Effect of carbonization temperature on the basic properties of woodceramics made from carbonized bamboo fiber and liquefied wood

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In this paper, a new woodceramics from carbonized bamboo fiber and liquefied wood was developed and the effects of carbonization temperature on the dimension shrinkage, weight loss, density, compressive strength, and volume electrical resistivity of the woodceramics were investigated. The results show that, with the increase in the carbonization temperature, liquefied wood decompose and then combine with carbonized bamboo fiber, the dimension of sample almost keep constant, but its compressive strength was improved, and its electrical resistivity was reduced.

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1. Introduction

Woodceramics are made from wood or woody material impregnated with phenolic resin being carbonized in vacuum at high temperatures. During the carbonization process, the wood or woody material changes into soft amorphous carbon and the phenolic resin changes into hard glassy carbon. Pores, which originally exist in the wood and woody material, still remain in the woodceramics. Accordingly, substantially woodceramics is a kind of composite of porous amorphous carbon and glassy carbon. The woodceramics show high stiffness, high resistance to corrosion and friction, and electromagnetic shielding ability [1–5].

Regretfully, it is still difficult to produce woodceramics board with a large size (maximum size is $30 \text{ cm} \times 30 \text{ cm} \times 4 \text{ cm}$) for two reasons: first, twist and cracks will be induced when gas produced from decomposition of raw materials rush out from inside; second, phenolic resin, which is main impregnation agent in manufacture of woodceramics, can not release inner stress induced in carbonization because of its thermosetting characteristic. To solve the problem, an attempt was made in this paper that carbonized bamboo fiber and liquefied wood (thermoplastic resin) was used as raw material of woodceramics.

On the other hand, in the past several decades, considerable researches have focused on conversing the waste lignocellulosic resources mentioned above into useful materials, which included "wood liquefaction" technique to converse wood chemically or thermochemically into liquid material [6, 7]. H. Ono et al. described a simple wood liquefaction method, where phenol was applied in the presence of conc. H₂SO₄ to obtain completely liquefied wood [8,9]. In the manufacturing of woodceramics, the wood liquefaction techniques are also of interest because if liquefied wood instead of phenolic resin was used as impregnation agent, the amount of phenol compound used in the production of woodceramics will decrease greatly and the lignocellulosic waste resources could be utilized with maximum efficiency.

In the present study, a new woodceramics which was made from carbonized woody material and liquefied wood, and the effects of carbonization temperature on dimension shrinkage, weight loss, compressive strength and volume electrical resistivity of the woodceramics were also investigated.

2. Experimental

2.1. Material preparation

Fig. 1 shows the schematic illustration of the synthesis of liquefied wood. At 150°C, hinoki (*Chamaecyparis obtusa* Endl.) wood powder and phenol compound was mixed while the sulfuric acid was added as catalyst. The beech wood powder was not performed any treatments such as coating and painting. Fig. 2 shows the external appearance of synthesized liquefied wood. Fiber from bamboo pulp was used in this experiment. It was carbonized at 800°C for 2 hours into soft carbon fiber.

Fig. 3 shows the woodceramics manufacturing methods. Carbonized bamboo fiber at 800°C, was mixed



Figure 1 A schematic illustration of the synthesis of liquefied wood.



Figure 2 Physical appearance of liquifed wood.

with the liquefied wood diluted with ethanol (ratio 2:1), and then, after being dried, it was made into board under the pressure of 5 MPa for 10 minutes at 160°C. The ratio of carbonized bamboo fiber to liquefied wood is 1:1.3. The board was carbonized in vacuum furnace to form woodceramics. Because when being carbonized, the material generated a considerable amount of decomposition products, for example, pyroligneous acid and wood tar, in order to make sure the woodceramics quality, the carbonization was carried out in the vacuum furnace where the decomposition products could be vented off continuously. Carbonization temperatures are 400°C, 500°C, 650°C, and 800°C, respectively.

2.2. Characterization

In order to investigate the effect of the process on dimension shrinkage and weight loss of woodceramics from carbonized bamboo fiber and liquefied wood, the dimension of 25 samples are measured using a pair of digital slide calipers (Mitutoyo, CD-15CP Co., Ltd.) before and after carbonization, respectively. Similarly, the weight changes are also measured using an electrical balance (Kensei, ER120-A Co., Ltd.).

The compressive strength of woodceramics is measured by Shimadzu material testing system. The crosshead speed is 1 mm/minute. The deflection is measured with a dial gauge. The test direction is perpendicular to the pressing direction.

The volume electrical resistivity of woodceramics is measured by Mitsubishi MCP-PR02 resistivity system. In order to avoid the influence of the water content on the electrical resistivity of woodceramics, the specimens are dried at 105°C for 8 hours and keep in dried box at 20°C for 24 hours before measurement.

Microstructures of the resultant woodceramics are observed in Scanning Electron Microscope (S-2250N) operated at 20 kV and 20 mA.

3. Results and discussion

Fig. 4 shows the relationship between the carbonization temperature and the decrease in dimensions and weight. In the length and width direction, the dimension shrinkage of woodceramics carbonized at 400°C,



A Material B Cooling Tube C Pressure Gauge D Themometer E Heater F Finsulation Material G Water Trap H Cold Trap I Activated Carbon Filter J Air Filter K Vacuum Pump

Figure 3 Woodceramics manufacturing methods.



Figure 4 Dimensional and weight changes of woodceramics made from carbonized bamboo fiber and liquefied wood.

500°C, 650°C and 800°C are 1.4%, 1.8%, 2.9% and 4.1%, respectively, while in the thickness direction, shrinkage of woodceramics are -3.8%, -2.3%, -0.7%and 3.3%, respectively. The results show that the dimension is not almost changed although it is carbonzed at different carbonization temperature, even thickness is increased a little when the carbonization temperature is below 650°C. Liquefied wood is a kind of thermoplastic resin. During the course of carbonization, it will melt firstly and then decompose into carbon. The melt of liquefied wood releases the remaining stress in the board, which make the sample expand in the direction of thickness. On the contrary, the decomposition of liquefied wood brings the shrinkage of the samples. Therefore, the expansion of thickness will take place when the shrinkage of the size for carbonization is smaller than expansion for melt of liquefied wood. Generally the ratio of dimension shrinkage of woodceramics from uncarbonized woody materials being impregnated with resin and then carbonized at 800°C is about 30% [10, 11]. The shrinkage of woodceramics in this study is much lower than previous woodceramics. Such a large shrinkage is reason of twist and cracks being induced in board during carbonization. However, the shrinkage of the woodceramics in this paper is very low, so that this material may have much less defects than traditional one. The weight loss of woodceramics carbonized at 400°C, 500°C, 650°C and 800°C are 22%, 27%, 27% and 33%, respectively.

Fig. 5 shows the relationship between carbonization temperature and the apparent density of the wood-ceramics. The apparent density of the woodceramics carbonized at 400°C 500°C, 650°C and 800°C are 0.71×10^3 Kg/m³, 0.68×10^3 Kg/m³, 0.71×10^3 Kg/m³ and 0.69×10^3 Kg/m³, respectively. The result shows that the apparent density almost don't change with increasing the carbonization temperature, which lies in that the dimension shrinkage has a similar tendency to the weight loss rate with increasing carbonization temperature.

Fig. 6 shows the relationship between carbonization temperature and the compressive strength of the



Figure 5 The relationship between carbonization temperature and the apparent density of the woodceramics made from carbonized bamboo fiber and liquefied wood.



Figure 6 The relationship between carbonization temperature and the compressive strength of the woodceramics made from carbonized bamboo fiber and liquefied wood.



Figure 7 The relationship between the carbonization temperature and the ambient volume electrical resistivity of the woodceramics made from carbonized bamboo fiber and liquefied wood.

woodceramics made from carbonized bamboo fiber and liquefied wood. The compressive strength is a little decreased at the temperatures between 400 and 500°C, and increase with carbonization temperature above 500°C. For phenol compound, it was known that depolymerization occurs between 300 and 400°C, and that the aromatic polynuclear structure starts to form above 400°C and develops above 500°C [12, 13]. As a result, combining phenol compound advances as the carbonization temperature rises, which result in the increase in the strength. Fig. 7 shows the relationship between the carbonization temperature and the ambient volume electrical resistivity of the woodceramics made from carbonized bamboo fiber and liquefied wood. It is shown that the electrical resistivity of this material depends on the carbonization temperature. There is a rather large difference in the volume resistivities of woodceramics carbonized between 650°C and 800°C. As mentioned above, during this temperature period, the phenol compound cause a big structural changes, which result in rapid decrease in electric resistivity. It has the same



Figure 8 SEM photographs of woodceramics made from carbonized bamboo fiber and liquefied wood. (a) SEM photograph of woodceramics (400° C). (b) SEM photograph of woodceramics (500° C). (c) SEM photograph of woodceramics (500° C). (d) SEM photograph of woodceramics (800° C). A: liquefied wood in continuous state, B: carbonized bamboo fiber, C: decomposed liquefied wood, D: spaces between fibers, E: composite of liquefied wood carbon/carbonized bamboo fiber, F: spaces between fibers. (*Continued.*)



(d)

Figure 8 (Continued.)

tendency as the woodceramics made from woody materials being impregnated with phenol resin and liquefied wood directly [5, 11].

Fig. 8 shows the SEM photographs of surface of samples carbonized respectively at 400°C, 500°C, 650°C and 800°C. In photo (a), liquefied wood is still continuous matrix of carbonized bamboo fiber, but in photo (b), liquefied wood is not in continuous state for its decomposition. After being carbonized at 500°C, liquefied wood was decomposed into small particles as

shown in photo (b). In photo (c), those particles have begun to combine with carbonized bamboo fiber, and spaces between fibers emerge. In photo (d), the combination of carbon from liqufied wood has finished combination with carbonized bamboo fiber, and formed a network of carbon composite of liquefied wood carbon/carbonized bamboo fiber. Spans exist obviously between the carbon.

This figure suggest that during carbonization, liquefied wood begin to decompose at a temperature about 400° C, 500° C and combine with carbonized bamboo fiber from about 650° C, finally form a composite of liquefied wood carbon/bamboo fiber carbon.

4. Conclusions

According to the results of the study, conclusions as follows can be got:

1. Woodceramics can be prepared from carbonized bamboo fiber and liquefied wood.

2. Dimension of the woodceramics is almost constant during carbonization, which means the application of carbonized material in manufacturing woodceramics may reduce intensively the shrinkage of dimension.

3. Higher the carbonization temperature is, higher the compressive strength of the woodceramics is, and lower its electrical resistivity is.

4. In this new kind of woodceramics, liquefied wood carbon combine with carbonized bamboo fiber and form a network structure.

References

- 1. T. OKABE and K. SAITO, J. Porous Materials 2 (1996) 215. 2. Idem., ibid. 2 (1996) 223.
- 3. K. HOKKIRIGAWA, T. OKABE and K. SAITO, *ibid.* 2 (1996) 229.
- 4. K. SHIBATA, K. KASAI, T. OKABE and K. SAITO, J. Society Material Science 44 (1995) 284.
- 5. T. OKABE, K. SAITO, H. TOGAWA and Y. KUMAGAI, *ibid.* 44 (1995) 284.
- 6. T. HARAGUCHI, in "New Materials of Woody Handbook" (Gihoudou, Tokyo, 1996) p. 131.
- 7. R. YAMAI, in "Wood Industry Handbook" (Maruzen, Tokyo, 1982) p. 620.
- 8. H. ONO and T. YAMADA, J. Adhensive **59** (1996) 135.
- 9. T. YAMADA and H. ONO, *Bioresource Technology* **70** (1999) 61.
- 10. T. OKABE, K. SAITO, M. FUSHITANI and M. OTSUKA, J. Porous Materials 2 (1996) 223.
- 11. T. HIROSE, T. X. FAN, T. OKABE and M. YOSHIMURA, *J. Mat. Sci.*, accepted.
- 12. K. OUCHI and H. HONDA, Fuel 38 (1959) 429.
- 13. Y. YAMASHITA and K. OUCHI, Carbon 19 (1981) 89.

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