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Dynamic Viscoelastic Measurements of Polyethylene Terephthalate

- The Effects of Thermal History -

Nobuaki.Okubo, Application Engineering Section

1. Introduction

Various properties of polymers, in particular the thermal properties of crystalline polymers, are affected by thermal history. Differences in the fabrication, preservation and usage of polymers change their thermal history and thermal analysis and viscoelasticity measurements are often used to investigate these changes.

In this brief, we measure differences in the viscoelasticity properties of Polyethylene Terephthalate (PET) caused by different thermal histories.

2. Experiment

A 0.5mm thick extruded PET sheet was heated (1st heating) and measured until cold crystallization was completed and then slowly cooled. The sample was then heated again (2nd heating) and measured until just before melting.

SDM5600H Rheol. station was connected to a DMS200 dynamic mechanical spectrometer (tension module) for the measurements. The deformation mode was tension and the measurement frequencies were 0.5, 1, 2, 5, and 10Hz. The measurement temperature range was -150°C to 220°C for the 1st heating and -150°C to 250°C for the 2nd heating. The heating rate was 1°C/min.

3. Measurement results

Figure 1 shows the dynamic viscoelasticity spectrum from the 1st heating. These results are the simultaneous measurement of temperature and frequency dispersions and show the E' , E'' and $\tan\delta$ curves for 5 frequencies from 0.5Hz to 10Hz. The 1st heating (Figure 1) measurement results show α -dispersion around 80°C to 90°C and β -dispersion over -150°C to 20°C. The α -dispersion is attributed to the main dispersion (glass transition) and β -dispersion is attributed local mode relaxation¹⁾. In addition to these dispersions, peaks were observed in the E'' and $\tan\delta$ curves accompanying an increase in E' near the 120°C to 140°C region. Because this PET is fabricated by quickly cooling from a melted state, it is largely amorphous. The cold crystallization of this amorphous portion likely occurred between 120°C and 140°C of the 1st heating (Figure 1).

Figure 2 shows the viscoelastic spectrum of the 2nd heating. Figure 3 shows the $\tan\delta$ and E' curves from the 1st (Figure 1) and 2nd heating (Figure 2). In Figures 2 and 3, α - and β -dispersions are observed in the 2nd heating but the cold crystallization seen in the 1st heating is not visible. It is likely that cold crystallization did not occur in the 2nd heating because the sample was heated until completion of cold crystallization in the 1st heating and then gradually cooled. The portion that was cold crystallized remained crystallized as it was cooled to the glassy region.

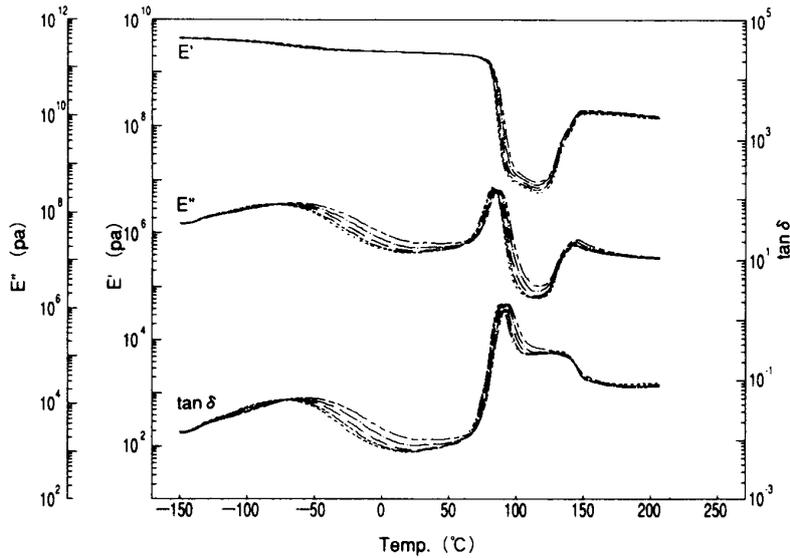


Figure 1 Dynamic viscoelasticity spectrum for 1st heating of PET

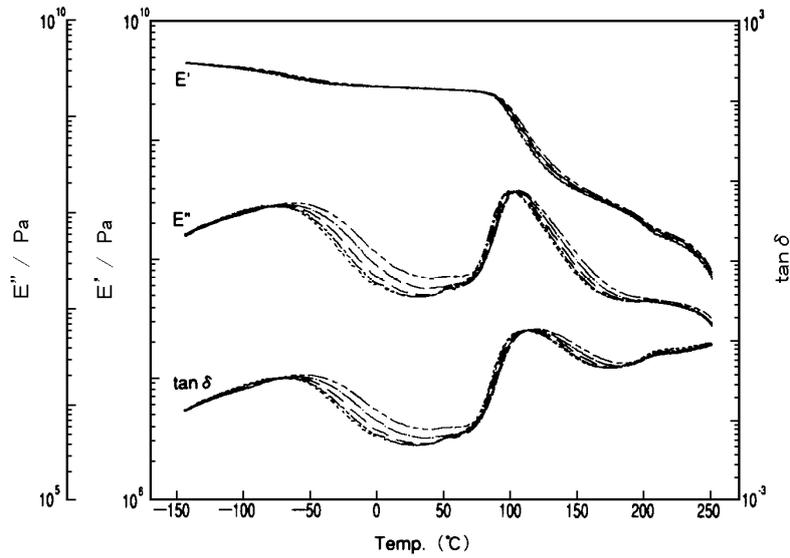


Figure 2 Dynamic viscoelasticity spectrum for 2nd heating of PET

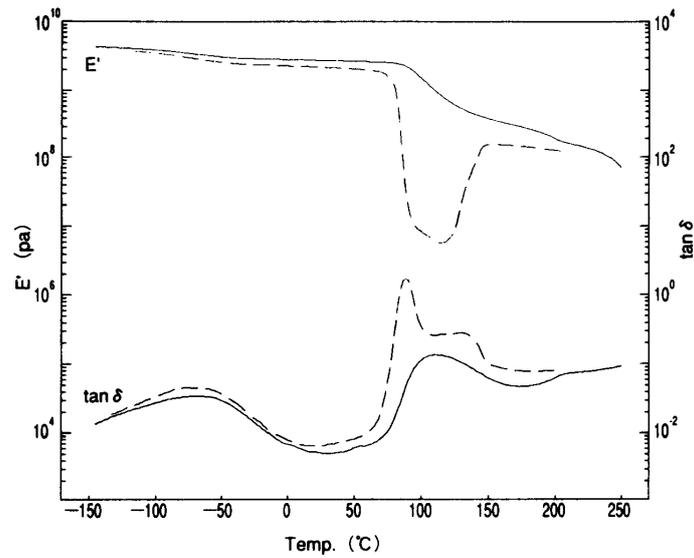


Figure 3 Comparison of E' and $\tan\delta$ Curves from the 1st and 2nd Heating
 Frequency: 1Hz
 ----- : 1st heating
 ————— : 2nd heating

4. Conclusion

In this brief, the effects of thermal history on the dynamic viscoelastic behavior of PET were measured. In particular, a large behavior difference was seen between the 1st heating and 2nd heating regarding cold crystallization.

References

- 1) Yasaku. Wada, "Solid Properties of Polymers", Baihukan (1971)