FIBER FORMATION FROM COAL TAR PITCH MODIFIED WITH P-BENZOQUINONE

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INTRODUCTION

Molecular size and shape of the pitch are determining factors of the spinnability and the readiness of pitch fiber. Thermal stabilization polymerization through heat treatment is a method of high energy consumption, which leads to an uncontrollable of molecular structure. Though the air blowing method is an effective method in increasing softening point of the pitch (1), it also introduces also the side effect of reducing in the molecular weight and carbon yield (2). The chemical reaction of the coal tar pitch (CP) was performed with p-benzoquinone (BQ) at 180 ^{o}C. The molecular weight of the product was increased by about three times of that of the original pitch. When the pitch was carbonized at 500 ^{o}C, the carbon yield was increased by 20%, showing an alternation of the anisotropic to isotropic phase at the concentration of 50 wt.% BQ (3).

In the present study, preparations of carbon fibers have been performed from the pitch reacted with 10 wt % of BQ. The molecular weight distribution of the reaction product was determined, and characterizations were performed for the pitch fibers, the stabilized fibers and the carbon fibers.

EXPERIMENTAL

The THF soluble fraction of the coal tar pitch (softening point, 85 ^{o}C) was reacted with 10 wt. % of p-benzoquinone under nitrogen flow at 132 ^{o}C holding for one hour. Both CP and the chemically modified pitch (MCP) were heat treated by bubbling the pitch with a nitrogen flow of 20mL/min at 350 ^{o}C for one hour. Apparent viscosity of the CP and MCP were measured by using a capillary rheometer at the temperatures 30 ^{o}C higher than the softening points. The softening points of CP and MCP were 234 ^{o}C and 264 ^{o}C. The MCP was spun through a nozzle (L/D=1; diameter, 0.3 mm) at 4kg/cm^2 and at 280^{o}C after soaking at 290 ^{o}C.

The pitch fiber was stabilized by blowing air heated at 0.5 ^{o}C/min upto 330 ^{o}C. The stabilizing process of the pitch fiber was monitored thermally and gravimetrically by using a DSC and TGA respectively. The stabilized fiber was carbonized at 1200^{o}C with a heating rate of 10 ^{o}C/m, holding for one hour under argon gas flow. The carbon fiber was characterized by using SEM.

RESULTS AND DISCUSSION

The softening point of the pitch reacted with BQ increased with an increase in BQ concentration (Fig.1). The increment of 15 ^{o}C by addition of 10% BQ was widened to 50 ^{o}C after heat treatment at 350 ^{o}C. The BQ introduced in the CP would give an effect of accelerating condensation reaction on the heat treatment. Fig. 2 represents that molecular weight distribution of the THF soluble portions of the CP, the CP reacted with 10 wt.% BQ and the product heat treated at 350^{o}C. Though they are soluble fractions in THF, the pitch reacted with BQ polymerized showed a shoulder in upper region representing heterogeneous reaction with BQ. Also the heat treatment gave an effect of increase in molecular weight and also an effect of homogenizing.

Fig. 3 shows the comparisons of the rheological behaviors of CP and CP/BQ product after heat treatment at 350 ^{o}C. Both the CP and CP/BQ show Non-Newtonian behavior showing the decrease in viscosity with shear rate. The viscosity of MCP decreased more sharply than than that of CP with shear rate. This may represent that the enhanced interactions introduced by BQ deformed more sensitively by the shear.

The SEM photograph (Fig. 4) indicates that the fiber has isotropic texture with diameter of 18μm.

CONCLUSIONS

Isotropic carbon fibers were obtained from the source of MCP. The MCP showed good spinnability and stabilizability.

REFERENCES

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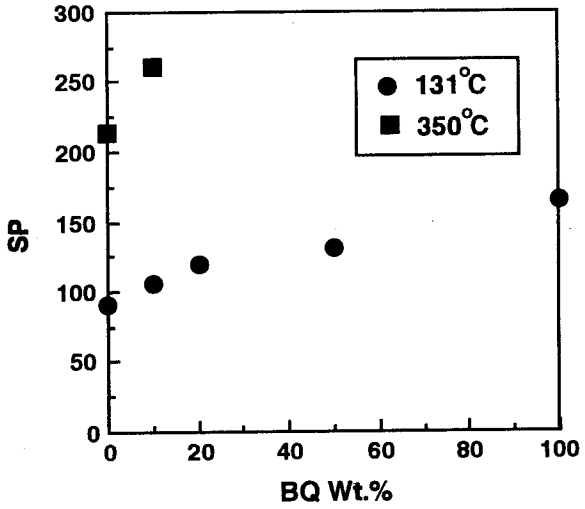

Fig. 1. The dependence of softing point on the BQ wt %.

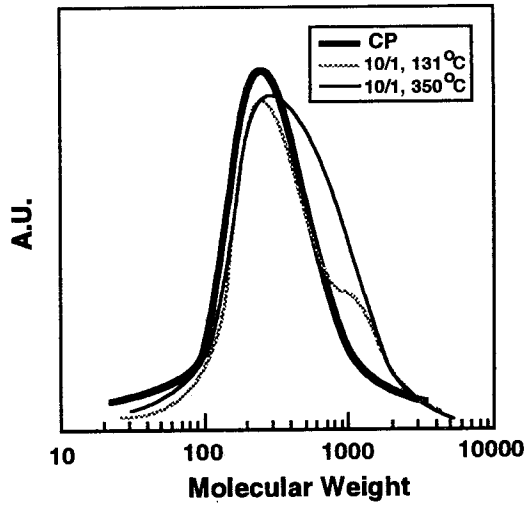

Fig. 2. Molecular weight distribution of the THF soluble fraction of the samples prepared at various conditions.

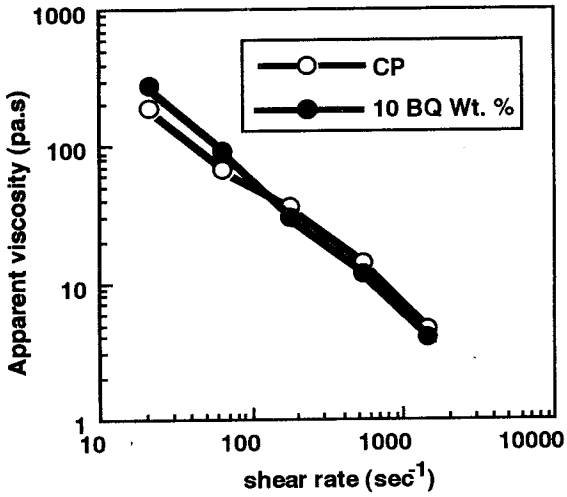

Fig. 3. The relationship between apparent viscosity and shear rate for the samples heat-treated at 350°C.

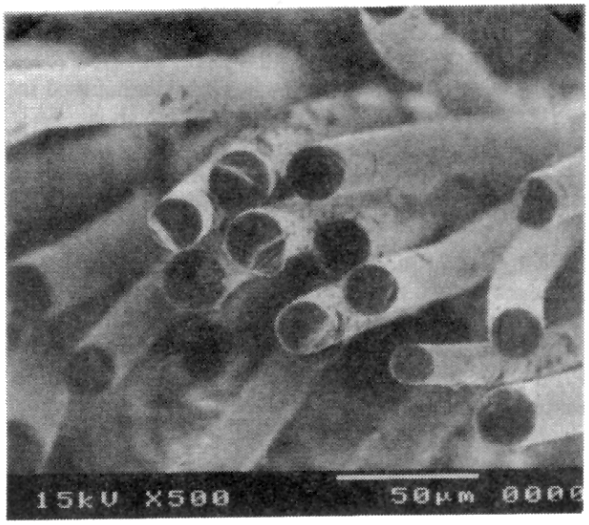

Fig. 4. SEM microphotographs of carbon fibers prepared at 1200°C; 10 wt% of BQ.