Carbonization of Glassy Carbon derived from Furan Resin and its Microstructural Analyses

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Introduction

Glassy Carbon has been recognized as an excellent carbon in terms to its hardness and precise shape stability. Such characteristics have been highly evaluated in the semi-conductor manufacture and fabrication such as an electrode for the plasma etching. Nevertheless Glassy Carbon has a drawback of producing a small dust. Recently, it was reported that Glassy Carbon carrying larger micrograin tended to decrease the dust formation.

The aim of the present study is to enlarge the microgel diameter of furan resin by adding solvent at curing, which leads to the increase of the micrograin diameter after the carbonization.

Experimental

Furan resin (HITACHI CHEMICAL Co.Ltd. VF303 Mw=11,700) used in this study was viscous liquid, carrying 40% monomer. The furan resin was cured with 12wt% of curing agent (trichloroacetic acid : ethylene glycol = 1:1) adding 50 wt% of each solvents (Acetone, THF, DMF, Phenol). Some samples were cured under ultrasonic irradiation for 15min. This mixture was cured at 50°C for 7h and then 90°C for 3h. After curing, the cured resin was further treated under vacuum at 90°C for 12h to remove the residual solvent. Cured sample was carbonized at 1200°C for 1h under Ar atmosphere by a heating rate of 1°C/min. The samples were examined by SEM, XRD and Raman spectroscopy. Average size of micrograins was calculated from diameter of fifty micrograins found in SEM micrograph.

Results

Table 1 shows the size of micrograins in the fractured surface of Glassy Carbon derived from furan resin cured in solvents and under ultrasonic irradiation. The size increased by addition of solvent at the curring. Ultrasonic irradiation further increased the size.

Table 2 shows the numbers of pores found in fifty SEM micrographs (6μm x 5μm) of fractured surfaces. Addition of acetone, THF, DMF as solvent at the curing decreased the number of pores.

Figures 1 and 2 show XRD profiles of glassy carbons carbonized at 1200°C. All carbons exhibit a very distinct difraction of (10) regardless of the solvents in addition to a broad diffraction around 26°.

Table 3 shows the intensity ratio of $1350cm^{-1}/1580cm^{-1}$ peaks in Raman spectrum of glassy carbon carbonized at 1200 ° C. Adding solvent and ultrasonic irradiation increased the ratio, indicating decrease of graphitizability.

Discussion

The present study clarified that the curing of the thermosetting polymer influenced strongly the miroscopic as well as mesoscopic structure of resultant glassy carbon. The micrograin of the carbon basically inherits the microgel of the cured polymer, although some swelling as well as shrinkage of the grain unit takes place. The graphitization of the carbon is slightly lowered by the solvent at the curing. In contrast, particular (10) band is observed regadless of the solvents.

The solvent at the curing dissolves the small microgels to precipitate larger ones, increasing their average size. The stacking of aromatic planes which are produced at curing may be disturbed by the solvent. The growth of aromatic plane is observed in the present carbon obtained from furan resin. Since such a diffraction is not always observed in the glassy carbon, important structural information may be present there.

The solvent can remove the water produced at the curing, decreasing the formation of bubble pores at the carbonization.

Table 1 Micrograin diameter of glassy carbon derived from furan resin

Solvent	—	Acetone	THF	DMF	Phenol
—	24	33	38	42	28
with ultrasonic irradiation	36	45	37	44	42

Table 2 The number of pore in fifty SEM micrographs with the area of 6 μmx 5μm

Solvent	—	Acetone	THF	DMF	Phenol
—	7	3	3	3	46
with ultrasonic irradiation	3	<1	1	2	many

Table 3 Raman spectra of glassy carbon prepared with solvents

Solvent	—	Acetone	THF	DMF	Phenol
—	1.51	1.71	1.70	1.76	1.31
with ultrasonic irradiation	1.77	1.76	1.73	1.65	1.76

※ Showing the values of R=(I_{1350}/I_{1580})

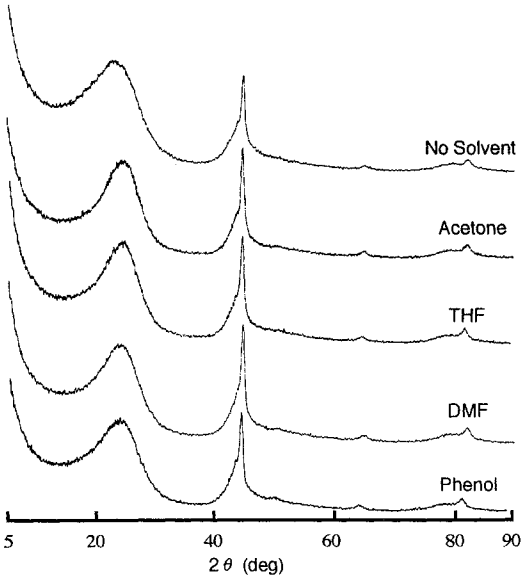

Fig.1 XRD Profiles of Glassy Carbon Prepared from Furan Resin Cured in Solvent HTT. 1200°C

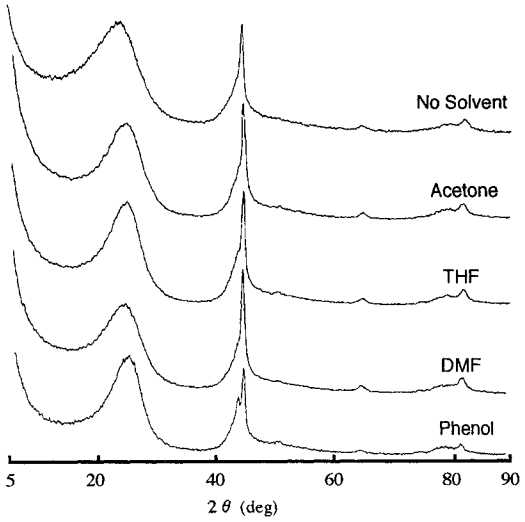

Fig.2 XRD Profiles of Glassy Carbon Prepared from Furan Resin Cured in Solvent under Ultrasonic Irradiation HTT. 1200°C