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Application News

Thermomechanical Analyzer TMA-60

Measurement of Polymer Materials Using Three TMA Measurement Modes

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User Benefits

- By switching the measurement mode, various material properties can be measured, such as the expansion coefficient, softening temperature, and glass transition temperature, regardless of the shape of the test sample.
- The sample support tube and detection probe can be detached and replaced with one-touch operation, so maintainability is excellent.

■ Introduction

The TMA series thermomechanical analyzer can vary the temperature of a sample in accordance with a program and measure changes in its dimensions while the sample is subjected to constant pressure. The TMA-60 has three measurement modes that enable it to perform various measurements, such as expansion coefficient and penetration measurements, regardless of the shape of the test sample. The sample support tube and detection probe can be detached and replaced with one-touch operation, so the measurement methods can be easily switched and maintainability is excellent. Fig. 1 shows the three measurement modes of the TMA. This article provides examples of measuring several types of polymer using the three measurement modes.

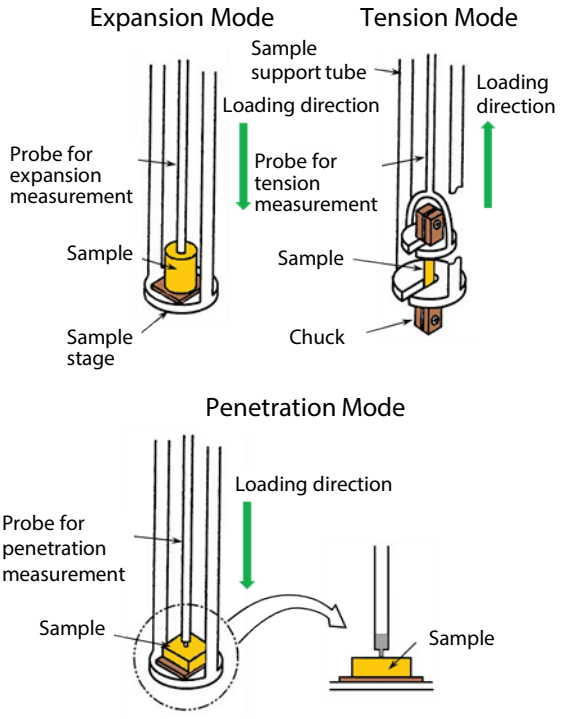

Fig. 1 Measurement Modes of the TMA

■ Measurement of Expansion of Sheet-like Samples

In expansion mode, the expansion coefficient can even be measured for the thickness direction of sheet-like samples with a thickness of less than 1 mm. In this example, a polyethylene sheet with a thickness of about 0.5 mm was measured. The measurement conditions are shown in Table 1 and the measurement results in Fig. 2.

Fig. 2 shows that the sample expands linearly as the temperature rises. The coefficient of thermal expansion of the sample obtained over the temperature range of 40 to 80 °C was 215.4×10^{-6}/K.

Table 1 Measurement Conditions

Measurement Mode:	Expansion mode
Heating Rate:	2 °C/min
Temperature Conditions:	Room temperature to 100 °C
Compression Load:	5 g
Atmosphere:	Air

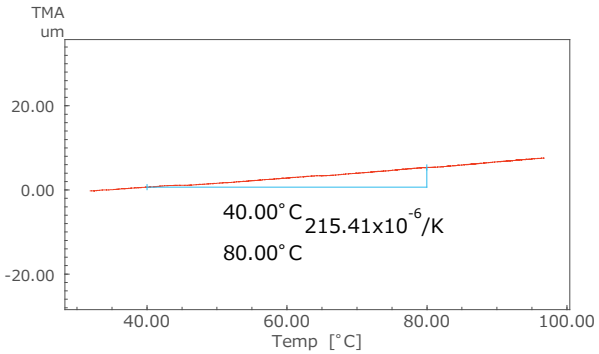

Fig. 2 Polyethylene Sheet Expansion Measurement Results

■ Measurement of Expansion Coefficient of Film-like Samples

By performing measurements of film-like or fiber-like samples while applying a load in the tension direction in tension mode, their properties, such as the expansion coefficient or the shrinkage temperature, can be measured. In this example, two types of separator film for a lithium-ion battery were measured. The measurement conditions are shown in Table 2, a sample set in the chucks is shown in Fig. 3, and the measurement results are shown in Fig. 4.

Table 2 Measurement Conditions

Measurement Mode:	Tension mode
Heating Rate:	10 °C/min
Temperature Conditions:	Room temperature to 200 °C
Compression Load:	1 g
Atmosphere:	Air

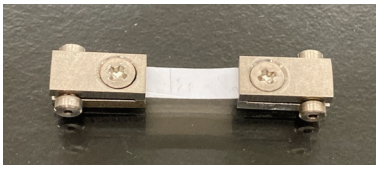

Fig. 3 View of the Sample and Chucks

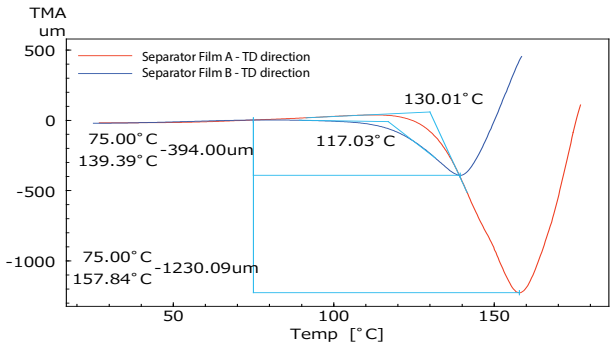

Fig. 4 Measurement Results of the TD Direction for Two Types of Separator

The separator film in a lithium-ion battery is located between the positive electrode and the negative electrode. It prevents short circuits and ensures conductivity by allowing the passage of ions. The properties of the separator film greatly affect the performance and safety of the battery. When a battery generates abnormal heat, the film melts causing the fine pores to close, preventing the passage of ions. This has the function of controlling the thermal runaway of the battery. However, when the temperature rises above the melting point of the film, the whole film shrinks, and the positive electrode and negative electrode come into contact, causing a short circuit. Comparing the two types of film from the point of view of preventing short circuits, Fig. 4 shows that the temperature when shrinkage occurs is higher in Film A than Film B, so Film A is safer in terms of heat resistance. However, the amount of shrinkage is smaller for Film B than Film A, so Film B is actually safer for a device. Evaluation of the thermal shrinkage process in this way is effective for evaluating the separator films in lithium-ion batteries.

■ Measurement of Expansion Coefficient of Fiber-like Samples

In tension mode, not only film-like but also fiber-like samples can be measured by using the optional chucks for fibers. In this example polyester fibers used in textiles were measured. The measurement conditions are shown in Table 3; a sample set in the chucks for fibers is shown in Fig. 5; and the measurement results are shown in Fig. 6.

Fig. 6 shows that as the temperature rises, there is a small amount of shrinkage up to about 100 °C. It is considered that this is due to the evaporation of moisture contained in the sample. Thereafter the sample expands, and at 173.0 °C softening followed by shrinkage occurs. Then at 265.2 °C rupturing occurs. During manufacturing, film or fiber is often formed while tension is applied, so when it melts and softens shrinkage occurs.

Table 3 Measurement Conditions

Measurement Mode:	Tension mode
Heating Rate:	5 °C/min
Temperature Conditions:	Room temperature to 300 °C
Compression Load:	1 g
Atmosphere:	Air

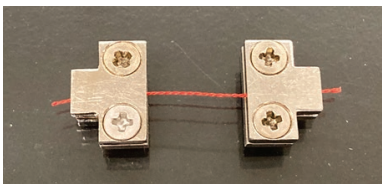

Fig. 5 View of the Sample and Chucks

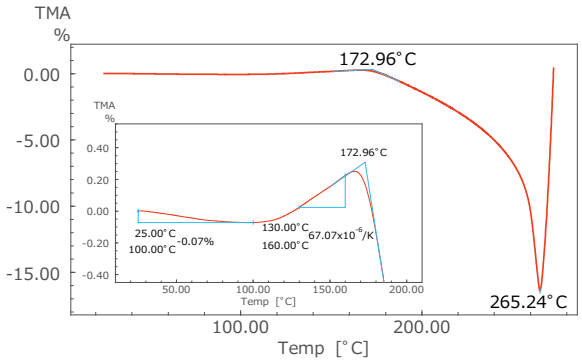

Fig. 6 Polypropylene Fiber Measurement Results

■ Measurement of Penetration of Film-like Samples

Penetration mode can be used to obtain the softening point not only for solid samples but also for extremely thin samples, such as film. In this example, the softening point was measured for polyethylene wrapping film with a thickness of about 10 µm. The measurement conditions are shown in Table 4, and the measurement results are shown in Fig. 7.

Table 4 Measurement Conditions

Measurement Mode:	Penetration mode
Heating Rate:	5 °C/min
Temperature Conditions:	Room temperature to 135 °C
Compression Load:	10 g
Atmosphere:	Air

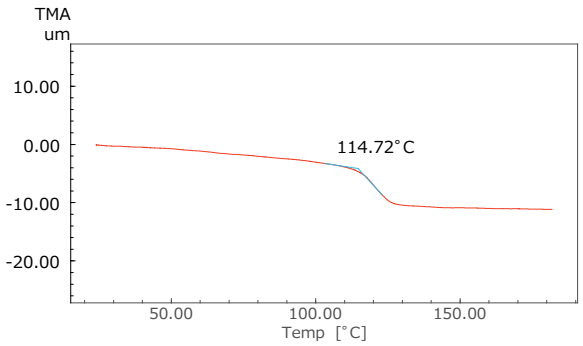

Fig. 7 Polyethylene Film Penetration Measurement Results

Fig. 7 shows that the softening point is at 114.7 °C. The softening point of polyethylene depends on its density, so it is considered that this wrapping film sample is low-density polyethylene.

■ Conclusion

Various polymers were measured using the three different measurement modes of the TMA-60. By switching the measurement mode, various thermal material properties can be measured, such as the expansion coefficient and softening temperature, regardless of the shape of the sample.

01-00416-EN First Edition: Sep. 2022

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