Short Communication

The Effect of Carbonizing Temperature on Friction and Wear Properties of Hard Porous Carbon Materials Made from Rice Husk

Tuvshin Dugarjav*, Takeshi Yamaguchi, Shohei Katakura and Kazuo Hokkirigawa

Graduate School of Engineering, Tohoku University,
6-6-01 Aramaki Aza-Aoba, Aoba-ku, Sendai, Miyagi 980-8579, Japan
*Corresponding author: tuvshin@gdl.mech.tohoku.ac.jp

(Manuscript received 24 September 2008; accepted 7 January 2009; published 15 January 2009)

The new hard porous carbon material called the RH (rice husk) ceramics has been developed by carbonizing rice husk as the main raw material. In order to clarify the friction and wear properties of RH ceramics, a study of the effect of carbonization temperature on the mechanical and tribological properties of RH ceramics was undertaken. RH ceramics samples carbonized at 900, 1400, and 1500 °C were prepared for this study. The RH ceramics samples were inspected using X-ray diffraction, EDX and scanning electron microscopy. Mechanical properties of RH ceramics such as Vickers hardness, compressive strength, and bulk density were also studied. The RH ceramics carbonized at 1500 °C had much higher porosity compared to the ones carbonized at lower temperatures. In addition, the bulk density and compressive strength decreased with increasing carbonization temperature. Whereas, the hardness was higher for the RH ceramics carbonized at higher temperatures. Ball-on-disk sliding tests showed that the RH ceramics carbonized at the conventional 900 °C have better friction and wear properties compared to the ones carbonized at 1400 and 1500 °C under dry condition sliding against austenitic stainless steel (JIS SUS304). The friction coefficient and specific wear rate took very low values of 0.07 and less and 5.0×10^-10 mm³/N and less, correspondingly, for the RH ceramics carbonized at 900 °C. Based on the experimental results, friction and wear mechanisms have been summarized for each carbonization temperature.

Keywords: friction, wear, carbonization temperature, RH ceramics

1. Introduction

The worldwide production of rice husk is about 120 million tons per year\(^1\). That makes rice husk one of the largest readily available but also one of the most under-utilized resources.

Increase of environmental awareness has led to a growing interest in researching ways of an effective utilization of rice by-products, from which rice husk is particularly valuable due to its high content of amorphous silica\(^1,2\).

The new hard porous carbon material called the RH (rice husk) ceramics has been developed by carbonizing rice husk as the main material at 900 °C\(^2\). It was found that RH ceramics contain amorphous silica in addition to amorphous carbon as the main constituents\(^2\). The silica in the rice husk undergoes structural transformations depending on the temperature of a heat treatment. At temperatures around 1400 °C a phase change of silica from amorphous to crystalline takes place\(^3,4\). It is possible that such phase transitions have significant effect on mechanical and tribological properties of RH ceramics.

The purpose of this study is to clarify the effect of carbonization temperature on the tribological properties of RH ceramics sliding against austenitic stainless steel (JIS SUS304) under dry condition.

2. Preparation of RH ceramics

Figure 1 shows a schematic diagram of the preparation process of RH ceramics. First, the mixture of milled rice husk and phenol resin in powder form was carbonized in nitrogen gas. In this study 900, 1400, and 1500 °C were selected for the initial carbonization temperature \((T_1)\). The carbonized powder was then crushed and the resulting powder was screened with 145 (JIS) mesh screen to a particle size of below 106 µm. Subsequently, the carbonized powder was mixed again with the phenol resin and was carbonized once more (at \(T_2=900\) °C) in nitrogen gas after being formed. The final product is the hard porous carbon material RH ceramics.
Table 1 Friction test experimental conditions

<table>
<thead>
<tr>
<th>Normal load $W$, N</th>
<th>0.98, 1.96, 4.9, 9.8</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sliding velocity $v$, m/s</td>
<td>0.02, 0.05, 0.1, 0.5, 1.0</td>
</tr>
<tr>
<td>Number of repeat passages $N$, cycles</td>
<td>$2 \times 10^4$</td>
</tr>
<tr>
<td>Lubrication condition</td>
<td>Dry</td>
</tr>
</tbody>
</table>

3. Experimental results

Compressive strength measurements were carried out with $5 \, \text{mm} \times 5 \, \text{mm} \times 12.5 \, \text{mm}$ size samples using a compression testing apparatus. Hardness of the RH ceramics specimens was determined by the Vickers indentation method under the load of 0.245 N. The X-ray diffraction patterns of the RH ceramics specimens were collected using Cu Kα radiation. The tube current and voltage were 40 mA and 40 kV, respectively. The scan step time was 1 s and the step size was 0.02°. The 2θ range examined was 20 to 90°.

Friction and wear tests were conducted using a ball-on-disk tribometer, as shown in Fig. 2, under dry condition in air. The tests were carried out under a wide range of sliding velocities and normal loads, as shown in Table 1. The RH ceramics disk specimens were cut to $35 \, \text{mm} \times 35 \, \text{mm} \times 5 \, \text{mm}$ size and pre-polished to a surface roughness of 0.7 µm $R_a$ to 1.0 µm $R_a$. Austenitic stainless steel SUS304 balls with a diameter of 8 mm and a surface roughness of 0.14 µm $R_a$ were used as a counterpart material for the sliding tests.

Fig. 1 Schematic diagram of preparation process of RH ceramics

Fig. 2 Schematic diagram of friction test apparatus

(a) $T_1=900 \, ^\circ\text{C}$  (b) $T_1=1400 \, ^\circ\text{C}$  (c) $T_1=1500 \, ^\circ\text{C}$

Fig. 3 Surface profiles, SEM images, and EDX mappings of C, O, and Si of RH ceramics carbonized at different temperatures

Table 1. The RH ceramics disk specimens were cut to $35 \, \text{mm} \times 35 \, \text{mm} \times 5 \, \text{mm}$ size and pre-polished to a surface roughness of 0.7 µm $R_a$ to 1.0 µm $R_a$. Austenitic stainless steel SUS304 balls with a diameter of 8 mm and a surface roughness of 0.14 µm $R_a$ were used as a counterpart material for the sliding tests.
The Effect of Carbonizing Temperature on Friction and Wear Properties of Hard Porous Carbon Materials Made from Rice Husk

4. Results and discussion

4.1. Structural and mechanical properties

Figure 3 shows the scanning electron micrographs (SEM), the surface profiles, and EDX analysis of RH ceramics carbonized at different temperatures. It can be seen in Fig. 3 that the RH ceramics samples carbonized at 1500 °C have highly porous structure in contrast to the samples carbonized at 900 and 1400 °C. The EDX analysis, as shown in Fig. 3, suggest existence of silica and carbon as the main constituents in RH ceramics carbonized at different temperatures.

Figure 4 illustrates X-ray diffraction profiles of the RH ceramics samples carbonized at different temperatures. The appearance of a broad peak centered at the 2θ angle of 24° in the XRD patterns of the sample carbonized at 900 °C indicates the presence of amorphous silica. On the other hand, the XRD patterns of the RH ceramics samples carbonized at 1400 and 1500 °C show sharp diffraction peaks, characteristic to cristobalite, the high temperature polymorph of silica, superimposed on a broad background caused by the remaining amorphous carbon and silica.

The Vickers hardness of RH ceramics increased with an increase of the carbonization temperature as shown in Fig. 5. The changes in hardness values are likely the consequence of the alteration in a crystalline structure of the silica. The hardness increase of RH ceramics carbonized at 900 and 1400 °C resulted from the change of crystalline structure of amorphous silica to its relatively harder crystalline form. Consequently, with higher carbonizing temperature of 1500 °C, the amount of crystalline silica is believed to increase, therefore resulting in further increase of overall hardness value.

The bulk density of RH ceramics decreased with increasing carbonization temperature. It was $1.35 \times 10^3$ kg/m$^3$ for the RH ceramics carbonized at 900 °C, $1.28 \times 10^3$ kg/m$^3$ and $1.2 \times 10^3$ kg/m$^3$ for the RH ceramics carbonized at 1400 and 1500 °C, correspondingly, as shown in Fig. 6. This decrease in density and, consequently, the increase in porosity, such as the particularly large pores observed in Fig. 3 for the RH ceramics samples carbonized at 1500 °C, were

Fig. 4 X-ray diffraction patterns of RH ceramics carbonized at different temperatures

Fig. 5 Vickers hardness of RH ceramics carbonized at different temperatures

Fig. 6 Bulk density of RH ceramics carbonized at different temperatures

Fig. 7 Compressive strength of RH ceramics carbonized at different temperatures

Fig. 8 The variation of friction coefficient with number of repeat passages for RH ceramics carbonized at different temperatures ($W=9.8$ N, $v=1.0$ m/s)
Fig. 9  Distribution of friction coefficient of RH ceramics carbonized at different temperatures as a function of sliding velocity and normal load

Friction coefficient was the lowest for the RH ceramics carbonized at 900 and 1400 °C. Whereas, the friction coefficient was the highest for the RH ceramics carbonized at 1500 °C. Whereas, it decreased for the specimens carbonized at 1500 °C well corresponding with the much lower bulk density as shown in Fig. 7.

4.2. Friction and wear properties under dry condition

Figure 8 demonstrates the variation of the friction coefficient with number of repeat passages of RH ceramics carbonized at different temperatures. The friction coefficient after starting at rather high level rapidly decreased and reached its steady state condition. It can be seen in Fig. 8 that the friction coefficient was less than 0.1 for the RH ceramics carbonized at 900 and 1400 °C and it was at relatively high value around 0.15 for the RH ceramics carbonized at 1500 °C.

Figure 9 shows the distribution of friction coefficient as a function of sliding velocity and normal load for RH ceramics carbonized at different temperatures. The friction coefficient was the lowest for the RH ceramics carbonized at 900 °C displaying very low values of less than 0.07 at low sliding speeds. The friction coefficient, however, mostly stayed within 0.09 to 0.11 for the RH ceramics carbonized at 900 and 1400 °C. The friction coefficient was the lowest for the RH ceramics carbonized at 1400 °C. Whereas, the specific wear rate was comparable for the RH ceramics carbonized at 1400 °C at most conditions.

Fig. 11 Effect of carbonization temperature on friction coefficient of RH ceramics under dry condition
only reaching $5.0 \times 10^{-9}$ mm$^2$/N and more at lower normal loads. And the specific wear rate was highest for the RH ceramics carbonized at 1500 °C with the low values from $1.5 \times 10^{-9}$ to $2.5 \times 10^{-9}$ mm$^2$/N and going as high as $5.0 \times 10^{-9}$ mm$^2$/N and over.

Figures 11, 12 and 13 show consolidated results of the sliding tests of the effect of the carbonizing temperature $T_1$ on the friction coefficient, specific wear rate of RH ceramics disk and specific wear rate of SUS304 ball specimens, correspondingly. The friction coefficient and specific wear rate of RH ceramics and the counterpart material increased with increased carbonization temperature. That increase was small from 900 to 1400 °C showing the effect of the change in the crystalline structure of the silica in RH ceramics. On the other hand, the friction coefficient and specific wear rate drastically increased from 1400 to 1500 °C showing the effect of the very high porosity increase and the reduction in mechanical strength.

Figure 14 shows the typical appearance of the worn surfaces of the SUS304 ball specimens. The SEM observations revealed that the worn surfaces of the counterpart SUS304 steel balls of the RH ceramics samples carbonized at 900 and 1400 °C were covered with a film like formation of transferred materials. Though, in case of the RH ceramics carbonized at 1500 °C, the corresponding worn surfaces of the steel balls displayed little to no deposit of such transferred materials and were larger and seemingly much rougher with deep scratch marks, indicating abrasive wear of the ball.

Figure 15 shows the typical appearance of worn
Fig. 16 Schematic diagram of sliding contact interface of RH ceramics carbonized at different carbonizing temperatures

surfaces of RH ceramics specimens after the friction tests. As it can be seen from the SEM images in Fig. 15, the worn surface of the RH ceramics carbonized at 1500 °C appeared much rougher than that of the RH ceramics specimens carbonized at 900 and 1400 °C. The much rougher surface with higher hardness produced abrasive wear of the counterpart ball surface resulting in little to no deposit of film like transferred materials.

Based on the above mentioned results, the friction and wear mechanisms of RH ceramics sliding against SUS304 under dry condition were summarized as shown in Fig. 16. It can be concluded that the friction and wear properties of RH ceramics under dry condition were closely related with the presence of the film like formation of transferred materials on the worn surfaces of ball specimens. And it is considered that the film was transferred from RH ceramics and, therefore, consists of amorphous carbon and amorphous silica, and is formed more consistently on the counterpart steel surfaces sliding against the RH ceramics carbonized at 900 and 1400 °C, resulting in very low friction and wear. And in case of the RH ceramics carbonized at 1500 °C, the film is not able to form regularly due to increased wear and is constantly removed by much rougher counterpart surface.

It is also believed that tribochemical reactions of silica in the contact interface greatly benefits low friction of RH ceramics sliding against a steel counterpart, similarly to Si-DLC/steel contact interfaces[5]. However, the detailed mechanisms of such interactions are subject of further studies. On the other hand, the inclusion of the crystalline structured silica in the contact interface possibly causes slight increase of the coefficient of friction and specific wear rate for the RH ceramics carbonized at 1400 °C due to increase in overall hardness. Whereas, in case of the RH ceramics carbonized at 1500 °C, the increased amount of crystalline silica; the particularly high porosity, as was shown in Fig. 3; and, consequently, the low mechanical strength, as was shown in Fig. 7, lead to increased wear, particularly of the steel counterpart surfaces, and, hence, to inability to form a consistent transferred film which results in much higher friction coefficient values.

5. Conclusions

(1) The hardness of RH ceramics increased, whereas, the bulk density and compressive strength decreased with increasing carbonization temperature.

(2) The RH ceramics carbonized at 900 °C demonstrated the lowest friction coefficient of 0.07 and less and the specific wear rate of 5.0×10⁻¹⁰ mm²/N and less under dry condition than the ones carbonized at 1400 and 1500 °C due to the presence of the transferred film consisting of amorphous silica and amorphous carbon from RH ceramics. The increased friction coefficient and wear rate of the RH ceramics carbonized at high temperatures were closely related with the higher hardness of crystalline silica as well as with low mechanical strength and high porosity in case of the RH ceramics carbonized at 1500 °C.

6. References


