Insight — Application Note 3.15

Cure Monitoring of Epoxy Molding Compound (EMC)

Epoxy molding compound for electronic packaging

The curing behavior of epoxy molding compound (EMC) was observed using the LTF-631 High Speed Dielectric Cure Monitor.[1] Data from dielectric cure monitoring (DEA) clearly show:

- Critical Points identify characteristic features of the cure such as minimum ion viscosity, maximum slope of log(ion viscosity) and the time to a chosen end of cure.
- Cure time decreases and reaction rate increases as cure temperature increases, as expected for a reaction that is thermally driven.
- Dielectric cure monitoring of EMC samples show very high repeatability.

Normally available in either B-staged powders or pellets, as shown in Figure 15-1, for transfer molding, EMC is essential for electronics packaging and is used to encapsulate billions of integrated circuits each year.

Figure 15-1
Granular (left) and pelletized (right) epoxy molding compound[2]

Definitions

This application note presents and discusses data for *log(ion viscosity)* and *slope of log(ion viscosity)*, which indicate the state of cure. The plots show characteristic features such as minimum ion viscosity, maximum slope of log(ion viscosity) and the time to a chosen end of cure. For brevity, log(ion viscosity) will be called *log(IV)* and slope of log(ion viscosity) will simply be called *slope*.

Electrical conductivity (σ) has both frequency independent (σ_{DC}) and frequency dependent (σ_{AC}) components. In an oscillating electric field, σ_{DC} arises from the flow of mobile ions while σ_{AC} arises from the rotation of stationary dipoles. These two responses act like electrical elements in parallel and are added together as expressed below:

(eq. 15-1) $\sigma = \sigma_{DC} + \sigma_{AC}$ ($ohm^{-1} - cm^{-1}$)

Resistivity (ρ) is the inverse of conductivity and is defined as:

(eq. 15-2) $\rho = 1/\sigma$ (ohm-cm)

From its relationship to conductivity, resistivity also has both frequency independent (ρ_{DC}) and frequency dependent (ρ_{AC}) components. The amount of polymerization or crosslink density, which are measures of cure state, affect both mechanical viscosity and the movement of ions, and therefore influence ρ_{DC}. As a result, the term *Ion Viscosity* was coined to emphasize the relationship between mechanical viscosity and ρ_{DC}. Ion viscosity (*IV*) is defined as:

(eq. 15-3) $IV = \rho_{DC}$ (ohm-cm)

Although the strict definition of ion viscosity is frequency independent resistivity, ρ_{DC}, for convenience ion viscosity may also be used to describe resistivity in general, which has both frequency independent (ρ_{DC}) as well as frequency dependent (ρ_{AC}) components. ***Note, however, that cure state and mechanical viscosity relate best to frequency independent resistivity, ρ_{DC}, which is true ion viscosity***.

Spiral flow test

The viscosity and curing behavior of EMC vary with the hundreds of available formulations and the high-speed, high-volume requirements of manufacturing demand consistent, reliable material performance. Presented schematically in Figure 15-2, the spiral flow test is one of the principal methods for evaluating EMC. This test uses a mold with an Archimedean spiral channel and EMC is injected into it with a specified charge mass, mold temperature and transfer plunger speed.

As material flows through the channel, it cures, undergoes gelation and finally solidifies enough to stop flow. The length of the cured portion, shown in Figure 15-3, is a measure of the combined characteristics of fusion under pressure, melt viscosity and gelation rate under the test conditions.

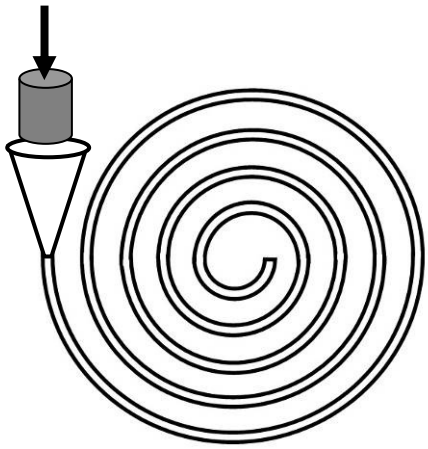

Figure 15-2
Spiral flow test of epoxy molding compound with initial charge

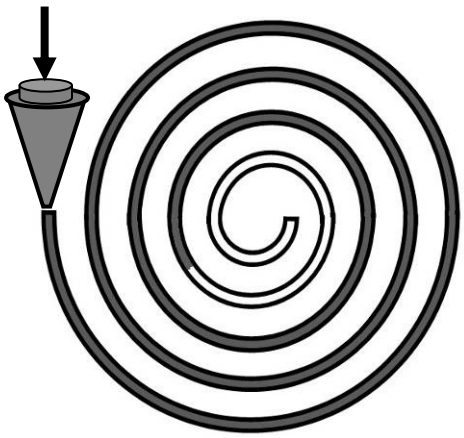

Figure 15-3
Spiral flow test after solidification and flow has stopped

Integrated circuits are manufactured with steps similar to those of Figure 15-4, in which EMC is heated until it melts then is injected into a mold with cavities for each integrated circuit. With a process temperature usually between 160 °C and 180 °C, the EMC must flow with low viscosity to avoid damaging delicate wire bonds and must fill the mold before gelation.

To maximize productivity, parts are removed from the mold when the EMC has reached about 40% epoxy conversion and is rigid enough to retain its form. Then they are baked in ovens at about 175 °C for an additional three or four hours until full cure.

Although the spiral flow test roughly emulates this process up to the gel point, it gives little insight into how the material cures. Nevertheless, if a manufacturer has established that consistent, specific results of the spiral flow test correspond to well molded products, then any change in test results is a warning that the EMC is not behaving as required.

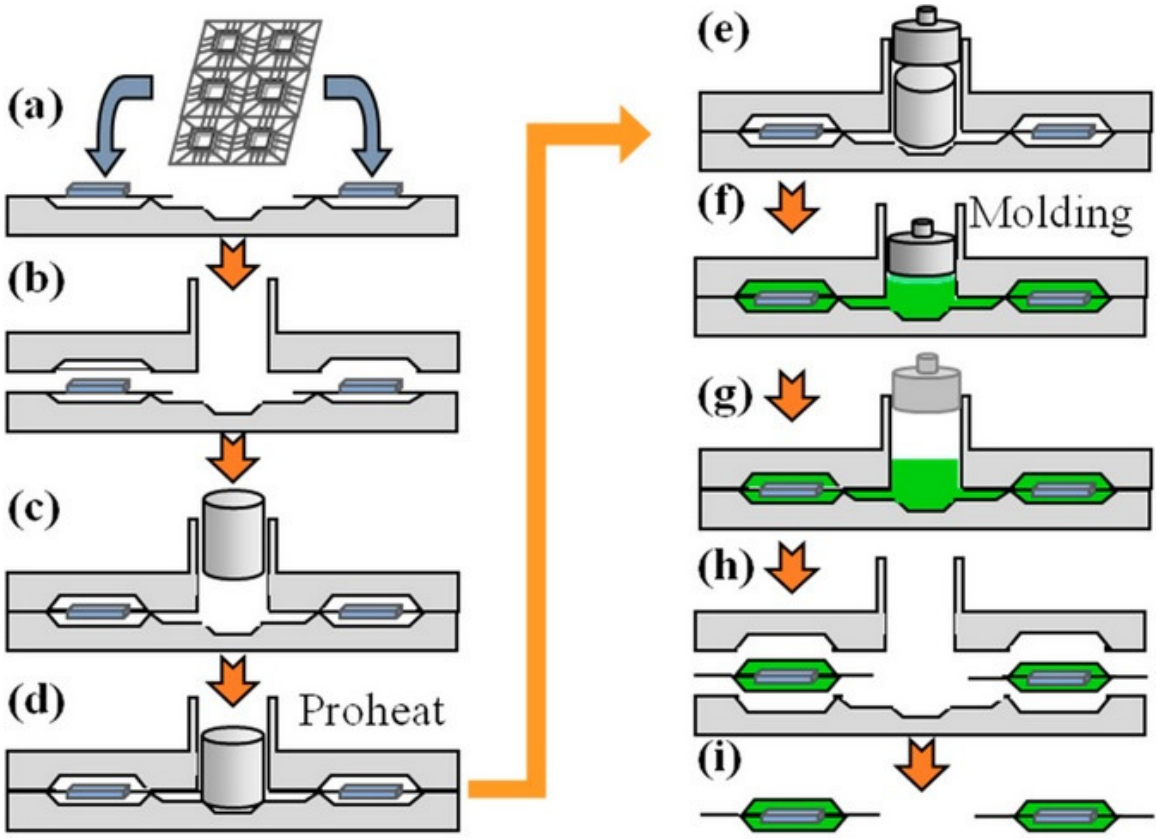

Figure 15-4
Steps in the EMC transfer molding process for electronic packaging[3]

Dielectric cure monitoring (DEA) can provide valuable additional information for quality control and manufacturing. The viscosity of EMC and the result of the spiral flow test depend on how the resin interacts with the filler, which often consists of spherical silica particles. The cure of EMC, however, depends only on the resin, which is commonly epoxy cresol novolac with phenolic curing agents.

Procedure

Samples of EMC were placed on a reusable 1" Single-Electrode Sensor,[4] shown in Figure 15-5, then compressed and cured in an LTP-250 MicroPress[5], which applied pressure and heat for separate runs at 150 °C, 160 °C, 170 °C and 180 °C. Previous tests had identified 10 Hz as an optimum excitation frequency for cure monitoring.

The cure time for these samples can be less than three minutes so an LTF-631 High Speed Dielectric Cure Monitor[1] measured the dielectric properties of each sample. The measurement interval was 100 ms/data point and a trigger on the LTP-250 initiated data acquisition at a consistent point in the compression cycle. CureView software acquired and stored the data, and later performed Critical Point analysis and presentation of the results.

Figure 15-5
Single-Electrode reusable sensor

Results

Figures 15-6, 7, 8, 9 show data from the cures of EMC at 150 °C, 160 °C, 170 °C and 180 °C, respectively. For each cure *log(IV)* and *slope* follow the typical behavior of a thermoset during isothermal cure. The ability of dielectric

measurements to observe the effect of temperature on cure is apparent in this sequence of plots.

As expected for a thermally activated reaction, the *log(IV)* curves rise and flatten more quickly with increasing temperature. The ion viscosity minimum—CP(2)—and the peak slope—CP(3)—also occur sooner at higher temperatures. Furthermore, the peak value of CP(3), which is related to the maximum reaction rate, increases with temperature. After acquiring these data, CureView was able to extract the Critical Points that characterize each cure and allow direct comparison of their behavior across the temperature range.

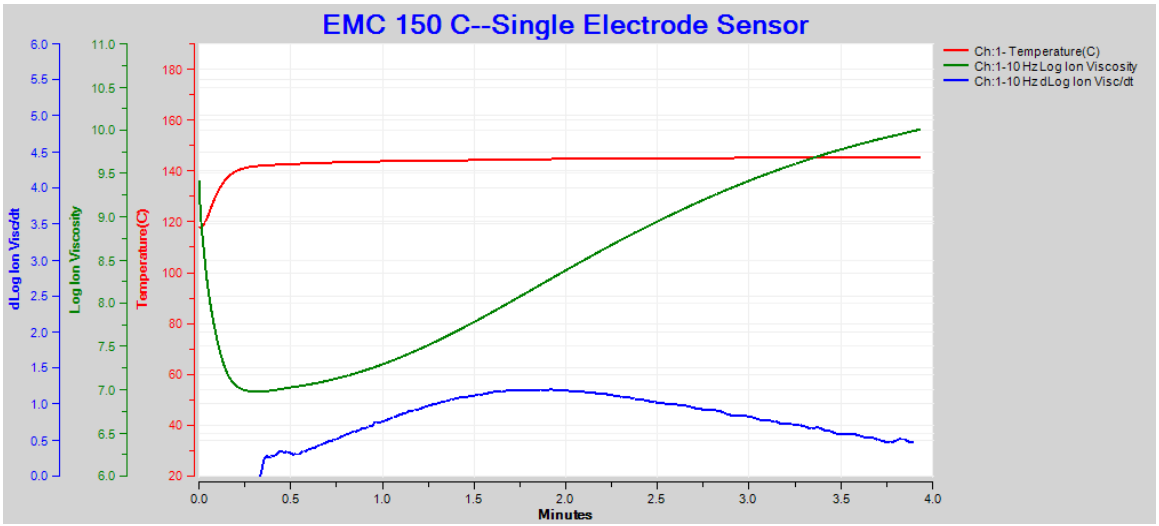

Figure 15-6
150 °C EMC cure data at 10 Hz

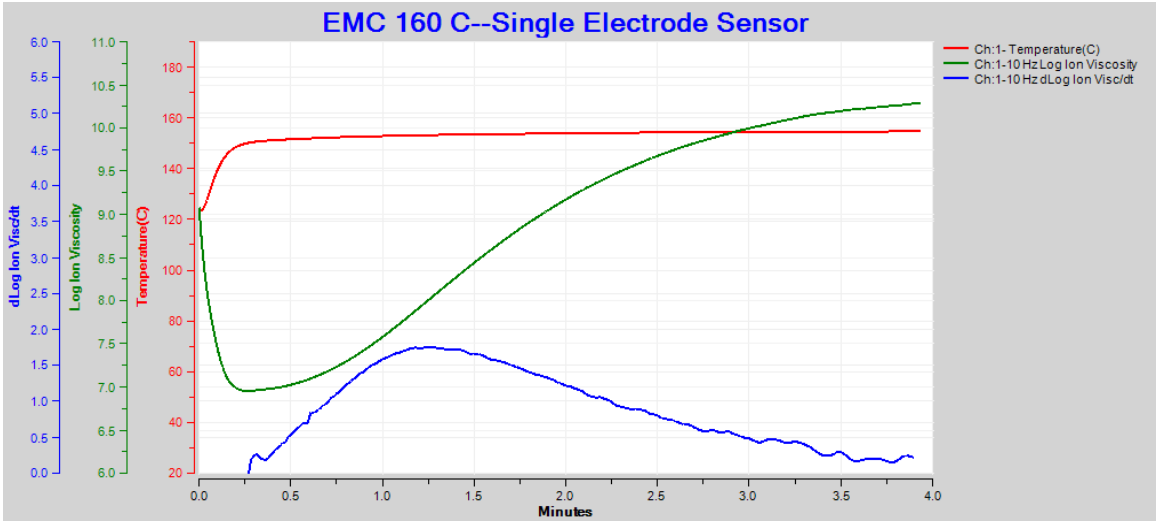

Figure 15-7
160 °C EMC cure data at 10 Hz

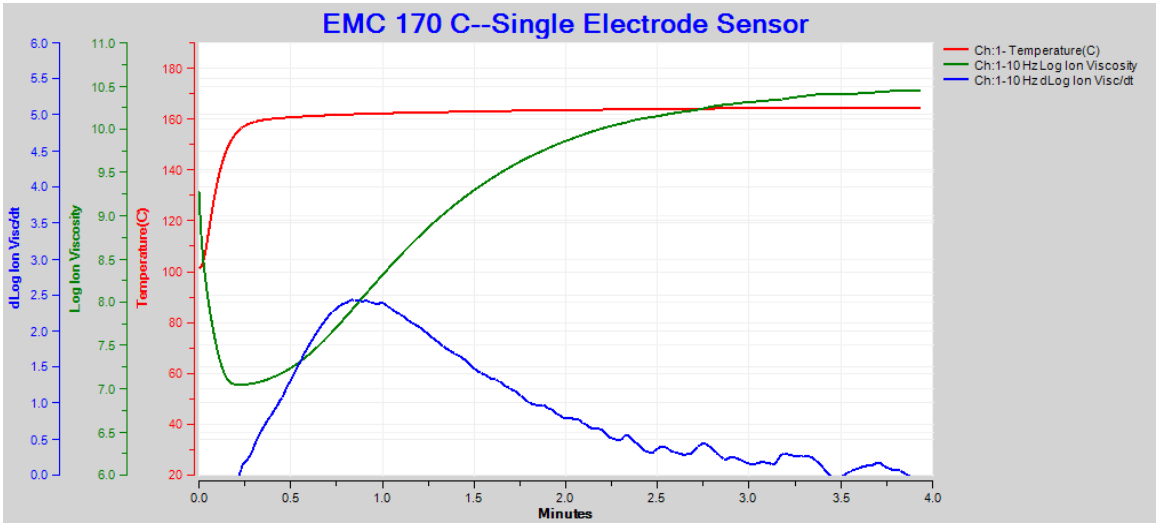

Figure 15-8
170 °C EMC cure data at 10 Hz

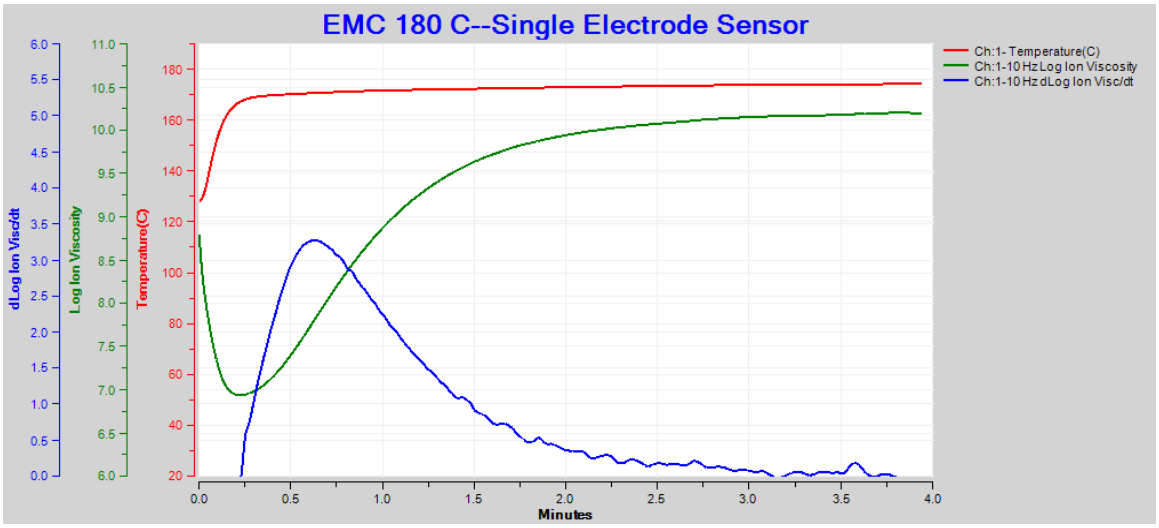

Figure 15-9
180 °C EMC cure data at 10 Hz

Figure 15-10 overlays the *log(IV)* and *slope* curves for these four cures. This comparison shows the sensitivity of dielectric cure monitoring to changes in cure due to temperature differences.

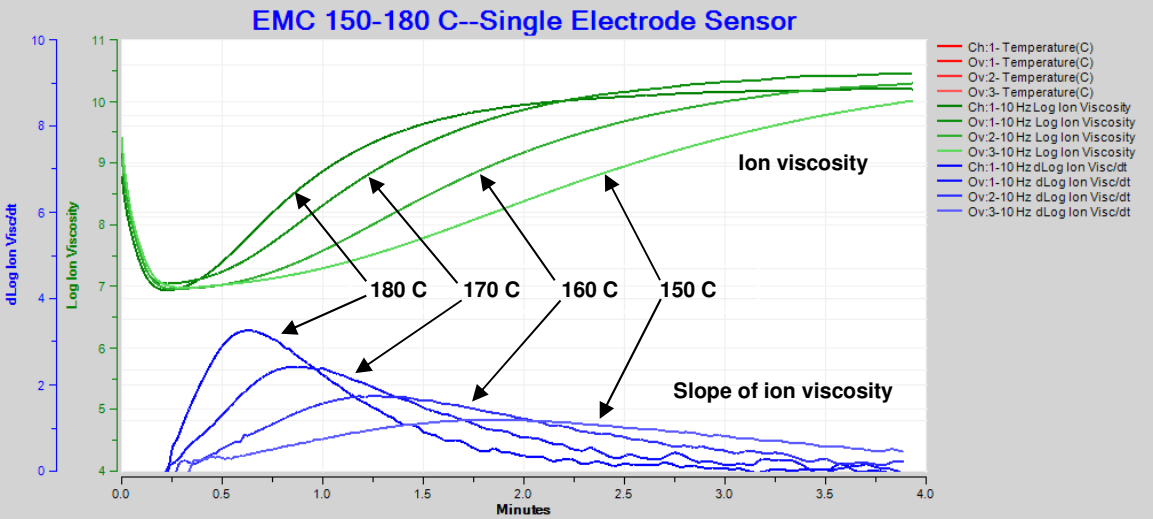

Figure 15-10
EMC *ion viscosity* (green) and *slope* (blue) at 10 Hz, cures at 150 °C, 160 °C, 170 °C and 180 °C

As expected, the maximum value of *slope* increases with process temperature and shows the relationship between reaction rate and temperature. Critical Points that characterize each cure are shown in Table 15-1, with the following notes:

- The time to CP(1) indicates onset of flow and is not a measure of cure, so CP(1) data are not shown
- The slope of 0.5 to define CP(4) was chosen arbitrarily; in fact, a user must determine a suitable slope based on the needs of the application to indicate end of cure.

Table 15-1
Critical Points from EMC cure monitoring

Cure Temp. (℃)	CP(1) Crit. Visc.		CP(2) Min. Visc.		CP(3) Max Slope		CP(4) End of Cure	
	Value	Time	Value	Time	Value	Time	Value	Time
150	---	---	6.99	0.334 m (20.0 s)	1.21	1.92 m (114.9 s)	0.50	3.728 m (223.7 s)
160	---	---	6.96	0.270 m (16.3 s)	1.76	1.26 m (75.9 s)	0.50	2.997 m (179.8 s)
170	---	---	7.06	0.218 m (13.1 s)	2.44	0.833 m (50.0 s)	0.50	2.282 m (136.9 s)
180	---	---	6.95	0.228 m (13.7 s)	3.28	0.636 m (38.2 s)	0.50	1.779 m (106.7 s)

The time to Critical Point 2—CP(2)—is the point when the EMC has the lowest mechanical viscosity. This information is often useful for identifying the optimum time to apply compression to squeeze out voids, consolidate the layers of a laminate or fill a mold.

The time to Critical Point 3—CP(3)—indicates the moment of fastest reaction. Before CP(3) the reaction is accelerating as temperature increases from the exotherm and external heating. After CP(3) the reaction slows as the network grows and monomers are depleted. **Although CP(3) is not the gel point, CP(3) is often used as a signpost *associated* with the gel point.**

The time to Critical Point 4—CP(4)—is the time to a user defined slope indicating end of cure. True end of cure occurs when the reaction stops and the material is no longer changing; at this time the slope is zero. The reaction may continue at a very low level for considerable time, so for practical purposes a small, non-zero slope is usually selected, with a value depending on the needs of the application.

The times to reach CP(2) depend largely on how quickly the EMC heats and do not significantly reflect the rate of cure, so it is not surprising to see fairly constant times at different process temperatures. As seen in Figure 15-11, the times to reach CP(3) and CP(4) decrease as temperature increases, which is expected for thermally driven reactions.

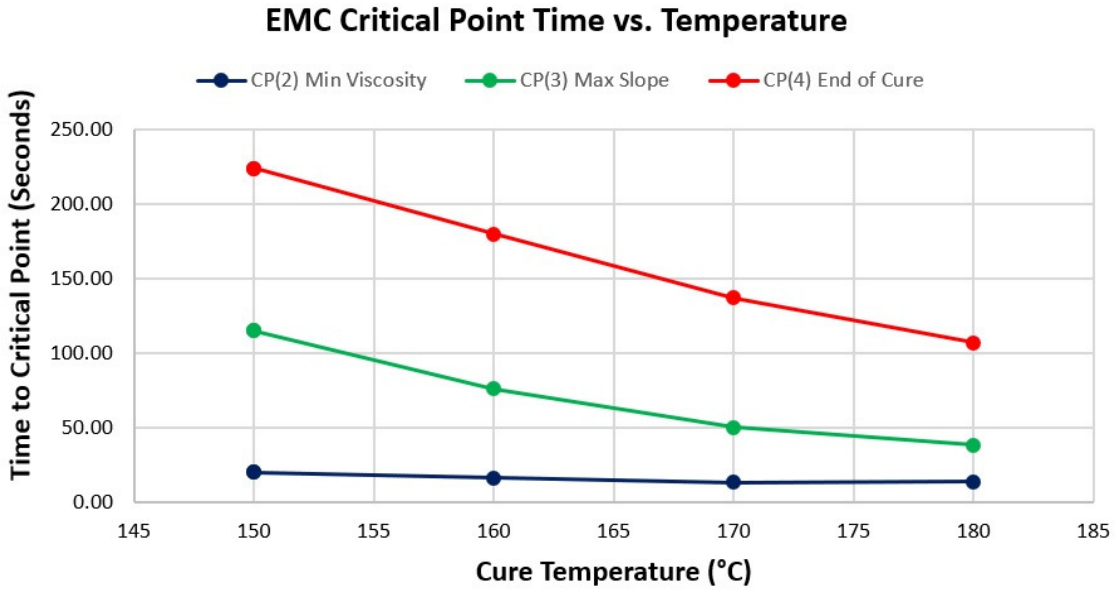

Figure 15-11
Critical Point time vs. cure temperature for BMC

Figure 15-12 shows how the maximum value of *slope* increases with temperature. Again, this relationship is expected because the height of CP(3) is a relative measure of the maximum reaction rate.

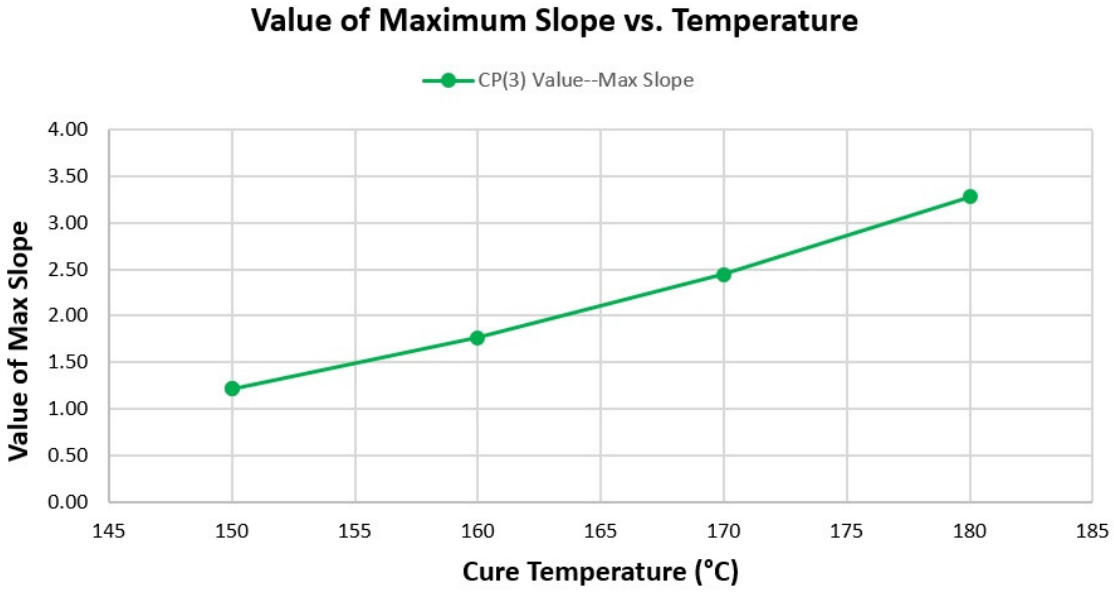

Figure 15-12
Value of maximum slope vs. cure temperature for EMC

Quality control of epoxy molding compound

The spiral flow test provides limited insight into a material because a single number—the length of the solidified EMC—can only partly characterize the combined behavior of cure and viscosity. In the high-speed, high-volume manufacture of integrated circuits, consistency is an encapsulant's most important quality. Dielectric cure monitoring can supplement the spiral flow test by providing information about the entire cure, allowing the documentation of consistency in far greater detail.

Figure 15-13 shows data from four consecutive tests of EMC at 170 °C. In this case the ion viscosity curves are virtually indistinguishable from one another, verifying high repeatability. When dielectric cure monitoring is used for quality control of outgoing material, formulators of EMC can assure customers their product has been fully tested for consistency.

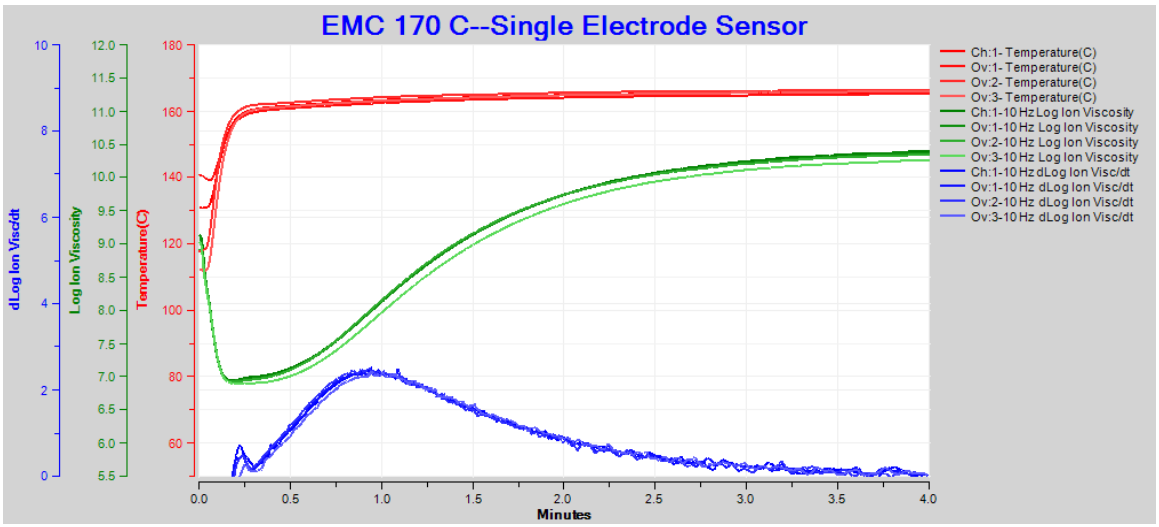

Figure 15-13
Repeatability of EMC cure at 170 °C (four consecutive tests)

Some electronics manufacturers have installed dielectric sensors in both the input and output ports of a mold, illustrated schematically in Figure 15-14.

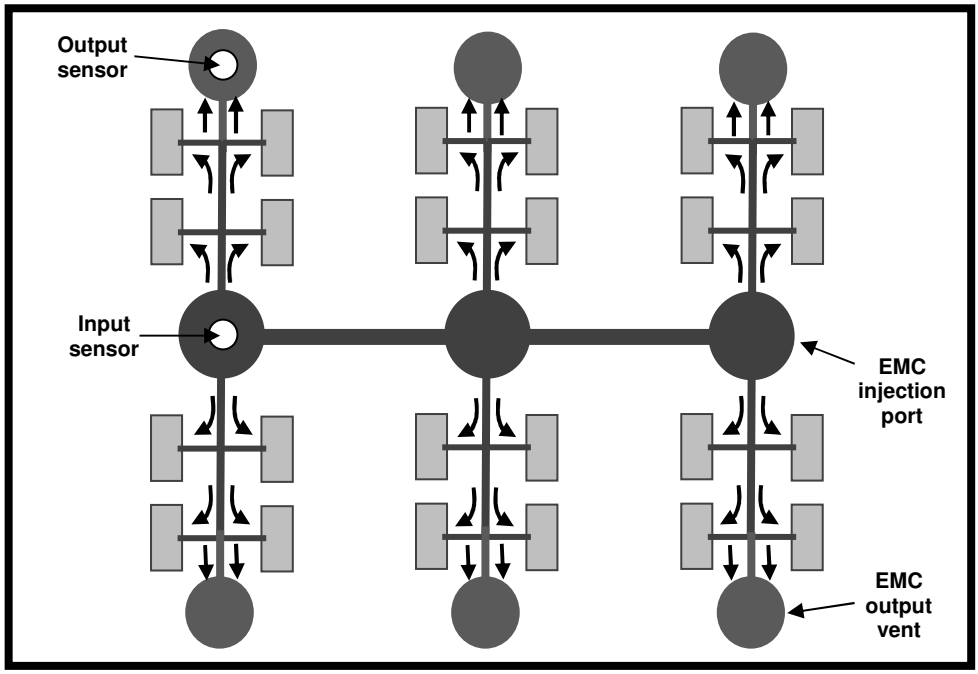

Figure 15-14
Mold with sensors for EMC cure monitoring

By continuously monitoring the ion viscosity of EMC at both the injection port and output vent, it is possible to understand the behavior of the material between these two points and determine:

- Flow time from input to output
- Variation of minimum viscosity at the mold cavities
- Cure rate at mold cavities
- Cure state at time of demold

These additional parameters provide important information for keeping the packaging process in control. Changes beyond specified limits warn the manufacturing engineer of potential problems with the epoxy molding compound.

Critical Points during thermoset cure

A thermoset cures when monomers react to form polymer chains then a network. The reaction is usually exothermic—generating heat—and may additionally be driven by the heat of a press or oven. A plot of log(*ion viscosity*) is a simple way to characterize the progress of cure and Figure 15-15 shows the behavior of a typical thermoset with one ramp and hold step in temperature.

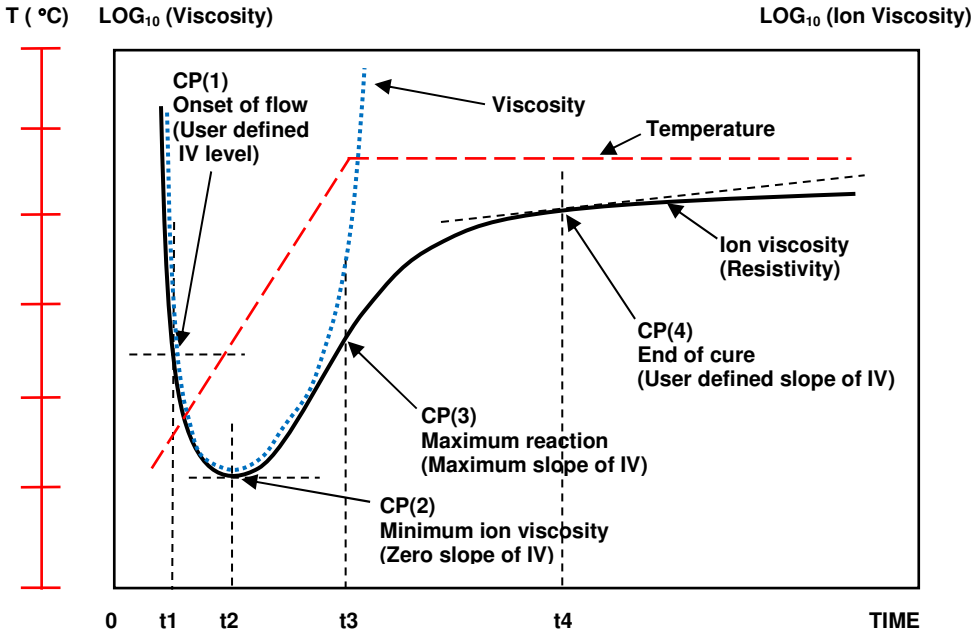

Figure 15-15
Typical ion viscosity behavior of thermoset cure during thermal ramp and hold

At first as temperature increases, the material softens or melts and mechanical viscosity decreases. Mobile ions also experience less resistance to movement and ion viscosity decreases. At this point the reaction is still slow.

As the material becomes hotter, the cure rate increases. At some time the accelerating reaction begins to dominate; mechanical viscosity reaches a minimum then the material becomes more viscous. Electrically, the increase in ion viscosity due to polymerization overcomes the decrease in ion viscosity due to higher temperature. Ion viscosity also reaches a minimum then increases due to chain extension, which presents a greater and greater impediment to the flow of ions.

After the minimum point, ion viscosity increases continuously until the concentration of unreacted monomers diminishes and the reaction rate decreases. Consequently, the slope of ion viscosity also decreases and eventually reaches a value of zero when cure has stopped completely.

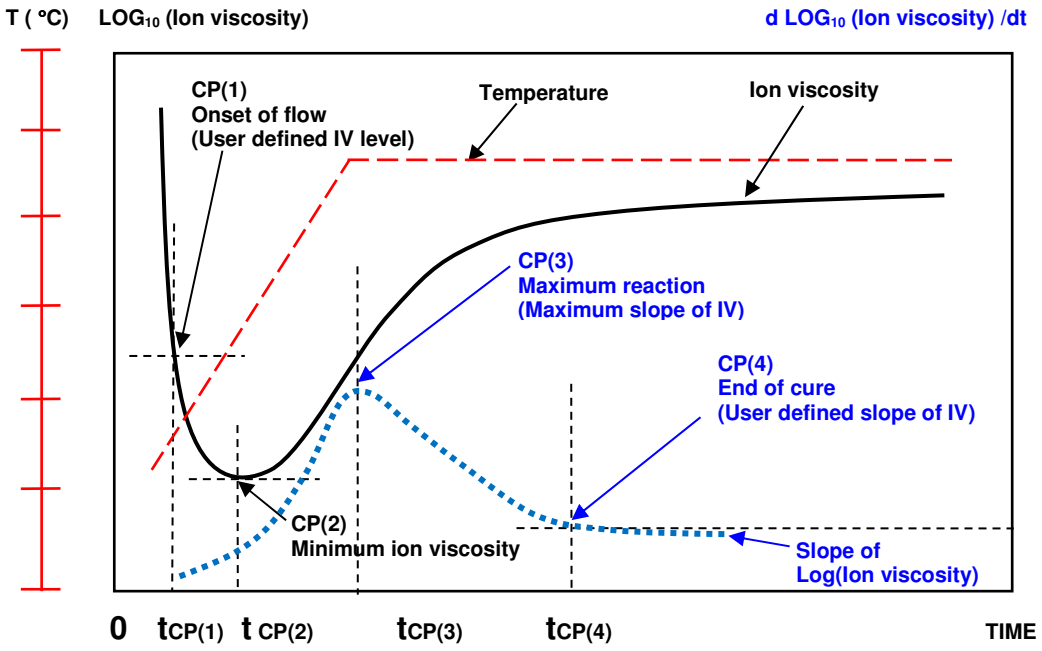

Figure 15-16
Ion viscosity curve and slope of ion viscosity of thermoset cure during thermal ramp and hold

As shown in Figure 15-16, four Critical Points characterize the dielectric cure curve:

- CP(1)—A user defined level of *log(IV)* to identify the onset of material flow.
- CP(2)—Minimum ion viscosity, which closely corresponds to minimum mechanical viscosity, indicating when polymerization and increasing viscosity begin to dominate the material's behavior.
- CP(3)—Maximum *slope*, which identifies the time of maximum reaction rate. The height of CP(3) is a relative measure of the reaction rate and CP(3) is often used as a signpost associated with gelation.
- CP(4)—A user defined *slope* that can define the end of cure. The decreasing *slope* corresponds to the decreasing reaction rate.

Figures 15-15 and 15-16 illustrate the typical behavior of curing thermosets when temperature gradually ramps to a hold value. The response is slightly different when the material under test is essentially isothermal, as shown in Figure 15-17.

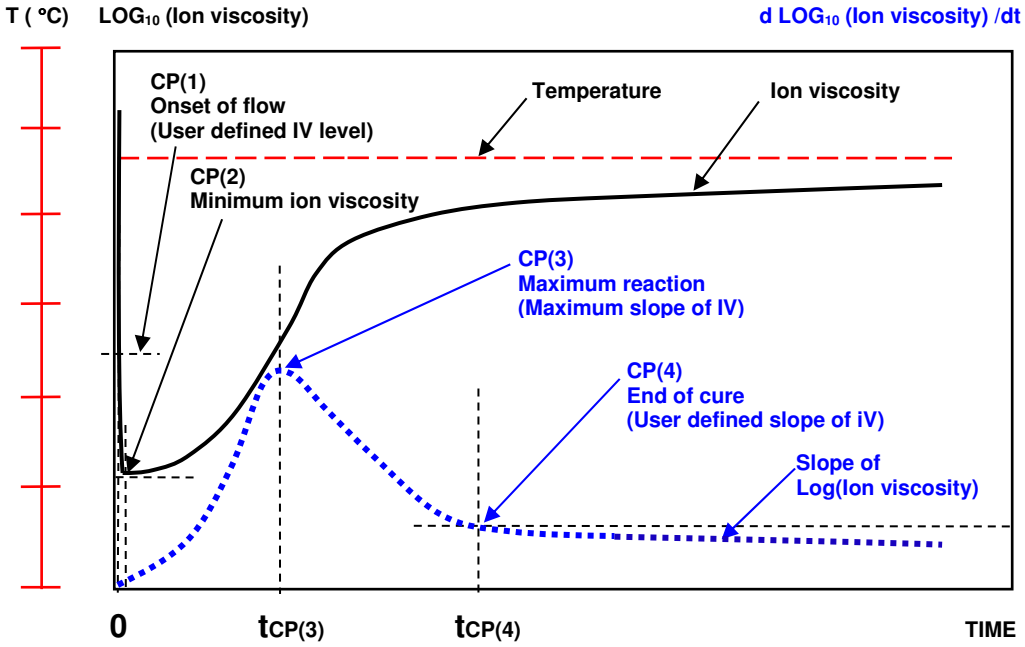

Figure 15-17
Ion viscosity curve and slope of ion viscosity of thermoset cure during isothermal processing

In this case CP(1) either is meaningless or occurs immediately after the application of heat, when material flows and contacts 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.

References

1. LTF-631 High Speed Dielectric Cure Monitor, manufactured by Lambient Technologies, Cambridge, MA USA. https://lambient.com

2. Epoxy molding compound picture credit: Shyama Construction & Waterproofing Co., Kolkata, West Bengal, India

3. Huang, Wan-Chiech; Hsu, Chao-Ming and Yang, Cheng-Fu, "Recycling and Refurbishing of Epoxy Packaging Mold Ports and Plungers," *Inventions* **2016**, *1*(2), 11; https://doi.org/10.3390/inventions1020011, June 6, 2016

4. 1" Single-Electrode sensor, manufactured by Lambient Technologies, Cambridge, MA USA

5. LTP-250 MicroPress, manufactured by Lambient Technologies, Cambridge, MA USA

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