Water Resistance of Porous Carbon Materials made from Rice Hull

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The authors have investigated a new utilization of rice-hull from a viewpoint of the recycling. The rice-hull carbon-material (RHS carbon) is manufactured by mixing the rice-hull particles with a phenol resin, pressure forming, drying, and then carbonizing in the temperature range from 1173K to 1773K. Since the most of the inorganic component is Si in the rice hull, the most of the produced inorganic component in the RHS carbon is SiO₂, which is stable under wet or water-ret conditions. Therefore, the RHS carbon is expected to be a high water-resistant material.

In this study, the authors measure the compressive strength of the RHS carbon after water immersing and hygroscopic expansion in a deionized water. The compressive strength did not decrease after the water immersing. Moreover, the hygroscopic expansion of the RHS carbon was considerably low. These results clearly showed the advantage of the RHS carbon, comparing to the similar porous carbon materials made from rice bran.

Key Words : Porous Carbon Materials, Rice Hull, Water Resistance

1. INTRODUCTION

Rice hull is a residual product of rice, and the amount of it is about 2.6 million tons per year in Japan. About 1.7 million tons of rice hull is effectively utilized as feed, fertilizer and soil conditioner, etc. Therefore, about 0.9 million tons of the remainder is disposed of without useful utilization.

The rice hull silica carbon material (RHS carbon) has been developed in order to utilize the rice hull from a viewpoint of recycling. The RHS carbon is expected as the sliding materials such as the linear motion bearing in future, because it has excellent low friction and abrasion resistance under unlubricating conditions. The present authors have already confirmed the high fracture strength and reasonable low Young's modulus of the RHS carbon [3]. The heat resistance is another important properties for the sliding materials. Moreover, since the high dimensional accuracy is important for the precision components such as sliding bearing and sliding guides, it is necessary to keep the low hygroscopic expansion.

The RHS carbon is manufactured by impregnating a phenol resin with the rice hull, and carbonizing it in a nitrogen gas atmosphere. Since the rice hull has a porous structure, the RHS carbon can be a porous carbon material. The present authors have discussed the RB carbon[1]. The rice bran contains P, K and Mg as the inorganic components. Therefore, the RB carbon includes consequently the water soluble chemical compounds such as the phosphate chemical compound and the carbonate chemical compound. These inorganic components reduce the mechanical strength and induce the high hygroscopic expansion. By contrast, the RHS carbon is expected to be a high water resistant material because almost of the inorganic component in it is the silica which is chemically stable under the aqueous environment.

In this study, the compressive strength of the RHS carbon after water immersing and hygroscopic expansion in deionized water were measured, and confirmed the high water resistance of the RHS carbon comparing to the RB carbon. Moreover, the effects of carbonizing temperature on the mechanical properties were discussed.

2. EXPERIMENTAL PROCEDURE

2.1 Materials

Rice hull contains about 20 mass% of inorganic constituent and 80 mass% of organic constituent. The inorganic constituent is silica, which amount is more than 96 mass%. The organic constituents are carbon and hydrogen. The amount of the former is about 90 mass% and the latter is 10 mass%.

Figure 1 shows the making process of the porous carbon material from the rice hull raw material. The RHS carbon is manufactured by impregnating a phenol resin with the rice hull, and carbonizing it at 1173K in a nitrogen gas atmosphere. Then, it is pressure formed, and carbonized again in the temperature range from 1173K to 1773K. Since the rice hull has a porous structure, the RHS carbon can consequently be a porous carbon material.

The geometry of the pressure-formed board was $150 \times 75 \times$

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 5 mm^3 . The test piece for the hygroscopic expansion and the compressive strength were prepared from the board. The geometry of the test piece for the hygroscopic expansion was $50 \times 5 \times 5 \text{ mm}^3$, and for the compressive strength was $10 \times 5 \times 5 \text{ mm}^3$.

Photo 1 shows the macrostructure of the RHS carbon. There are two types of voids in this structure. The large void, with approximately $100\,\mu m$ diameter, is formed among the rice-hull particles. The origin of the small voids, with less than $10\,\mu m$ diameter, is based on the natural porous structure of the rice hull. 2.2 Mechanical Tests

2.2.1 Hygroscopic expansion: Photo 2 shows the test instrument for hygroscopic expansion. The test piece was

maintained in deionized water at room temperature, and the expansion was measured with the immersing time. The displacement of test piece in the deionized water was measured using a laser displacement measuring system (resolution was $1 \mu m$). The displacement was measured by monitoring the position of the metallic column that was put on the head of the test piece.

2.2.2 Compression test after water immersing: Photo 3 shows the testing instrument for compressive strength. The test piece was maintained in deionized water for 1, 5, 10, 50, 100, 500 and 1000 hours, and then dried at 333K for over 12 hours. The compression test was carried out using a universal testing machine



Figure 1 Making process of porous carbon material from rice hull raw material



Photo 2 Test instrument for hygroscopic expansion

(maximum load was 50 kN). The crosshead speed was 0.5 mm/ min. The displacement of test piece was measured using a laser displacement measuring system (resolution was 1μ m).



Photo 3 Test instrument for compressive strength



Figure 2(a) Relation between immersing time and hygroscopic expansion of RHS carbon at 1173K carbonizing



Figure 2(b) Relation between immersing time and hygroscopic expansion of RHS carbon at 1773K carbonizing

3. EXPERIMENTAL RESULTS AND DISCUSSION

3.1 Water Resistance

Figure 2 (a)-(d) show the relationship between the immersing time and the hygroscopic expansion of the RHS carbons at the carbonizing temperature range from 1173K to 1773K. The hygroscopic expansion was about 0.080% for 1173K, and 0.012% for 1773K after about 100 hours. The saturated hygroscopic expansion is decreased with increasing the carbonization temperature. For comparison, Figure 2 (e) shows the hygroscopic expansion of the traditional RB carbon[1], which



Figure 2(c) Relation between immersing time and hygroscopic expansion of RHS carbon at 1573K carbonizing



Figure 2(d) Relation between immersing time and hygroscopic expansion of RHS carbon at 1373K carbonizing



Figure 2(e) Relationship between the immersing time and the hygroscopic expansion of traditional RB carbon [1]

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is made from the rice bran. The traditional RB carbon largely expands by the water immersing. Finally, the hygroscopic expansion is saturated with about 0.2%. The values of 0.015% or 0.012% in RHS carbon are considerably low comparing to the value of 0.2% in the traditional RB carbon.

Figure 3 shows the relationship between the immersing time and the residual compressive strength of the RHS carbon and traditional RB carbon. Before water immersing (0 hours), the compressive strength of the both carbons are almost equal. However, the compressive strength of the RB carbon decreases with increasing the water immersing time, and is approached to about 60% of the virgin value after about 1000 hours. By contrast, the compressive strength of the RHS carbon does not decrease



Figure 3 Relationship between immersing time and compressive strength of porous carbon material



Figure 4 XRD profiles of inorganic component in RHS carbons

even after 1000 hours water immersion. The similar stability was measured at all the carbonizing temperature range from 1173K to 1773K. Namely, the RHS carbon achieves high water resistance.

In general, the amount of clearance between the friction element and shaft is important for the transmission accuracy and the limiting speed for safety in linear motion bearings. Since the RHS carbon has stable under humidity and water-ret conditions, the RHS carbon is advantageous to the precision elements.

Moreover, the Young's modulus is about 16.7 GPa, which is considerably low comparing to many engineering ceramics, for example 310 GPa in silicon nitride. Namely, large elastic deformation is expected for the RHS carbon. This low modulus is also convenient as the material for the friction elements. 3.2

Effect of carbonizing temperature

Figure 4 shows the XRD profiles of the inorganic component in the RHS carbon for 1573K, 1673K and 1773K. Amorphous silica is the major inorganic component in the RHS carbon for 1573K. However, SiC and Si₂N₂O are apparently formed for 1673K and 1773K. Figure 5 shows the amount of inorganic component with



Figure 5 Amount of inorganic component in RHS carbons carbonized from 1173K to 1773K



Figure 6 Relationship between carbonized temperature and compressive strength of RHS carbon

the carbonizing temperature. It is proven that the amount of inorganic component largely decreases at 1773K. The reduction of the amount is considered to be the cause by the sublimation of the Si.

Figure 6 shows the compressive strength with the carbonizing temperature. The amount of the inorganic component was decreased at higher than 1673K as shown in Figure 4. The compressive strength is also decreased at higher than 1673K. The inorganic component is necessary to keep the strength high in the RHS carbon. Therefore, the high strength was obtained from 1173K to 1573K in the RHS carbon. In the RB carbon, the highest strength was obtained for 1173K[3]. The difference between the both materials is explained by the sublimination temperature of the inorganic components in the both materials.

4. CONCLUSIONS

The compressive strength after water immersing and the hygroscopic expansion were measured for RHS carbon, which is made from rice hull. The obtained results were summarized as follows :

(1) The RHS carbon has high water resistance, because the

inorganic component in the rice hull is almost silica.

(2) The hygroscopic expansion of the RHS carbon was considerably low of about 0.015% for about 100 hours.

(3) Compressive strength is kept high even after 1000 hours water immersing for RHS carbon.

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