TA Instruments

Thermal Analysis & Rheology

THERMAL SOLUTIONS

CHARACTERIZATION OF RESIN CURING USING DMA / DEA / CONTROLLED STRESS RHEOLOGY

PROBLEM

Suppliers of thermosetting materials are interested in rapidly assessing important processing (curing) parameters such as the glass transition temperature (Tg), the point of minimum viscosity, the onset of gelation, and the completion of cure. Since many of these thermosets are powders or viscous liquids before cure and highly rigid solids after cure, evaluation of all of the critical curing parameters is difficult with a single analytical technique.

SOLUTION

Dynamic mechanical analysis (DMA), dielectric analysis (DEA) and controlled stress rheology (CSR) are techniques which have been widely used to measure the curing phenomena in thermoset materials (1-5). DMA, which tracks the modulus and damping properties of a material, is particularly valuable for evaluating the latter stages of cure as well as the characteristics of the fully cured final product. On the other hand, DEA, which measures the permittivity (capacitance) and conductance properties of a material, and rheology, which measures the flow properties of materials, are more valuable for evaluating the early stages of cure when the material is more "fluid".

Figures 1-3 show the comparatiave DMA, DEA, and CSR results for the curing of an epoxy resin. All three techniques indicate the onset of cure. Furthermore, DMA and CSR also indicate gelation. DEA and CSR indicate the minimum viscosity achieved before cure, while DMA indicates completion of cure and the final modulus (stiffness).

Figure 1. EPOXY CURE - DMA

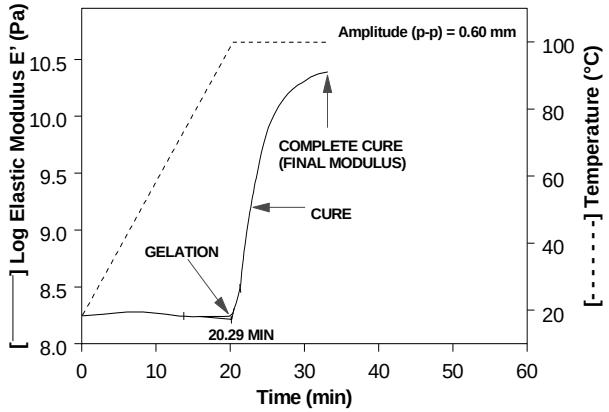
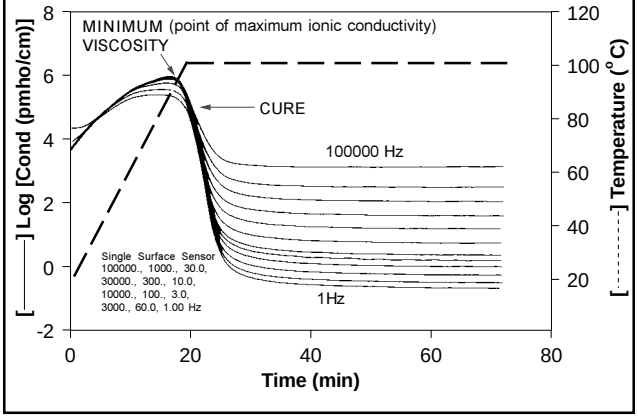


Figure 2. EPOXY CURE - DEA



In addition to the individual techniques, TA Instruments also supplies a remote DEA sensor which allows both DMA and DEA results to be obtained simultaneously on the same sample (Figure 4). Hence, in a single experiment complete information about the curing process can be obtained, including:

1. Glass Transition Temperature of the Uncured Material
2. Temperature of Minimum Viscosity
3. Onset of Gelation
4. Completion of Cure
5. Modulus when Fully Cured

By altering experimental conditions slightly to include isothermal segments and/or by running two different thermosets under identical experimental conditions, it is possible to expand the information obtained further to include:

1. "Working" time at the Minimum Viscosity before curing begins.
2. Time to achieve final cure.
3. Comparative data such as minimum viscosities and "working" times which enable selection of specific formulations for specific process situations.

REFERENCES

1. TA Instruments Applications Brief No. TA 100.
2. TA Instruments Applications Brief No. TA 101.
3. TA Instruments Applications Brief No. TA 103.
4. TA Instruments DMA 983 Product Brochure.
5. "DMA - A Versatile Technique for Characterization of Materials", I. Groves et al., Inter. Lab Mate, 1992.

Figure 3. EPOXY CURE - CSR

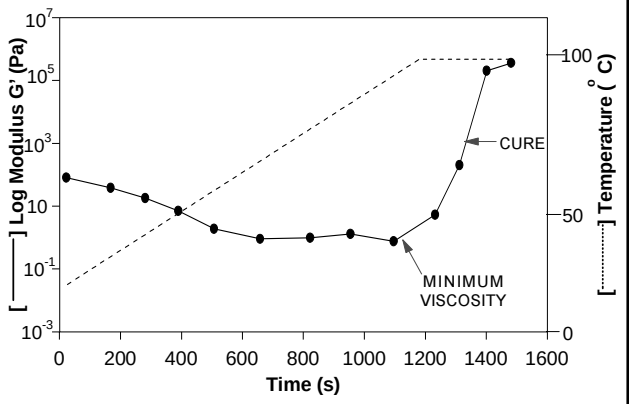
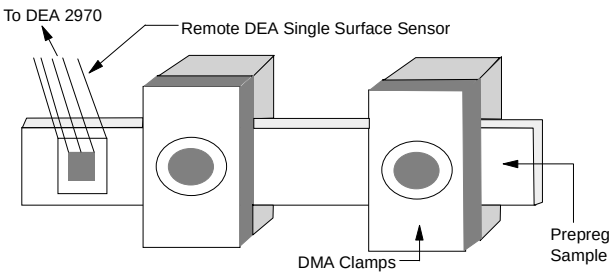


Figure 4. SIMULTANEOUS DMA / DEA CONFIGURATION



For more information or to place an order, contact:

TA Instruments, Inc.
109 Lukens Drive
New Castle, DE 19720
Telephone: (302) 427-4000
Fax: (302) 427-4001

TA Instruments N.V./S.A.
Gent, Belgium
Telephone: 32-9-220-79-89
Fax: 32-9-220-83-21

TA Instruments, Ltd.
Leatherhead, England
Telephone: 44-1-372-360363
Fax: 44-1-372-360135

TA Instruments S.A.R.L.
Paris, France
Telephone: 33-01-30489460
Fax: 33-01-30489451

TA Instruments GmbH
Alzenau, Germany
Telephone: 49-6023-30044
Fax: 49-6023-30823

TA Instruments Japan K.K.
Tokyo, Japan
Telephone: 813-5434-2771
Fax: 813-5434-2770

Internet: http://www.tainst.com

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