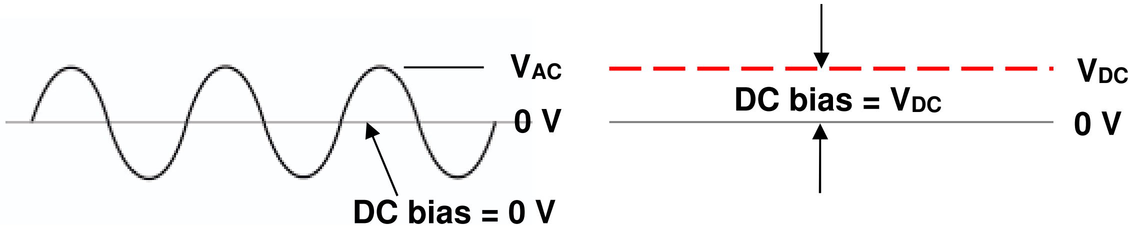
# *Insight* —Technical Overview 3.04

## AC and DC Cure Monitoring

### Introduction

Both AC and DC techniques can be used to study thermoset and composite cure. AC dielectric cure monitoring, also called *Dielectric Analysis* (DEA) excites a sensor with a sinusoidal signal of chosen frequency and amplitude, using a DC bias of zero volts. The measured frequency independent resistance, or *ion viscosity*, correlates with the cure state of the Material Under Test (MUT). DC resistance cure monitoring similarly probes a material but instead uses a constant excitation voltage $V_{DC}$.



**AC** **DC**

**Figure 4-1**
**AC and DC signals**

AC and DC cure monitoring are complimentary but electrochemical reactions can affect DC results. In contrast, AC measurements have no DC bias and zero average current, and do not experience electrochemical effects. While DC techniques can measure cure state, they must be used carefully and with an understanding of their capabilities.

### AC and DC cure monitoring

AC dielectric cure monitoring, also known as *Dielectric Analysis* (DEA), measures a polymer's resistance and capacitance, which are bulk properties used in the standard electrical model of a resin, shown in Figure 4-2. Resistance and capacitance can be scaled with a sensor's cell constant to yield the material properties of resistivity ($\rho$) and permittivity ($\varepsilon'$).

Resistivity itself has a frequency independent ($\rho_{FI}$) component due to the flow of mobile ions and a frequency dependent ($\rho_{AC}$) component due to the rotation of stationary dipoles.

Although often called DC resistivity, *frequency independent* resistivity actually extends across a range of frequencies that includes DC (0 Hz). The change in frequency independent resistivity is proportional to the change in viscosity before gelation and proportional to the change in modulus after gelation.

To emphasize the relationship with mechanical viscosity, the term *ion viscosity* (*IV*) was coined in the early 1980's as a synonym for frequency independent resistivity. Ion viscosity is defined below:

(Eq. 4-1) $$IV = \rho_{FI} \qquad \text{(ohm-cm)}$$

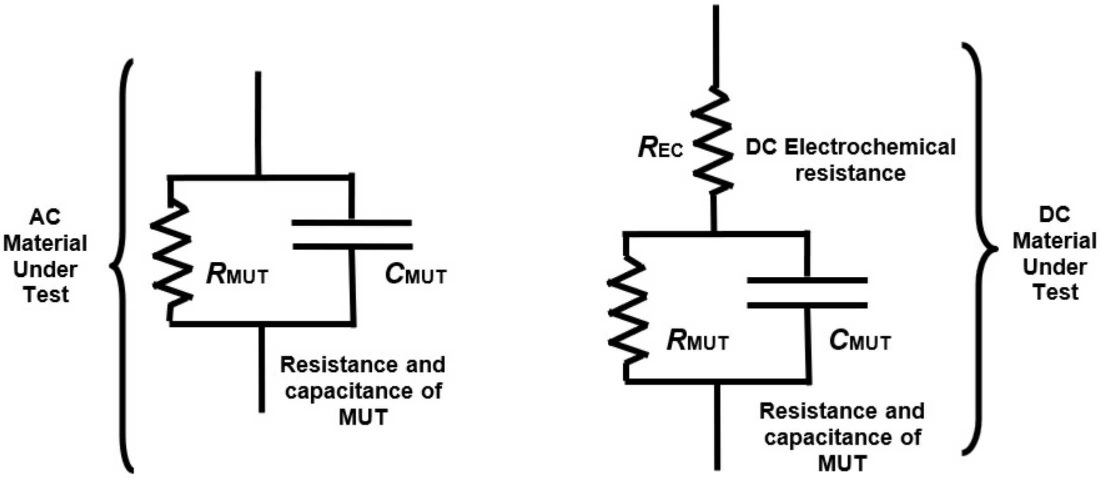


**Figure 4-2**
**AC and DC electrical models of thermoset resins**

During early to mid-cure, an electrochemical reaction under DC bias is the likely cause of discrepancies between DC and AC measurements. Although the nature of the electrochemistry is unknown, the DC response during cure can be reproduced by a *DC electrochemical resistance*[1] added to the standard model of a resin in Figure 4-2.

## Comparison of AC and DC measurements

By taking advantage of a wide frequency range, AC measurements can probe material state from beginning to end of cure. In contrast, simpler DC methods drive current with a constant voltage through the Material Under Test.

AC and DC techniques are complimentary but the choice of measurement requires understanding their capabilities and limitations:

- DC measurements can only obtain DC resistance
    - DC resistance includes an added DC electrochemical resistance
    - AC methods measure capacitance, frequency *independent* resistance and frequency *dependent* resistance
- DC measurements deviate from AC measurements during early and mid-cure
    - DC electrochemical resistance decreases as the material cures, reducing discrepancy over time as plotted in the example of Figure 4-3
    - DC and AC measurements agree during end of cure
    - AC ion viscosity avoids the effects of DC electrochemistry

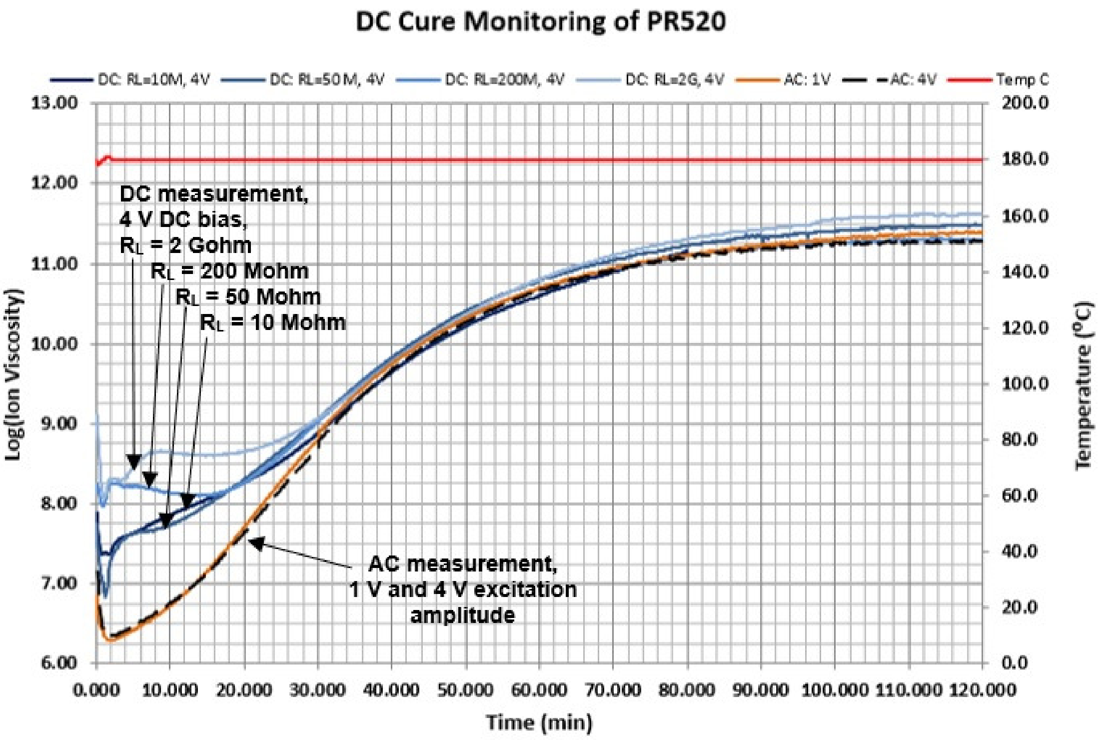


**Figure 4-3**
**AC and DC measurements during cure of PR520 epoxy**

- DC measurements are not possible through release films or vacuum bags
  - Insulating layers block DC current and prevent DC resistance measurement
  - AC sensors with the proper design can measure through release films and vacuum bags[2]
- DC measurements may have systematic errors caused by other DC signals
  - Offset voltage drifts, thermal drifts and leakage currents are DC in nature and cannot be distinguished from the true DC response
  - DC sensors with proper design can reduce or eliminate systematic errors
  - AC measurements are insensitive to DC systematic errors
- AC measurements may have distortions caused by *electrode polarization*
  - Electrode polarization may affect AC data at low frequencies during early cure
  - AC measurements at high frequencies do not have distortion caused by electrode polarization
  - The effects of electrode polarization can be corrected mathematically[3,4,5]
  - DC measurements have electrochemical effects that minimize electrode polarization
- AC measurements for extremely resistive materials may need long measurement times
  - AC measurements at higher frequencies (shorter measurement times) may be used for most thermosets at elevated temperatures
    - Elevated temperatures reduce the resistivity of thermosets
  - AC measurements with very low frequencies (long measurement times) may be necessary if materials become extremely resistive
    - Extreme resistivity may occur in thermosets at room temperature at end of cure
  - DC measurements are faster for extremely resistive materials when the AC measurement time would be very long

## Electrode polarization in AC measurements

At low frequencies the phenomenon of *electrode polarization* (EP) can create a blocking layer across sensor electrodes during early cure, when material is most conductive, and cause abnormally high *apparent* ion viscosities.[3,4,5]

Figure 4-4 is a plot of resistivity from AC measurements of five-minute epoxy. All data are plotted against an axis labeled *ion viscosity* and may collectively be called ion viscosity.

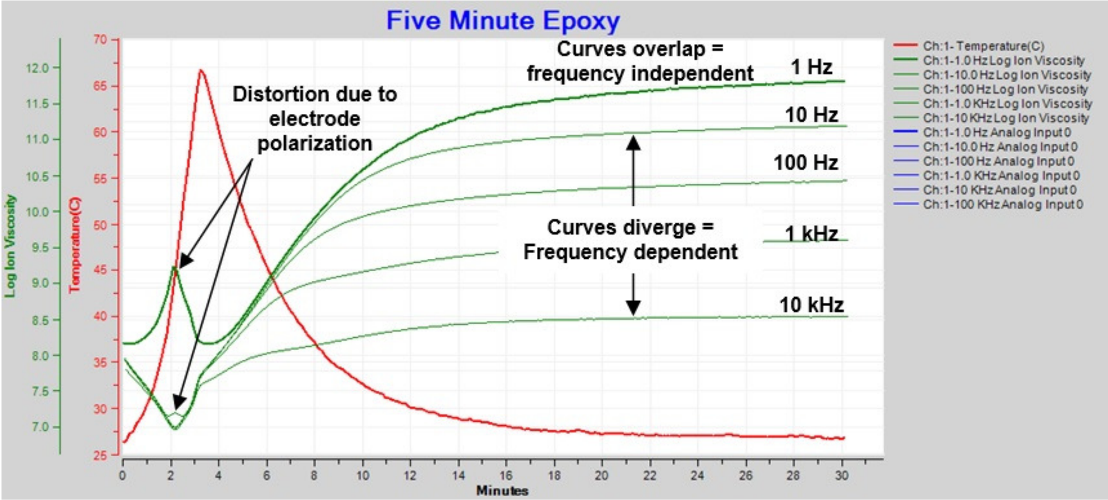


**Figure 4-4**
**Ion viscosity / resistivity during cure of 5-minute epoxy**

Three features are apparent in Figure 4-4:

- Curves that overlap or nearly overlap, indicating the dominance of frequency *independent* resistivity, or true ion viscosity
  - Caused by movement of mobile ions
  - Correlates well with cure state
- Curves that diverge, indicating the dominance of frequency *dependent* resistivity
  - Caused by rotation of dipoles
  - Does not correlate well with cure state
- Distortion of 1 Hz and 10 Hz curves around 2 minutes due to electrode polarization

At the beginning of cure, electrode polarization distorts the 1 Hz data in the expanded plot of Figure 4-5. Measurements at 10 Hz show distortion to a much lesser degree because the boundary layer effect decreases with increasing frequency. Furthermore, measurements at even higher excitation frequencies—1 kHz to 10 kHz—show no distortion and correctly identify the ion viscosity minimum.

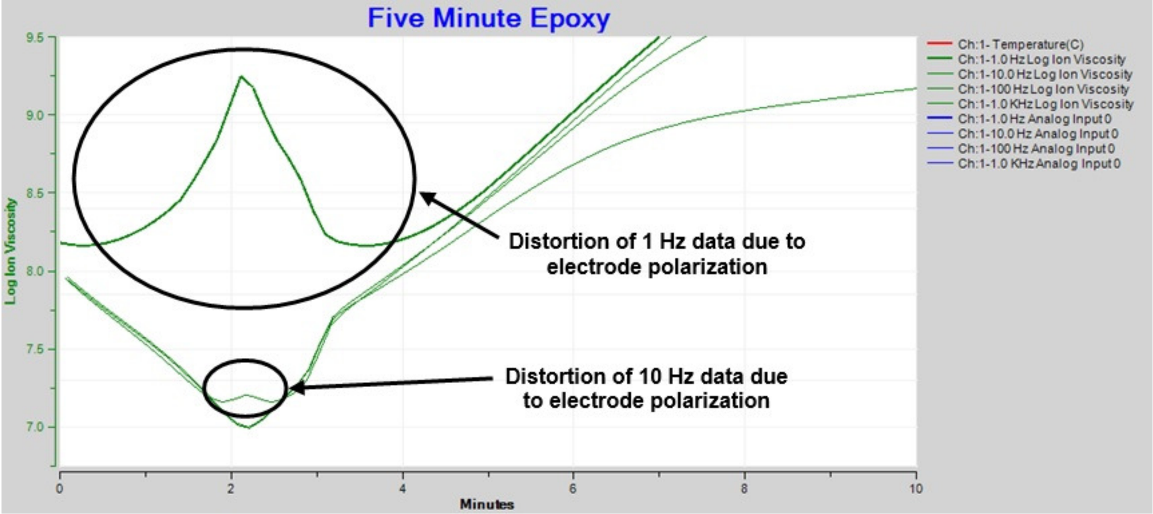


**Figure 4-5**
**Expanded ion viscosity / resistivity around time of minimum viscosity**

In many cases it is possible to mathematically restore information about cure. Figure 4-6 shows how boundary layer correction—also called *electrode polarization* (EP) correction—recovers affected data.

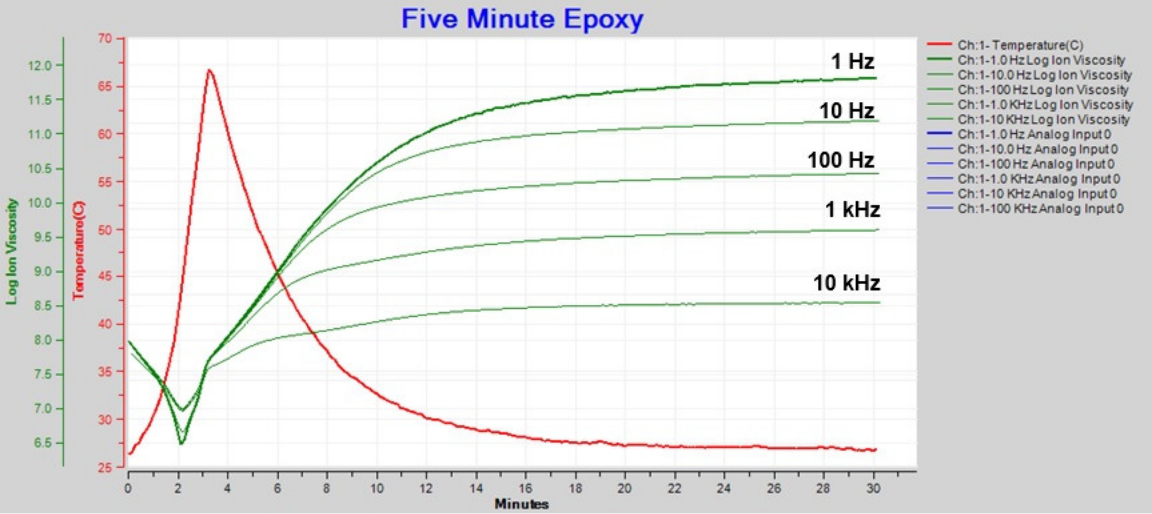


**Figure 4-6**
**Resistivity / ion viscosity with boundary layer (EP) correction**

After boundary layer correction, 1 Hz and 10 Hz ion viscosity show a proper minimum and are consistent with the higher frequency data. It is also possible to use only 1 Hz ion viscosity to follow the entire five-minute epoxy cure, as shown in Figure 4-7.

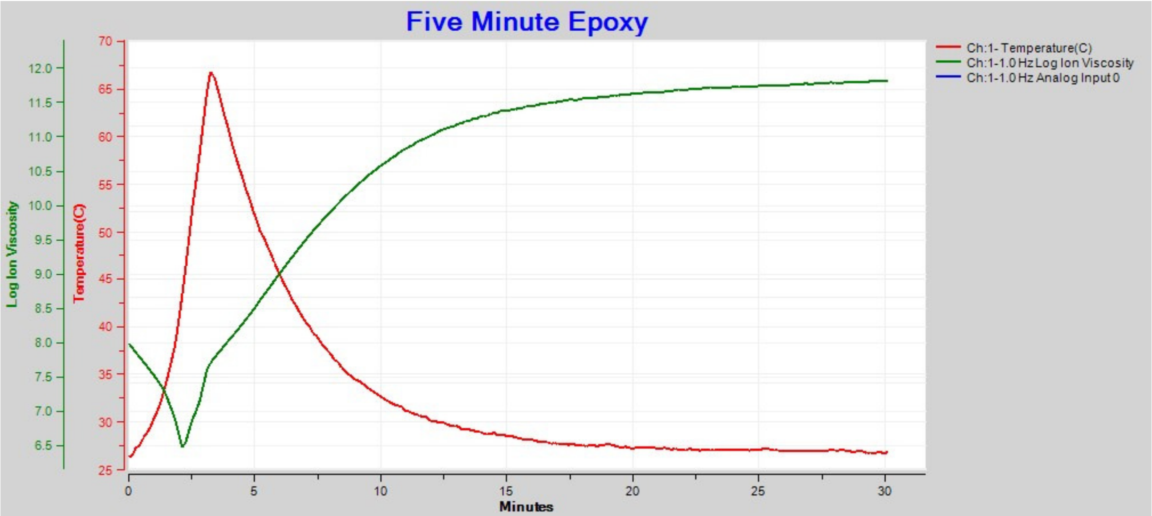


**Figure 4-7**
**1 Hz ion viscosity with boundary layer (EP) correction**

## AC and DC measurements with release films and vacuum bags

Release films prevent the adhesion of molded parts to a tool, and vacuum bags enable atmospheric pressure to compress composites in the Vacuum Assisted Resin Transfer Molding (VARTM) process. Figure 4-8 illustrates a sensor in a mold with release film, which is typically made of PTFE or other electrically insulating material. Because insulators block DC signals, cure monitoring in this situation is not possible with DC methods. A suitably designed dielectric sensor, however, enables AC cure monitoring through a release film or vacuum bag.

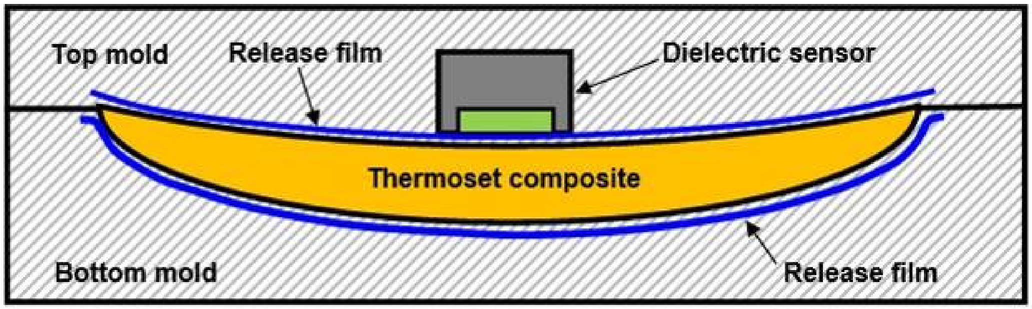


**Figure 4-8**
**Dielectric sensor with release film**

Figure 4-9 shows a Lambient Technologies 1" Single-Electrode dielectric sensor[6] installed in a press platen and covered with Northern Composites HTF-621[7], a PTFE-based release film that is only 0.001" thick.

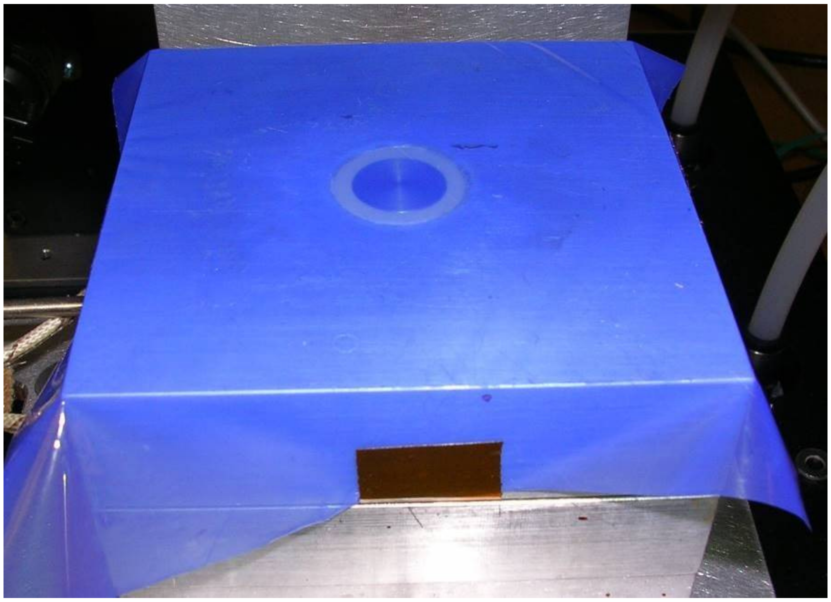


**Figure 4-9**
**1" Single Electrode Sensor in press platen with HTF-621 release film**

Figure 4-10 shows ion viscosity measured through the release film during cure of PR520[8], a pre-mixed epoxy used for resin transfer molding (RTM). Insulating films create a boundary layer that affects data like electrode polarization does. As a result the ion viscosity curves are distorted for all but the highest frequency.

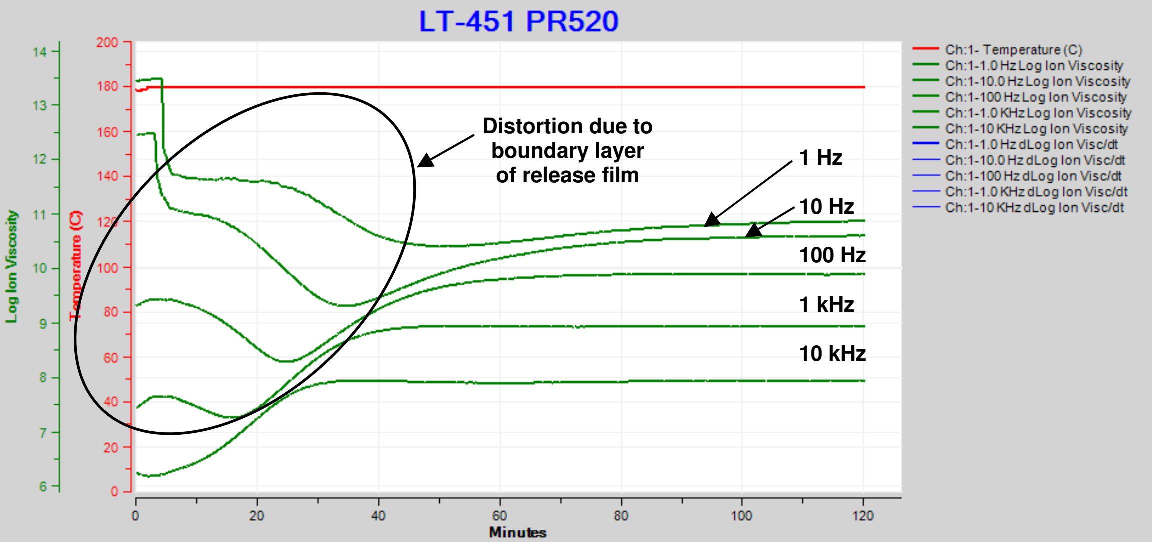


**Figure 4-10**
**Ion viscosity of PR520 during cure with release film**

In many cases it is possible to mathematically correct this distortion and restore information about the cure. Figure 4-11 shows how boundary layer correction recovers affected data.

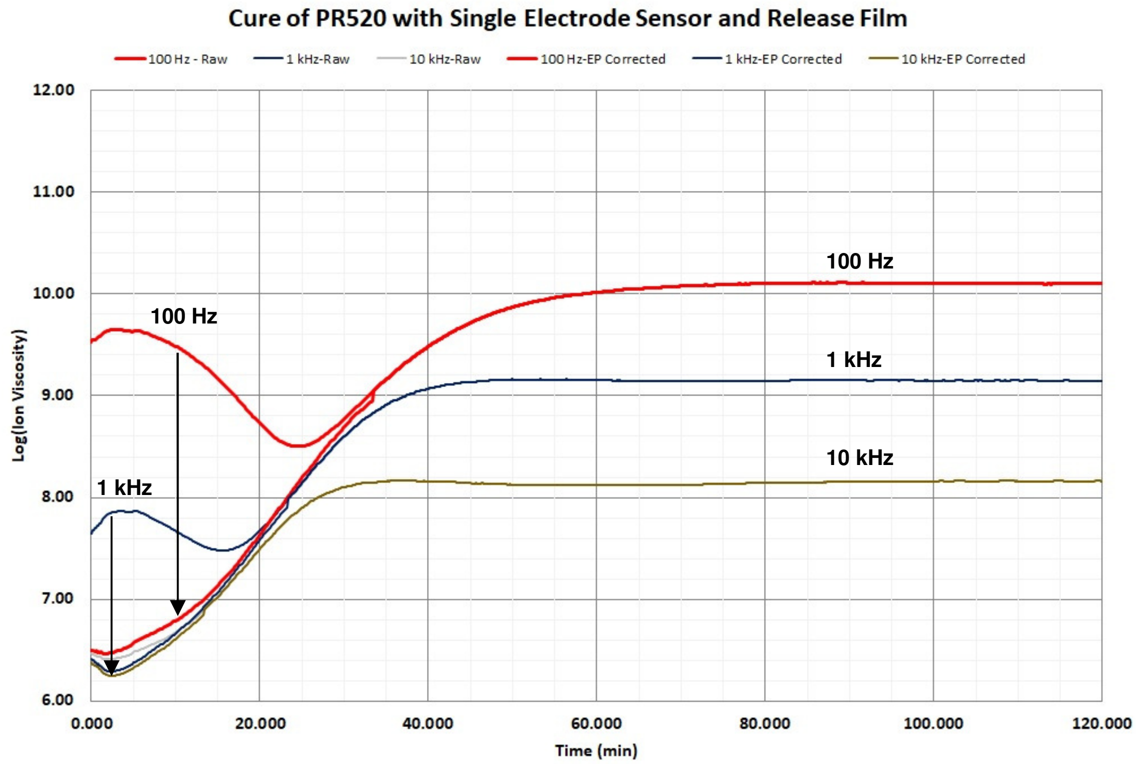


**Figure 4-11**
**Ion viscosity of PR520 before and after EP (boundary layer) correction, 1 Hz and 10 Hz not shown for clarity**

After boundary layer correction and after software removes data dominated by a dipolar response (frequency *dependent* resistivity), ion viscosity measured through the release film follows ion viscosity measured with direct sensor contact, as compared in Figure 4-12.

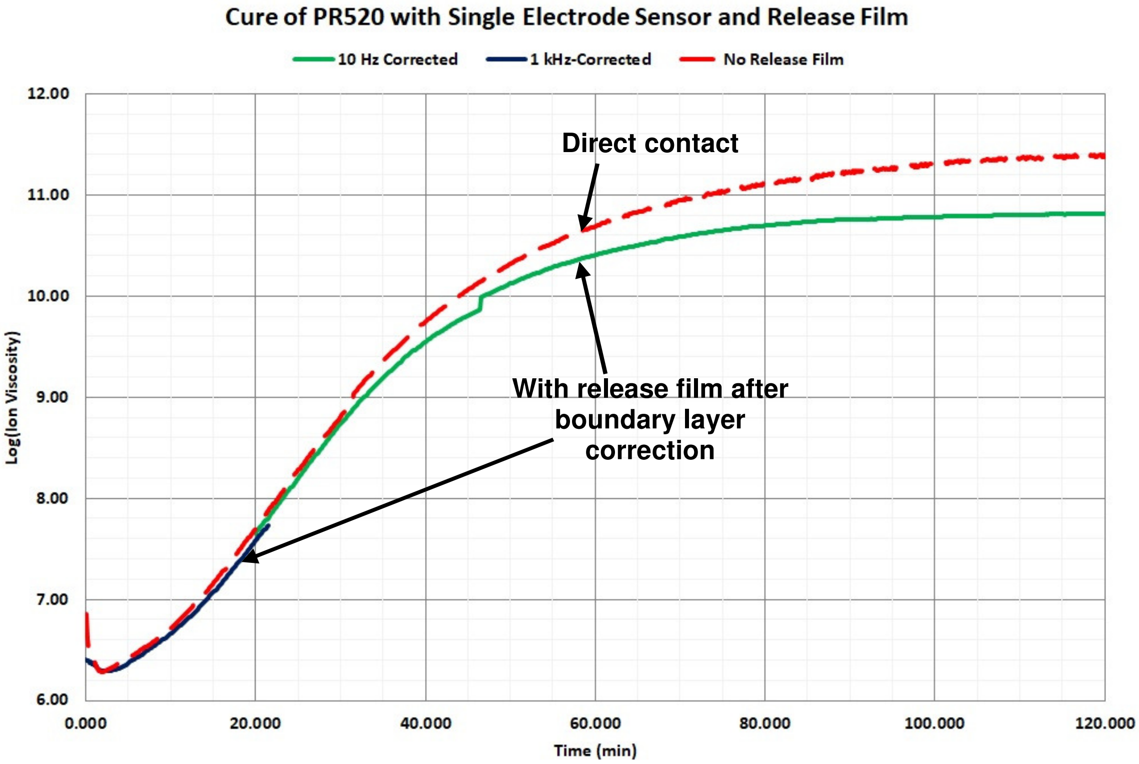


**Figure 4-12**
**Comparison of ion viscosity without release film and with release film after boundary layer correction**

Because insulators block DC signals, DC resistance measurements with release films or vacuum bags require placing the sensor in direct contact with the composite part. The release film or vacuum bag then must have a hole for the sensor and gaskets to seal the hole. A typical layup for a DC sensor is illustrated in the exploded view of Figure 4-13 and the photograph of Figure 4-14.

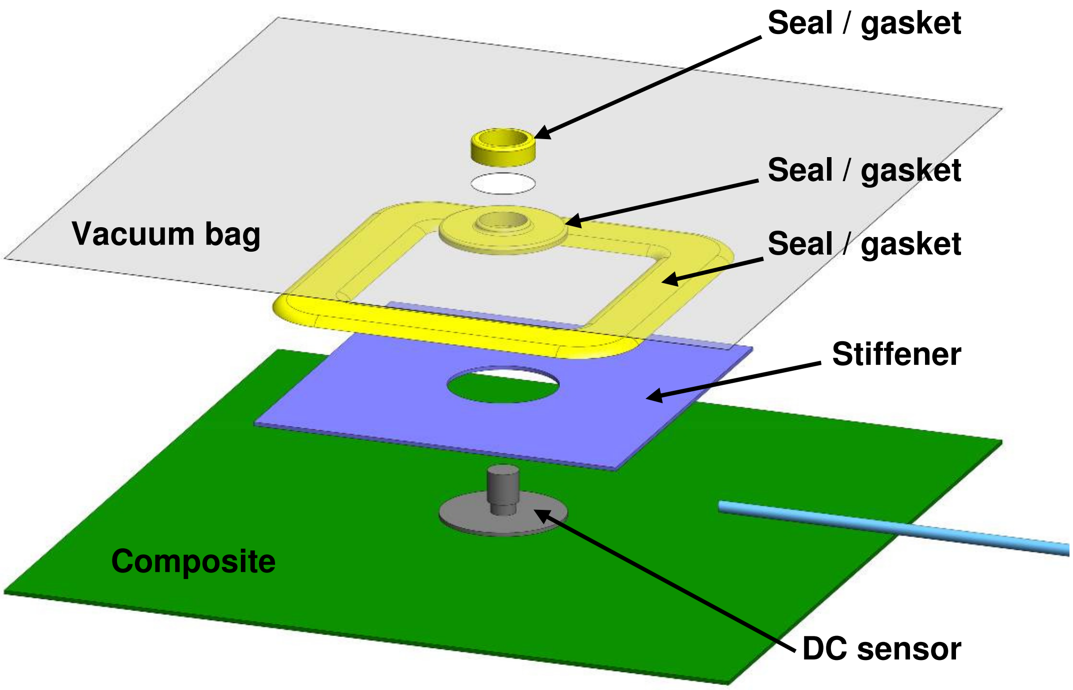


**Figure 4-13**
**Typical layup for cure monitoring with a DC sensor and vacuum bag**

**Figure 4-14**
**Layup for cure monitoring with a DC sensor and vacuum bag[9]**

In contrast, an AC dielectric sensor may be placed on top of the release film and vacuum bag, needing no hole in the film, as in Figure 4-15.

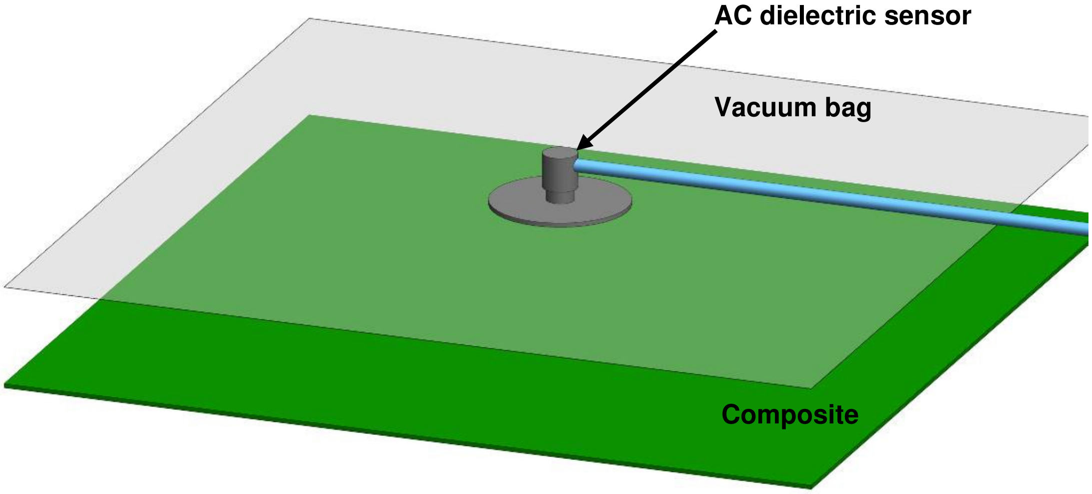


**Figure 4-15**
**Layup for cure monitoring through vacuum bag with an AC sensor**

## Conclusion

AC and DC measurements can be complimentary and it is important to be aware of the capabilities of each when deciding the method to use. While AC resistance or ion viscosity can accurately probe the entire cure of thermosets and composites, DC resistance shows discrepancies in comparison during early to mid-cure. During end of cure, however, AC and DC results agree.

The discrepancies in DC measurements are likely caused by a current-driven electrochemical reaction. Although the nature of the electrochemistry is unknown, the DC response during cure can be reproduced by an added *DC electrochemical resistance*.

For thermoset cure monitoring, electrode polarization may distort AC data at low frequencies during early cure. It is possible, however, to mathematically correct distorted data. DC methods allow faster measurement of extremely resistive materials, while AC measurements for these same materials might require very low frequencies and long data acquisition times.

DC methods require direct contact with the thermoset or composite, and are unable to measure through release films and vacuum bags. AC measurements therefore become especially useful in manufacturing. With the ability to monitor cure through insulators, AC methods enable the convenient use of sensors through a release film, preventing problems from adhesion of material to the sensor. In vacuum assisted resin transfer molding (VARTM) and similar processes, AC measurements through the vacuum bag avoid introducing breaks in the bag that could become a source of leakage.

## References

1. Lambient Technologies application note AN3.33, "Electrochemical Effects in AC and DC Cure Monitoring"

2. Lambient Technologies application note AN3.20, "Cure Monitoring Through Release Films and Vacuum Bags"

3. Day, D.R.; Lewis, J.; Lee, H.L. and Senturia, S.D., *Journal of Adhesion*, V18, p.73 (1985)

4. Lambient Technologies application note AN3.29, "Electrode Polarization and Boundary Layer Effects"

5. Lambient Technologies application note AN3.19, "Electrode Polarization with AC and DC Cure Monitoring"

6. 1" Single-Electrode sensor, manufactured by Lambient Technologies, Cambridge, MA USA. https://lambient.com

7. HTF-621 manufactured by Northern Composites, Greensboro, NC USA. https://northerncomposites.com

8. Cycom PR520N RTM manufactured by Solvay, Brussels, Belgium

9. Gardiner, Ginger, "DC Dielectric Sensors for Industrial Composites Production, *Composites World*, (Feb. 24, 2020). https://www.compositesworld.com/articles/dc-dielectric-sensors-for-industrial-composites-production

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