Insight — Application Note 3.39

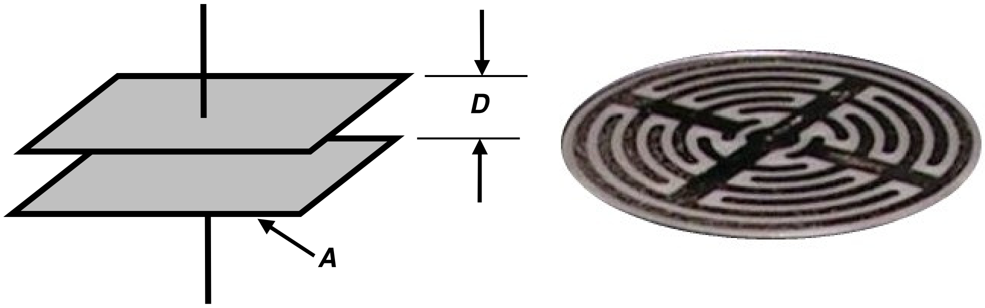
Sensor Geometry and Measurements Of Cure State

Introduction

Originally, studies of thermoset and composite cure used simple parallel plate electrodes like those of Figure 39-1 a. The ratio of electrode area *A* and the distance *D* between them—the *A*/*D* ratio—is the cell constant, which is a scaling factor between material and bulk properties, allowing calculation of resistivity from resistance, or permittivity from capacitance. As distance increases between parallel plate electrodes, the sensitivity and scaling factor decrease.

A common alternative is the interdigitated or comb sensor, which uses a rigid substrate to support its electrodes, as in Figure 39-1 b. Unlike parallel plates, as long as the Material Under Test (MUT) has a thickness greater than the electrode separation, the sensitivity of comb electrodes is constant.

The concept of an *A*/*D* ratio for parallel plate electrodes may be generalized to comb structures. But in this case *A* is not simply the area of the electrodes and *D* is not simply the distance between them. For interdigitated electrodes the *A*/*D* ratio is the term, by analogy with parallel plates, that defines the scaling factor between resistivity and resistance, or between permittivity and capacitance.



**Parallel plates—
A/D ratio depends on separation**

**Interdigitated electrodes—
A/D ratio is fixed**

a. **b.**

**Figure 39-1
Comparison of parallel plate and interdigitated electrodes**

Comparison of sensor geometries

Used for thermoset and composite cure monitoring, the Mini-Varicon[1], Ceramicomb-1″[2], Unitrode-1″[3] and Carbon+Unitrode-1″[4] sensors of Figure 39-2 have very different constructions and electrode configurations, as summarized in Table 39-1. Nevertheless, with the proper choice of A/D, different sensors measuring resistivity (ρ), also known as *ion viscosity* (*IV*), should produce the same result.

A second sensor parameter, called *Base Capacitance* (C_B), is related to the capacitance contributed by the substrate and cabling, and is subtracted from a raw measurement for calculations of permittivity. Base capacitance, however, does not affect the measurement of ion viscosity.

a. **b.**

c. **d.**

Figure 39-2
Mini-Varicon sensor (a.), Ceramicomb-1″ sensor (b.), Unitrode-1″ sensor (c.) and Carbon+Unitrode-1″ sensor (d.)

Table 39-1
Comparison of dielectric sensors

Sensor	Mini-Varicon	Ceramicomb -1”	Unitrode -1”	Carbon+ Unitrode-1”
Electric Field Type	Fringe	Fringe	Fringe + parallel plate	Fringe + parallel plate
Electrode Design	Interdigitated 2-electrode	Interdigitated 2-electrode	Disk 1-electrode	Coated Disk 1-electrode
Electrode Width	0.004” (100 µm)	0.020” (0.5 mm)	0.50” dia. (12.7 mm)	0.50” dia. (12.7 mm)
Electrode Spacing	0.004” (100 µm)	0.020” (0.5 mm)	0.062” for test (1.6 mm)	0.062” for test (1.6 mm)
Substrate	Polyimide ($\varepsilon_r = 3.6$)	Alumina ($\varepsilon_r = 9.8$)	High temp thermoplastic ($\varepsilon_r = 4.2$)	High temp thermoplastic ($\varepsilon_r = 4.2$)
***A*/*D* Ratio**	80 cm	10 cm	6 cm for 1.6 mm sample	5 cm for 1.6 mm sample
Relative Sensitivity	16.0	2.0	1.2	1.0
Base Capacitance	~30 pF	~30 pF	~13 pF	~20 pF

Procedure

To compare the results of different geometries, the four sensors of Table 39-1 were used to monitor the cure of epoxy molding compound (EMC). The Ceramicomb-1″, the Unitrode-1″ and the Carbon+Unitrode-1″ sensors were embedded in steel platens heated in an LTP-250 MicroPress[5]. For its tests, the Mini-Varicon was placed on a similar platen so its thermal environment was the same as the other sensors.

During cure at 170 °C, each sample was initially a powder. The LTP-250 MicroPress applied heat and pressure to mold the EMC to a final thickness of 0.062″ (1.6 mm), which was determined by a Teflon spacer. The experimental set up is shown in Figure 39-3.

The A/D ratios for the Mini-Varicon and Ceramicomb-1″ sensors are constant at 80 cm and 10 cm, respectively, because the 1.6 mm sample thickness is greater than their electrode spacing. The Unitrode-1″ and Carbon+Unitrode-1″

sensors, however, have a parallel plate configuration, and their A/D ratios depend on the separation between their electrode and the upper heater platen, which acts as the second electrode. Because fringing electric fields complicate the calculation of a cell constant, the A/D ratio for a parallel plate sensor is usually determined experimentally. Measurements for a 1.6 mm sample thickness found A/D = 6 for the Unitrode-1″ sensor, and A/D = 5 for the Carbon+Unitrode-1″ sensor.

- For more information about A/D ratio and base capacitance, see application note *AN 3.06, Sensors A-D Ratio and Base Capacitance*

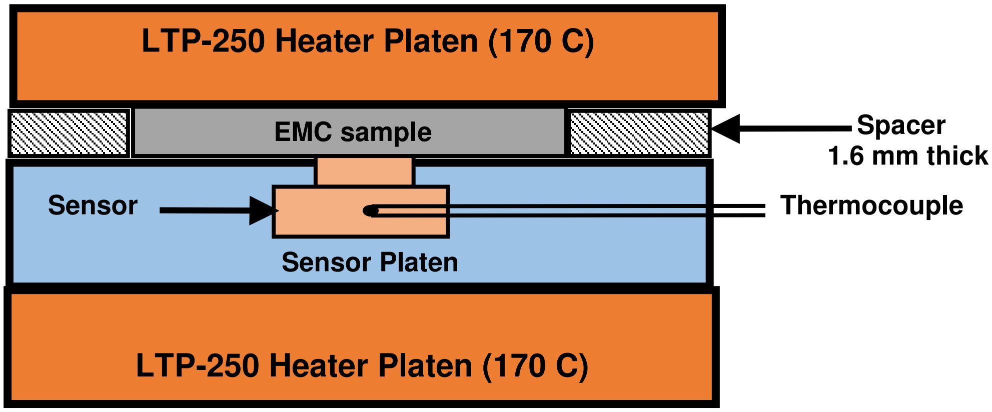


Figure 39-3
Experimental set up for EMC cure, 1.6 mm thickness

Unlike the other sensors of Table 39-1, the Carbon+Unitrode-1″ has a thin, insulating coating for use with carbon filled composites. This coating prevents carbon fibers from short circuiting the electrode and interfering with the measurement. The insulation, however, acts as a boundary layer, which is well understood to distort data during early cure when the Material Under Test is most conductive.

An LTF-631 High Speed Dielectric Cure Monitor[6] made measurements with an excitation frequency of 1 kHz for the Mini-Varicon sensor and 10 Hz for the others. These frequencies were previously determined to be optimum for the particular sensor when monitoring EMC cure. Tests with each sensor were conducted three times to observe repeatability. CureView[7] data acquisition and analysis software controlled the LTF-631 and plotted the ion viscosity and slope of ion viscosity in real time during each five minute test. For brevity, log*(ion viscosity)* will be called log*(IV)* and the slope of log*(ion viscosity)* will simply be called *slope*.

Results

Figure 39-5 shows the log(*IV*) and *slope* for EMC measured with the Mini-Varicon sensor. The curves for three consecutive tests are almost identical, confirming that well controlled experimental conditions yield repeatable results. Figures 39-7, 39-9 and 39-11 show similarly excellent consistency for the Ceramicomb-1", the Unitrode-1" and the Carbon+Unitrode-1" sensors, respectively.

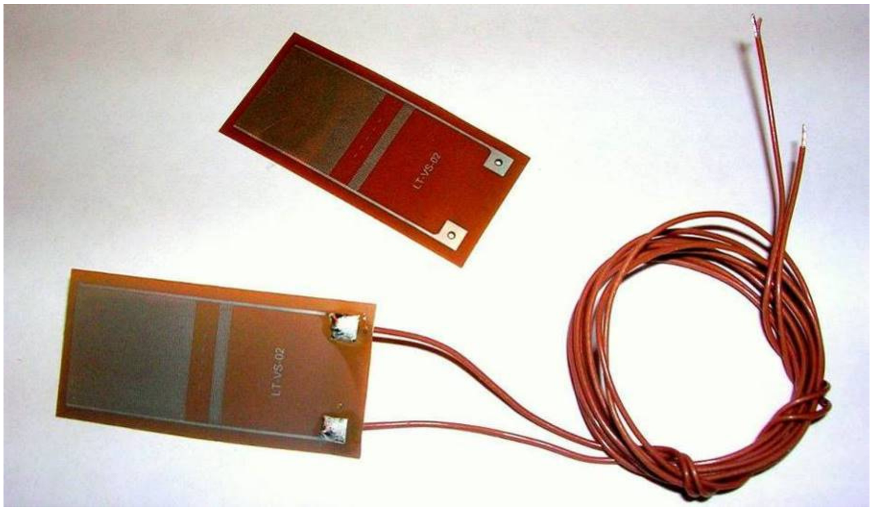


Figure 39-4
Mini-Varicon sensor, A/D = 80 cm

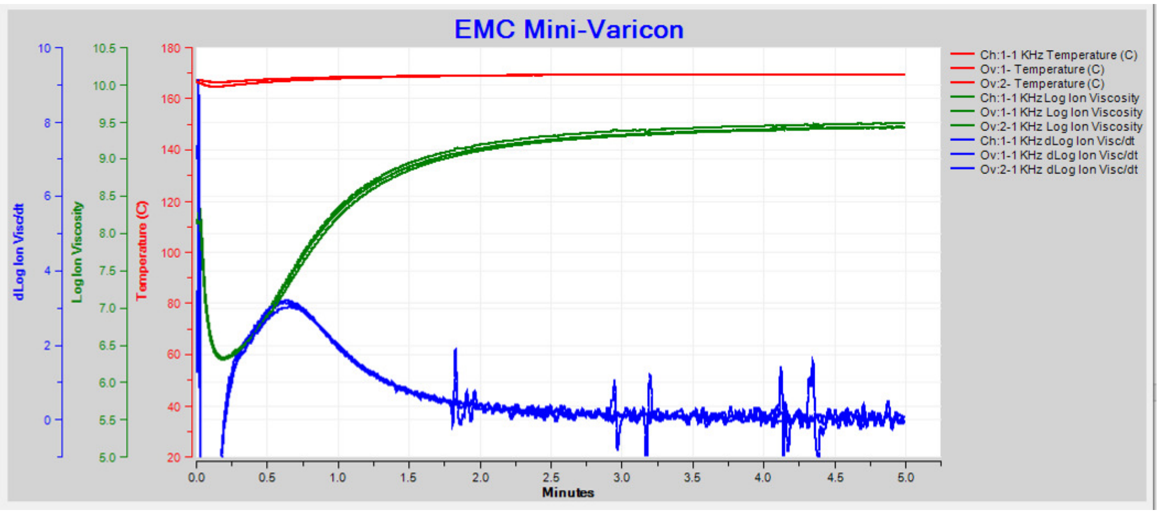


Figure 39-5
Mini-Varicon sensor, A/D = 80 cm, EMC cure at 170 °C,
three consecutive tests

Figure 39-6
Ceramicomb-1″ sensor in press platen, A/D = 10 cm

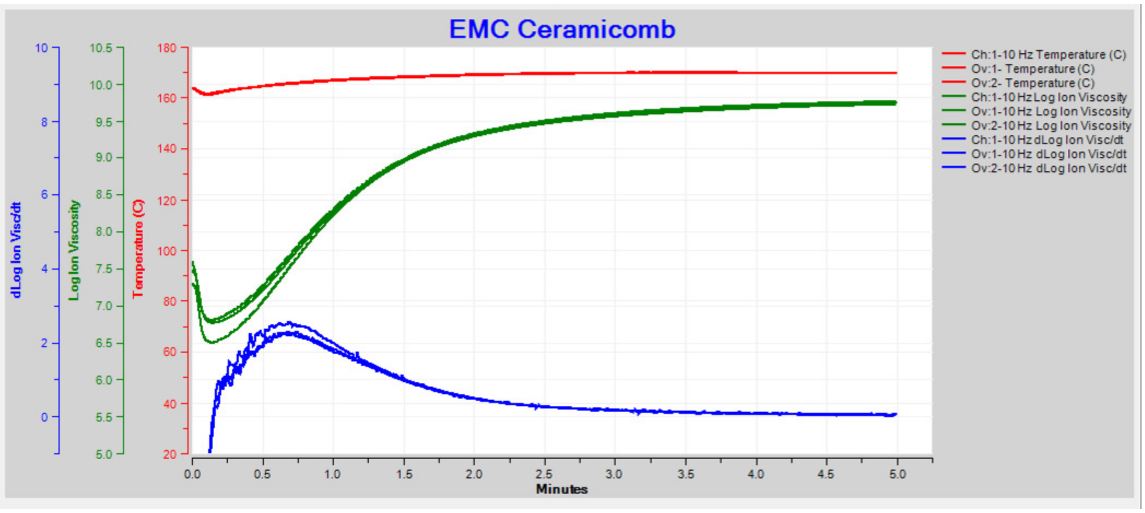


Figure 39-7
Ceramicomb-1″ sensor, A/D = 10 cm, EMC cure at 170 °C, three consecutive tests

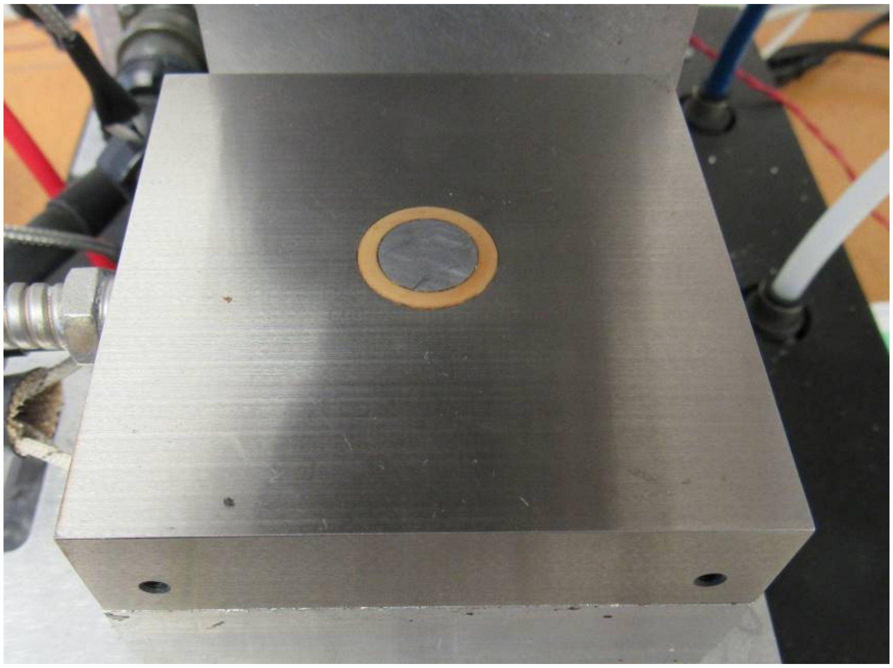


Figure 39-8
Unitrode-1" sensor in press platen, A/D = 6 cm

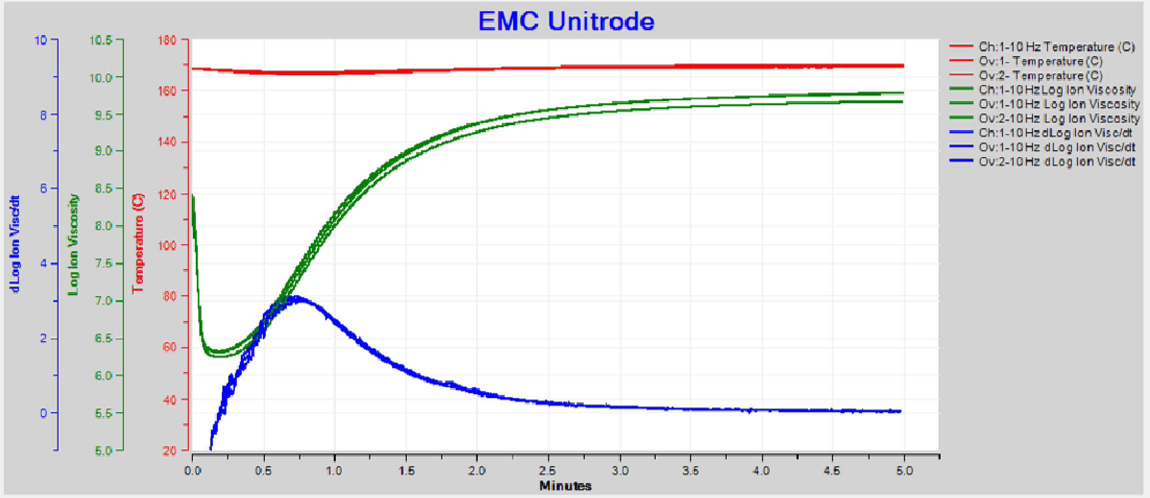


Figure 39-9
Unitrode-1" sensor, A/D = 6 cm, EMC cure at 170 °C, three consecutive tests

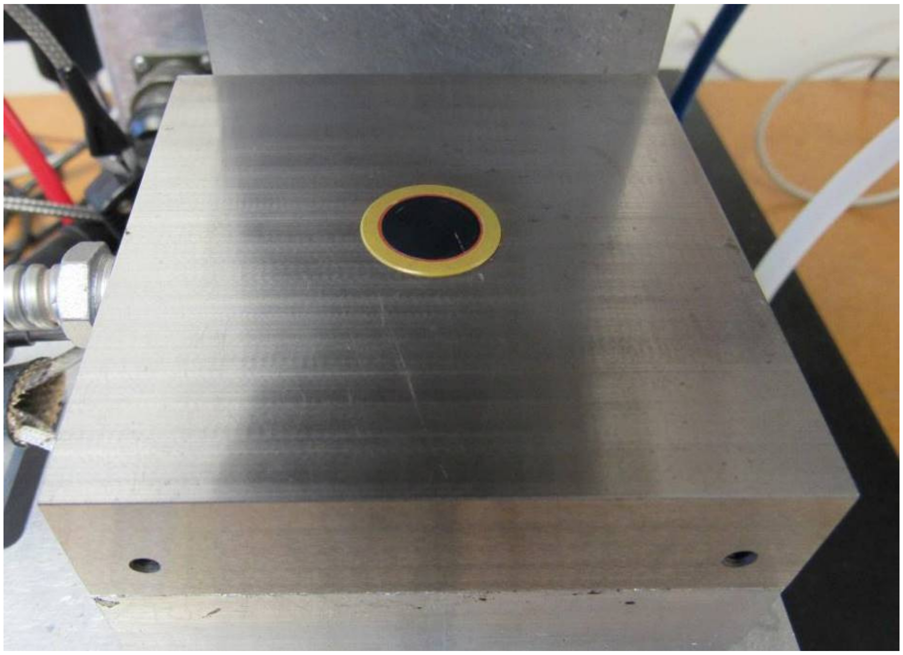


Figure 39-10
Carbon+Unitrode-1″ sensor in press platen, A/D = 5 cm

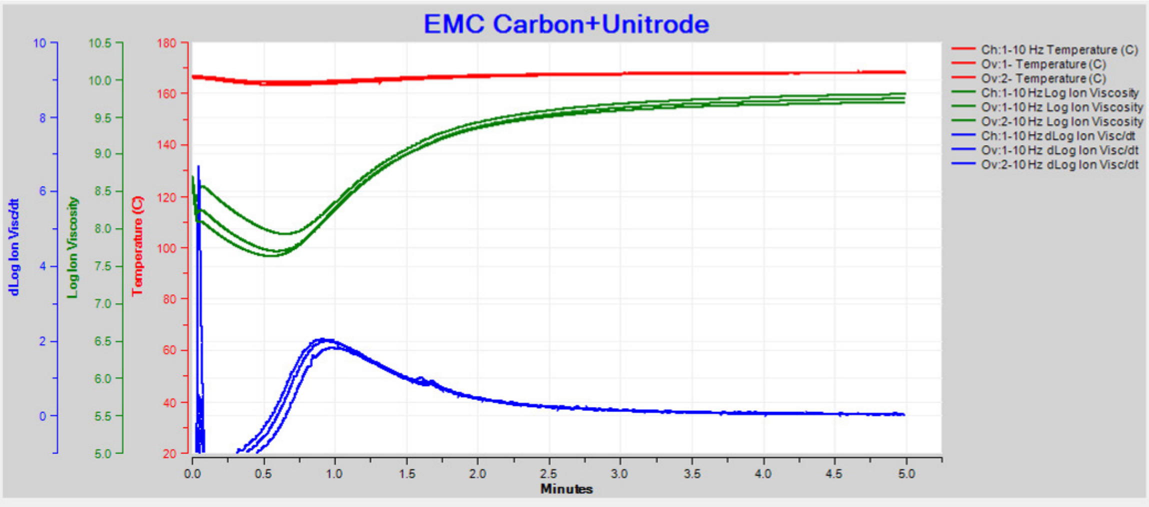


Figure 39-11
Carbon+Unitrode-1″, A/D = 5 cm, EMC cure at 170 °C,
three consecutive tests

Figure 39-12 overlays representative log(*IV*) and *slope* curves for the four sensors. Except for the Carbon+Unitrode-1″ during early cure, the log(*IV*) and slope data for all sensors overlap very well, indicating equivalent data across a wide range of geometries.

Data for the Carbon+Unitrode-1″ sensor deviates from the others during early cure because its insulating coating is a boundary layer that distorts measurements when the sample is most conductive. Even so, from mid-cure through end of cure, when the EMC is less conductive, data from the Carbon+Unitrode-1" agrees well with the other sensors.

- For more information about the Carbon+Unitrode-1" sensor and its coating, see application note *AN 3.36, Cure Monitoring of Carbon Fiber Composites with Coated Sensors*
- For more information about boundary layers, see application note *AN 3.29, Electrode Polarization and Boundary Layer Effects*

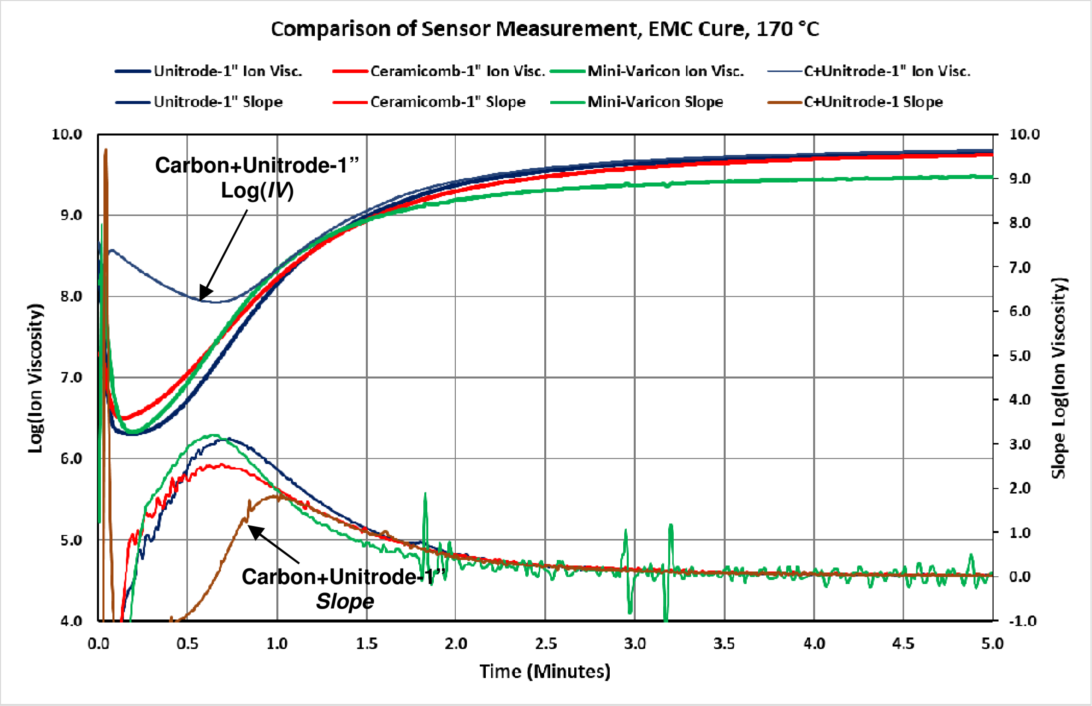


Figure 39-12
Comparison of log(*IV*) and *slope* for EMC cure at 170 °C
Mini-Varicon, Ceramicomb-1", Unitrode-1", Carbon+Unitrode-1" sensors

Behavior of isothermal cure

A thermoset or composite 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. Plots of log(*IV*) and *slope* are a simple way to characterize the progress of cure. Figure 39-13 shows the behavior of a typical thermoset cured at a constant temperature.

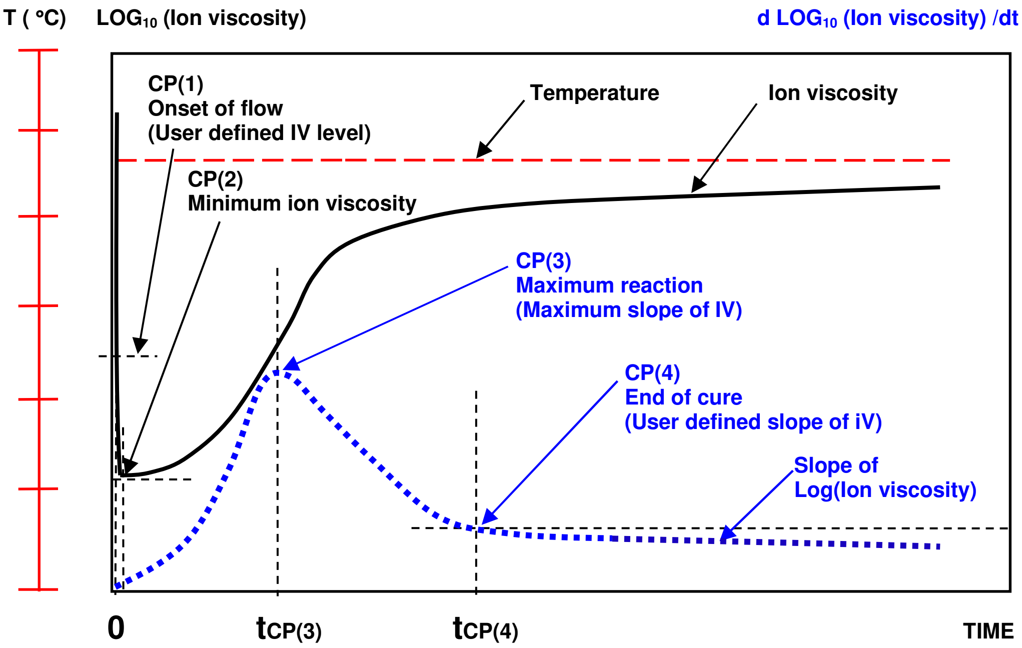


Figure 39-13
Log(*IV*) and *slope* during isothermal cure

With the initial, sudden application of heat, the resin quickly softens or melts, mechanical viscosity decreases and the material flows. Mobile ions also experience less resistance to movement and ion viscosity rapidly decreases.

At high temperature the accelerating cure dominates almost immediately; mechanical viscosity reaches a minimum and 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 as 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.

As shown in Figure 39-13, four Critical Points characterize the dielectric cure curve:

- CP(1)—A user defined level of *log(IV)* to identify the onset of material flow. For isothermal cures CP(1) occurs immediately or shortly after the application of heat, when ion viscosity suddenly decreases.

- CP(2)—Minimum ion viscosity, which closely corresponds to minimum mechanical viscosity, indicating when polymerization and increasing viscosity begin to dominate the cure state.
- 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*, based on the needs of the application, that can identify the end of cure. The decreasing *slope* corresponds to the decreasing reaction rate.

Conclusion

For the cure monitoring of thermosets and composites, available dielectric sensors have a wide range of geometries and constructions. Despite the variety of configurations, properly chosen A/D ratios enable different sensors to measure material properties like ion viscosity and produce the same results. This equivalence allows flexibility in the choice of sensor for a given application and material, with assurance the sensor can observe the true cure state.

References:

1. Mini-Varicon sensor, manufactured by Lambient Technologies, Cambridge, MA USA. https://lambient.com

2. Ceramicomb-1" sensor, manufactured by Lambient Technologies, Cambridge, MA USA

3. Unitrode-1" sensor, manufactured by Lambient Technologies, Cambridge, MA USA

4. Carbon+Unitrode-1" sensor, manufactured by Lambient Technologies, Cambridge, MA USA

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

6. LTP-631 High Speed Dielectric Cure Monitor, manufactured by Lambient Technologies, Cambridge, MA USA

7. CureView software, manufactured by Lambient Technologies, Cambridge, MA USA

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