimen (heat treated specimen) of silicon carbide having a crack healing ability were evaluated in terms of the bending strength by corrosion in acidic and alkaline solutions, while Vickers hardness was investigated from the probability statistical properties. For the bending strength and the Vickers hardness, the average value from a small amount of measurement data are usually used; however, this study reports a Weibull statistical analysis in order to evaluate the reliability of the Vickers Hardness measured data.

Materials and Experimental Method

Commercially available silicon carbide (90 wt.%, Ultrafine grade, Ibiden Co., Japan), aluminum oxide (6 wt.%, Sumitomo Chemical Co. Ltd., Japan), and yttrium oxide (4 wt.%, CI Chemical Co., Japan) were used as the starting materials. The mean particle sizes of the silicon carbide, aluminum oxide and yttrium oxide powders were 0.27 mm, 0.1 mm, and 31 nm, respectively. The mixtures were subsequently hot-pressed in N_2 gas for one hour via hot-pressing, conducted under 35 MPa at

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2053 K. The heat treatment was carried out for 1 hour at 1373 K, showing the optimum crack healing.

Corrosion was conducted using the acidic and alkaline corrosion test method of fine ceramics under the KS standard, KSL1607. Solutions of H_2SO_4 3 mol/L and NaOH 5 mol/L were used. For the corrosion test, a semi-circular crack was made of about 125 μm in the center of the specimen by a load of 29.4 N using a Vickers hardness tester (HV-114, Mitutoyo). Hereafter, the as-received specimen with the crack is callesd the cracked specimen, and the cracked specimen with healing at 1373 K for 1 hr is called the crack healed specimen. The two types of specimen were cleaned by ultrasound, and corrosion was carried out at room temperature for 400 hours in solution, after drying in a constant temperature drier at 383 K. 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.

Hardness was measured using a Vickers hardness tester (HV-114, Mitutoyo). The heat treated specimens for hardness measurement were heat treated for 1 hr at 1373 K after mirror polishing. The as-received specimen and the heat treated specimen were measured for 10 seconds from the indentation loads of 9.8 N and 29.4 N. Weibull statistical analysis was used as hardness data of 20 measured on each specimen.

Results and Discussion

Bending strength of corroded specimen

Fig. 1 shows the bending strength of the corroded specimen in an acidic solution (H_2SO_4) and an alkaline solution (NaOH). Solid symbols (■ , ● , ▲) in the figure represent the average strength of the as-received specimen, cracked specimen, and crack healed specimen, respectively. The strengths of the as-received specimen were found to be 674 MPa. The strength of the cracked specimens with a crack dimension of 2c 125 μm was lower than half of the smooth specimen strength. The strength of the crack healed specimen represented as 1,254 MPa, was increased by approximately 270% more than that of the cracked specimen. On the other hand, the strength of the corroded cracked specimen is 310 and 314 MPa in an acidic and alkaline solution, respectively, showing a slightly lower strength than that of the cracked specimen. However, the strength of the corroded cracked healed specimen is 661 and 384 MPa in the acidic and alkaline solution, respectively, and decreased by approximately 47% and 70% in an acidic and alkaline solution than that of cracked healed specimen, respectively. The strength of corroded cracked healed specimen in an acidic solution is similar to that of the as-received specimen, but that of the alkaline solution was similar to the strength of the cracked specimen. Therefore, the silicon carbide demonstrated that corrosion is faster in an alkaline solution than an acidic solution.

Fig. 2 shows the more fractured surface of the corroded cracked specimen and corroded crack healed specimen in an alkaline solution than in an acidic solution. Fig. 2(a) shows the cracked specimen, Figs. 2(b) and 2(c) show corrosion in an acid solution, and Figs. 2(d) and 2(e) show the corrosion in an alkaline solution. The cracked specimen in Fig. 2(a) shows the semi-elliptical crack. The corroded specimens in Figs. 2(b) and 2(d) could be confirmed by the corrosion in the portion of Vickers indentation and by the corrosion in the cracked part. However, the corroded crack healed specimens in Figs. 2(c) and 2(e) could hardly confirm the appearance of corrosion because the shape of the Vickers indentation is maintained due to the healing.

Weibull statistical analysis of Vickers hardness

Figs. 3(a) and 3(b) show the Vickers hardness obtained from the indentation load of 9.8 N and 29.4 N in the as-received specimen and corroded specimens. The as-received specimen and corroded specimen differ

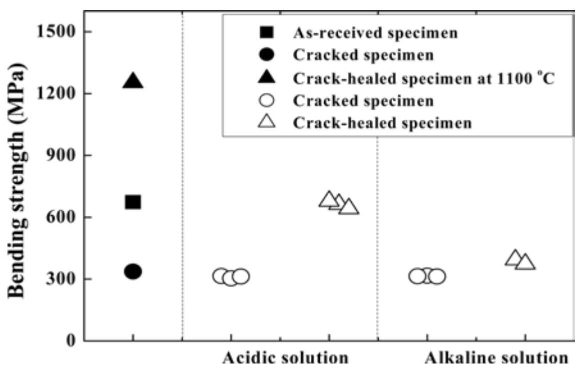

Fig. 1. Bending strength of the corroded silicon carbide in both solutions.

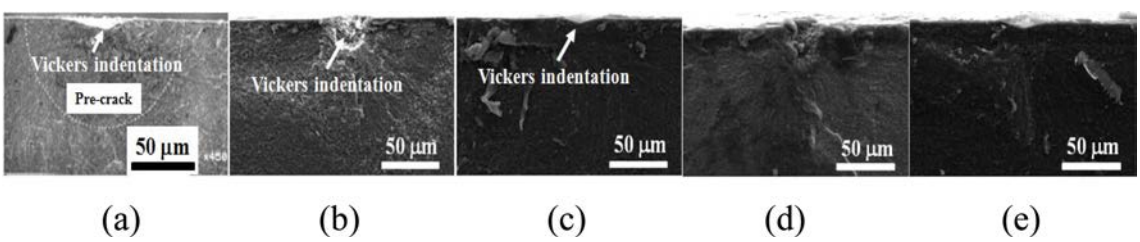

Fig. 2. SEM photograph of fracture surface; (a) cracked, (b) cracked in acidic, (c) crack healed in acidic, (d) cracked in alkaline, (e) crack-healed in alkaline.

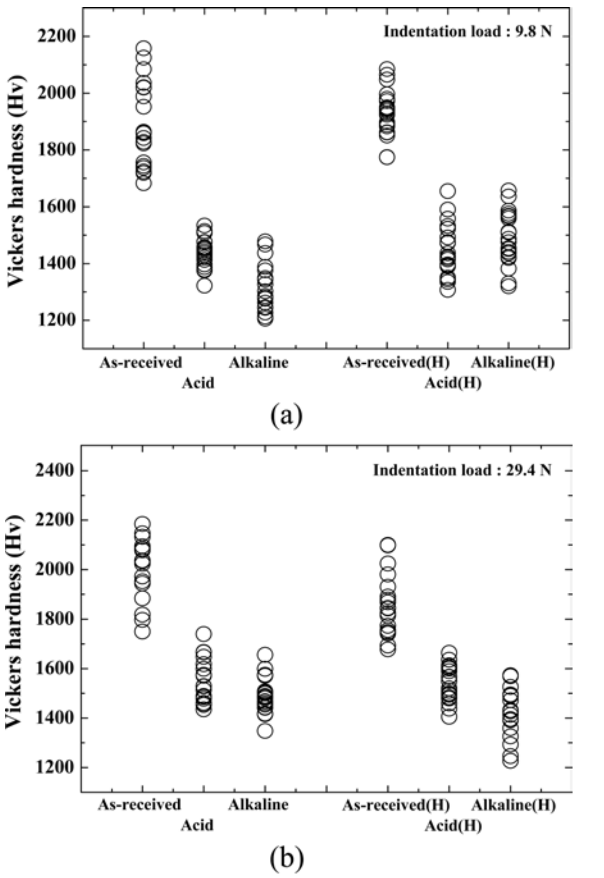

Fig. 3. Vickers hardness values according to indentation load for specimen conditions. (a) 9.8 N, (b) 29.4 N.

by the indentation load, but a variation can be seen.

In the strength evaluation of 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 statistically changes. Accordingly, considering the ease of analysis and the weakest link assumptions, a Weibull statistical analysis needs to be applied as a two-parameter Weibull distribution as shown below.

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

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

Fig. 4 shows the Vickers hardness according to Weibull probability. Since hardness is expressed as a straight line, it can be seen as applicable to the Weibull probability distribution. Figs. 4(a) and 4(b) show the Vickers hardness result of 9.8 N and 29.4 N, respectively. From Fig. 4(a), the Vickers hardness of the corroded as-received specimen in both solutions exhibited a significantly lower value than that of the as-received specimen. The Vickers hardness of the corroded as-received specimen in an alkaline solution

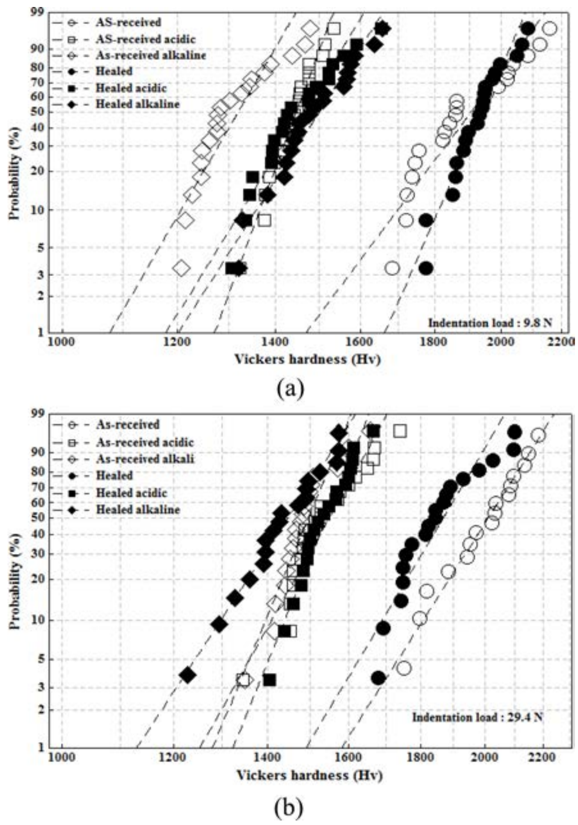

Fig. 4. Weibull plot of Vickers hardness values. (a) 9.8 N, (b) 29.4 N.

is lower than that of the corroded as-received specimen in an acidic solution. While this does not significantly differ in appearance from the corrosion of the acidic and alkaline solutions, it indicates that the specimens were degraded by corrosion. The Vickers hardness from an alkaline solution appeared to be lower than that of the acidic solution. This suggests that the silicon carbide is consistent with those having more corrosion in the alkaline solution. Meanwhile, the Vickers hardness of healed specimens was slightly higher than that of the as-received specimens. Moreover, the Vickers hardness of the corroded healed specimens in an acidic and an alkaline solution also exhibited lower values than those of the healed specimen. In addition, the Vickers hardness of the corroded healed specimen in an alkaline solution appeared to be slightly higher than that of the corroded healed specimen in an acidic solution.

The results shown in Fig. 4(b) obtained at 29.4 N were also similar to those obtained at 9.8 N. The Vickers hardness of the corroded as-received specimen in both solutions exhibited a significantly lower value than that of the as-received specimen. However, the Vickers hardness of the corroded as-received specimen in an alkaline solution was similar to that of the corroded as-received specimen in an acidic solution. While degradation occurred in an acidic and an alkaline solution, the corrosion occurred only on the surface part. Meanwhile, the Vickers hardness of the healed specimens

Table 1. Estimated Weibull parameters by 9.8 N of indentation load.

Parameter / Specimen	Shape parameter	Scale parameter	Mean/STD COV
As-received	16.18	1950	1891/147 0.078
As-received acidic	32.59	1463	1440/52 0.036
As-received alkaline	20.83	1344	1311/82 0.063
Healed	27.52	1965	1929/84 0.044
Healed acidic	19.52	1490	1452/92 0.063
Healed alkaline	18.93	1530	1489/94 0.063

Table 2. Estimated Weibull parameters by 29.4 N of indentation load.

Parameter / Specimen	Shape parameter	Scale parameter	Mean/STD COV
As-received	17.55	2054	1996/130 0.065
As-received acidic	23.10	1577	1538/96 0.062
As-received alkaline	26.89	1517	1488/70 0.047
Healed	18.79	1905	1855/126 0.068
Healed acidic	27.35	1564	1534/67 0.044
Healed alkaline	17.01	1479	1436/98 0.068

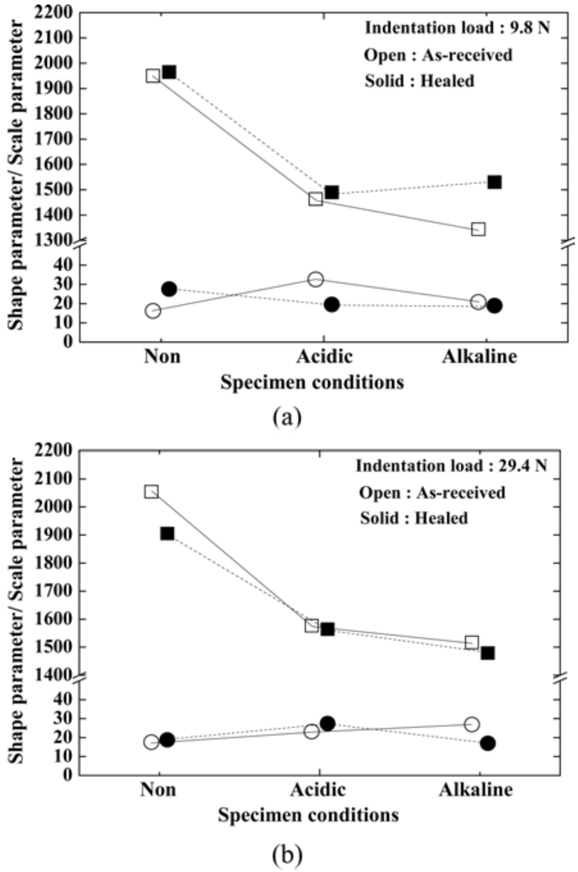

Fig. 5. Shape parameter and scale parameter from Weibull probability. (a) 9.8 N, (b) 29.4 N.

is lower than that of the as-received specimens, or were almost similar. The Vickers hardness of corroded healed specimens in both solutions is also lower than that of the healed specimen. However, the Vickers hardness of the corroded healed specimen in an acidic solution was higher than that of the corroded healed specimen in alkaline solution. It is determined that either the oxide layer or the cured layer of the surface part formed by heat treatment is destroyed by the large indentation load.

Tables 1 and 2 show the shape parameter and the scale parameters of the Weibull distribution function estimated from the Vickers hardness of the as-received specimen and healed specimen. The table also shows the average, standard deviation (STD), and coefficient of variation (COV) according to mathematical statistics.

Figs. 5(a) and 5(b) show the shape parameters and the scale parameters from Tables 1 and 2. The following results were compared with the non-corroded specimen. In Fig. 5(a), the shape parameters of the corroded as-received specimen in both solutions increased by about 100% and 30%, respectively. However, the shape parameters of the corroded healed specimen in both solutions reduced by approximately 30%. On the other hand, the shape parameters of the corroded as-received specimen in both solutions reduced by approximately 25% and 31%, respectively; those of the healed specimen reduced by 24% and 22%, respectively. In Fig. 5(b), the scale parameters of the corroded as-received specimen in both solutions increased by about 32% and 53%, respectively. However, the scale parameter of the corroded healed specimen in the acidic solution increased by approximately 46%, but that in the alkaline solution was reduced by about 10%. On the other hand, the scale parameters of the corroded as-received specimen in both solutions reduced by approximately 23% and 26%, respectively and those of the healed specimen reduced by 18% and 22%, respectively.

From Figs. 5(a) and 5(b), the shape parameters of the corroded as-received specimen in both solutions increased, but that of the scale parameters had reduced. However, the shape parameters of the corroded healed specimen in the acidic solution increased, but those of the alkaline solution had reduced. The scale parameters had also reduced.

Fig. 6 shows the average hardness shown in Tables 1 and 2 along with the standard deviation. Figs. 6(a) and

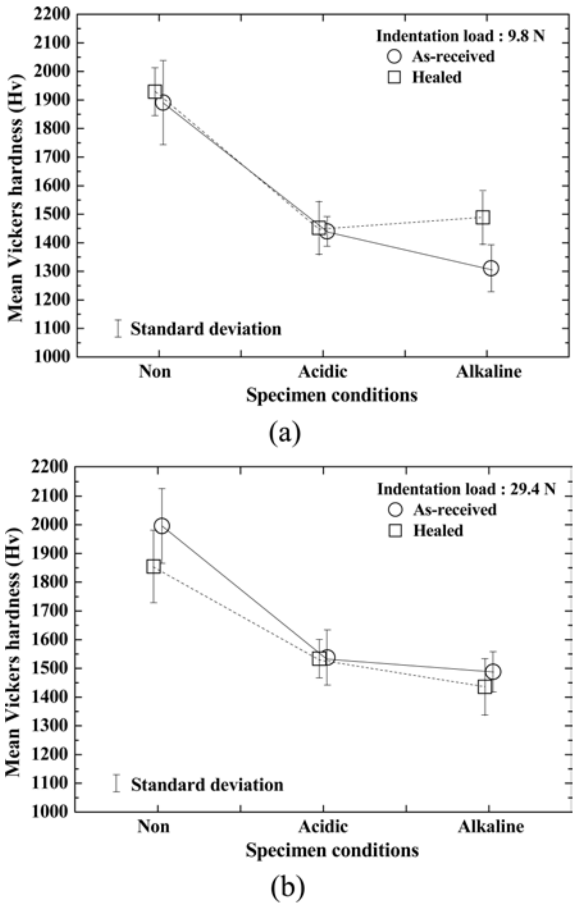

Fig. 6. Mean Vickers hardness values according to specimen conditions. (a) 9.8 N, (b) 29.4 N.

6(b) show an average hardness of 9.8 N and 29.4 N, respectively. The average hardness of corroded as-received specimen and corroded healed specimen in both solutions reduced by 20% ~ 24% and 22% ~ 31% compared to the non-corroded specimen, respectively. The average hardness of the specimen in the alkaline solution decreased slightly more than in the acid solution. The standard deviation of the as-received specimen and healed specimen is the largest, followed by the corroded specimen in an alkaline solution and an acid solution. That is, the as-received specimen and healed specimen showed the fine surface defect of the sintered state or binding properties between the particles. The corroded specimen in both solutions was due to the fine surface defect and the corrosion between particles.

Conclusions

This study evaluated the bending strength of silicon carbide having corrosion in an acid solution and an alkaline solution. Vickers hardness was performed from Weibull statistical analysis in order to evaluate the reliability of the measurement data. The results are obtained as follows. The strength of the corroded cracked specimen was similar to that of the cracked specimen, but the strength of the corroded healed specimen in both solutions decreased by 47% and 70% more than the healed specimen, respectively. Therefore, silicon carbide shows more rapid corrosion in the alkaline solution than in the acid solution. The Vickers hardness of the as-received specimen, healed specimen, and corroded specimen followed a two-parameter Weibull probability distribution. The shape parameters of the as-received specimen and healed specimen are large in the corrosion of acid and alkaline solutions, but the scale parameters were smaller. The mean Vickers hardness of the corroded specimen in both solutions was much lower than in the as-received specimen and healed specimen, and it was lower than the acidic solution in the alkaline solution.

Acknowledgments

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