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resulting in low specific wear rates (  $< 5 \times 10^{-9} \text{ mm}^2/\text{N}$ ).

# Tribological behavior of RH ceramics made from rice husk sliding against stainless steel, alumina, silicon carbide, and silicon nitride



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#### ARTICLE INFO

#### ABSTRACT

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#### 1. Introduction

The annual global production of rice is more than 650 million tons [1,2], and the main by-products of rice milling are rice husk and rice bran. Each ton of paddy rice yields approximately 220 kg of rice husk. Both rice husk and rice bran are rich in organic compounds such as cellulose and lignin, but only rice husk is rich in amorphous silica (15–22 mass% of the rice husk) [3–5]. Because of such a high percentage of silica blended with lignin, rice husk is resistant to biodegradation and is difficult to dispose of by conventional methods such as dumping or burning. Hence, rice husk presents an enormous disposal problem for industries worldwide and will continue to cause substantial ecological hazards until a better way to utilize it is discovered. Till date, traditional use of rice husk are related to agriculture, and a few notable industrial sectors have attempted to utilize rice husk, such as the steel industry as a lagging material, the water treatment industry as a clarifying filter, and the energy industry as biomass fuel. In contrast, Hokkirigawa et al. have developed a novel tribomaterial made from rice bran, called RB ceramics, by carbonizing a mixture of defatted rice bran and phenol resin in an inert gas atmosphere at 900 °C [6-8]. RB ceramics are a carbon-based material and show low friction and high wear resistance behavior under dry conditions. Because of the excellent tribological performance and the low cost of manufacturing, RB ceramics are used in

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several applications, including a dry linear sliding bearing and a sleeve of dry stainless chains. However, the presence of silicon (Si) greatly affects the tribological performance of some Si-containing tribomaterials, such as silicon nitride ( $Si_3N_4$ ) and Si-containing diamond-like carbon [9–15].

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The tribological behavior of rice husk (RH) ceramics, a hard, porous carbon material made from rice husk,

sliding against stainless steel, alumina, silicon carbide, and silicon nitride (Si<sub>3</sub>N<sub>4</sub>) under dry conditions

was investigated. High hardness of RH ceramics was obtained from the polymorphic crystallinity of silica.

The friction coefficients for RH ceramics disks sliding against  $Si_3N_4$  balls were extremely low ( < 0.1),

irrespective of contact pressure or sliding velocity. Transfer films from RH ceramics formed on Si<sub>3</sub>N<sub>4</sub> balls.

Wear-mode maps indicated that the wear modes were powder formation under all tested conditions,

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As an efficient use of rice husk, the development of a novel tribomaterial based on rice husk was considered to be promising. Therefore, rice husk ceramics, called RH ceramics, have been developed [16]. RH ceramics are manufactured by a two-stage carbonization of a mixture of rice husk and phenol resin in an inert gas atmosphere which is based on the RB ceramics preparation process. RH ceramics are a hard, porous carbon material containing silica. In a previous study, the tribological behavior of an RH ceramics disk against an austenitic stainless steel ball under dry and water-lubricated conditions was investigated [17]. The experimental results showed that RH ceramics show lower friction coefficients ( < 0.11) and lower specific wear rate (  $< 1.5 \times$  $10^{-9}$  mm<sup>2</sup>/N) under dry conditions. Slightly higher friction and wear were obtained under water-lubricated conditions. Furthermore, the effects of first-stage carbonization temperature on the friction and the wear behavior of an RH ceramics disk against a stainless steel ball were investigated [18]. RH ceramics manufactured at a first-stage carbonization temperature of 900 °C demonstrate superior tribological behavior than those manufactured at 900 °C, 1400 °C and 1500 °C. In addition, the effects of counterpart materials were revealed in another study [19]. Compared to alumina (Al<sub>2</sub>O<sub>3</sub>) balls, when sliding against high carbon-chromium bearing steel balls and austenitic stainless steel balls, RH ceramics manufactured at the first-stage carbonization

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temperature of 900 °C display superior properties, such as low frictions ( < 0.11) and low specific wear rates ( <  $1.0 \times 10^{-8}$  mm<sup>2</sup>/N). As described above, RH ceramics manufactured at first-stage carbonization temperature of 900 °C have been expected to be utilized as a dry sliding material against steel materials. However, with increasing carbonization temperature, they exhibit the graphitization of amorphous carbon and the crystallization of amorphous silica [19]. Therefore, RH ceramics carbonized at high temperatures will have low friction and low wear against hard materials. The effect of specific counterpart materials on the tribological behavior of RH ceramics carbonized at high temperatures remains unclear, and these are important issues to address for the further development of RH ceramics.

For these reasons, the aims of this study were to investigate the tribological behavior of RH ceramics carbonized at 1400 °C sliding against hard stainless steel and several ceramics under dry conditions, and to discuss the low-friction mechanism.

#### 2. Experimental details

#### 2.1. Material preparation

Fig. 1 shows a schematic diagram of the manufacturing process of RH ceramics. First, milled rice husk powder was mixed with liquid phenol resin. The mass fractions of milled rice husk and liquid phenol resin were 75 mass% and 25 mass%, respectively. Then, the powder mixture was dried at 150–180 °C. After drying, the dried mixture was carbonized in an argon atmosphere. The first-stage carbonization temperature  $(T_1)$  was 400 °C. The carbonized mixture was crushed and screened to a mean particle size of less than 150 µm. Subsequently, the carbonized powder (75 mass%) was mixed again with powdery phenol resin (25 mass%). Then, the mixture was pressed at 100 °C into a disk shape. The molding pressure was 19.6 MPa, and the pressing time was 5 min. Finally, the molded disk was re-carbonized in a furnace at 1400 °C ( $T_2$ ) in an argon atmosphere. The temperature of the furnace was raised to 500 °C at a heating rate of 30 °C/h, followed by 60 °C/h to reach the maximum temperature and held for 2 h. Then, the furnace was cooled to 600 °C at a cooling rate of 30 °C/h, followed by natural cooling.

#### 2.2. Microstructural analysis and mechanical property testing

The microstructural morphologies of RH ceramics samples were observed by scanning electron microscopy (SEM) coupled

with energy dispersive X-ray spectroscopy. Raman spectroscopic analysis was used for the carbon characterization of RH ceramics. Raman spectra were obtained using a micro-Raman spectrophotometer system. X-ray diffraction (XRD) analysis was used to characterize the phase of the constituents of RH ceramics. The XRD patterns were collected using Cu K $\alpha$  radiation.

Mechanical properties of RH ceramics such as density, elasticity, hardness, and surface roughness were measured. The bulk density was obtained as a ratio of the measured weight and the bulk volume of the samples. The compression tests were conducted using a uniaxial compression testing apparatus to obtain Young's modulus. RH ceramics samples were cut to  $4.9 \text{ mm} \times 5.0 \text{ mm} \times 4.9 \text{ mm}$  size, with flat end. The rate of loading of the compression tests was 0.01 mm/s. The hardness values of RH ceramics were determined by micro-Vickers indentation method under the load of 0.98 N, the loading time of 20 s, and the number of measurements of 20 data points. The values of surface roughness were measured using a profilometer.

#### 2.3. Tribological testing

Friction tests were conducted using a ball-on-disk-type friction tester under dry conditions in a laboratory. Fig. 2 illustrates the testing apparatus, which consisted of a moving stage, an arm with a ball holder, and a controller. A disk specimen was attached to the underlying stage. A ball specimen was fixed to the upper arm with a ball holder. Then, the stage was moved linearly and perpendicularly against the arm. RH ceramics samples were used in the form of disk specimens. High carbon martensitic stainless steel (JIS SUS440C), Al<sub>2</sub>O<sub>3</sub>, SiC, and Si<sub>3</sub>N<sub>4</sub> were used as ball specimens. Table 1 lists the mechanical properties of the ball specimens. Before each sliding test, the ball and the disk specimens were ultrasonically cleaned in hexane for 5 min, and then dried under a low-vacuum atmosphere.

Table 2 lists the experimental conditions. Each test was conducted with a constant ball specimen having a radius of 4 mm. The normal loads were 1.0, 2.0, 4.9, and 9.8 N. The sliding velocities were 1, 5, and 10 mm/s. Each test was conducted for  $2 \times 10^4$  cycles of repeat passages under dry conditions with a laboratory temperature of 22 °C and relative humidity of 46%.

Friction force was measured using a load cell coupled to the upper arm, and each friction coefficient was calculated on the basis of the friction force. The values of wear volume and specific wear rate for each disk specimen were calculated on the basis of



Fig. 1. Manufacturing process for RH ceramics.

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