Application Brief

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Dynamic Viscoelastic Measurements of Carbon Fiber Reinforced Epoxy Prepreg

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1. Introduction

Prepreg is a kind of fiber reinforced resin based composite material. It is non-adhesive/ semi-cured molding material made of reinforced fiber material such as glass fiber and carbon fiber impregnated with matrix resin, for example epoxy or polyester.

Carbon fiber reinforced epoxy resin prepreg is made by impregnating epoxy resin (curing agent added) with carbon fiber. It is semi-cured material designed for easy lamination or processing and is used as the material used in, for example, sporting goods like golf club and aircraft parts.

In this brief, Carbon fiber reinforced epoxy resin prepreg is introduced as an example of Dynamic Viscoelastic measurement of composite material. Comparison to DSC measurement for the same material is also introduced for reference.

2. Experiment

The sample used in the experiment is carbon fiber reinforced epoxy resin prepreg in film shape with thickness of 150μm. It is continuous fiber made of carbon fiber stretched in one direction.

DMS6100 Dynamic Mechanical Spectrometer and DSC6220 Differential Scanning Calorimeter, both connected to a PC station are used for the measurement.

In dynamic viscoelastic measurement, using film shear mode (Figure 1) the sample is measured from -120 to 280°C , heating rate 2°C/min, at 5 different frequencies, 05, 1, 2, 5 and 10Hz.

In DSC measurement, approx.10mg of the sample is measured from -30 to 240°C at 10°C/min.

Both in DMA and DSC measurements, epoxy resin is cured in 1st heating, and 2nd heating is carried out with same condition as 1st heating.

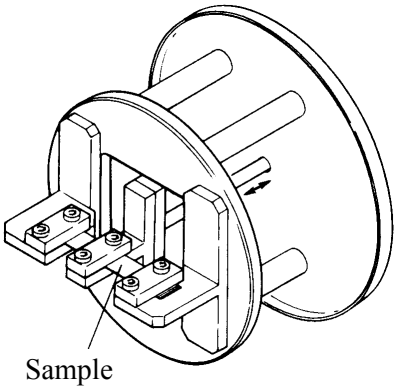

Figure 1 Film shear measurement head

3. Measurement results

Figure 2 and 3 show the viscoelastic spectrum of 1st and 2nd heating. Those are *G'*, *G"* and tanδ curves at 1Hz.

In 1st heating (Figure 2), decrease of *G'* and dispersion peak in *G"* and tanδ curves from 0 to 50°C are observed and it attributes to the primary dispersion (glass transition) of epoxy resin. Increase of *G'* observed from 130 to 150°C is assumed to be caused by curing of uncured portion of epoxy resin. There is a shoulder peak at higher temperature side of the primary dispersion peak (0 to 50°C) of tanδ curve. There is a report [1)] that in case of fiber reinforced composite material, molecule motion is restrained more in the polymer that are caught between fibers than in the one

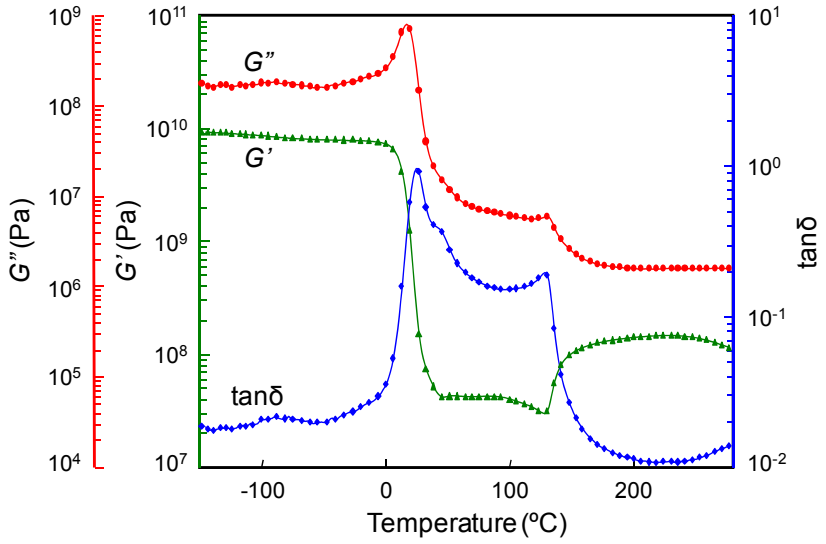

Figure 2 Viscoelastic spectrum of Carbon fiber reinforced epoxy prepreg (1st heating)
Frequency: 1Hz

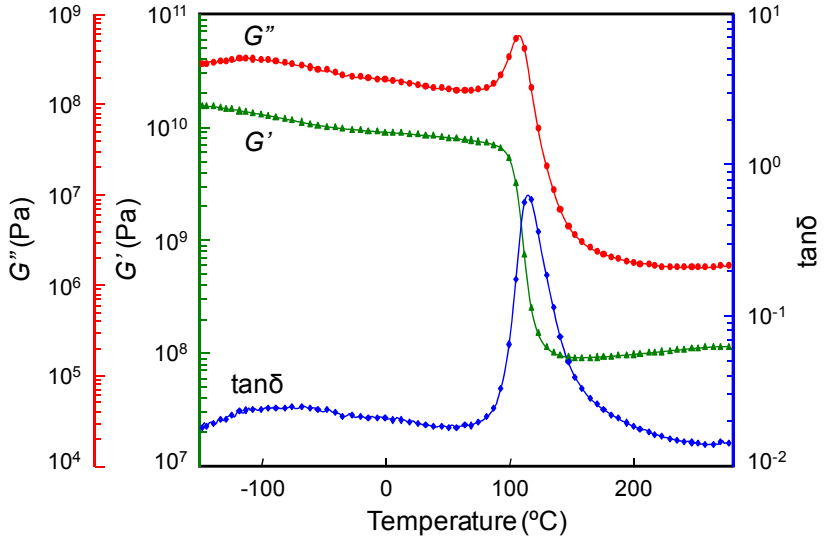

Figure 3 Viscoelastic spectrum of Carbon fiber reinforced epoxy prepreg (2nd heating)
Frequency: 1Hz

that is not, and because of the restrain, a shoulder peak appears in *G"* and tanδ peaks at its higher temperature range. The shoulder peak of the primary dispersion peak observed at its higher temperature side in Figure 2 is assumed to be caused by the same reason.

In 2nd heating (Figure 3) approx. from 80 to 120°C, decrease of *G'* and dispersion peak in *G"* and tanδ curves are observed due to the primary dispersion (glass transition) of cured epoxy resin.

Figure 4 shows the apparent activation energies [2)] calculated from tanδ peak of the primary dispersion of Figure 2 and 3. The activation energy of the primary dispersion in 1st heating is 325.4kJ/mol, and it is 626.8kJ/mol for 2nd heating. Those results suggest also that all the dispersion peaks are primary dispersion (glass transition) of epoxy resin.

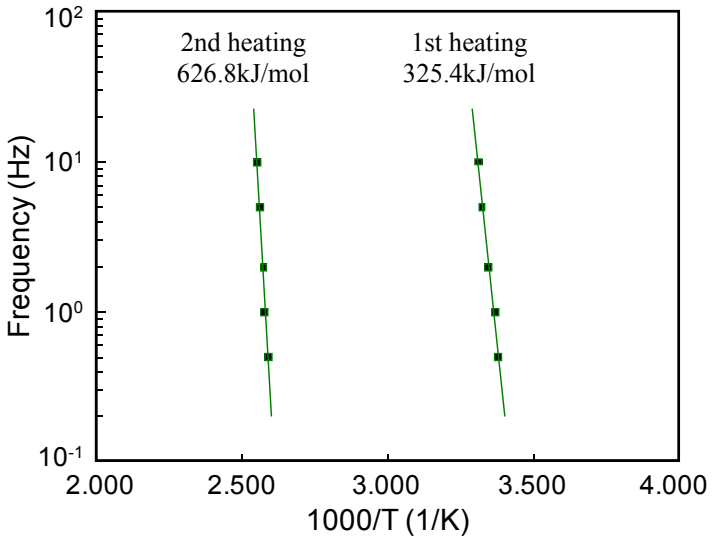

Figure 4 Apparent activation energy of primary dispersion of 1st and 2nd heating

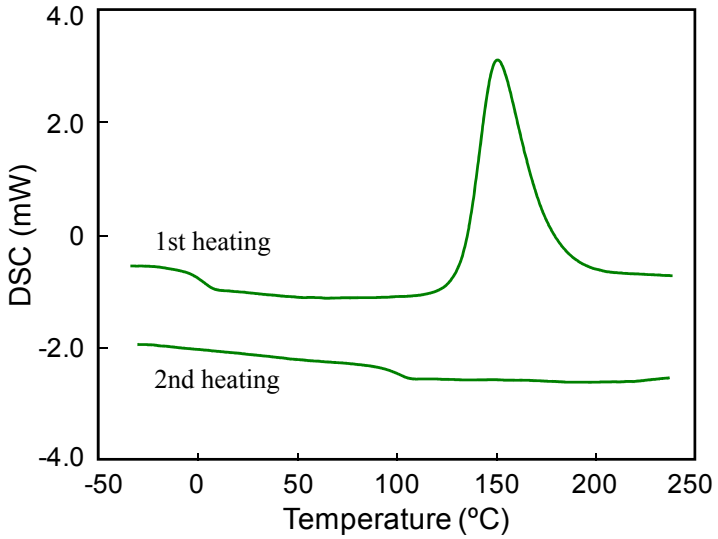

Figure 5 DSC curves of Carbon fiber reinforced epoxy prepreg
Heating rate: 10°C/min

Figure 5 shows 1st and 2nd heating of DSC measurement. In 1st heating, in the area between -5 to 10°C baseline shift due to glass transition is observed and curing of uncured portion of epoxy resin can be identified from 120 to 200°C. In 2nd heating, baseline shift due to glass transition after curing of epoxy resin is observed at temperature 90 to 110°C. It is assumed from the result that decrease of *G'* (0 to 50°C) and dispersion peaks of *G"* and tanδ are of the primary dispersion (glass transition), and increase of *G'* (130 to 150°C) is due to epoxy curing. In the same way, decrease of *G'* (80 to 120°C) and dispersion peaks of *G"* and tanδ in 2nd heating can be identified as the primary dispersion (glass transition).

4. Conclusion

In this brief, Carbon fiber reinforced epoxy prepreg is introduced as an example of Dynamic Viscoelastic measurement for composite material. In 1st heating, curing of uncured portion of epoxy resin is observed apart from the primary dispersion (glass transition). In 2nd heating, primary dispersion of epoxy resin after curing is observed. Same results are identified in DSC measurements.

References

1) M.Kodama, J.Appl.Polym.Sci., **20**, 2853（1971）
2) Nobuaki Okubo, Application Brief DMA No.7, Hitachi High-Tech Science Corporation (1990)