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Thermogravimetric and differential thermal analysis of woodceramics

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The relatively new material called "woodceramics" has been developed. This material is made from a composite precursor of natural lignum and phenol resin. In order to clarify the thermophysical properties of it, we have carried out thermogravimetric and differential thermal analysis.

1. INTRODUCTION

Replacing an infrastructure of the traditional industry of mass production, consumption, and waste systems, a sustain-able technology system is expected considering limitation of environment, resources, and market [1]. As a part of this trend, woodceramics harmonizing with an ecological system have been now focused from the view point of global environmental issues [2-7].

In order to clarify the thermal properties of woodceramics, we have carried out thermo-gravimetric and differential thermal analysis [8].

2. EXPERIMENTAL METHOD

Following three types of specimens were used.

1. White ceder in Aomori Prefecture
2. Phenol resin for infiltration (PX-1600 made by Honen Corp.)
3. Woodceramics from medium density fiber board (MDF) infiltrated with phenol resin at mass ratio 1:1 using ultrasonic vibration.

Before curing, it was dried and harden-treated in a oven at 135 °C for 10 hours.

Each specimen cured at 400 °C, 600 °C, and 800 °C. For only woodceramics, a 2800 °C treated specimen was added. Resultant decomposing gases were led to a water tank and completely removed through a cold trap at -80 °C and charcoal filter. The powdered specimen with a grain size of 50 to 200 microns was measured with thermo-gravimetric and differential thermal analysis using a TG-DTA 8101D analyzer manufactured by Rigaku-Denki Kogyo Co. Ltd. Measurement temperature ranged from room temperature to 1,000 °C at the temperature rising rate of 10 K/min. Just before measurement, caloric calibration for temperature and quantification of DTA was conducted using In, Pb, Al, and Au specimens.

The powdered specimen of (25 ± 0.5) mg was filled in an open platinum container and placed in atmosphere flowing dry nitrogen gas at 100 ml/min. Alumina was used as the standard specimen to convert a relative mass change and caloric change to an electric signal for recording.

3.RESULTS AND DISCUSSION

Figure 1 shows mass reduction and endo- and exothermic behavior at heat up white ceder and Figure 2 those of phenol resin and Figure 3 wood ceramics. In each case, the mass reduction was minor and stable with less endo- and exothermic changes at higher curing temperature. TG in the figure means thermogravimetry and DTA differential thermal analysis.

The specimen chamber was evacuated before switching to nitrogen gas flow. Mass reduction during evacuating process due to separation of adsorbed element (mainly water) was about 3% for white ceder, 5% for phenol resin, and 10% for woodceramics. The reason why mass reduction of woodceramics was large is supposed due to its porosity. Mass reduction at heating process to 1,000 °C was up to 70% for non-cured white cedar but after curing it lowered to 20% or less. Phenol resin and woodceramics show similar results and after curing their mass reduction were about 10%. As an endothermic phenomenon, each specimen shows some mass reduction and endothermic reaction due to dehydration at about 100 °C. At about 400 °C, the specimen indicates mass reduction and endothermic reaction due to heat decomposition.

This suggests that white ceder shows evaporation of lignum oil due to dehydration reaction and cutting reaction of cellulose carbon fiber, and phenol resin indicates dehydration reaction and bridge-building

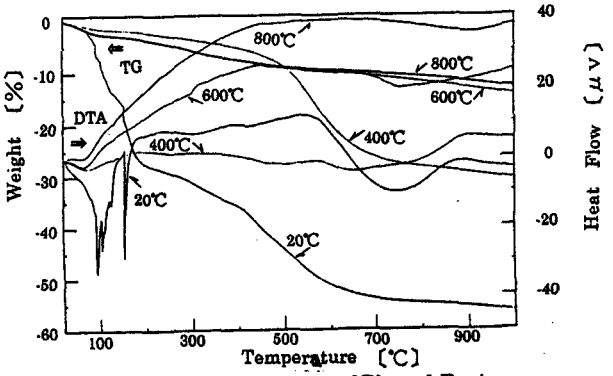

Fig.2 TG-DTA curves of Phenol Resin

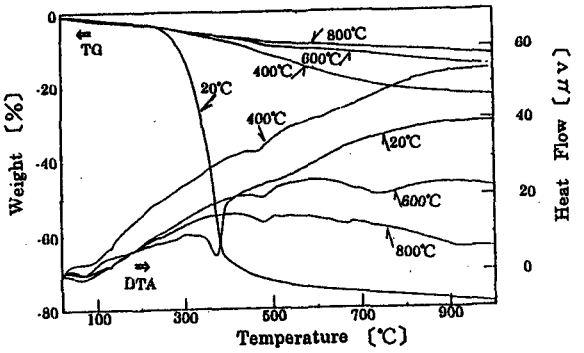

Fig.1 TG-DTA curves of white ceder

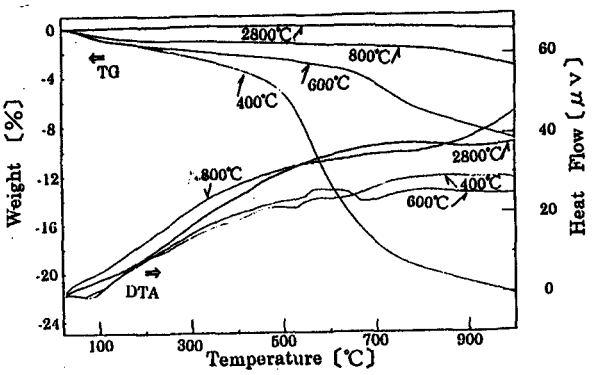

Fig.3 TG-DTA curves of Woodceramics

reaction within and between molecules. Woodceramics are composite materials resulted from their reactions, but are not simple composition of the reactions of white ceder and phenol resin. In addition to dehydration reaction above 400 °C, slow separation reaction of elements and endo- and exothermic reactions due to carbonization process might occur.

Each specimen was stable at higher curing temperature, especially wood ceramics cured at 2,800 °C show excellent stability. Practically, the behavior in the atmosphere is important, so Figure 4 and 5 indicate both measurement results in nitrogen flowing condition without evacuation just before measurement and in atmospheric condition without nitrogen flowing. The specimen is woodceramics powder cured at 800 °C. Figure 4 and 5 show measurement results in nitrogen flowing condition (1000 ml/min in ten minutes) without evacuation before measurement then to change the nitrogen atmosphere of 100 ml/min and measurement in the atmospheric condition. DTG in figures indicate differential curve of a TG curve.

At nitrogen gas flowing without evacuation, mass reduction and endothermic reaction due to dehydration was apparently observed before 100 °C and about three times reduction heat was measured compared with the condition of without evacuation process. At atmospheric condition, the starting temperature of mass reduction due to dehydration is nearly equal to the one which shows a peak of reduction heat but at about 400 °C severe heat decomposition reaction was observed. This contributes, in addition to carbonization reaction due to condensation polymerization of aromatic rings, largely to reduction reaction of components due to oxygen in the atmosphere. This reduction reaction is supposed to be production reaction of carbon dioxide.

4. CONCLUSIONS

Upon heating woodceramics, there is first a reduction in mass and an endothermic reaction around 100 °C caused by dehydration from the materials. This is followed by an

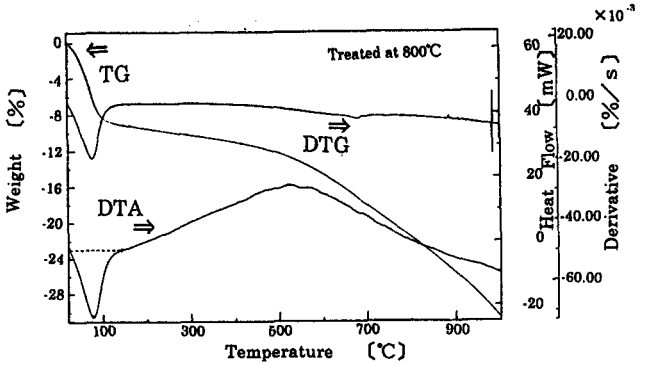

Fig. 4 TG-DTA curves of Woodceramics under the condition of nitrogen gas flow

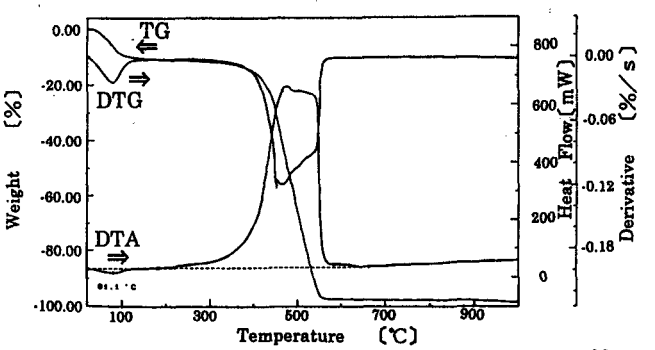

Fig.5 TG-DTA curves of Woodceramics sintered at 800℃

endothermic and an exothermic reaction around 400 °C from the decomposition of the components material and by carbonization of the lignum and phenol resin. The material become stable when heated above about 400 °C. Woodceramics cured at 800 °C are very thermally stable in the noble gas atmosphere up to 1,000 °C with slight mass reduction. Especially, the specimen cured at 2,800 °C showed highest stability. The behavior is remarkable characteristics not obtaining by only thermal treatment of wood and phenol resin.

However, at a condition including oxygen such as atmosphere and others, the severe reduction reaction of components was observed between 400 °C and 550 °C to cause heat generating reaction of about 2,000 J/g and mass reduction of 80% or more. On the contrary, at dehydration at around 80 °C endothermic reaction was about 300 J/g and mass reduction was about 10%. Adsorption of water is attributed to porous carbon material of woodceramics. However, this endothermic reaction is very lowered in re-temperature rising measurement immediately after measurement, therefore, it is likely largely to depend on the storage period and condition of the specimen.

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