

§12. Study on Degradation Process of Organic Insulation Materials for Fusion Superconducting Magnet by Exposure to Radiation

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i) Introduction

Organic electric insulation materials are the most radiation-sensitive among the materials used in superconducting magnet of the nuclear fusion reactor. In order to maintain the reactor safety, it is important to investigate radiation effect and improve radiation durability. In this study, we investigated the degradation process of organic insulation materials to clarify the feasible molecular structure to realize higher irradiation durability. The sample was fabricated with two types of resin; epoxy resin and cyanate ester (CE) which is known for its good irradiation durability. Dynamic mechanical analysis (DMA) was conducted to acquire the information about changes in molecular structure by irradiation.

ii) Experiment

The sample was fabricated by CE (CTD403, Composite Technology Development) mixed with epoxy resin (Epikote828, Mitsubishi Chemical Corporation) in the ratio of 20, 40, 60, 80, and 100 wt.%.

After processing the shape of cured specimens suitable for each analytical method, gamma ray irradiation was conducted. Irradiation was conducted at room temperature and air atmosphere (dose rate 42 kGy/h) up to 10 MGy in 60-Co gamma-ray irradiation facility.

After the irradiation, the glass transition temperature (T_g) of polymer samples before and after the irradiation was examined with DMA and the change in the cross-link density was investigated, based on the fact that T_g increases as increase in the cross-link density of the polymer. The dynamic viscoelasticity was measured using Advanced Rheometer AR1000 (TA Instruments). The resin sample was processed to the rectangular pieces (1 × 10 × 50 mm). Subsequently torsional vibration (1 Hz, $\epsilon = 0.5\%$) was applied to them with heating at 2 °C/min, and storage elastic modulus and $\tan\delta$ were logged. FT-IR measurement was also conducted to investigate the molecular structure.

iii) Results and discussion

In this experiment, T_g was defined as the peak temperature that exists most on the high temperature side among peaks of $\tan\delta$ in the area where the storage modulus decreases rapidly. Fig.1 shows the temperature dependence of storage modulus and $\tan\delta$ of un-irradiated CE/epoxy resin samples. The higher CE content is, the higher T_g obtained by DMA is; e.g. T_g of CE100 wt.% is higher than that of CE40 wt.% by 99 °C. This suggests that the network structure of triazine rings much more contribute to increase in crosslink density than that of oxazoline and oxazolidinone.

Fig. 2 shows the changes in T_g of each sample before and after irradiation measured by DMA. The T_g values for all the samples decreased after irradiation, which indicates that radiation causes the decrease in crosslink density.

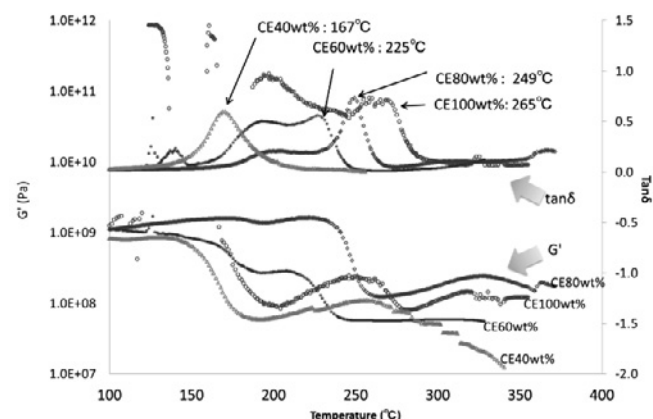


Fig. 1 Dynamic viscoelasticity of intact CE/epoxy resin compounds.

Intact CE 20 wt.% sample showed the lowest T_g (116 °C) among all the un-irradiated samples, and the largest drop (36 °C) with 5 MGy irradiation. These results suggest that the crosslink density of the CE/epoxy compound depends on the amount of triazine rings in the resin. In our previous study, the mechanical strength of samples with high epoxy content degraded after 5 MGy irradiation, and it is considered that it is due to decrease in crosslink density.

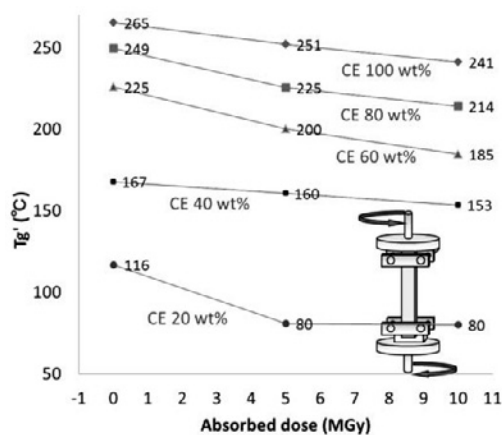


Fig.2 T_g of CE/Epoxy resin compounds before and after irradiation.

iv) Conclusion

In order to examine the degradation process of organic insulation materials, the samples of CE/epoxy resin compound was irradiated up to 10MGy, and the dynamic viscoelasticity measurement were conducted to measure T_g . The results showed that the crosslink density of CE/epoxy resin depends largely on the amount of triazine rings. From FT-IR spectra, it was also confirmed that triazine rings becomes harder to be formed with increase in the percentage of epoxy resin. From these results, it was shown that the formation of triazine rings is important to improve the irradiation durability.