Application Brief

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Dynamic Viscoelastic Measurements of Glass-Fiber Reinforced Epoxy Resin

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1. Introduction

Composite material has the characteristics which the resin itself could not have by mixing the reinforcement such as glass fiber and inorganic filler with plastic. Composite material is widely used in the field such as home electric appliances, automobile, and architectural material.

Resin components used in the composite materials are the thermoset resin such as unsaturated polyester resin, phenol resin and epoxy resin, thermoplastic resin such as polyethylene and polypropylene, synthetic rubber, and elastomer. Reinforcement materials in the composite materials are the fibrillar systems such as glass fiber and carbon fiber, and the powdery substance such as carbon black and calcium carbonate.

Glass-fiber reinforced epoxy resin (GFEP) has the advantages in the mechanical strength, the dimensional stability because of the small molding shrinkage, chemical resistance, and electrical insulation properties. GFEP, as a typical fiber-reinforced plastic (FRP), is used in the industrial parts and the printed circuit boards.

In this brief, glass-fiber reinforced epoxy resin which is used in the printed circuit boards and the insulating material is measured as an application of the dynamic viscoelasticity measurement.

2. Experiment

Two kinds of GFEP samples with 1mm thickness are used.

DMS110 Dynamic Mechanical Spectrometer (Bending Module) connected to a SDM5600H Rheol. Station is used for the measurements. Measurement condition is bending mode and 5 frequencies of 0.5, 1, 2, 5, and 10Hz. The measurement temperature range is -150 to 230°C and the heating rate is 2°C/min.

3. Measurement results

Figure 1 and 2 shows the viscoelastic spectrum of GFEP-A and GFEP-B. The results are simultaneous measurements of temperature dispersion and frequency dispersion. It shows the *E*', *E*", and tanδ curves for 5 frequencies from 0.5Hz to 10Hz. Several peaks on the *E*" and tanδ curves are observed in both GFEP-A (Figure 1) and GFEP-B (Figure 2) measurement results. As the frequency dependence is shown on all curves, they are considered as the relaxation phenomena for the different attributions. Dispersion peaks are observed on the *E*" curves and tanδ curves at 100 to 120°C in GFEP-A (Figure 1) and at 120～140°C in GFEP-B (Figure 2) while *E*' is decreasing.

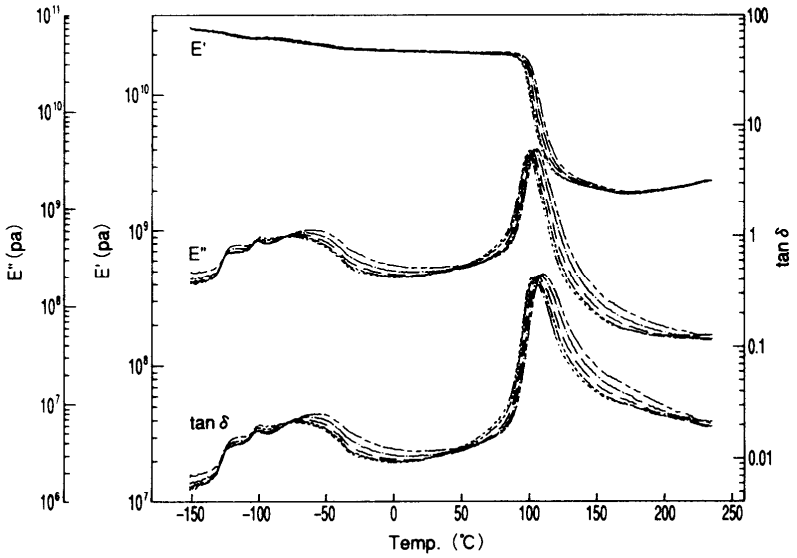

Figure 1 Dynamic viscoelasticity spectrum of GFEP-A

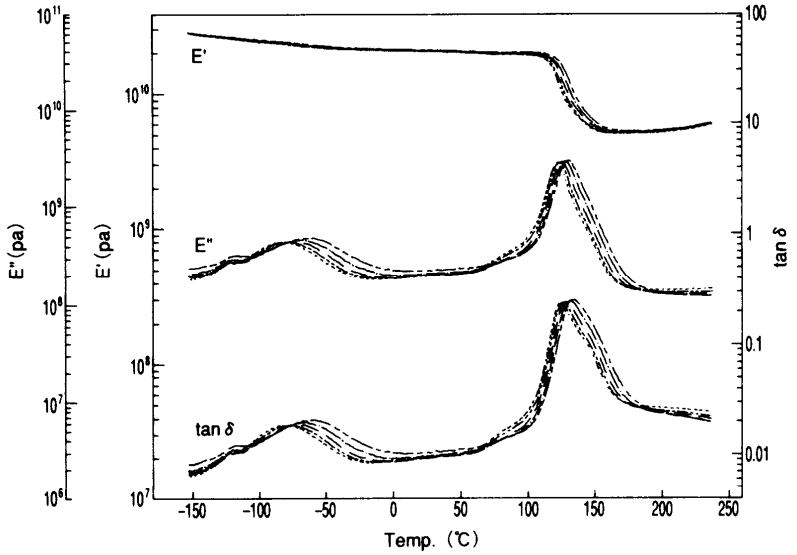

Figure 2 Dynamic viscoelasticity spectrum of GFEP-B

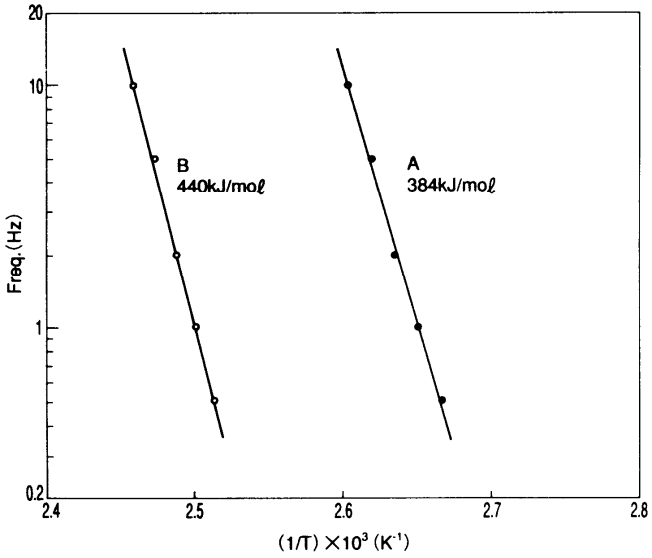

Figure 3 Apparent activation energy of main dispersion of GFEP-A and -B

Figure 3 shows the calculation result of the apparent activation energy ΔE[1)] from each tanδ peak. The result shows 384kJ/mol in GFEP-A (Figure 1) and 440kJ/mol in GFEP-B (Figure 2). It means that both dispersion peaks show the main dispersions (glass transition) of the epoxy resin component[2)].

Figure 4 shows the comparison data of the *E*' and tanδ curve of the measurement data of GFEP-A (Figure 1) and GFEP-B (Figure 2). The comparison of *E*' shows that the different start temperature of storage modulus of each sample has the different glass transition temperature. Both samples show almost the same value in the storage modulus at room temperature, however, it shows a big difference in storage modulus of rubbery region after glass transition. Both samples shows the increase of *E*' in rubbery region. This is likely due to the curing reaction of the uncured area in the epoxy resin component. In the comparison of the tanδ curves, the difference in the peak temperature and peak value at the main dispersion region is seen. Secondary dispersion region at the lower temperature shows the difference in the number of the peaks and their profiles.

Figure 5 shows the measurement result of GFEP-B under the same condition again after the 1st heating. Viscoelastic spectrum of the 2nd heating (Figure 5) does not show the increase in *E*' after glass transition. This is considered that the curing reaction at uncured region in the epoxy resin component occurred when the temperature is increased up to 230°C at the 1st heating. Figure 6 shows the comparison of the *E*' and tanδ curves of the 1st heating (Figure 2) and 2nd heating (Figure 5) measurement results. *E*' curve of the 2nd heating is observed higher than that of 1st in all temperature range. This is considered the curing degree of all sample area increases because the uncured area cures in the epoxy resin component at the 1st heating. In the comparison of the tanδ curves, the main dispersion of the 2nd heating curve is shown at higher temperature. It is known that the glass transition of the thermoset resin is gradually shifted by the repeated heating up to the higher temperature. Figure 6 shows the same tendency.

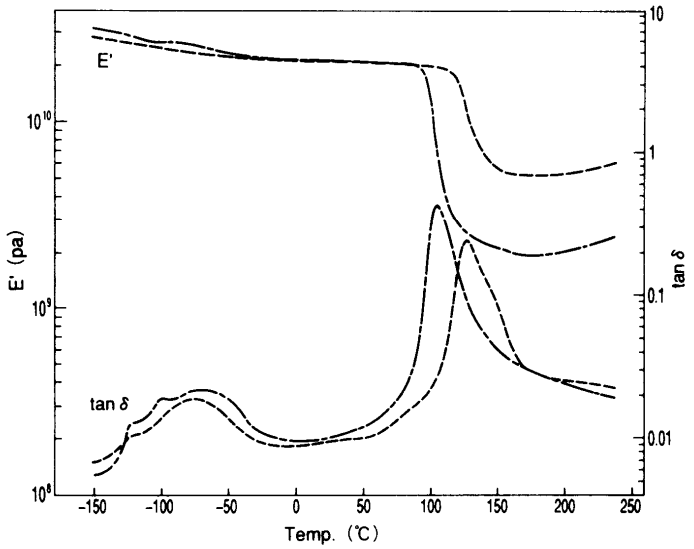

Figure 4 Comparison of *E'* and tanδ curves of GFEP-A and GFEP-B
Frequency: 1Hz
– · – · – · – : GFEP-A
– – – – – – – – : GFEP-2

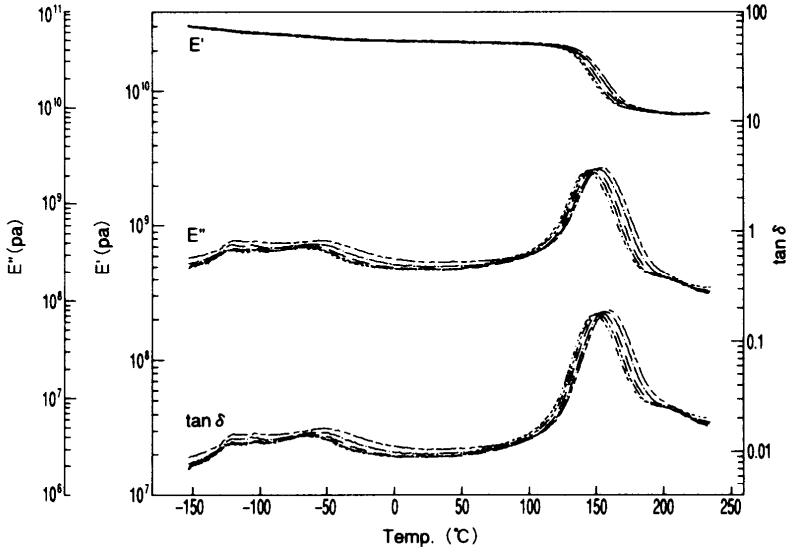

Figure 5 The 2nd heating result of GFEP-B

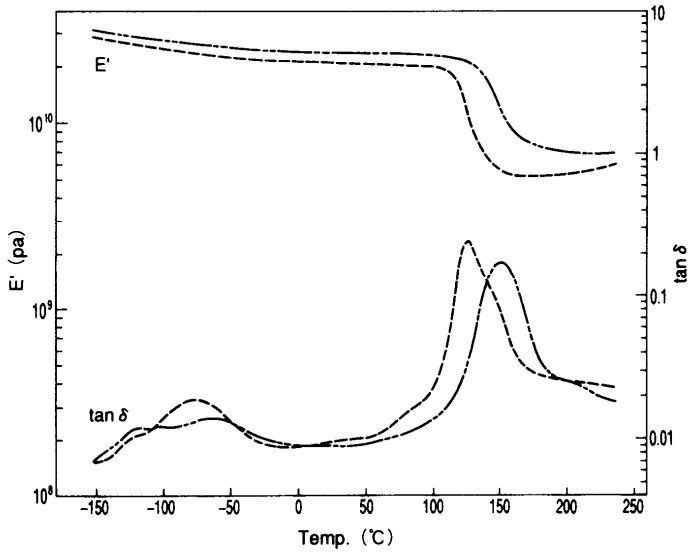

Figure 6 Comparison of *E'* and tanδ curves from the 1st and 2nd Heating
Frequency: 1Hz
- - - - - - - - : 1st heating
- · - · - · - · - : 2nd heating

4. Conclusion

In this brief, GFEP is measured as an application of dynamic viscoelastic measurements of the composite material. As a result of the measurement of the two makers' GFEPs, the difference in the glass transition temperature and the storage modulus of the rubbery region are observed. The 1st heating result of viscoelastic spectrum of GFEP-B is compared with the 2nd heating result. The difference in the storage modulus by the difference in the curing degree of resin component and the shift of the glass transition to the higher temperature is observed.

References

1) Nobuaki Okubo, Application Brief DMS No.7, Hitachi High-Tech Science Corporation (1990)
2) The Society of Rheology, Japan, "Rheology course", Koubunshikankoukai (1992)