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Friction and wear properties of spark-plasma-sintered α -AlB₁₂ and SiB₆ powder compacts in water



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ABSTRACT

In our present study, α -AlB₁₂ and SiB₆ powder compact disk specimens were prepared by spark plasma sintering α -AlB₁₂ and SiB₆ powders, respectively, and their X-ray diffraction patterns, microstructures, Knoop hardnesses were examined. Also, the friction and wear properties of the α -AlB₁₂ and SiB₆ powder compact disk specimens were investigated when they were slid against Si₃N₄ ball specimens in water. Both the α -AlB₁₂ and SiB₆ powder compact disk specimens exhibited friction coefficients lower than 0.1 while the blended elemental Al-92.3 at% B and Si-85.7 at% B powder compact disk specimens exhibited friction coefficients between 0.1 and 0.2. The specific wear rates of the α -AlB₁₂ and SiB₆ powder compact disk specimens were lower than those of the blended elemental Al-92.3 at% B and Si-85.7 at% B powder compact disk specimens, respectively.

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1. Introduction

Lubrication with water has been receiving attention in industries related to water pumps, food machinery and medical devices [1] because water is an ecological material. Currently, Si₃N₄-, α-Al₂O₃-, SiC-based ceramics are generally used as the sliding materials in such water-lubricated systems. In particular, a report has shown that Si₃N₄-Si₃N₄ and SiC-SiC tribopairs exhibited friction coefficients lower than 0.01 in water after sliding 1000 m and 4000 m, respectively [2]. However, these Si₃N₄-Si₃N₄ and SiC-SiC tribopairs exhibited friction coefficients higher than 0.5 immediately after the sliding began [2]. These tribopairs also exhibited friction coefficients higher than 0.5 at low sliding speeds under boundary lubricating conditions. Moreover, it has been reported that α -Al₂O₃- α -Al₂O₃ tribopairs exhibited relatively high friction coefficients in water. Saito et al. have reported that hexagonal BN-hexagonal BN tribopairs exhibited friction coefficients lower than 0.1 because low friction H₃BO₃ films with layered crystal structures had been formed on the worn surfaces through the following reactions (1) and (2) [3,4].

$$2BN + 3H_2O \rightarrow B_2O_3 + 2NH_3 \tag{1}$$

$$B_2O_3 + 3H_2O \rightarrow 2H_3BO_3$$
 (2)

However, the hexagonal BN-hexagonal BN tribopairs exhibited low wear resistance in water. Their specific wear rates were as high as 10^{-4} – 10^{-5} mm³/N m [3,4].

On the other hand, it was found in our previous study that α -AlB₁₂- and SiB₆-based ceramic disk specimens, which were prepared by spark plasma sintering blended elemental Al-92.3 at% B and Si-85.7 at% B powders, respectively, exhibited friction coefficients lower than 0.2 when they were slid against the Si₃N₄, α -Al₂O₃ and SiC ball specimens in water [5]. Also, the α -AlB₁₂- and SiB₆-based ceramic disk specimens in our study exhibited specific wear rates as low as 3.0×10^{-6} mm³/N m. It is considered that the low friction coefficients of the α -AlB₁₂- and SiB₆-based ceramic disk specimens were due to the formation of low friction H₃BO₃ and Al(OH)₃ films with layered crystal structures [6] and low friction Si(OH)₄ gels on the worn surfaces. On the other hand, it is considered that the low specific wear rates of the α -AlB₁₂- and SiB₆-based ceramic disk specimens were due to their hardness higher than that of Si₃N₄ and Al₂O₃ [7–11].

However, the α -AlB₁₂- and SiB₆-based ceramic disk specimens contained a large amount of oxide phases because the blended elemental active Al-92.3 at% B and Si-85.7 at% B powders absorbed a small amount of oxygen during the spark plasma sintering although the sintering was performed in an Ar gas atmosphere. It is considered that sintering α -AlB₁₂ and SiB₆ powders, not blended elemental powders, reduces the oxygen contamination because the α -AlB₁₂ and SiB₆ forming reactions from the blended elemental Al-92.3 at% B and Si-85.7 at% B powders may promote their respective oxidation reactions. If the α -AlB₁₂ and SiB₆ forming reactions are exothermic, the temperatures of the blended elemental Al-92.3 at% B and Si-85.7 at% B powders increase. The increase in the powder temperature generally promotes the oxidation reaction of the powder sample. On the other hand,

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 $\rm H_3BO_3$ films could not be detected on the worn surfaces of the $\alpha\textsc{-}AlB_{12}\textsc{-}$ and $SiB_6\textsc{-}based$ ceramic disk specimens by x-ray photoelectron spectroscopy (XPS). Before the XPS analyses, both the $\alpha\textsc{-}AlB_{12}\textsc{-}$ and $SiB_6\textsc{-}based$ ceramic disk specimens were cleaned in a mixture of 50 vol% acetone and 50 vol% petroleum benzene for 1.2 ks using an ultrasonic cleaner [12,13], and this cleaning might have removed the H_3BO_3 films.

In our present study, α -AlB₁₂ and SiB₆ powder compact disk specimens were respectively prepared by spark plasma sintering commercially available α -AlB₁₂ and SiB₆ powders, and their x-ray diffraction patterns, microstructures, Knoop hardnesses and friction and wear properties in water were examined.

2. Experimental procedures

The α -AlB₁₂ and SiB₆ powder compact disk specimens with a diameter of 15 mm and a thickness of 3 mm were prepared in our present study by spark plasma sintering 99.8% pure α -AlB₁₂ and 98% pure SiB₆ powders (Furuuchi Chemical Co., Ltd.), respectively, using a Sumitomo Coal Mining SPS-515 spark plasma sintering machine under the conditions shown in Table 1. Blended elemental Al-92.3 at% B and Si-85.7 at% B powder compact disk specimens were also prepared by spark plasma sintering as references. The Al-92.3 at% B and Si-85.7 at% B correspond to the compositions of α -AlB₁₂ and SiB₆, respectively [14,15]. The blended Al-92.3 at% B powder was prepared by blending 99.9% pure Al powder (Rare Metallic Co., Ltd.) with a particle size of 3 µm and 99.9% pure B powder (Furuuchi Chemical Co., Ltd.) with a particle size of less than 1 μm in a hexane-filled standard ball mill for 18 ks. In the same way, the blended Si-85.7 at% B powder was prepared by blending the B powder and 99.99% pure Si powder (Furuuchi Chemical Co., Ltd.) with a particle size of less than 104 μm . After the spark plasma sintering, the surface of each disk specimen was polished using diamond paste with a particle size of $1 \mu m$. Then all of the disk specimens were cleaned in a mixture of 50 vol% acetone and 50 vol % petroleum benzene (Kanto Chemical Co., Inc.) for 1.2 ks using an ultrasonic cleaner [12,13]. The Knoop hardness of each disk specimen was measured 20 times using a Shimadzu HMV-1 hardness tester at a load of 9.8 N and a loading time of 15 s while the x-ray diffraction pattern of each disk specimen was investigated using a Rigaku RINT-2000 X-ray diffractometer. In addition, the cross sections of each disk specimen were observed using a JEOL JSM-7400F scanning electron microscope (SEM) with an energy dispersive spectroscopy (EDS) attachment. Furthermore, differential thermal analysis (DTA) curves of the blended elemental Al-92.3 at% B and Si-85.7 at% B powders in an Ar gas atmosphere were obtained using a SETARAM TG-DTA 96 instrument at a heating speed of 10 °C per minute in order to investigate the respective AlB₁₂ and SiB₆ forming reactions.

The friction and wear properties of each disk specimen sliding against the Si_3N_4 ball specimen were investigated twice using a Rhesca FPR 2001 rotating ball-on-disk tribometer. The diameters of the Si_3N_4 ball specimens were 9.52 mm, and their micro-Vickers hardness at a load of 9.8 N for a loading time of 15 s was 16.4 GPa. In each friction and wear test, the disk specimen was immersed in 100 ml of water, and the water was not replenished during the

test. The friction and wear testing conditions were as follows; load: 9.8 N, sliding speed: 1.57 mm/s, testing time: 3.6 ks, and temperature: 25 °C. The specific wear rates of the disk specimens and the ball specimens were calculated using a Tokyo Seimitsu SURFCOM surface profilometer and a Shodensya GR3400J optical microscope [12,13,16–18]. In addition, the worn surfaces of each disk specimen were observed using a JEOL JSM-7400F SEM with an EDS attachment. Also, the worn surfaces of the α -AlB $_{12}$ and SiB $_{6}$ powder compact disk specimens were examined using a Thermo VG Theta Probe XPS.

3. Results

Fig. 1 shows the DTA curves of the blended elemental Al-92.3 at 8 B and Si-85.7 at 8 B powders. Both of the DTA curves contained exothermic reactions, which indicate that the sintering of the α -AlB₁₂ and SiB₆ powders, not the blended elemental powders, would reduce the oxygen contamination because the α -AlB₁₂ and SiB₆ forming exothermic reactions are thought to increase the sample temperature and promote the oxidation reactions of the blended elemental powders. Al, B and Si contents oxidize very easily, and that their oxidation rates increase with the temperature as shown in an Ellingham diagram [19].

Fig. 2 shows the XRD patterns of the α -AlB₁₂ and SiB₆ powder compact disk specimens and the blended elemental Al-92.3 at% B and Si-85.7 at% B powder compact disk specimens. The blended elemental Al-92.3 at% B powder compact disk specimen was composed of α -AlB₁₂ and α -Al₂O₃ phases. It is considered that the α -Al₂O₃ phase was formed by the oxidation reaction of Al content in the blended elemental powder during sintering. On the other hand, only the α -AlB₁₂ phase was observed in the α -AlB₁₂ powder compact disk specimen, which indicates that sintering the α -AlB₁₂ powder restrained the oxygen contamination more than

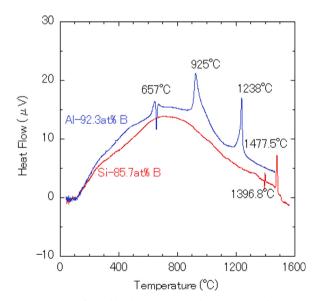


Fig. 1. DTA curves of the blended elemental Al-92.3 at% B and Si-85.7 at% B powders in an Ar gas atmosphere. The heating rate was 10 $^{\circ}$ C per minute.

Table 1 Spark plasma sintering conditions of α -AlB₁₂ and SiB₆ powder compact disk specimens.

Specimen	Sintering temperature (°C)	Sintering pressure (MPa)	Sintering time (min)	Atmosphere
α-AlB ₁₂ powder compact Blended elemental Al-92.3 at% B powder compact	1800	40	20	Ar gas at 0.10 MPa
SiB ₆ powder compact Blended elemental Si-85.7 at% B powder compact	1700			

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