Insight —Technical Overview 3.01

What is Dielectric Cure Monitoring (DEA)?

Introduction

Dielectric cure monitoring, also known as Dielectric Analysis (DEA) is the only method that can measure thermoset or composite cure state in real time under actual process conditions. Consequently, DEA can generate results in the laboratory that are directly applicable in manufacturing.

In brief, DEA measures a thermoset's resistivity and permittivity, which are the material's dielectric properties. Resistivity itself has a frequency independent component due to the flow of mobile ions and a frequency dependent component due to the rotation of stationary dipoles.

Although often called DC resistivity for brevity, frequency independent resistivity actually extends across a range of frequencies that includes DC (0 Hz). Because frequency independent resistivity correlates with cure state, it is a useful material probe. By taking advantage of optimal frequencies, AC measurements—unlike DC methods—can deal with distortion of data caused by electrode polarization and can work through release films and vacuum bags.[1]

The term *ion viscosity* (*IV*) is a synonym for frequency independent resistivity and this technical overview will use *ion viscosity* instead of the more unwieldy *frequency independent resistivity*, and *viscosity* instead of *mechanical viscosity*.

Characteristics of thermoset cure

In many cases the change of *log(IV)* is proportional to the change of viscosity before gelation and proportional to the change of modulus after gelation. Consequently, DEA can observe cure throughout a process.

Figure 1-1 illustrates the relationship between ion viscosity and viscosity for a curing thermoset with one temperature ramp and hold. At first, as temperature increases, ion viscosity decreases because the thermoset becomes more fluid and therefore less resistive. The reaction rate increases as the material becomes hotter.

At some time, the increase in ion viscosity due to polymerization overcomes the decrease in ion viscosity due to rising temperature. This point is

the ion viscosity minimum, which also occurs at about the time of minimum viscosity.

After the minimum, ion viscosity increases as the reaction accelerates and the material becomes more viscous. At gelation, with the start of crosslinking and network formation, viscosity increases rapidly until it is unmeasurable. Ion viscosity, however, continues to provide information about cure state past the gel point.

As the concentration of unreacted monomers diminishes and crosslinking becomes more extensive, the reaction rate decreases; consequently, the slope of ion viscosity also decreases and eventually ion viscosity will have zero slope when cure has stopped completely.

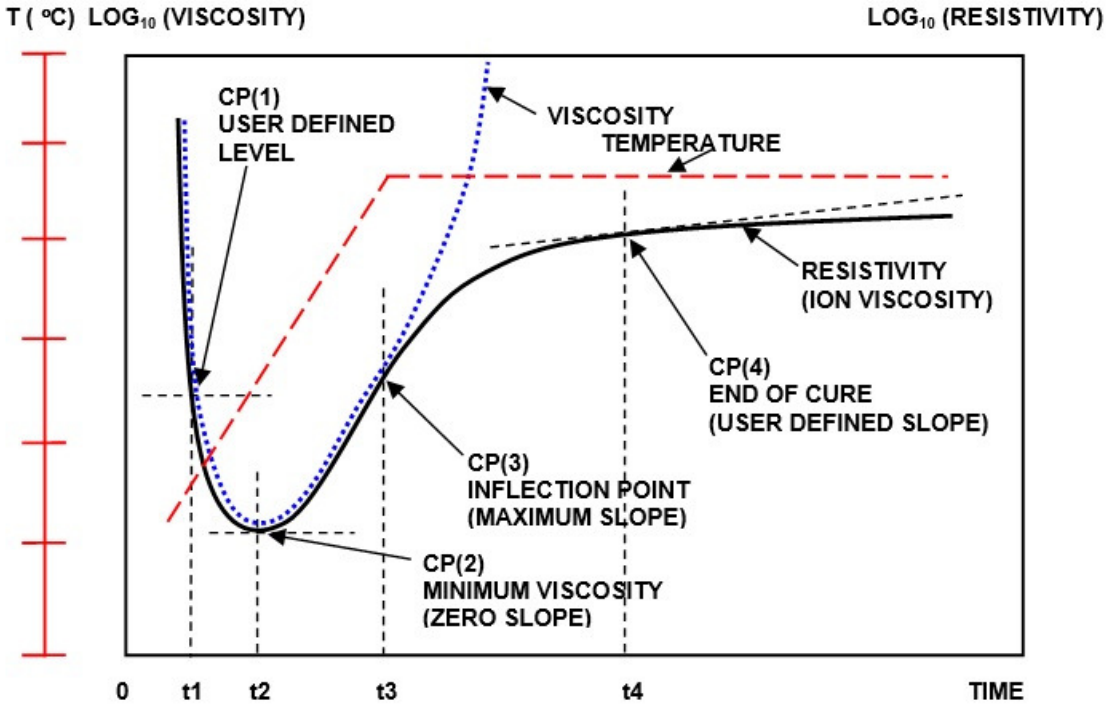

Figure 1-1
Ion viscosity and mechanical viscosity for thermoset cure

Figure 1-2 shows data from a typical thermoset cured with a temperature ramp and hold.

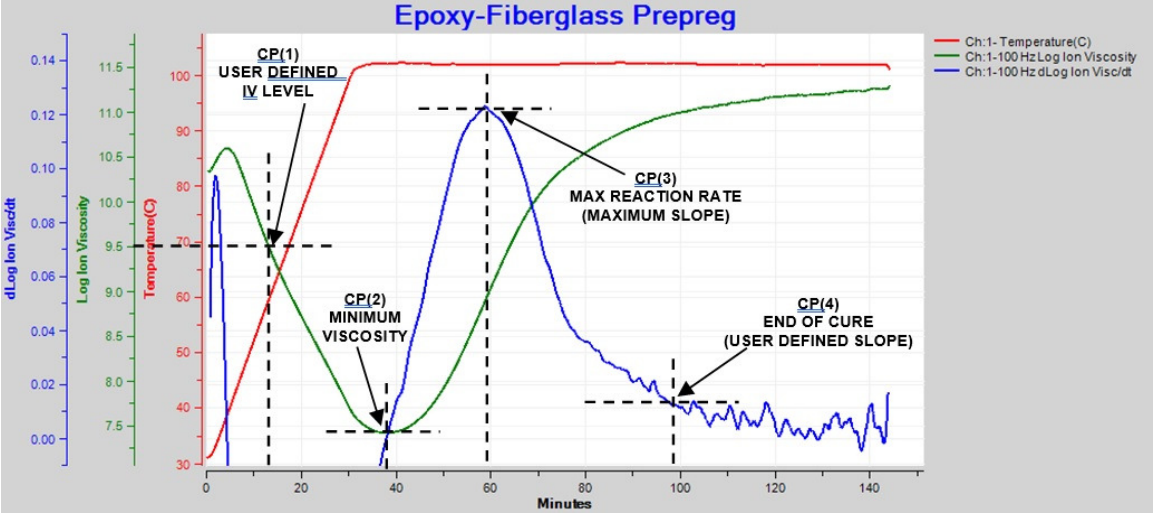

Figure 1-2
Ion viscosity and slope of ion viscosity for thermoset cure during thermal ramp and hold

As shown in Figures 1-1 and 1-2, four Critical Points characterize the data:

- CP(1)—A user defined level of log(*IV*) to identify the onset of material flow.
- CP(2)—Minimum ion viscosity to identify the time of minimum viscosity.
- CP(3)—Inflection point of log(*IV*) to identify the time of maximum reaction rate. The height of CP(3) is a relative measure of the reaction rate and the time of CP(3) is often used as a signpost associated with gelation.
- CP(4)—A user defined *slope* of log(*IV*) to identify end of cure.

The response is slightly different when the processing conditions are essentially isothermal, as shown in Figure 1-3. In this case, CP(1) either is meaningless or occurs at t = 0, immediately after the application of heat, when material flows to make contact with the sensor. Minimum ion viscosity also occurs at t = 0 or shortly afterwards because cure begins immediately.

For isothermal cures, CP(3) and CP(4) are conceptually the same as for ramp and hold conditions.

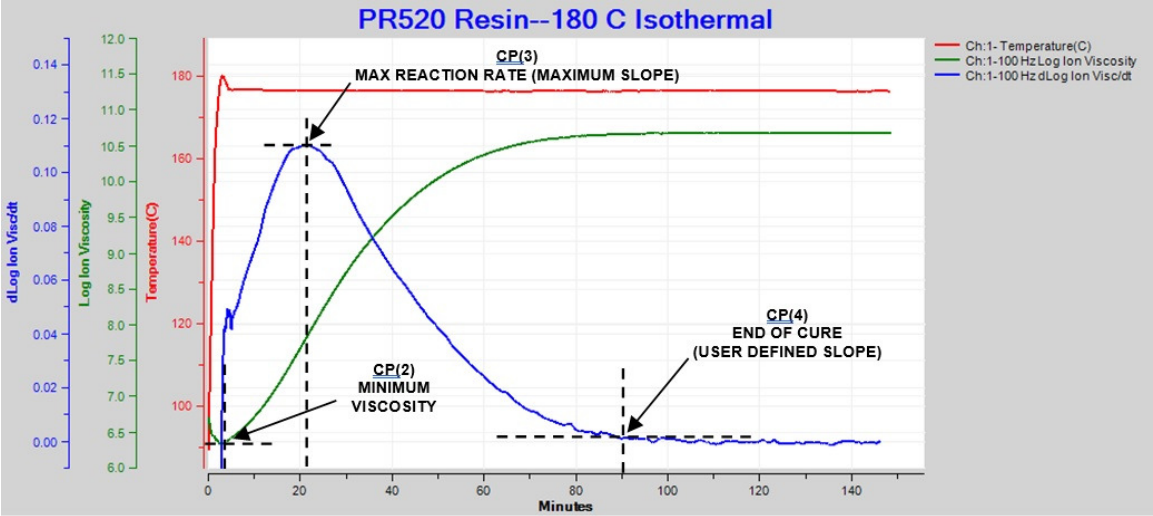

Figure 1-3
Ion viscosity and slope of ion viscosity for thermoset cure during isothermal processing

Correspondence of DEA with other properties

Figures 1-4, 1-5 and 1-6 show some examples from published research, comparing ion viscosity with viscosity, glass transition temperature and sound speed, respectively.

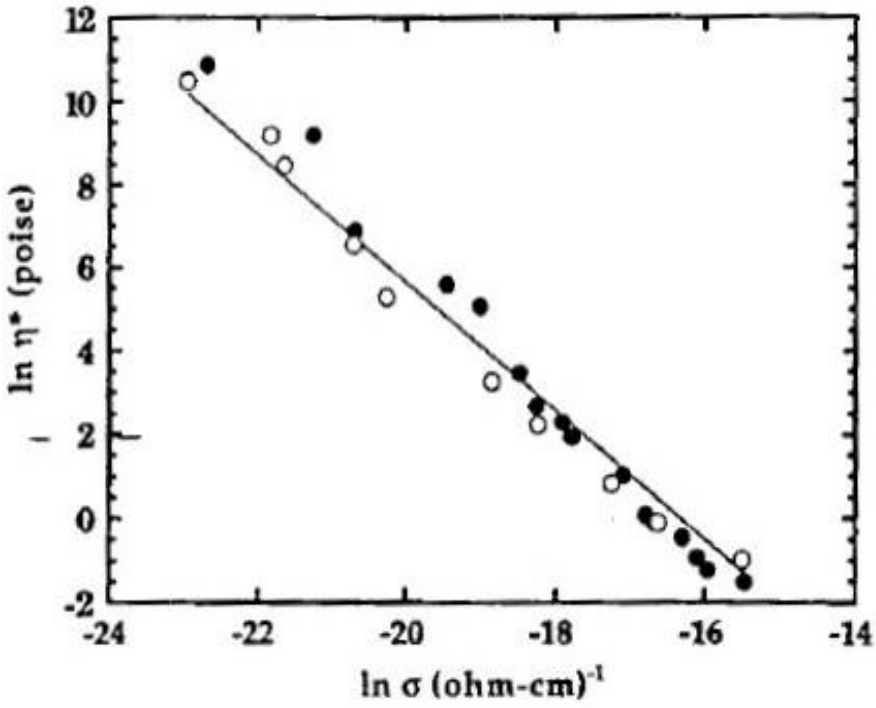

Figure 1-4
Viscosity vs. conductivity (ion viscosity = 1/conductivity) for an epoxy[2]

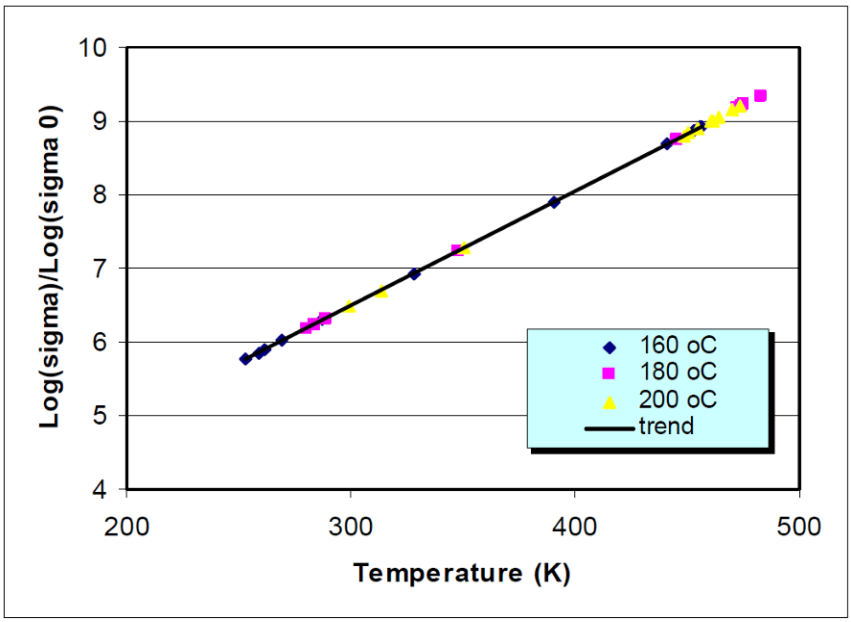

Figure 1-5
Conductivity (ion viscosity = 1/conductivity) vs. glass transition temperature for a carbon fiber-epoxy composite[3]

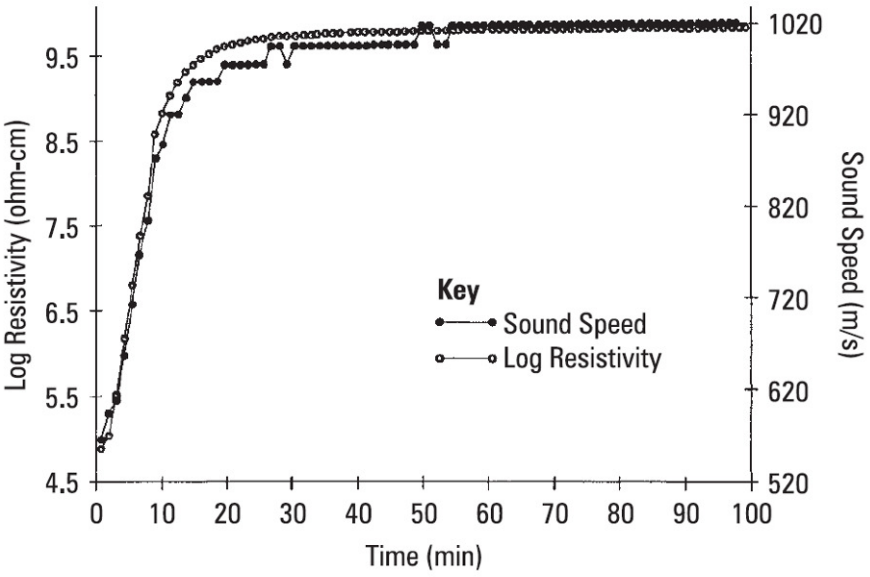

Figure 1-6
Resistivity (ion viscosity) vs. sound speed for a curing epoxy-fiberglass prepreg[4]

Note that every material is different and users should always confirm correlations of ion viscosity with a material's other properties. Nevertheless, dielectric cure monitoring (DEA) complements other thermal analysis techniques like differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA) as a valuable tool for studying curing thermosets and composites.

References

1. Application note AN2.39, "AC and DC Cure Monitoring Through Release Films and Vacuum Bags," Lambient Technologies, Cambridge MA

2. J. Simpson and S. Bidstrup, "Rheological and Dielectric Changes During Isothermal Epoxy-Amine Cure," J. of Polymer Science Part B: Polymer Physics, vol. 33, issue 1, pp 55-62 (1.1995)

3. J.H. Chen, M.A. Octeau, M. Hojjati and A. Yousefpour, ''Cure Cycle Optimization for Composite Panels Fabricated by RTM Using Dielectric Sensors,'' National Research Council Canada, Institute for Aerospace Research, ICCM International Conferences on Composite Materials (2009)

4. D. Shepard and K. Smith, ''Ultrasonic Cure Monitoring of Advanced Composites,'' Sensor Review, vol. 19 Issue: 3, pp. 187-194 (1999)

Lambient Technologies, LLC
649 Massachusetts Ave., Cambridge MA 02139, USA
(857) 242-3963
https://lambient.com
info@lambient.com

