Journal of Ceramic Processing Research. Vol. 19, No. 3, pp. 183~188 (2018)

J O U R N A L O F
Ceramic Processing Research

Mechanical properties and wear characteristics of $ZrO_2/SiC/TiO_2$ composite ceramics

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$ZrO_2/SiC/TiO_2$ composite ceramics were sintered. These specimens were heat treated at 1073 K and 1173 K for 1, 3 and 10 hrs, and evaluated the mechanical properties and the wear characteristics. The bending strengths of ZST specimens were almost the same. ZST specimens were able to crack-healing due to addition of SiC, and the strength was increased by addition of TiO_2. The Vickers hardness of the as-received ZST specimen was higher than that of the heat treated specimens. The Vickers hardness of the heat treated specimens decreased with increasing temperature and time. The coefficient of friction of the ZST specimens increased with increasing heat treatment temperature and heat treatment time. The friction coefficients of ZST specimens were inversely related to Vickers hardness. The wear loss increased with the heat treatment time at each heat treatment temperature. The wear loss of all specimens was inversely proportional to the Vickers hardness, but the wear loss was proportional to the friction coefficient.

Key words: $ZrO_2/SiC/TiO_2$ composite ceramics, Heat treatment, Vickers hardness, Friction coefficient, Wear loss.

Introduction

Ceramic material has been actively studied as a high temperature structural material due to its excellent resistance to heat, corrosion, and abrasion [1]. The application of materials with these characteristics to various structures is expected to improve the strength of such structures. However, ceramics have low fracture toughness, low reliability, and poor processability. In particular, the disadvantage of ceramic materials is that they are easily fractured even with in case of micro cracking. Many researchers have studied the self-healing of microcracks. [2-6] The crack-healed part of ceramics with silicon carbide (SiC) added showed a fracture strength equal to or higher than that of the as-received specimen [7-10]. Cracks of a certain width or less can be cured, even if the cracks are long [11].

Zirconium dioxide (ZrO_2) adopts a monoclinic crystal structure at room temperature, and transitions to a tetragonal and cubic structure at higher temperatures [12]. This substance can be manufactured as a ceramic with high toughness and high strength. Excessive transition has been shown to possibly lower the strength of ceramics. Also, research has shown the crack healing ability of ZrO_2 when SiC is added [13, 14]. Since ZrO_2 has excellent mechanical strength, it is widely used in dental materials, milling, grinding, mixer, and cutter. Many researchers have reported on the mechanical and wear characteristics of ceramics [15-18] for use in such devices. On the other hand, titanium dioxide (TiO_2) can also be used as a ceramic additive. TiO_2 can be divided into two types: anatase and rutile. The rutile type TiO_2 has a melting point of 1,830 ^{o} and has high strength properties as a sintering additive. [18] However, to the best of our knowledge, no studies have been reported on the evaluation of the mechanical properties and wear characteristics of ZrO_2 composite ceramics containing SiC and TiO_2.

In this study, $ZrO_2/SiC/TiO_2$ composite ceramics were sintered by adding SiC and TiO_2. The specimens were heat treated at 1073 K and 1173 K for 1, 5, and 10 hrs each. The specimens were evaluated for their mechanical properties and wear characteristics, and the results were reviewed.

Materials and test Methods

The ZrO_2 powder used in this study was TZ-3Y-E, including stabilizer Y_2O_3 3 mol.% (0.26 μm mean particle size, Tosho Co., Japan). The SiC powder was ultrafine (0.27 μm mean particle size, Wako Pure Chemical Industries, Ltd., Japan). To evaluate the characteristics of ZrO_2 according to the addition amount of TiO_2, an average particle diameter of 0.3 μm (Anatase) was used. Isopropanol and a silicon nitride (Si_3N_4) ball (φ5) were added to the mixture, which was then completely blended for 24 hrs. The mixture was

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Table 1. Batch composition and processing.

Specimen	ZST1	ZST2	ZST3
Batch Composition (wt.%)	ZrO_2 89.6 wt.%, SiC 10.0 wt.%, TiO_2 0.4 wt.%	ZrO_2 89.2 wt.%, SiC 10.0 wt.%, TiO_2 0.8 wt.%	ZrO_2 88.8 wt.%, SiC 10.0 wt.%, TiO_2 1.2 wt.%
Hot Pressing	30 MPa, 1723 K, 1 hour in vaccum		
Heat Treatment	1073 K and 1173 K during 1 hr, 5 hrs and 10 hrs in air		
Relative Density (%)	98.26	98.49	98.45

placed in desiccators to extract the solvent and to make a dry powder mixture. Plates were sintered in vacuum for 1 hr via a hot press at under 35 MPa at 1,723 K. Hereafter, the ZrO_2/SiC/TiO_2 composite specimens are referred to as the ZST specimens. Table 1 shows the composition of the ZST1~3 specimens.

The bending strength was measured in a three-point bending test at room temperature with a span of 16 mm and a crosshead speed of 0.5 mm / min. The hardness was measured using a Vickers hardness tester (HV-114, Mitutoyo). The as-received specimen and the heat treated specimen were measured for 10 seconds with an indentation load of 9.8 N. The type of wear tester (BRW140, Neoplus) was a "block on ring". The ring material was SKD 11 of 35 mm diameter and 7 mm thickness. The block was a rectangular shaped specimen. The test conditions were as follows: (1) the rotation speed of the ring was 50 rpm; (2) the load was 9.8 N; (3) the total wear distance was 500 m; and (4) the tests were performed at room temperature in a dry condition. To obtain high reliability, 55,000 data were used, which were obtained at 10 data per second.

Test results and Discussion

Fig. 1 shows the bending strengths for the as-received ZST specimen. The as-received Z and ZS specimens are shown for comparison. [19] Table 2 shows the mean, standard deviation (Std), and coefficient of variation (COV) according to mathematical statistics. The bending strengths of the as-received ZST1, ZST2, and ZST3 specimens are 1342, 1256, and 1410 MPa, respectively. These are smaller than the as-received Z specimen, but larger than the as-received ZS specimen. The as-received Z specimen did not show healing of cracks, but the as-received ZS specimen with SiC showed crack-healing properties. The as-received ZST specimen with the addition of SiC showed crack-healing, and the strength was increased by the addition of TiO_2.

Fig. 2 shows the Vickers hardness of the as-received ZST specimen and the heat-treated ZST specimen. Table 3 shows the mean, standard deviation (Std), and coefficient of variation (COV) according to mathematical statistics. The Vickers hardness of each as-received specimen was higher than that of the heat treated specimens. The heat treated specimens were smaller at a higher temperature and for a longer time. The Vickers hardness was the highest for the ZST3 specimen like

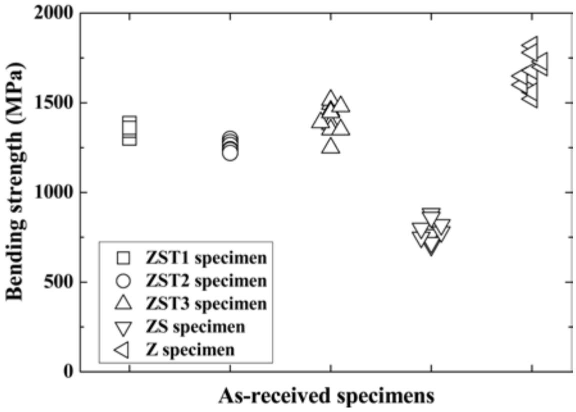

Fig. 1. Bending strength of as-received Z, ZS and ZST specimen.

Table 2. Mean, standard deviation(SD) and coefficient of variation(COV) by arithmetic statistics for bending strength of as-received specimen.

Parameter / Specimen	Mean	SD	COV
Z	1668	94.22	0.056
ZS	779	60.90	0.078
ZST1	1342	29.53	0.022
ZST2	1256	27.11	0.022
ZST3	1410	81.85	0.058

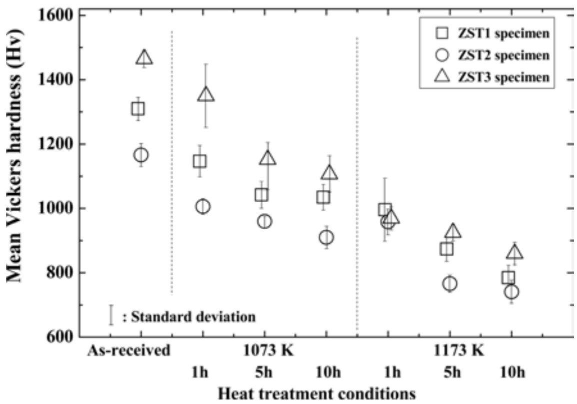

Fig. 2. Mean Vickers hardness according to heat treatment conditions.

the bending strength.

Fig. 3 shows the coefficient of friction of the as-received ZST specimen. Table 4 shows the mean and standard deviation of the as-received ZST specimen. The Z and ZS specimens are shown for comparison.

Table 3. Vickers hardness of ZST1, ZST2 and ZST3 specimen.

Z Specimen	ZST1	ZST2	ZST3
As-received	1310 ± 36	1166 ± 36	1465 ± 27
1073 K-1 hr	1147 ± 49	1006 ± 24	1350 ± 98
1073 K-5 hr	1042 ± 42	960 ± 16	1152 ± 53
1073 K-10 hr	1035 ± 40	910 ± 35	1107 ± 57
1173 K-1 hr	996 ± 98	958 ± 40	969 ± 36
1173 K-5 hr	874 ± 38	766 ± 27	925 ± 26
1173 K-1 0hr	785 ± 39	741 ± 36	860 ± 35

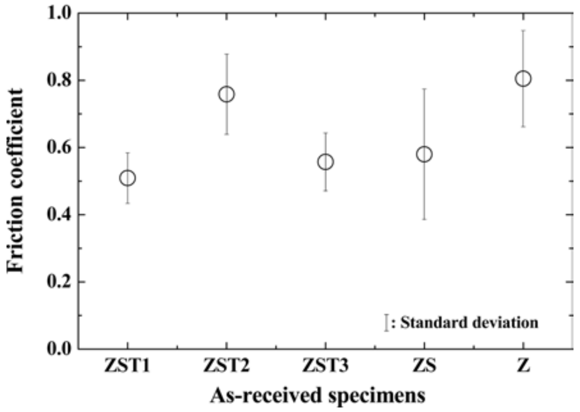

Fig. 3. Friction coefficient according to types of as-received specimen.

Table 4. Friction coefficient of as-received specimens.

ZST1	ZST2	ZST3	ZS	Z
0.5093 ± 0.0753	0.7585 ± 0.1195	0.5573 ± 0.0861	0.580 ± 0.013	0.805 ± 0.143

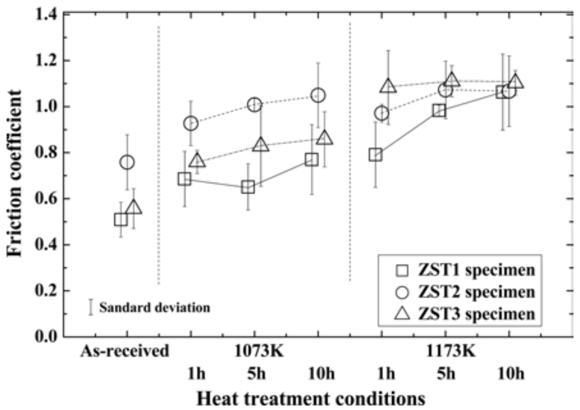

Fig. 4. Friction coefficient according to heat treatment conditions of ZST specimens.

The ZST2 specimens with the smallest bending strength showed the greatest coefficient of friction, while the coefficients of friction of the ZST1 and ZST3 specimens were similar. The Z specimens showed a large coefficient of friction, even though the bending strength was high. The bending strength of the ZS specimen was small, but the coefficient of friction was similar to that of the ZST1 and ZST3 specimens. This demonstrates the effect of the crack-healing property of the SiO_2 oxide formed on the surface of the specimens [20].

Table 5. Friction coefficient of ZST1 specimen.

Specimen	Mean	Std	COV
As-received	0.5093	0.0753	0.148
1073 K-1 hr	0.6865	0.1199	0.183
1073 K-5 hr	0.6510	0.1004	0.154
1073 K-10 hr	0.7703	0.1519	0.197
1173 K-1 hr	0.7915	0.1421	0.179
1173 K-5 hr	0.9836	0.0227	0.023
1173 K-10 hr	1.0630	0.1649	0.155

Table 6. Friction coefficient of ZST2 specimen.

Specimen	Mean	Std	COV
As-received	0.7585	0.1195	0.158
1073 K-1 hr	0.9270	0.0961	0.104
1073 K-5 hr	1.008	0.0280	0.028
1073 K-10 hr	1.0490	0.1402	0.122
1173 K-1 hr	0.9710	0.0389	0.040
1173 K-5 hr	1.0720	0.1253	0.117
1173 K-1 0hr	1.0670	0.1530	0.143

Table 7. Friction coefficient of ZST3 specimen.

Specimen	Mean	Std	COV
As-received	0.5573	0.0861	0.15
1073 K-1 hr	0.7595	0.0502	0.05
1073 K-5 hr	0.8280	0.1739	0.21
1073 K-10 hr	0.8583	0.1195	0.12
1173 K-1 hr	1.0830	0.1609	0.24
1173 K-5 hr	1.1100	0.0682	0.06
1173 K-10 hr	1.1030	0.0536	0.05

Fig. 4 shows the coefficient of friction of the heat treated ZST specimen. The open square (□), open circle (○), and open triangle (△) symbols represent the ZST1, ZST2, and ZST3 specimens, respectively. Tables 5-7 show the mean, Std, and COV according to mathematical statistics. Regardless of the type of specimen, the heat treatment temperature is high and the friction coefficient increased as the heat treatment time increased. The friction coefficient of the as-received ZST1 specimen was 0.5093, and the coefficient of friction of the heat treated specimens at 1073 K and 1173 K was 1.28 to 1.51 times and 1.55 to 2.9 times greater than that of the as-received ZST1 specimen, respectively. Thus, the coefficient of friction was increased according to the heat treatment temperature and time. This increase is caused by the oxides formed on the surface of the specimens.

Fig. 5 shows the relationship between the mean friction coefficient and the mean Vickers hardness of the ZST specimen. The symbol nomenclature are the same as those for Fig. 4 above. The ZS (▼) and Z (◆) specimens are shown for comparison with the ZST specimen. The friction coefficients of all specimens were inversely related to Vickers hardness. In the

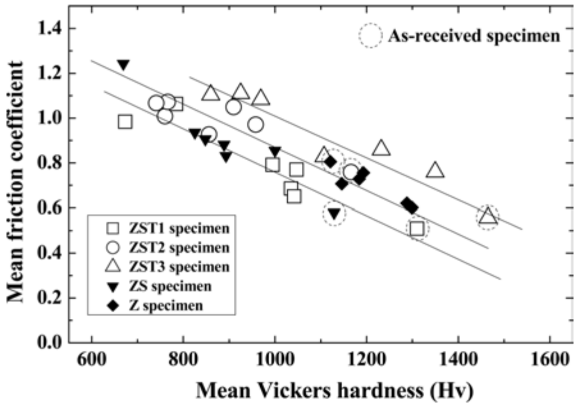

Fig. 5. Relationship of between mean friction coefficient and mean Vickers hardness.

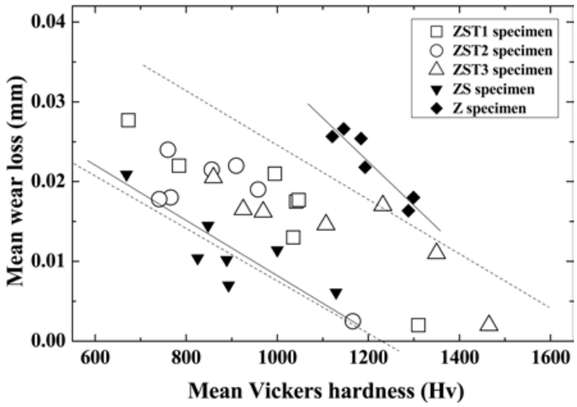

Fig. 7. Relationship of between mean wear loss and mean Vickers hardness.

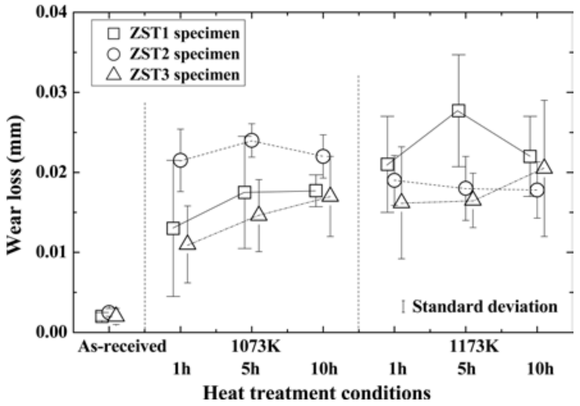

Fig. 6. Wear loss according to heat treatment conditions.

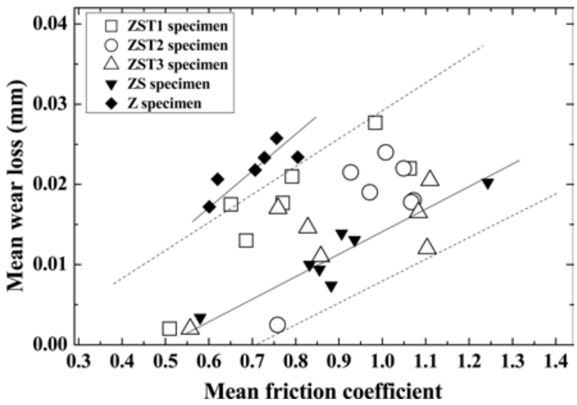

Fig. 8. Relationship of between mean wear loss and mean friction coefficient.

Table 8. Wear loss of ZST1, ZST2 and ZST3 specimen.

Z Specimen	ZST1	ZST2	ZST3
As-received	0.0020 ± 0.0005	0.0025 ± 0.0005	0.0020 ± 0.0010
1073 K-1 hr	0.0130 ± 0.0085	0.0215 ± 0.0021	0.0110 ± 0.0048
1073 K-5 hr	0.0175 ± 0.0070	0.0240 ± 0.0039	0.0146 ± 0.0045
1073 K-10 hr	0.017 ± 0.0020	0.0220 ± 0.0027	0.0170 ± 0.0050
1173 K-1 hr	0.0210 ± 0.0060	0.0190 ± 0.0031	0.0162 ± 0.0070
1173 K-5 hr	0.0277 ± 0.0070	0.0180 ± 0.0040	0.0165 ± 0.0034
1173 K-10 hr	0.0220 ± 0.0050	0.0178 ± 0.0035	0.0205 ± 0.0085

figure, the dotted circle symbol shows each as-received specimen. The Vickers hardness of the as-received specimen was the largest except for the Z specimen, and the coefficient of friction was the smallest. The coefficient of friction of the ZST1 specimen was smaller than that of the ZST2 and ZST3 specimens. On the other hand, the ZST3 specimen showed the largest friction coefficient distribution. The friction coefficient of the Z specimen was large compared to the size of the Vickers hardness. The ZS specimen showed low bending strength and low Vickers hardness due to the addition of SiC. It also had a lower coefficient of friction than the ZST specimen. This result is due to the effect of the oxide formed on the surface by heat treatment.

Fig. 6 shows the relationship between the heat treatment conditions and the wear loss of the ZST specimen. The symbol nomenclature are the same as those for Fig. 4 above. Table 8 shows the mean, Std, and COV according to the mathematical statistics. The wear loss of the three types of as-received specimens was very small, and the standard deviation was also small. However, the wear loss and deviation of the heat-treated specimens were large due to the presence of oxides. Since the amount of oxide produced on the surface differs depending on the specimen, the deviation in the wear loss also differed. The wear loss increased with the heat treatment time at each heat treatment temperature. The wear loss at 1073 K was the smallest in the ZST3 specimen, and the ZST2 specimen was the most abrasive. On the other hand, the wear loss at 1173 K was the highest in the ZST1 specimen, and it decreased in the 10 h specimen. This is because the oxides lubricated and suppressed the progress of wear. The wear losses of the ZST2 and ZST3 specimens were almost the same regardless of the heat treatment time, while the wear losses of the 10h specimens were similar to those of the ZST1~3 specimens.

Fig. 7 shows the relationship between the mean

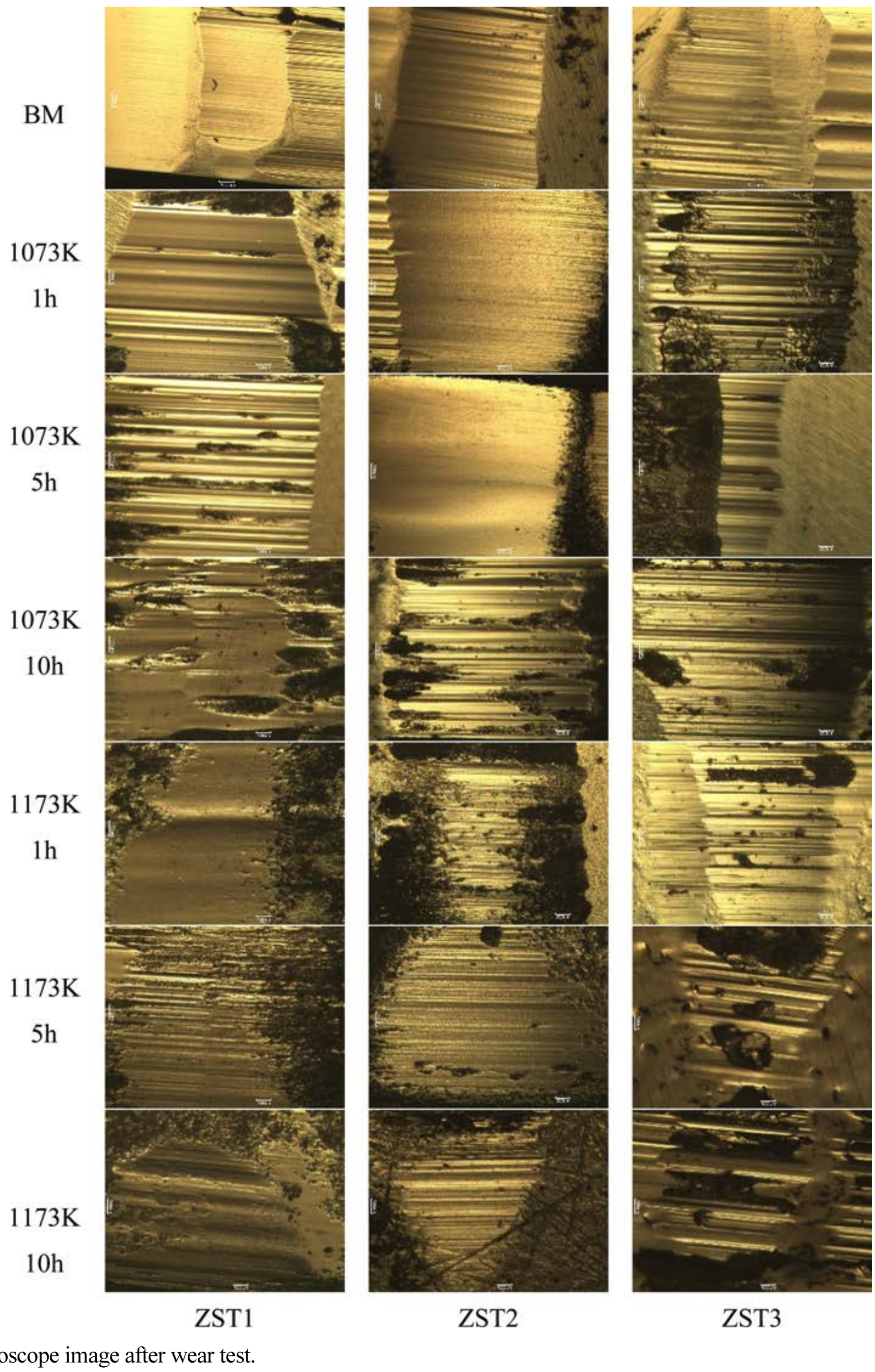

Fig. 9. Optical microscope image after wear test.

Vickers hardness and the mean wear loss of the ZST specimen. The symbol nomenclature are the same as those for Fig. 4 above. The ZS (▼) and Z (◆) specimens are shown for comparison with the ZST specimen. [19] The wear losses of all specimens were inversely proportional to the Vickers hardness. That is, the wear loss of each specimen decreased as the hardness increased. The wear loss of the ZST specimen was concentrated in the band indicated by the dotted line. However, the ZS and Z specimens showed a hardness tendency similar to that of the bending strength; the wear loss also showed the same tendency.

Fig. 8 shows the relationship between the mean friction coefficient and the mean wear loss of the ZST specimen. The wear loss of all specimens was proportional to the friction coefficient. The coefficient of friction of the ZST specimens was concentrated in the band indicated by the dotted line. The Z specimen with the highest bending strength showed higher wear loss at the same friction coefficient, but the wear loss of the ZS specimen was the most small. The high strength of the Z specimen is due to the abrasive adhesion wear caused by friction. The friction coefficient of the ZS specimen decreased due to the lubricating action of the oxide layer.

Fig. 9 shows the optical microscope images of the specimens after wear testing. Figs. 9(a), 9(b), and 9(c) show the ZST1, ZST2, and ZST3 specimens, respectively. Only few worn areas were found in the counterpart materials. However, the wear areas in the specimens were easily identified, as shown in the figures. The scratches and small dent marks shown in one direction on the worn area indicates abrasive wear behavior. The abrasive wear is the main mechanism (which is caused by micro-shear) that accounts for about 50% of the causes of wear loss.

Conclusions

This study evaluated the mechanical properties and wear characteristics of heat treated $ZrO_2/SiC/TiO_2$ composite ceramics at 1073 K and 1173 K. The following results were obtained. The bending strengths of the ZST specimens were almost the same. The ZST specimens showed a crack-healing ability due to the addition of SiC, and the strength was increased by the addition of TiO_2. The Vickers hardness of the as-received ZST specimen was higher than that of the heat treated specimens. The Vickers hardness of the heat treated specimens decreased with increasing temperature and time. The coefficient of friction of the ZST specimens increased with increasing heat treatment temperature and heat treatment time. The friction coefficients of the ZST specimens were inversely related to Vickers hardness. The wear loss increased with the heat treatment time at each heat treatment temperature. The wear loss of all specimens was inversely proportional to the Vickers hardness, but the wear loss was proportional to the friction coefficient.

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