CURING PROCESS OF FURAN RESIN WITH CARBONACEOUS FINE PARTICLES APPLYING THE HIGH POWER ULTRASOUND

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Introduction

Furfuryl alcohol condensate (furan resin) is commonly uses as a matrix precursor [1] of carbon fiber/carbon matrix composites because of the convenience at carbonization process due to thermosetting properties. However, its curing and carbonization process take a long period to prevent the formation of cracks and to remove out the induced pores. Recently, on the other hand, Park et al. [2] added small amount of inorganic fine particles in furan resin to control the microstructure of the furan resin derived carbon or to improve the oxidation resistance of carbon. They reported that even though mixed furan resin with additives were ball-milled for 24 hours, powder sedimented after pouring into polystyrene container. Increasing in the reaction temperature and the amount of catalyst to furan resin did not result in homogeneous and crack free carbon materials. To solve these problems, the authors applied high power ultrasound for the curing process of furan resin. The purpose of this study is to clarify the effect of ultrasonic treatment (sonication) on the curing process in furan resin, and to show the potential method for the homogeneous preparation of cured resin.

Experimental

Hitafuran 302 (Hitachi Chemical Co., Ltd.) was used as furfuryl alcohol condensate. Various carbonaceous fine particles were added to furan resin to investigate the change of polymerization degree. The amount of additives was fixed at 1 mass% of furan resin. Curing catalyst (p-toluenesulfonic acid) was added to the furan resin in the proportion of 0.3 mass%. After stirred at room temperature for 30 minutes, temperature of the furan resin was controlled at 50 ℃. In this condition, high power ultrasound was applied through the curing process by using an ultrasonic homogenizer (Branson, model 450) at the frequency of 20 kHz and the various intensities (0-90 W/cm^2). The viscosity change of furan resin in the curing process was determined at the constant temperature (50℃) by viscometer (Brookfield, DV-II+). The rotation speed of the viscometer was 100 rpm. Polymer molecular weights were measured by Gel Permeation Chromatograph (GPC: JASCO, GULLIVER SERIES, column; Shodex GPC K802 + K803 + K804 + K805, eluent; Chloroform), with polystyrene as a standard material. After the sonication, these samples were hardened at 50℃ for 48 hours. The hardened samples were post-cured at 160℃ for 6 hours. The structural change of cured resin was determined with X-ray diffractmeter (Rigaku, RINT2100, Mo-K α radiation).

Results and Discussion

Sonication was effective for remove out the included pores. Large size of carbonaceous fine particles of 15μm could be dispersed uniformly by sonication without any sedimentation. Fig.1 shows the degree of polymerization (number of furan ring) plotted against curing time with and without sonication. The rate constants of curing reaction for with and without sonication were 1.4 x 10^{-1} and 2.2 x 10^{-2} s^{-1}, respectively [3,4]. When the sonication was stopped after 10 minutes, the curing rate was the same as that of non-sonication. Fig.2 gives the XRD profiles of cured resin at 160℃. The profile of the sonicated sample was the same as that of the non-treated one. Fig.3 shows the change of polymerization degree as a function of ultrasound intensity. Curing rate of furan resin increased with an increase in ultrasound intensity. Fig.4 shows change of polymerization degree of furan resin with various carbonaceous fine particles. The increase of curing rate was also observed by addition of various carbonaceous fine particles. In this case, curing rate was increased with an increase of surface area in additives.

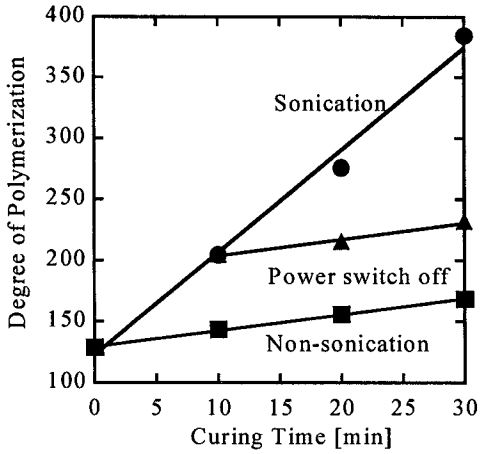

Fig.1. Change of polymerization degree with and without sonication.

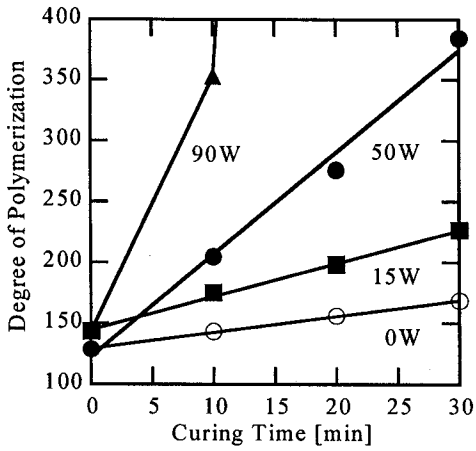

Fig.3. Change of polymerization degree as a function of ultrasound intensity.

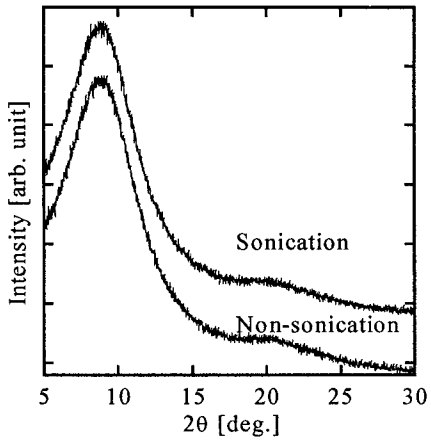

Fig.2. XRD profiles of (002) plane for the with and without sonicated sample (HTT 160°C, Mo-K α).

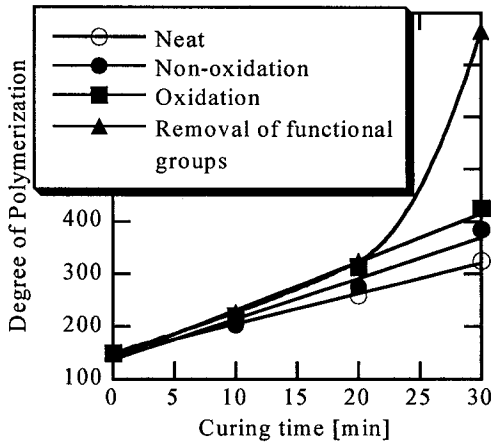

Fig.4. Change of polymerization degree of furan resin with various carbonaceous fine particles.

Conclusions

In this study, high power ultrasound was applied to the curing process of furan resin. The results are summarized as follows. (1) Sonication was effective for remove out the included larger pores. (2) Curing process of furan resin increased with an increase in ultrasound intensity. (3) The increase of curing rate was also observed by the addition of carbonaceous fine particles.

Acknowledgment

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References

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