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Constant Test Force Extrusion Type Capillary Rheometer Flowtester CFT-EX Series

Application News

Evaluation of Flowability of Thermosetting Resins

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User Benefits

- Easily evaluate viscosity of thermosetting resins.
- Easily measure and clean thermosetting resin, which can easily clog the nozzle (by using a split nozzle).

■ Introduction

Thermosetting resins (including fillers) are often used in package encapsulants for semiconductors such as ICs and LSIs as well as printed circuit boards. In particular, in recent years, demand for semiconductors has been increasing as society as a whole has turned to reducing CO2 emissions, including the EV shift in automobiles. As a result, the demand for assessment of flowability of semiconductor encapsulants has increased. Knowing the physical properties of materials and controlling the quality of viscosity, curing time, curing temperature, etc., is very important to increase productivity and reduce defect rate.

This article presents an example of evaluating the flow characteristics of epoxy resin, a thermosetting resin.

■ Samples and Test Conditions

A CFT-500EX model Flowtester (see Fig. 1) was used for the tests. This device is a canalicular rheometer that measures the viscous resistance of the melt as it passes through the canaliculus.

Three different kinds of thermosetting resins were used. The test conditions are shown in Table 1. The test pressure was chosen so that the sample melts, flows, hardens and stops flowing.

Fig. 1 CFT-500EX

Table 1 Test Conditions

Test Method:	Constant temperature method
Die Diameter:	0.5 mm
Die Length:	10 mm
Test Temperature:	185 °C
Test Pressure:	2.45 MPa
Preheating Time:	15 s
Sample Size:	2.5 g (formed into pellets))

In order to prevent hardening of the sample from progressing due to the time loss when the sample is loaded, the sample was granulated in advance so that it could be easily loaded (see Fig. 2). The granulation method was as follows.

1. Measure the sample and insert it into the hole of (1) the preforming die unit.
2. Set (2) the plunger in the hole of (1) the preforming die unit.
3. Operate the handle of the hand press and press the top edge of (2) the plunger with the press shaft to compress and shape the specimen.
4. Place (3) the sample extraction die at the bottom of the granulator.
5. Push the top edge of (2) the plunger with the hand press to extrude the molded sample.

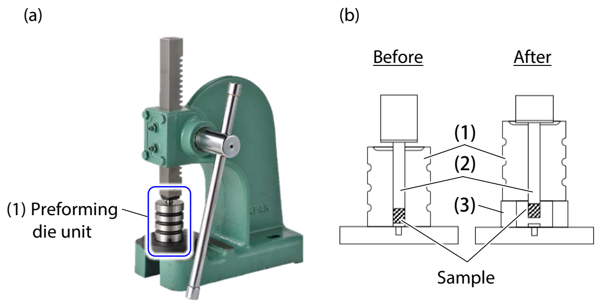

Fig. 2 Preforming Attachment
(a) Hand Press (b) Before and After Preforming

The minimum melt viscosity for thermosets is very low, so it is necessary to use a long die with a small diameter. Therefore, the test was carried out using a split nozzle (see Fig. 3 (a)) that could be cleaned even if the sample hardened after the test. During the test, the split nozzle was inserted into the die holder (see Fig. 3 (b)) and attached to the cylinder. After the test, a split nozzle disassembly tool was inserted into the die holder and pressed the upper end to remove the split nozzle.

Since the nozzle is split in two at the center, the resin can be easily removed, and cleaning and measuring can be done efficiently.

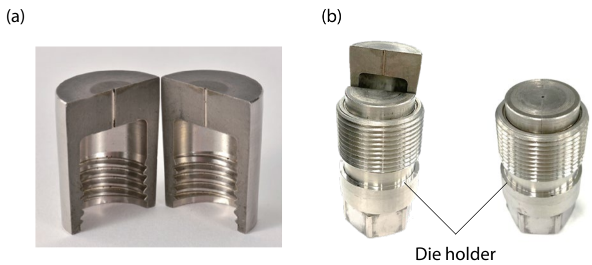

Fig. 3 Split Nozzle
(a) Split (b) Inset to Die Holder

■ Measurement Method

Constant temperature tests are often used to measure the flow characteristics of thermosets. There are two methods for calculating the flowrate in tests where the temperature is constant: the limited method and the automatic method (see Fig. 4).

Limited Method:	The descent positions of the upper and lower points on the piston are set beforehand, and the flowrate is obtained from the slope.
Automatic Method:	The stroke of the flow curve is divided into 20 parts and the slope is determined in each zone except the test start and end. The flowrate is determined from the maximum of these values.

Thermosetting resins differ from thermoplastic resins in that their viscosity changes constantly, so the automatic method of constant temperature testing was used. The lowest value of the melt viscosity can be automatically determined by calculating the viscosity in the section where the slope of the flow curve is the highest.

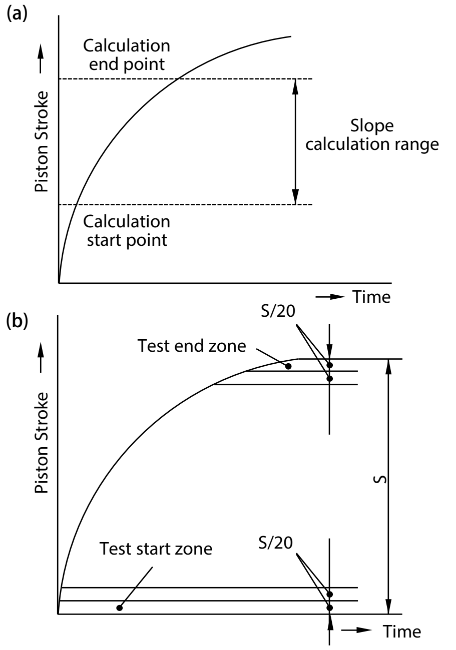

Fig. 4 Flow Curve
(a) Limited Method (b) Automatic Method

Viscosity is calculated by calculating the flowrate, obtaining the shear rate and shear stress, the ratio of which corresponds to viscosity, as shown in the following equation:

(1) Flowrate Q

$$Q = \frac{X}{10} \cdot \frac{A}{t}\ (cm^3/s)$$

t : Measurement time(s)
X : Apparent displacement(mm)
A : Piston cross sectional area (cm^2)

(2) Apparent shear rate γ

$$\gamma = \frac{32Q}{\pi D^3} \cdot 10^3\ (s^{-1})$$

D : Die orifice diameter (mm)

(3) Apparent shear stress τ

$$\tau = \frac{PD}{4L}\ (Pa)$$

P : Test pressure(Pa)
L : Die length(mm)

(4) Apparent viscosity η

$$\eta = \frac{\tau}{\gamma} = \frac{\pi D^4 P}{128LQ}\ (Pa \cdot s)$$

■ Result

Fig. 5 shows the resulting graph and Table 2 shows the numerical values. The viscosity relationship was as follows:

No. 3 < No. 2 < No. 1

As shown in Fig. 5, the amount of stroke per unit time differed depending on the sample. The difference in stroke is due to the outflow of the sample. The stroke was reduced due to material from several specimens having hardened in the cylinder due to heating and remaining there without being discharged.

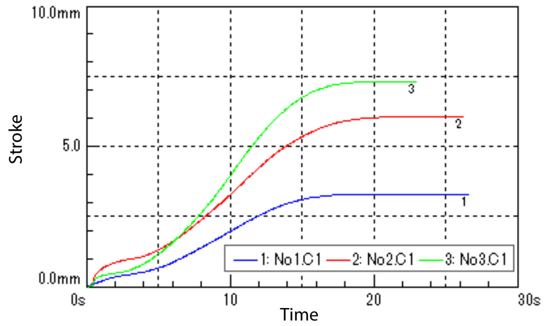

Fig. 5 Stroke-Time Graph

Table 2 Test Results

Sample No.	Shear Rate (s^{-1})	Viscosity (Pa•s)
1	2,471	12.4
2	4,073	7.5
3	5,810	5.3

Fig. 6 shows a viscosity-time graph showing the change in viscosity over time. The polymer melts and begins to flow at about 3 seconds from the start of measurement, reaches its lowest viscosity at about 10 seconds, and stops flowing at about 18 seconds.

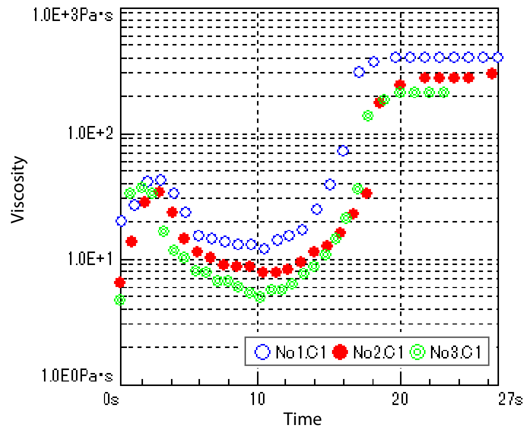

Fig. 6 Viscosity-Time Graph

■ Conclusion

The Shimadzu Flowtester calculates viscosity by measuring the amount of movement (movement speed) of the piston using the “Constant Test Force Extrusion Method.” Even if the specimen is hardened by heating, the piston displacement only stops and does not affect the control of the test force, which makes it possible to obtain very stable and highly reproducible test data.

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