

## TA NO.44 SEP.1987 Viscoelastic Measurement of Polymer Material by TMA/SS

### 1. Introduction

The interest of thermal property and mechanical property of functional polymeric material is growing.

In this brief, the viscoelastic measurement of soft material such as film, rubber, and gel is measured by TMA/SS100.

### 2. Instruments

Figure 1 shows the block diagram of TMA/SS100. TMA/SS100 load range is  $\pm 500\text{g}$  ( $\pm 4.9\text{N}$ ) and the load resolution is  $1.5\text{mg}$  ( $1.5 \times 10^{-5}\text{N}$ ). This system can control sinusoidal force (0.001 to 1Hz) and liner force. Also, it can control sinusoidal displacement and linear displacement. The frequency of the dynamic viscoelastic measurement of TMA/SS100 is less than 1Hz low frequency range. In this low frequency range, inertial effect of probe mass can be neglected, thus the force is applied to the sample without any loss.

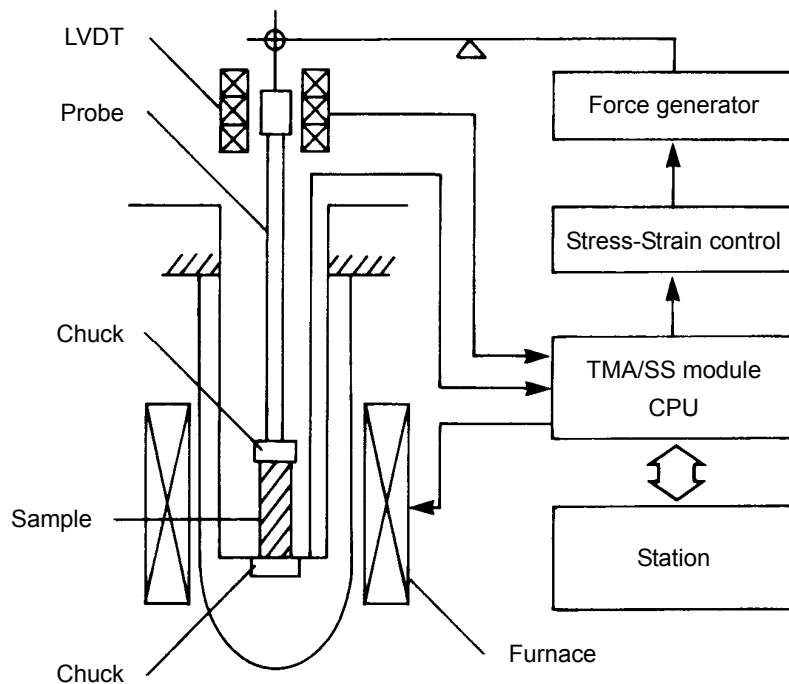


Figure 1 Block diagram of TMA/SS100

TMA/SS100 can measure creep, stress relaxation, thermal shrinkage stress, stress-strain property, and thermal curing. There are the following features in case TMA/SS100 is used for dynamic viscoelastic measurement.

- 1) Dynamic viscoelastic measurement of film material can be easily performed and sub dispersion as well as main dispersion can be obtained.
- 2) Low frequency region is easily measured. Accuracy of modulus is  $\pm 5\%$ . Accuracy of  $\tan\delta$  is  $\pm 0.002$ .
- 3) Low modulus sample such as rubber and gel is easily measured.

### 3. Analysis Method

Sine waves of stress and strain are shifted shown in Figure 2 when sinusoidal stress or strain is applied to viscoelastic body. Normally, the modulus is calculated from amplitude ratio of sine waves. Also,  $\tan\delta$  is calculated from phase difference of sine waves; however, this system uses the following methods:

Figure 2 data is shown as Lissajous on stress-strain graph in Figure 3. Length ratio of Line A and B of Figure 3 is  $\tan(\delta/2)$ . From this  $\tan$  value,  $\tan\delta$  is obtained. From the slope of line A, the modulus of sample can be calculated.

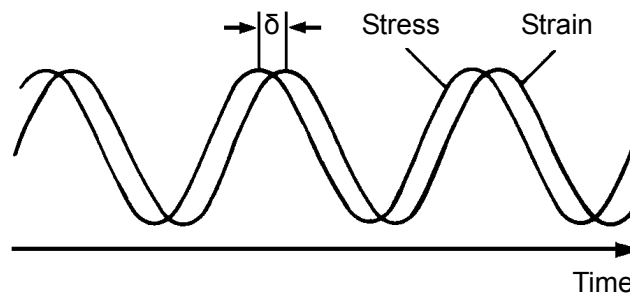


Figure 2 Stress-Strain data

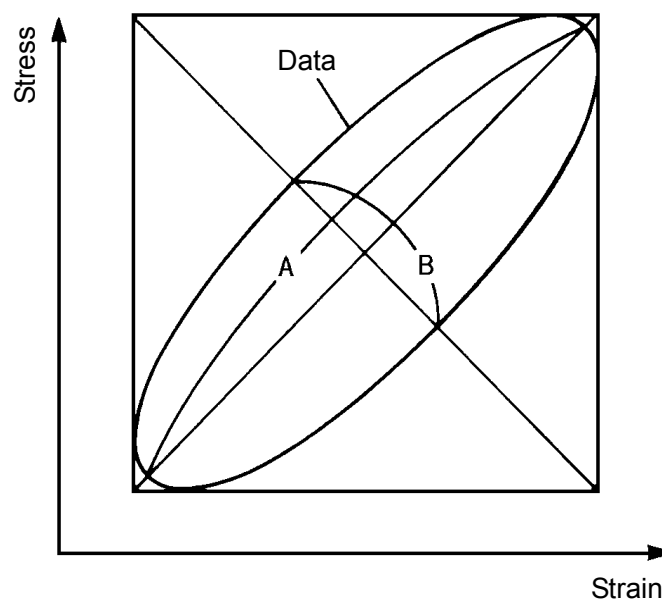


Figure 3 Lissajous on stress-strain graph

## 4. Measurement Examples

Figure 4 and 5 show the analysis results of high-density polyethylene (HDPE) film and polystyrene (PS) film by this method. These film samples are HDPE 10mm×3mm×25μm and PS 10mm×3mm×172μm. The measurement condition is tensile mode, frequency 0.1Hz, heating rate 2°C/min, and N<sub>2</sub> 200mℓ/min.

The  $\tan\delta$  is used as a factor which sensitively acquires the change of the molecular motion. The  $\tan\delta$  curve of HDPE in Figure4 shows the peak in the vicinity of -120°C and 70°C. These peaks show  $\gamma$ - and  $\alpha$ -dispersion.

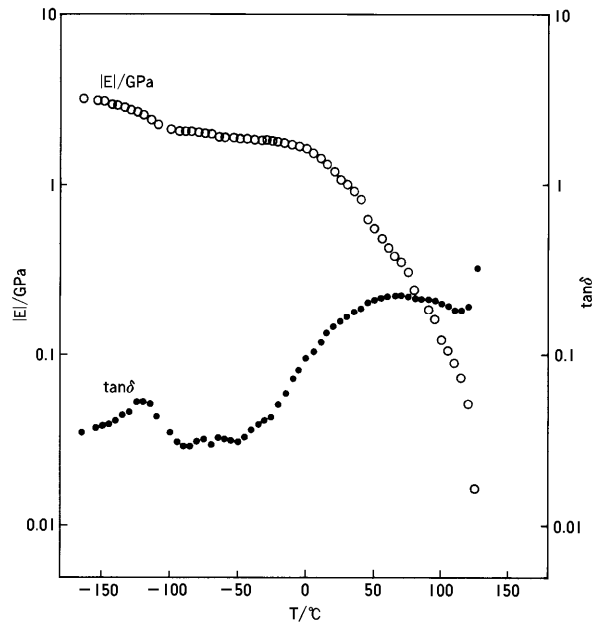


Figure 4 Dynamic viscoelastic measurement result for HDPE

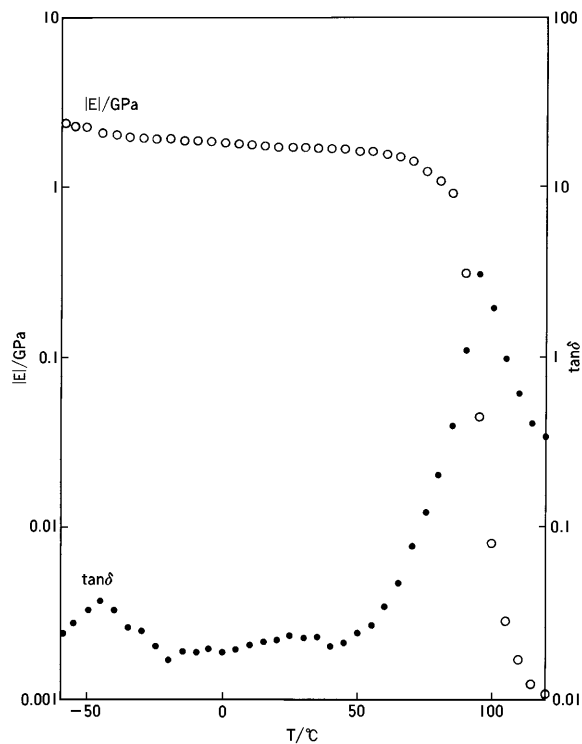


Figure 5 Dynamic viscoelastic measurement result for PS