Application Brief

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DMA NO. 29 MAR.1995

Dynamic Viscoelastic Measurements of Carbon Fiber Reinforced PEEK

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1. Introduction

PEEK (Polyether ether ketone, Figure 1) is one of the engineering plastic. It is the material which is able to be melt processing and has the highest capability in thermal resistance among the crystalline thermoplastic polymers. It is widely used in the field such as electrical and electronic engineering, machine or auto industry as it has exellent properties in the fatigue endurance, the weathering resistance, the solvent resistance, and the flame resistance.

Compared to the existing epoxy resin composite material, carbon fiber reinforced PEEK is easier for the molding process and has the higher resistance against the damage because of the high breaking elongation rate. Also as some of the carbon fiber reinforced PEEK has the possibility to recycle in the future, the application development is expanded as the material for the leading-edge industries such as the aerospace field.

In this brief, the carbon fiber reinforced PEEK is measured as an application of the dynamic viscoelastic measurement for the composite materials.

2. Experiment

The sample is the long carbon fiber-oriented carbon fiber reinforced PEEK. The thickness is 200um.

DMS110 Dynamic Mechanical Spectrometer (Bending Module) connected to a SDM5600H Rheol. Station with the film shear measurement head (Figure 2) is used for the measurements. Two measurements are done. One is for the stress parallel to the carbon fiber orientation (Figure 3.a) and the other is for the stress vertical to the carbon fiber orientation (Figure 3.b). For the case of the parallel stress (Figure 3.a), after the 1st heating, it is cooled down and 2nd heating is done.

Measurement condition is bending mode and 5 frequencies of 0.5, 1, 2, 5, and 10Hz. The measurement temperature range is 25 to 250°C and the heating rate is 2°C/min.

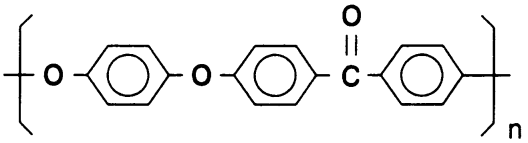

Figure 1 PEEK chemical structural formula

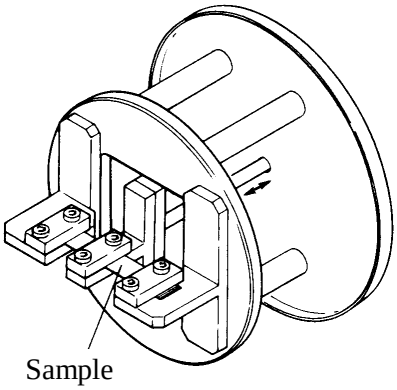

Figure 2 Film shear measurement head

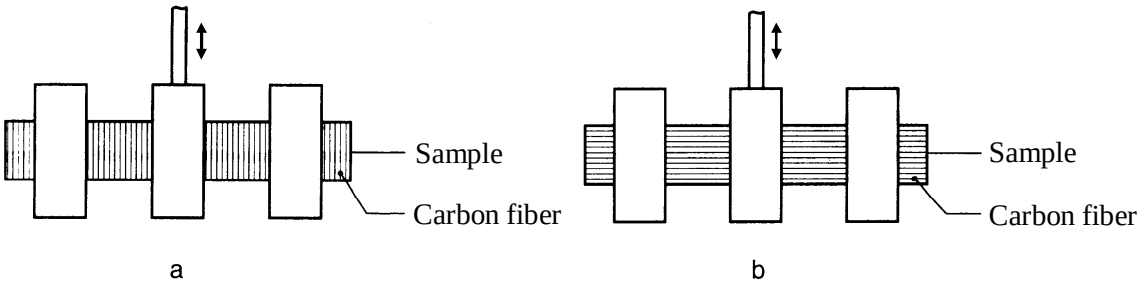

Figure 3 Sample set
a: Stress parallel to the carbon fiber orientation
b: Stress vertical to the carbon fiber orientation

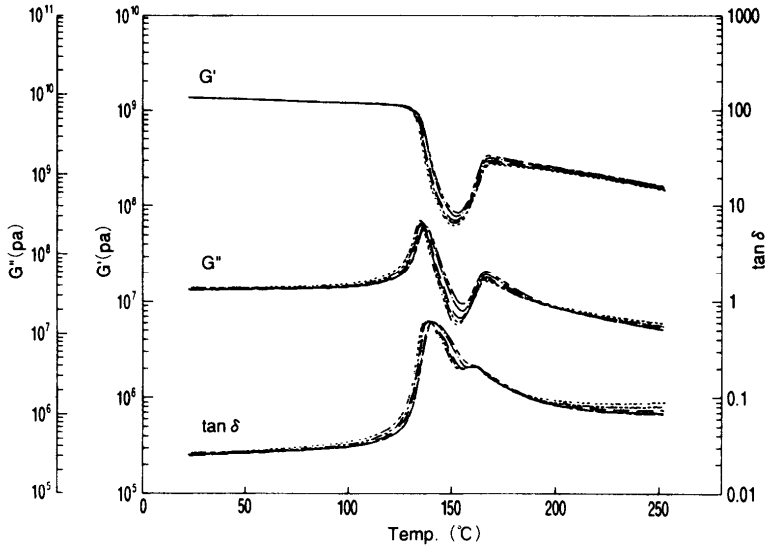

Figure 4 Dynamic viscoelasticity spectrum of carbon fiber reinforced PEEK
Stress parallel to the carbon fiber orientation (Figure 3.a)

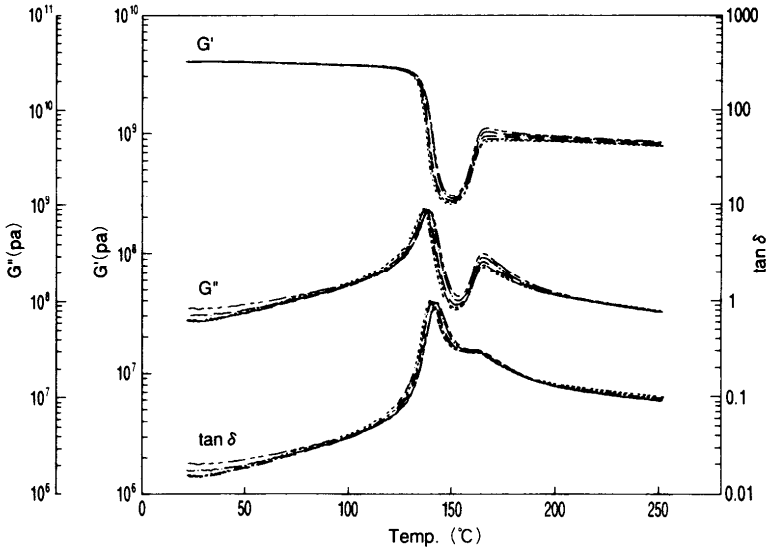

Figure 5 Dynamic viscoelasticity spectrum of carbon fiber reinforced PEEK
Stress vertical to the carbon fiber orientation (Figure 3.b)

3. Measurement results

Figure 4 shows the viscoelastic spectrum for the case of the stress parallel to the orientation (Figure 3.a). The results are simultaneous measurements of the temperature dispersion and frequency dispersion. They show *G'*, *G"*, and tanδ curve for 5 frequencies from 0.5Hz to 10Hz. Decrease in *G'* which is likely caused by the main dispersion (glass transition) of PEEK in the vicinity of 130 to 250°C and the peaks on *G"* and tanδ curves are observed. In the vicinity of 150 to 170°C, increase in *G'* which is likely caused by the cold crystallization of PEEK and the peaks on *G"* and tanδ curves are observed.

Figure 5 shows the viscoelastic spectrum for the case of the stress vertical to the orientation (Figure 3.b). Same as Figure 4, the change in *G'* which is caused by the main dispersion (glass transition) and the cold crystallization of PEEK and the peaks on *G"* and tanδ curves are observed.

Figure 6 shows the *G'* curves for the measurements result for the stress parallel to the orientation (Figure 4) and for vertical (Figure 5). In this comparison data, the data in Figure 3.b (stress vertical to the orientation) shows the higher values in all range than those of Figure 3.a (stress parallel to the orientation). This is because that in case of the stress parallel to the orientation, the PEEK is only deformed compared to the vertical case which the carbon fiber is deformed.

With regard to the case of the stress parallel to the orientation, Figure 7 shows the 2nd heating results after the 1st heating and the cooling down to the room temperature. Figure 8 shows the *G"* and tanδ curves of the measurement results of the 1st heating (Figure 4) and the 2nd heating (Figure 7). Measurement result of the 2nd heating shows the peak on the tanδ curve and the *G'* decrease by the glass transition in the vicinity of 140 to 170°C. However the peaks of the tanδ curve and the *G'* increase by the cold crystallization are not observed which are observed in the 1st heating. This difference is due to the thermal history[1)]. The reason why the cold crystallization does not occur is because of the cooling down after the 1st heating until the cold crystallization is completed. It is cooled down to the glassy region while the crystalline state is kept.

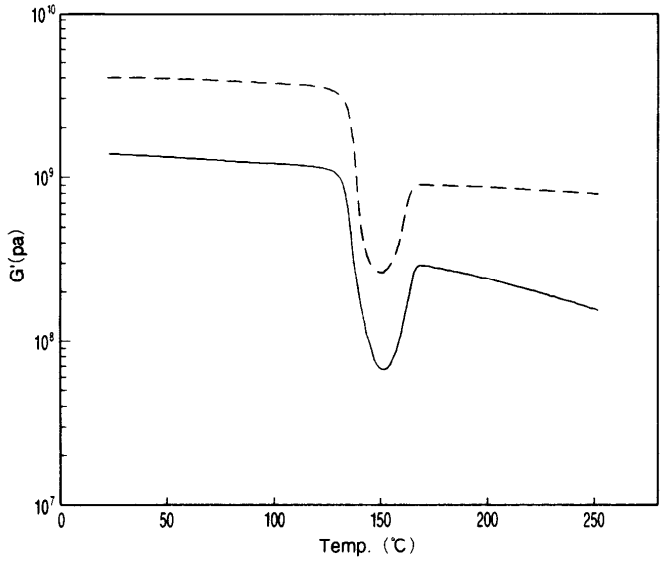

Figure 6 Comparison of *G'* curves for the measurement result for the stress parallel to the orientation and for the stress vertical
Frequency: 1Hz
———— : Parallel direction
- - - - - - - - : Vertical direction

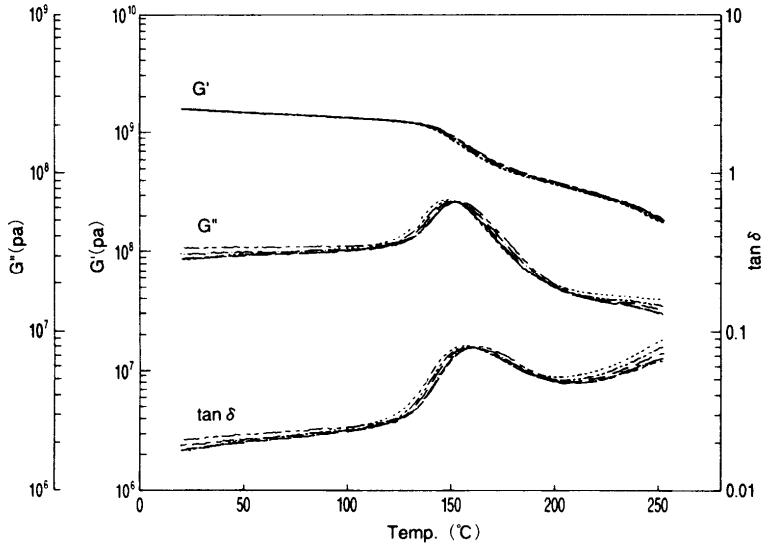

Figure 7 2nd heating dynamic viscoelasticity spectrum of carbon fiber reinforced PEEK
Stress parallel to the carbon fiber orientation (Figure 3.a)

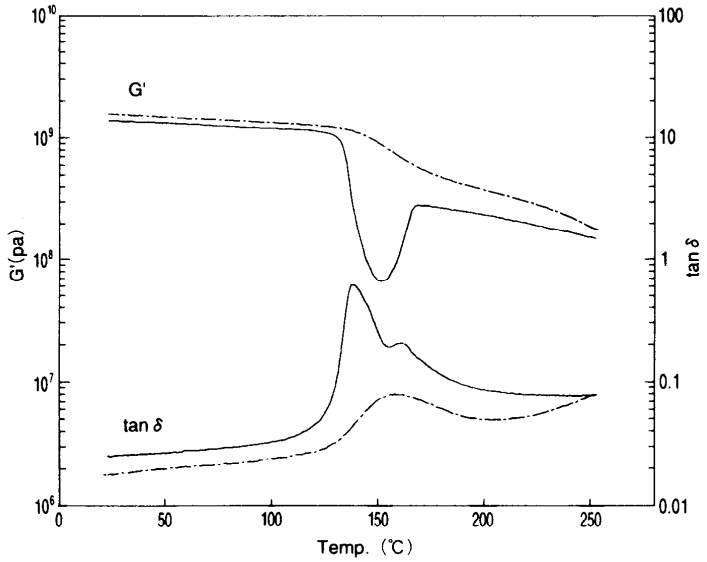

Figure 8 Comparison of *G*" and tanδ curves of the measurement results of the 1st and 2nd heating
Frequency: 1Hz
———— : 1st heating
- - - - - - : 2nd heating

4. Conclusion

In this brief, the carbon fiber reinforced PEEK is measured as an application example of viscoelastic measurements of the composite materials. The difference in the storage modulus is observed in the case of the stress parallel to the orientation of the carbon fiber and of the stress parallel to the orientation. Also as a difference in the 1st and 2nd heating, the behavior especially for the cold crystallization due to the thermal history is observed.

References

1) Nobuaki. Okubo, Application Brief DMS No.26, Hitachi High-Tech Science Corporation (1994)