Application Brief

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Dynamic Viscoelasticity Analysis Using TMARHEO Software

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1. Introduction

In previous briefs[1, 2], TMA and TMA/SS measurements were performed by applying cyclical sine waves. Those briefs showed that elastic modulus and tanδ (loss tangent) could be calculated from Lissajous figure analysis of stress-strain coordinates.

At the same time, dynamic mechanical measurements are widely used to evaluate the characteristics of high-polymer material. A sine wave is applied to the sample and *E'* (storage modulus), *E"* (loss modulus), tanδ and other values are acquired from stress, strain and their phase difference.

Since both of these methods apply sine waves to the sample, it is possible to calculate *E'*, *E"* and tanδ from TMA/SS measurement results.

In this brief, dynamic mechanical analysis is performed using TMA/SS measurement results.

2. Analysis Principles

As shown in Figure 1, when a sine wave stress is applied to a viscoelastic material such as high-polymer material, the strain phase is a delayed sine wave. The relation of stress, strain, and elastic modulus is shown below. TMARHEO software calculates values from TMA/SS measurement results based on the following relational expressions.

E' measures the energy to recover completely from the stress stored from sample deformation and represents the hardness of the material. E" measures the energy lost as heat from sample deformation and expresses the viscosity of the material.

$$E' = \frac{\text{Stress}}{\text{Strain}} \cos\delta$$

$$E'' = \frac{\text{Stress}}{\text{Strain}} \sin\delta$$

$$\tan\delta = \frac{E'}{E''}$$

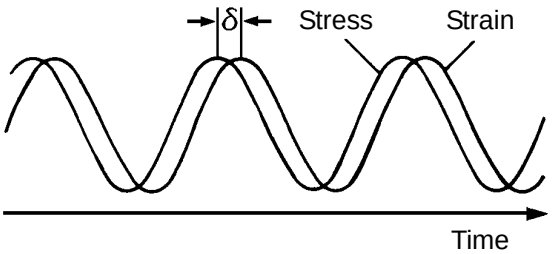

Figure 1 The Phase Relation of Stress and Strain

3. Measurements

The measurement sample was polyimide film with a thickness of 50μm.

For the measurements, a TMA/SS120C Thermal Mechanical Analyzer with Stress Strain was connected to a SSC5200H Disk station.

The test piece dimensions were 10mm (length) × 4mm (width), the load was 100±50g and the measurement frequency was 0.01Hz.

The measurement temperature range was room temperature to 400°C and the heating rate was 2°C/min.

TMARHEO software was used for analysis of elastic modulus and tanδ.

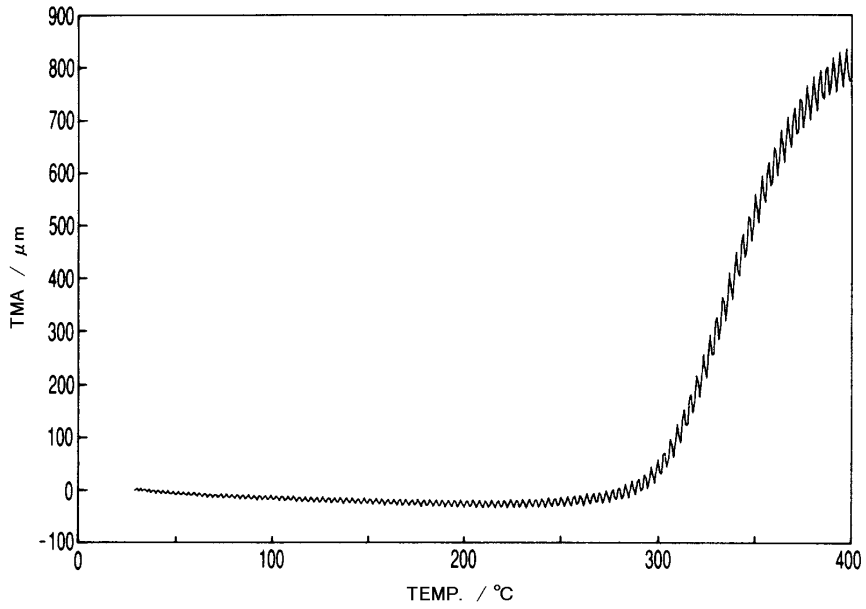

Figure 2 TMA/SS Measurement Results for Polyimide Film

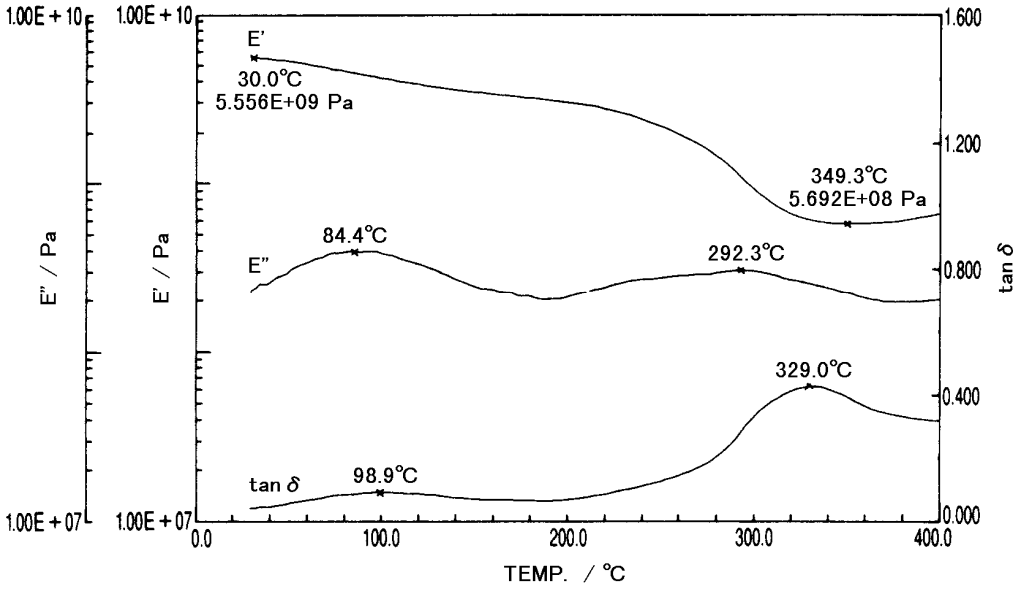

Figure 3 Dynamic Mechanical Analysis Results

4. Results

Figure 2 shows the TMA /SS measurement results for the polyimide film. As the load changes, the sample takes a sine wave shape. Around 300°C, the sample transformation increases, and sample dimensions stretch. This occurs because polyimide starts glass transition and the softening causes stretching. In conventional TMA data analysis, the glass transition temperature is acquired from the temperature at which this deformation occurs.

TMARHEO software was used to analyze the measurement results of Figure 2 and these results are shown in Figure 3. The results show the temperature dependence of *E'*, *E"* and tanδ. In the glassy region (room temperature) and rubbery region (350°C), *E'* was 5.6×10^{9}Pa and 5.7×10^{8}Pa, respectively. Furthermore, tanδ peaks are observed when *E'* decrease around 100°C and 300°C in Figure 3. The large decrease in elastic modulus near 300°C was due to glass transition, where main chain molecular motion is reduced. Furthermore, the small *E'* decrease near 100°C is likely due to reductions in local molecular movement. While it is difficult to detect this change near 100°C using DSC or other means, these results show that it is possible with dynamic mechanical analysis.

5. Summary

As shown in this brief, the dynamic mechanical analysis of the results from TMA/SS sine wave loading measurements can obtain the temperature dependence of elastic modulus and broaden the information about thermophysical properties. Furthermore, this analysis method can measure reductions in molecular motion, which conventional DSC cannot measure, and is effective in evaluating the physical properties of film and fiber samples.

Reference

1) Application Brief TA No.32,Hitachi High-Tech Science Corporation (1986)
2) Application Brief TA No.44, Hitachi High-Tech Science Corporation (1987)