



## ***Insight* — Application Note 3.20**

### **Cure Monitoring Through Release Films and Vacuum Bags**

#### **Release films and vacuum bags in manufacturing**

In manufacturing, release films prevent the adhesion of molded parts to a tool and vacuum bags enable atmospheric pressure to compress composite material in the Vacuum Assisted Resin Transfer Molding (VARTM) process. Dielectric sensors typically must have direct contact with the material under test but both release films and vacuum bags prevent contact and create challenges for dielectric cure monitoring (DEA).

In fact, a suitably designed sensor, such as the 1-inch Single Electrode<sup>1</sup> reusable sensor, can measure cure state through thin films and enable the use of DEA in a wider range of applications.

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

$$(eq. 20-1) \quad \sigma = \sigma_{DC} + \sigma_{AC} \quad (\text{ohm}^{-1} - \text{cm}^{-1})$$

Resistivity ( $\rho$ ) is the inverse of conductivity and is defined as:

$$(eq. 20-2) \quad \rho = 1/\sigma \quad (\text{ohm-cm})$$

From its relationship to conductivity, resistivity also has both frequency independent ( $\rho_{DC}$ ) and frequency dependent ( $\rho_{AC}$ ) 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  $\rho_{DC}$ . As a result, the term *Ion Viscosity* was coined to emphasize the relationship between mechanical viscosity and  $\rho_{DC}$ . Ion viscosity ( $IV$ ) is defined as:

(eq. 20-3) 
$$IV = \rho_{DC} \quad (\text{ohm-cm})$$

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

### AC Cure monitoring with direct contact

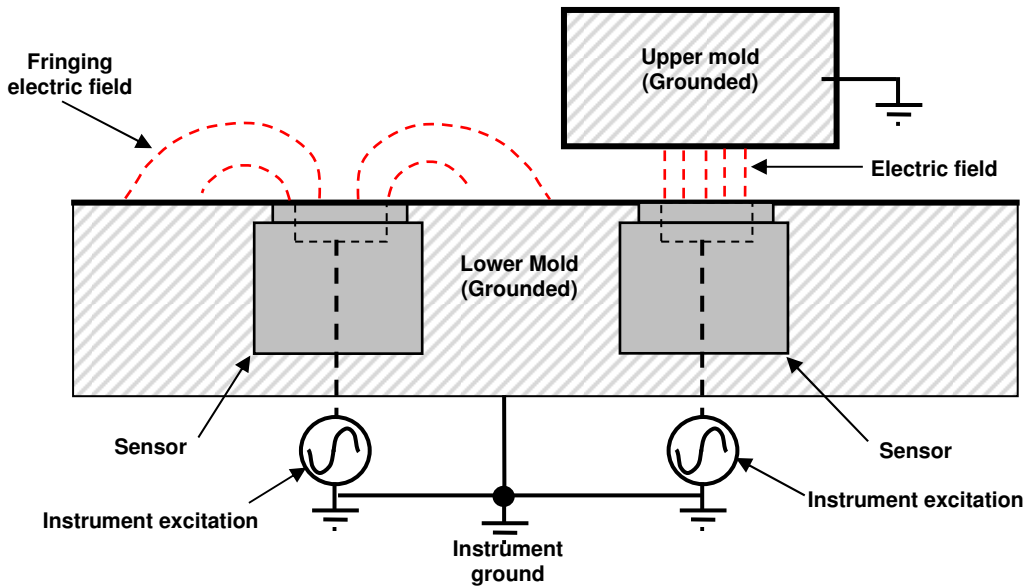
Figure 20-1 shows how a 1-inch Single Electrode sensor would be installed in a press or mold.



**Figure 20-1**  
**1" Single Electrode Sensor in press platen**

For proper operation the tooling around the Single Electrode sensor must be grounded. This ground must be connected to the chassis of the instrumentation, illustrated schematically in Figure 20-2, to make a closed circuit

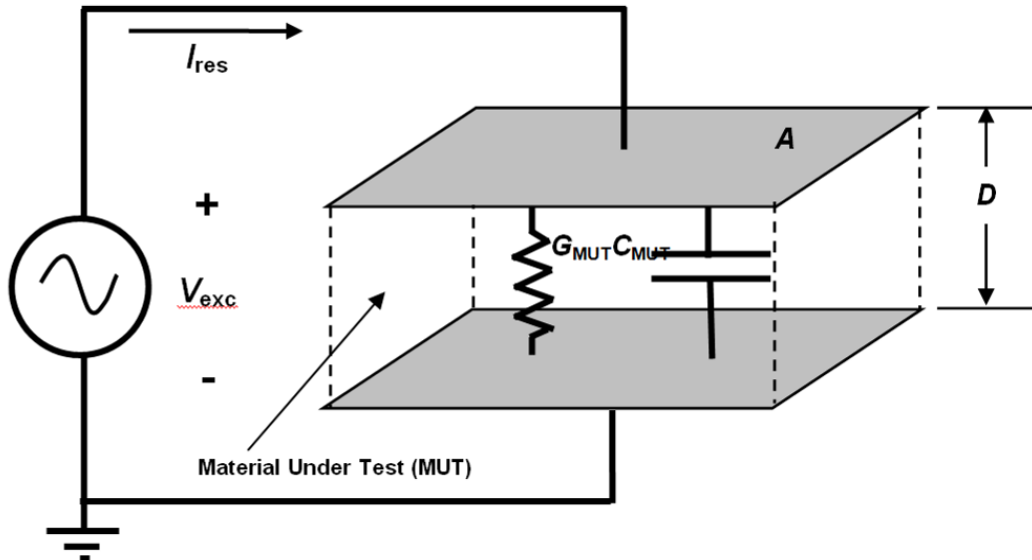
for the sensing current. If an upper platen or mold is used, it should also be grounded.



**Figure 20-2**

**Proper grounding of platens or mold for single electrode sensors**

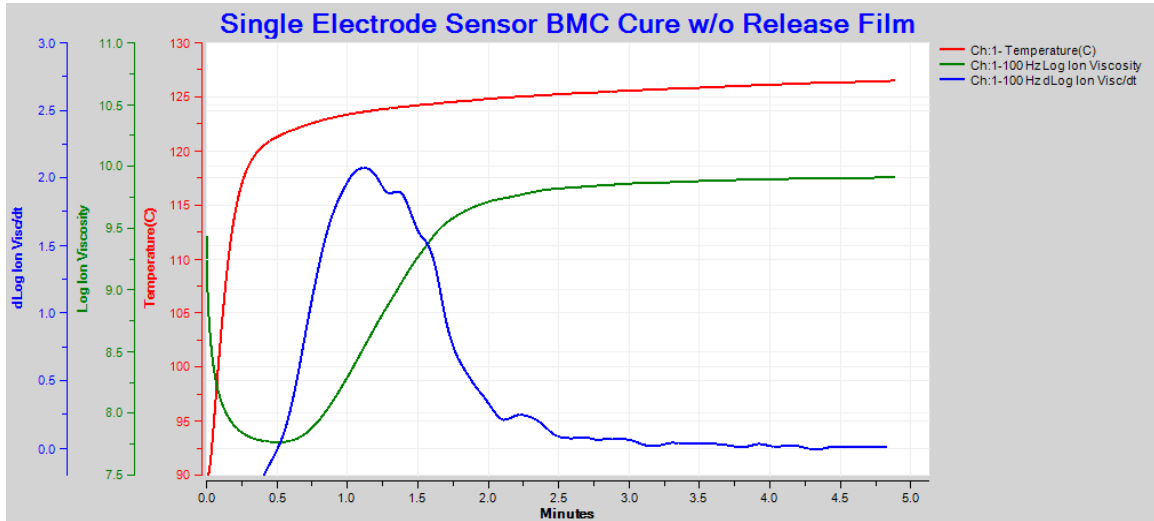
For applications that do not use a release film or vacuum bag, resin flows upon heating and compression and makes direct contact with the sensor. Figure 20-3 shows the electrical model of this resin-sensor system.



**Figure 20-3**

**Electrical model of resin in direct contact with sensor**

Figure 20-4 shows ion viscosity during a 130 °C cure of bulk molding compound (BMC). The BMC was in direct contact with the 1-inch Single Electrode sensor and an LT-451 Dielectric Cure Monitor<sup>2</sup> made measurements with a 100 Hz excitation. The response follows typical behavior for thermosets. As temperature increases, the resin's mechanical viscosity decreases, as does its ion viscosity. Then the material is at minimum mechanical and ion viscosity until the curing reaction dominates and they both increase.



**Figure 20-4**  
**Cure of BMC in direct contact with 1-inch Single Electrode Sensor,**  
**100 Hz AC measurement**

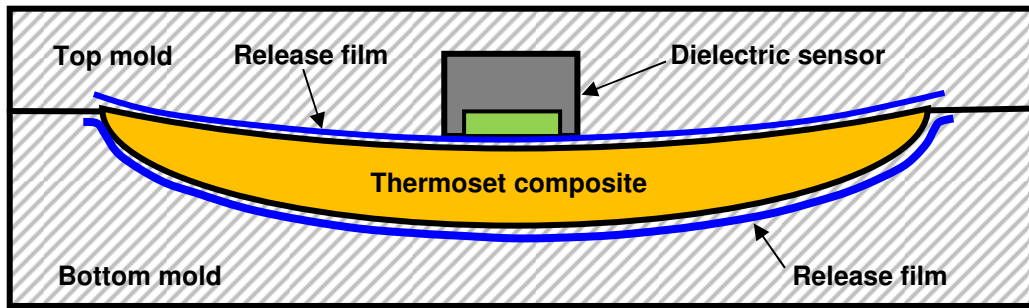
At gelation, mechanical viscosity increases rapidly until it becomes unmeasurable. After gelation ion viscosity diverges from mechanical viscosity and often correlates with modulus until the end of cure. Because gelation is a mechanical—not an electrical—event, no dielectric feature indicates the gel point. Instead, ion viscosity continues to increase past gelation, enabling the continual measurement of material state to the end of cure

As the reaction ends, the ion viscosity curve flattens and its slope approaches zero. In practice, end of cure is a user defined slope that depends on the requirements of the application.

### AC Cure monitoring with release films

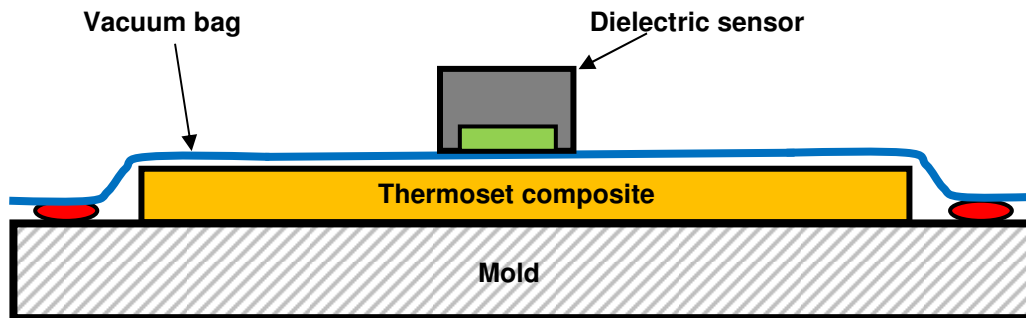
Figure 20-5 illustrates a dielectric sensor in a mold with release films. When the mold is closed, the applied pressure ensures close contact between the

sensor and the release film and between the release film and the composite material.



**Figure 20-5**  
**Cross section of mold with release film and dielectric sensor**

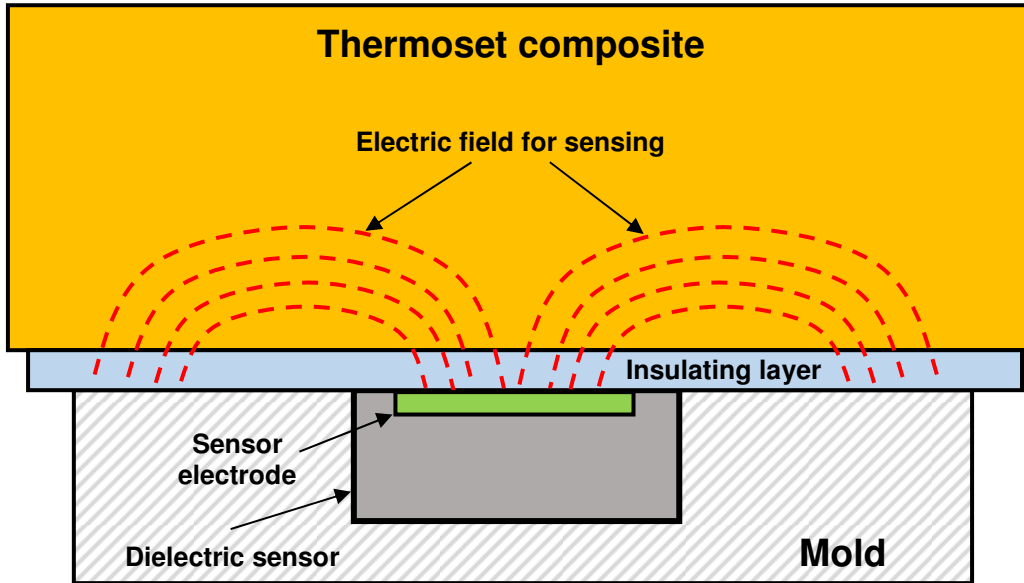
Figure 20-6 shows a dielectric sensor positioned for cure monitoring of material inside a vacuum bag. Once a vacuum is pulled, the bag is in close contact with the composite and dielectric measurements are possible.



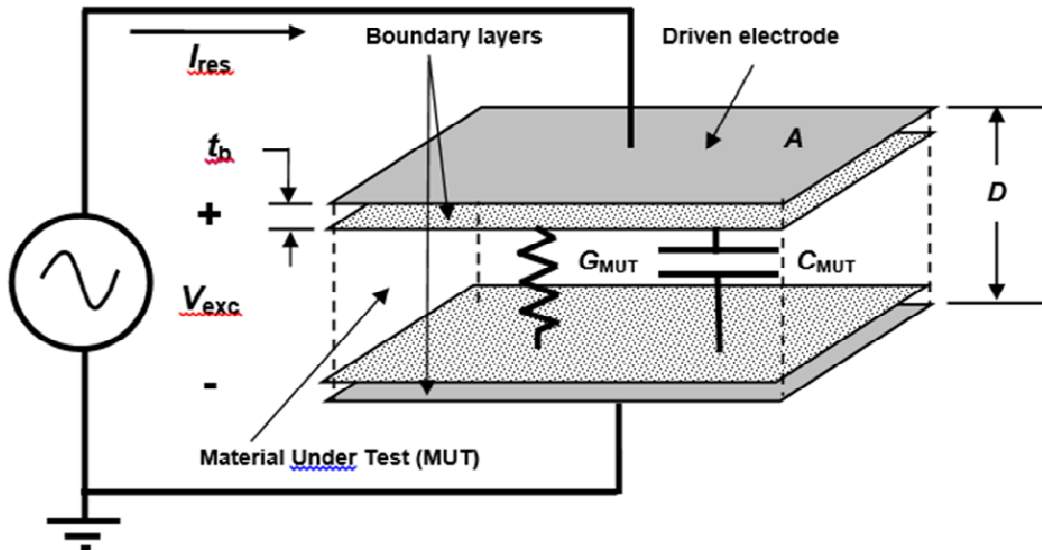
**Figure 20-6**  
**Cross section of lay-up with vacuum bag and dielectric sensor**

Figure 20-7 is a cross-section of these configurations with a release film or vacuum bag, showing how the electric field from the sensor's electrode passes through an insulating layer into the composite and to the mold.

Figure 20-8 is an electrical model of the composite-insulator-sensor system. The insulator introduces a pair of capacitors in series between the electrodes and the Material Under Test (MUT). These capacitors act as boundary layers, an extra element not included in the model of Figure 20-3. Because capacitors pass only AC signals, cure monitoring is not possible with DC methods



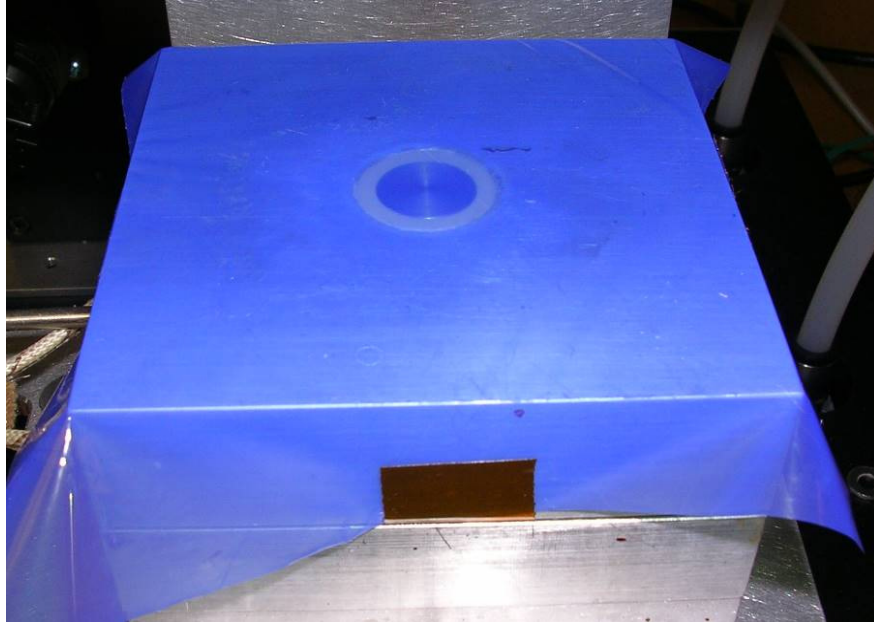
**Figure 20-7**  
**Cross section of lay-up with insulating layer, showing electric field**



**Figure 20-8**  
**Electrical model of resin on sensor with insulating layer**

When the Material Under Test is very conductive, usually around the time of minimum viscosity, the presence of boundary layers may distort ion viscosity measurements. To minimize the boundary layer effect, an insulating film must be thin compared to the separation between electrodes.<sup>3,4,5</sup>

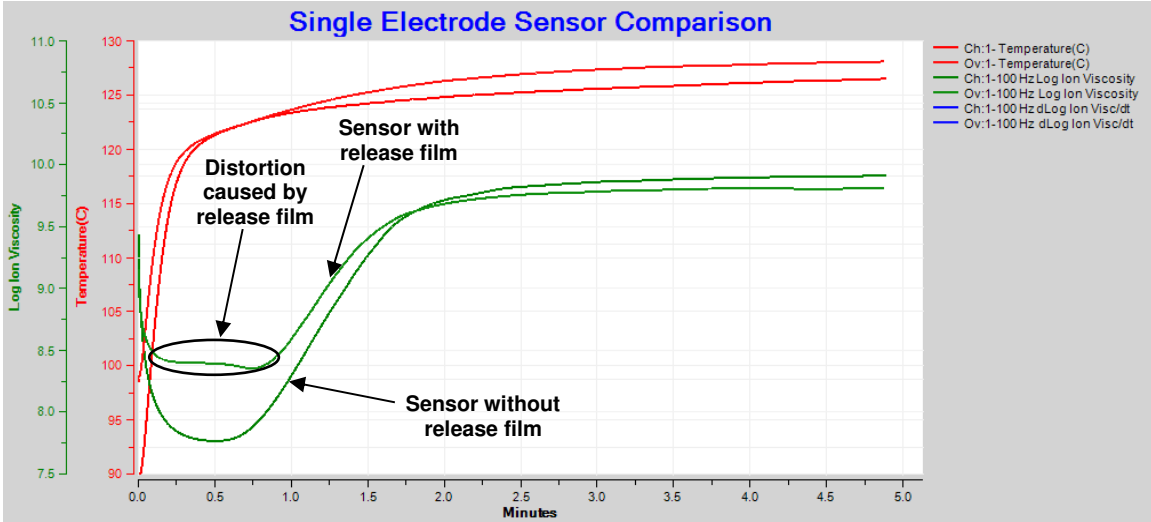
Figure 20-9 shows the 1-inch Single Electrode sensor covered with HTF-621, a PTFE-based release film from Northern Composites. The HTF-621 layer is only 0.001" thick, is chemically inert and non-conductive.



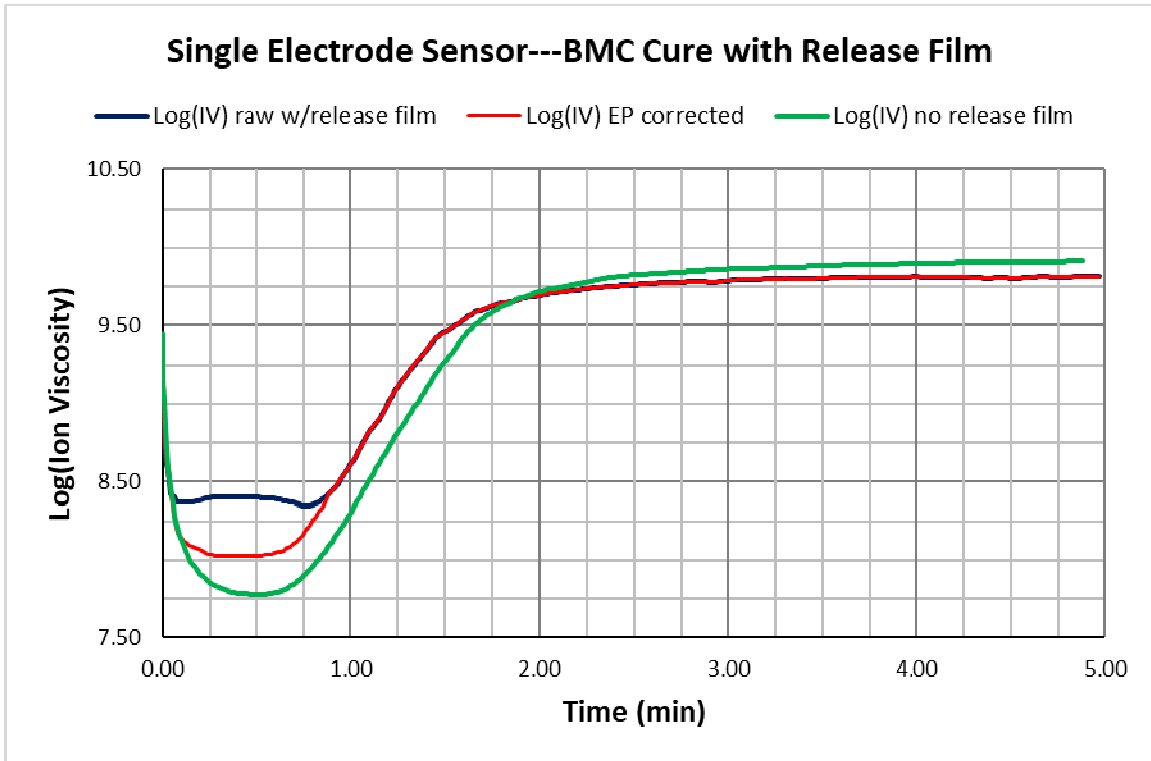
**Figure 20-9**  
**1" Single Electrode Sensor in press platen with HTF-621 release film**

Figure 20-10 compares ion viscosity with and without a release film during the 130 °C cure of BMC. The curves are substantially the same except around the time of minimum ion viscosity, when the boundary layer effect distorts measurements through the release film. In many cases it is possible to mathematically correct this distortion and restore information about the cure.

Figure 20-11 shows how boundary layer correction—also called *electrode polarization* (EP) correction—recovers affected data. After EP correction, ion viscosity measured with the release film correctly follows ion viscosity measured without the release film. The minor discrepancy between curves is largely due to temperature differences in the tests.



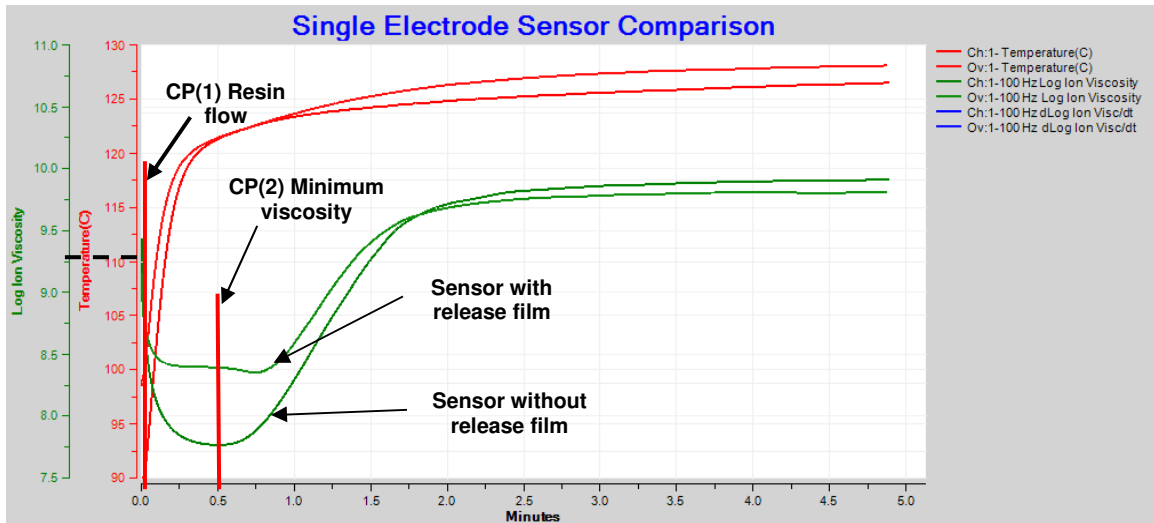
**Figure 20-10**  
**Comparison of BMC cure with and without release film,**  
**100 Hz AC measurement**



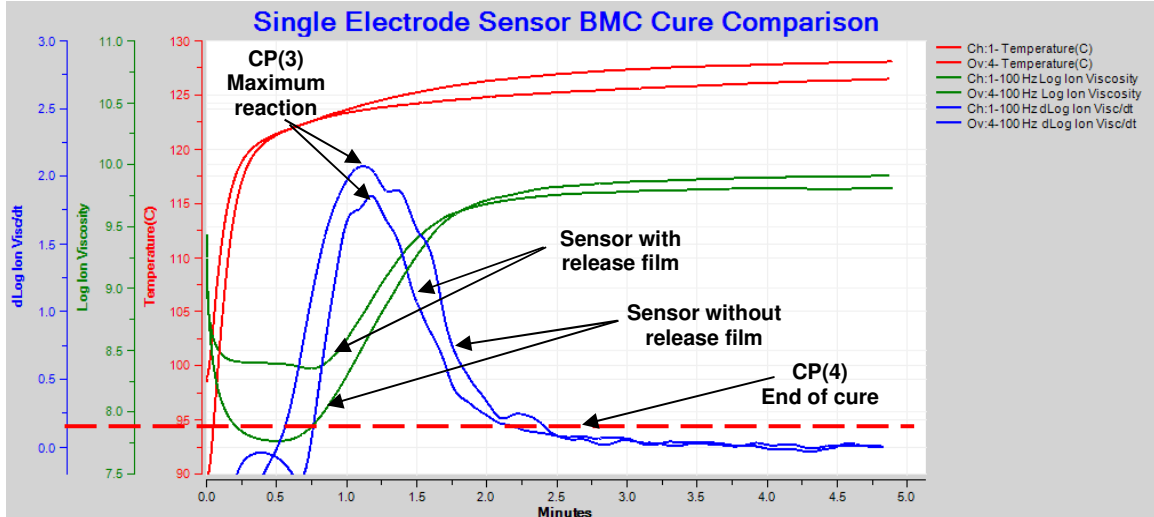
**Figure 20-11**  
**Comparison of raw ion viscosity and ion viscosity**  
**with EP (boundary layer) correction**



Figures 20-12 and 20-13 show the correspondence between Critical Points detected by sensors with and without a release film.



**Figure 20-12**  
**Comparison of ion viscosity with and without release film,**  
**100 Hz AC measurement**



**Figure 20-13**  
**Comparison of slope with and without release film,**  
**100 Hz AC measurement**

The slope for CP(4) was arbitrarily chosen to illustrate the determination of end of cure. In reality, the user selects a slope based on the requirements of the application. Because AC measurements through a release film correspond to cure

state, Figure 20-13 shows that the appropriate slope enables reliable detection of end of cure.

### **DC Cure monitoring with release films**

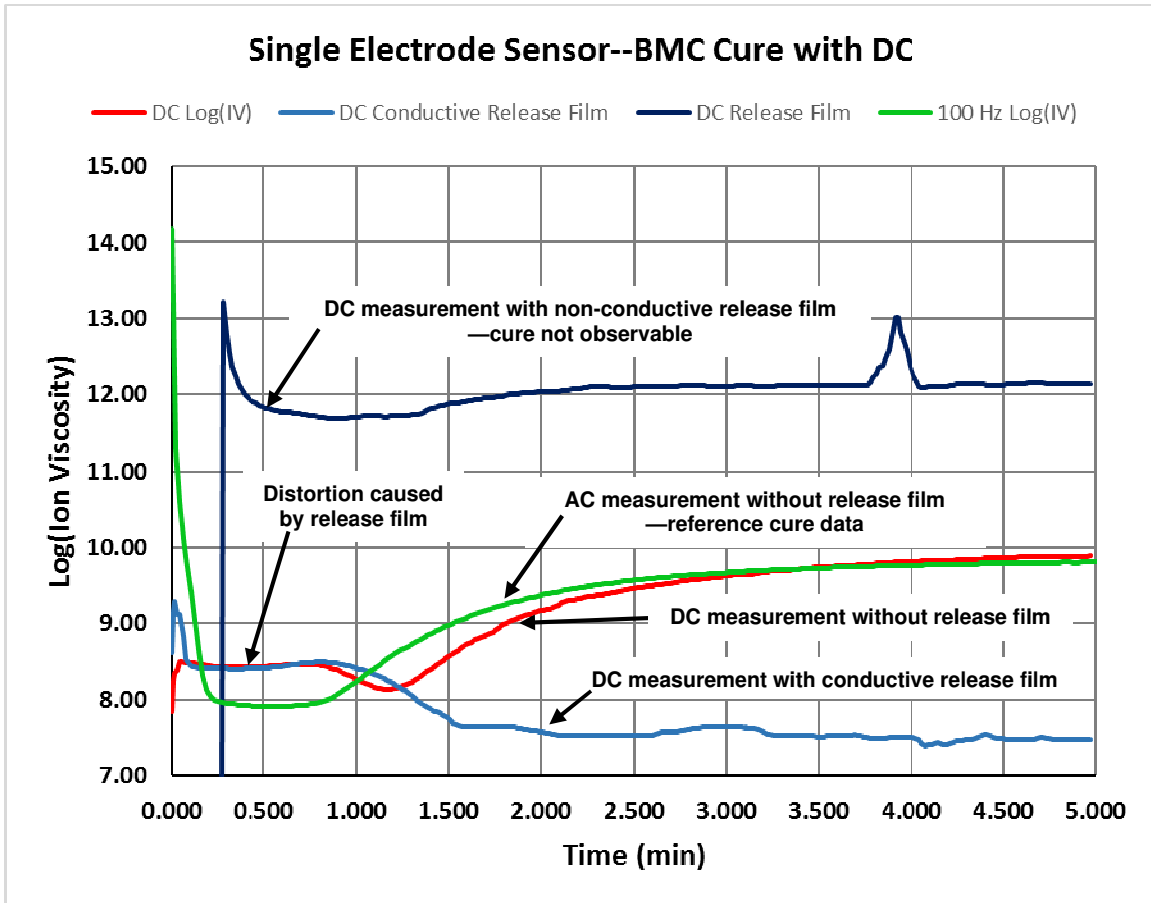
DC measurement of resistance or ion viscosity is a simple method of probing cure state; however, it has the following limitations:

- Possible distortion of data, due to electrode polarization, around the time of the viscosity minimum
- Inability to measure cure state through non-conductive release films or vacuum bags

Typical high temperature release films are made from PTFE or similar non-conductive materials. As a result, they act like the capacitive blocking layers of Figure 20-8 and prevent the passage of DC signals.

Some low-grade release films are conductive; however, the conductivity varies with temperature and may not be consistent from batch to batch. While it is possible to make DC measurements through such films, the results unpredictably combine the resistivity of the film and the material under test. Furthermore, the data depend on temperature, are unreliable and therefore not useful.

The LT-451 Dielectric Cure Monitor has a DC measurement option, which was used with the Single Electrode sensor to monitor BMC cure at 130 °C. Figure 20-14 compares DC results with no release film, with the non-conductive HTF-621 release film and with a conductive release film. For reference, data from 100 Hz AC measurements without a release film are also plotted.



**Figure 20-14**  
**Comparison of AC and DC measurements of ion viscosity**

Toward the end of cure, AC and DC measurements without a release film are similar. However, DC measurements with no release film show distortion caused by electrode polarization around the time of the ion viscosity minimum. While it is possible to correct this distortion with AC data (see Figure 20-11), it is not possible to correct DC data.

DC measurements through the conductive release film are possible, as shown, but still subject to distortion due to electrode polarization. Toward the end of cure the release film's conductivity dominates and prevents useful probing of cure state.

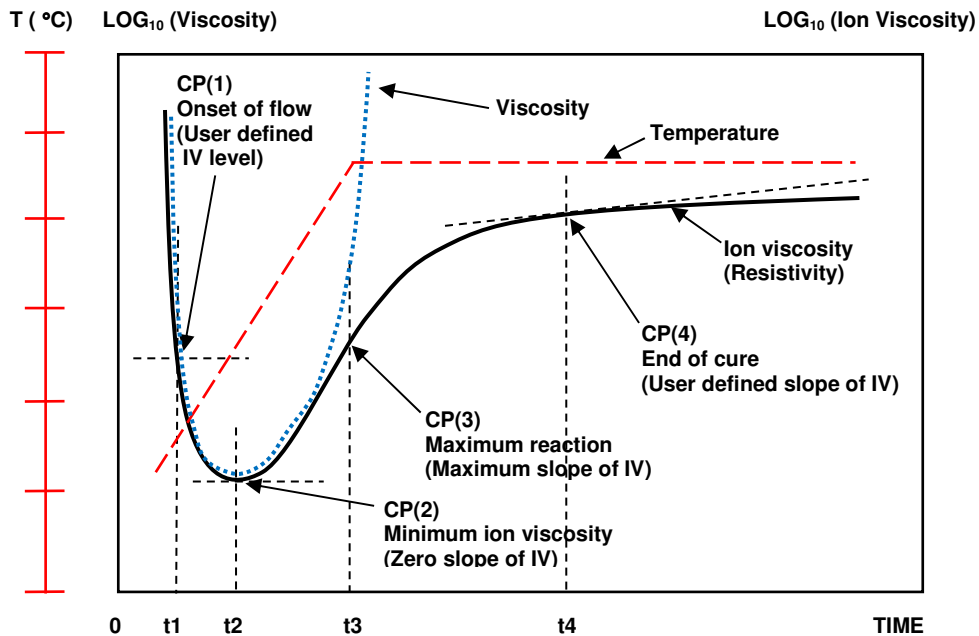
DC measurements through the non-conductive HTF-621 release film are not possible at all, as shown by very high, essentially constant ion viscosity at the measurement limit under these conditions.

With a suitable sensor, AC measurements through a release film or vacuum bag can follow cure and provide the same information as a sensor in direct contact with the material under test. The insulating layer distorts ion viscosity data around the time of the viscosity minimum, when the material is most conductive, but in many cases software can mathematically correct this distortion. DC measurements, however, are not possible through a release film and are a serious limitation of DC cure monitoring techniques.

### 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  $\log(\text{ion viscosity})$  is a simple way to characterize the progress of cure and Figure 20-15 shows the behavior of a typical thermoset with one ramp and hold step in temperature.

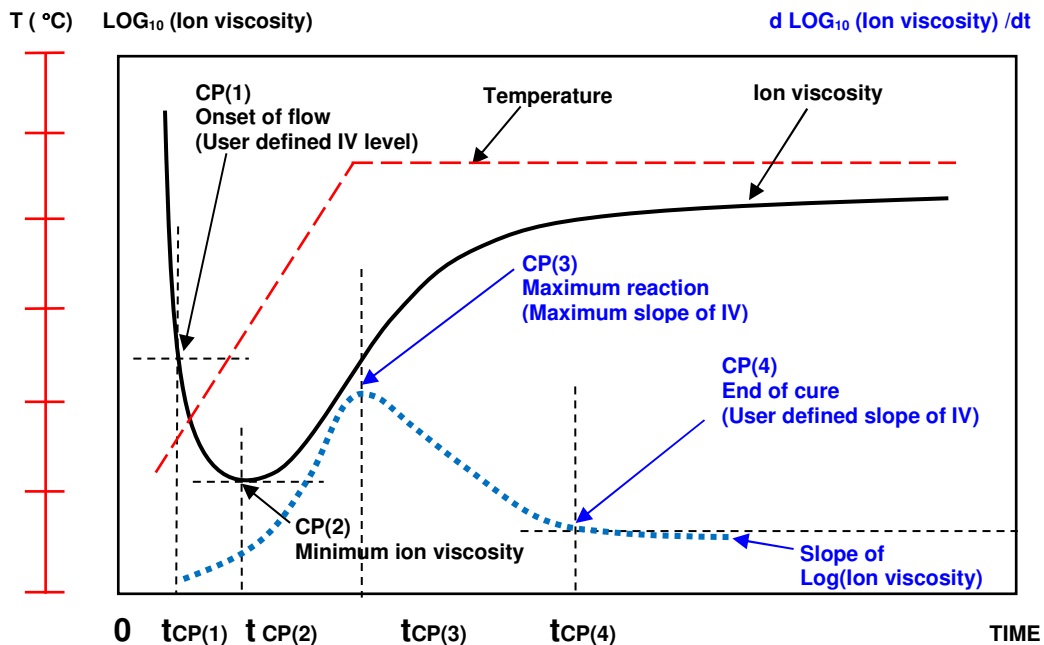
At first as temperature increases, the material softens or melts and mechanical viscosity decreases. Mobile ions also experience less resistance to movement and ion viscosity decreases. At this point the reaction is still slow.



**Figure 20-15**  
**Typical ion viscosity behavior of thermoset cure**  
**during thermal ramp and hold**

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.

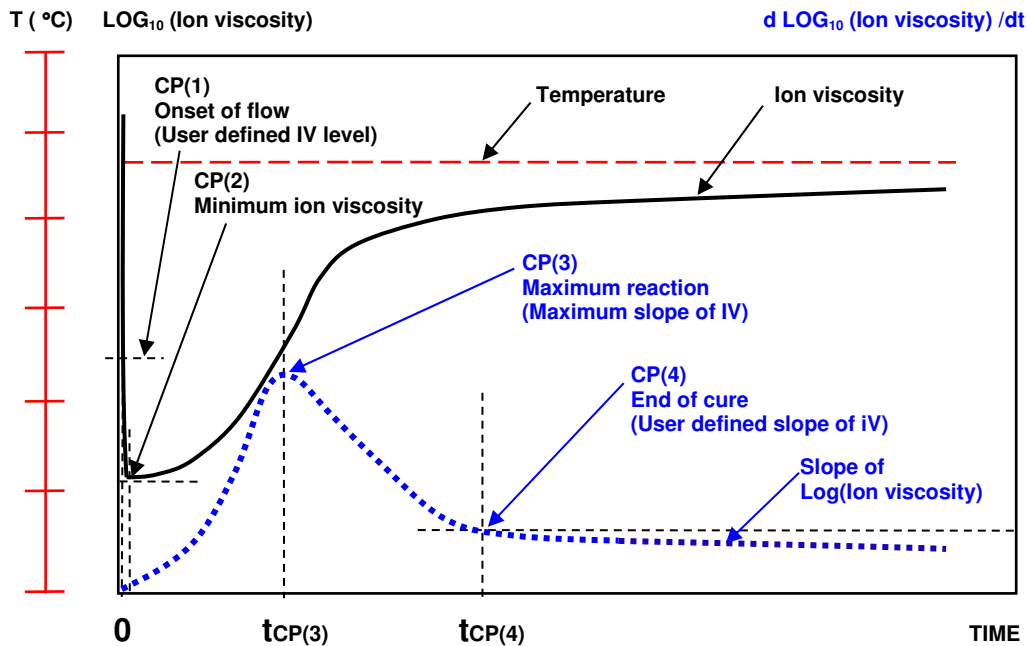


**Figure 20-16**  
**Ion viscosity curve and slope of ion viscosity of thermoset cure**  
**during thermal ramp and hold**

As shown in Figure 20-16, 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 20-15 and 20-16 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 20-17.



**Figure 20-17**  
**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. 1" Single-Electrode sensor, manufactured by Lambient Technologies, Cambridge, MA USA.  
<https://lambient.com>
2. LT-451 Dielectric Cure Monitor, manufactured by Lambient Technologies, Cambridge, MA USA
3. Day, D.R.; Lewis, J.; Lee, H.L. and Senturia, S.D., "The Role of Boundary Layer Capacitance at Blocking Electrodes in the Interpretation of Dielectric Cure Data in Adhesives," *Journal of Adhesion*, V18, p.73 (1985)
4. Lambient Technologies application note AN2.16, "Electrode Polarization and Boundary Layer Effects"
5. Lambient Technologies application note AN2.40, "Electrode Polarization and Boundary Layer Effects with AC and DC Cure Monitoring"



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