Application News

SHIMADZU

THERMAL ANALYSIS T117

Evaluation of Physical Properties of Thermosetting Resins

Thermosetting resins are widely used for electronic parts such as printed circuit boards, connectors and switches, for molding materials, and for adhesive agents.
Resins are pressed with reinforcing agents, hardening members and hardening agents to any required shapes through a hardening process utilizing the curing reaction.
Resins are heated to shorten the time for curing reaction, which is largely dependent on the set temperature and the length of time for temperature holding. State of curing must be measured as it is a very important factor to the mechanical strength and physical properties of resin.
Thermal analysis is a useful method for measurements of thermal expansion coefficient, glass transition temperature and for determination of quantity of reinforcement agents required for quality control of resins.
The following is a list of major measuring items for thermal analysis.

DSC (differential scanning calorimeter)

(1) State of curing
(2) Kinetic analysis of hardening reaction
(3) Glass transition temperature

TMA (Thermo-mechanical analyzer)

Thermal expansion coefficient

TGA (Thermogravimetric analyzer)

(1) Quantitative analysis of inorganic substances
(2) Heat resistivity

Influence of water to glass transition temperature (DSC)

It is already known that the glass transition temperature (Tg) is lowered by the presence of low polymer substances. If high polymer products, for example, are stored in an ambience containing a lot of moisture, a similar change occurs. Fig.1 shows a result of measurements of a mixture of resins with moisture. As shown in Fig.1, the glass transition temperature Tg is 55.13˚C at moisture content 0%, 52.16˚C at 2.2% and 38.95˚C at 4.2% shifting from high to low temperatures.

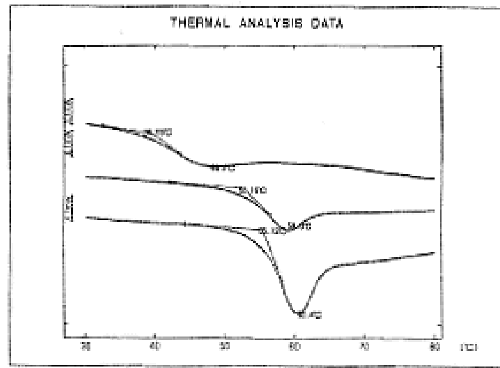

Fig.1 Shift of Glass Transition Temperature

Measurement of glass transition temperature (DSC)

Glass transition temperature is an important index to estimate the characteristic property of a thermosetting resin, as its hardness, thermal expansion coefficient, and specific heat are changeable according to the glass transition temperature. The temperature measured with DSC appears in a stepwise curve of base line. The measured temperature of glass transition Tg is 114.2˚C.

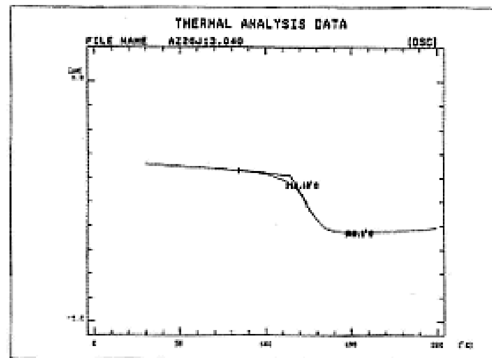

Fig.2 Tg of Epoxy Resin

Analysis of reacting speed of curing reaction (DSC)

The hardening process of epoxy resins must be measured as it is hardened in different ways depending on the curing conditions.
Determination of activation energy (ΔE) by an analysis of reacting speed is useful for predicting a possibility of a reaction to occur and the curing ratio under a thermostatic condition.
Fig3 is a result of an analysis on the reacting speed of epoxy resin. ΔE is 50.5KJ/mol.
Fig 4 is the result of the isothermal analysis of resin for predicting curing speed. It is known from this measurement that 7.8 hours was spent for 50% curing at 20˚C, 3.9 hours at 30˚C and 1.1 hours at 50˚C in each isothermal analysis.

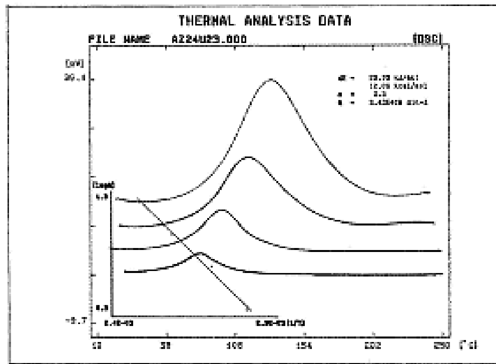

Fig.3 Reacting Speed Analysis

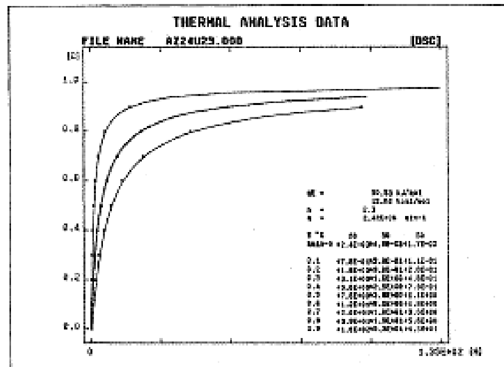

Fig.4 Isothermal Analysis

Determination of thermal expansion coefficient (TMA)

Thermal expansion of printed circuit boards is one of the most important points to watch in their production. This is why attention must be paid to the expansion coefficient (α). It should also be noted that α gets larger after Tg. Fig5 shows that the α after Tg is about four times the a before Tg, increasing from 5.71 × 10**-5/K to 2.36 × 10**-4/K. Excessive expansion in use may cause flexion and wiring breakage, for example.

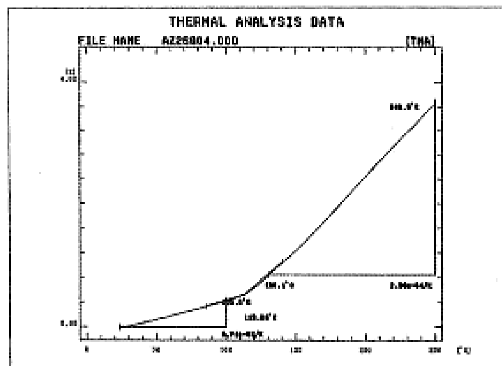

Fig.5 Thermal Expansion

Quantitative analysis of inorganic substance (TG)

Many molded products of thermosetting resin are made of composite materials containing an inorganic reinforcement substance for the enhancement of mechanical strength. Printed circuit boards, for example, are reinforced with glass fibers. If such composite materials consisting of organic and inorganic substances are heated in air with TG, organic substances are decomposed and inorganic substances are not. Fig.6 shows that 34.5% is shared by organic substances and the other 65.5% is by inorganic substances for reinforcement.

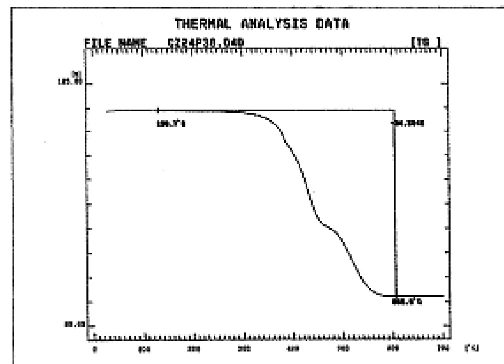

Fig.6 TG Curve (in Air)

* Please be advised that data obtained before the implementation of the current Weights and Measures Law may be presented in terms of gravimetric unit.

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