Insight — Technical Overview 3.04

AC and DC Cure Monitoring

Introduction

Dielectric cure monitors measure the electrical properties of a thermoset with AC signals across a range of frequencies. Measurement of resistance with DC methods can also reveal information about a material, but has disadvantages and limitations that the user must consider. In particular, the phenomenon of electrode polarization can distort DC resistance data and cause misinterpretation of cure state.

Dielectric cure monitoring

Dielectric cure monitoring, also known as *Dielectric Analysis* (DEA), measures a polymer's resistivity (ρ) and permittivity (ε'), which are a material's dielectric properties. Resistivity itself has a frequency independent (ρ_{DC}) component due to the flow of mobile ions and a frequency dependent (ρ_{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). Because frequency independent resistivity correlates with cure state, it is a useful material probe of epoxies, polyurethanes, polystyrenes, bulk molding compounds (BMC), sheet molding compounds (SMC) and other thermosets

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

(Eq. 4-1) $$IV = \rho_{DC}$$

DC resistance measurements

AC measurements of thermosets can obtain the full range of information about cure state. Simpler DC methods provide data that are limited but still useful. Resistance monitors are essentially highly sensitive ohmmeters that use a DC voltage source to drive current through the material between a pair of electrodes. Frequency independent resistivity, also known as ion viscosity, differs from resistance by only a scaling factor that depends on sensor geometry, so issues about ion viscosity apply equally to resistance.

Instruments using DC measurements achieve simplicity while sacrificing flexibility, and possibly accuracy, because of the following disadvantages:

- DC measurements can only obtain DC resistance
    - AC methods measure capacitance, frequency *independent* resistance and frequency *dependent* resistance
    - Frequency independent resistance, also known as *ion viscosity*, is the more accurate term that describes DC resistance
    - Frequency independent resistance can be measured across a range of frequencies that includes DC (0 Hz)
- DC measurements may produce distorted data caused by *electrode polarization*
- DC measurements are not possible with release layers
    - Release layers are very thin insulating sheets used to prevent material from adhering to a mold or platen
    - Release layers block DC current and prevent DC resistance measurement
- DC measurements may have systematic errors
    - Offset voltage drifts, thermal drifts and leakage currents in circuits cannot be distinguished from the true DC signal

Electrode polarization in AC and DC measurements

It is often *not* useful to measure frequency independent resistance (ρ_{DC}) using DC signals. 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.[1]

Figure 4-1 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.

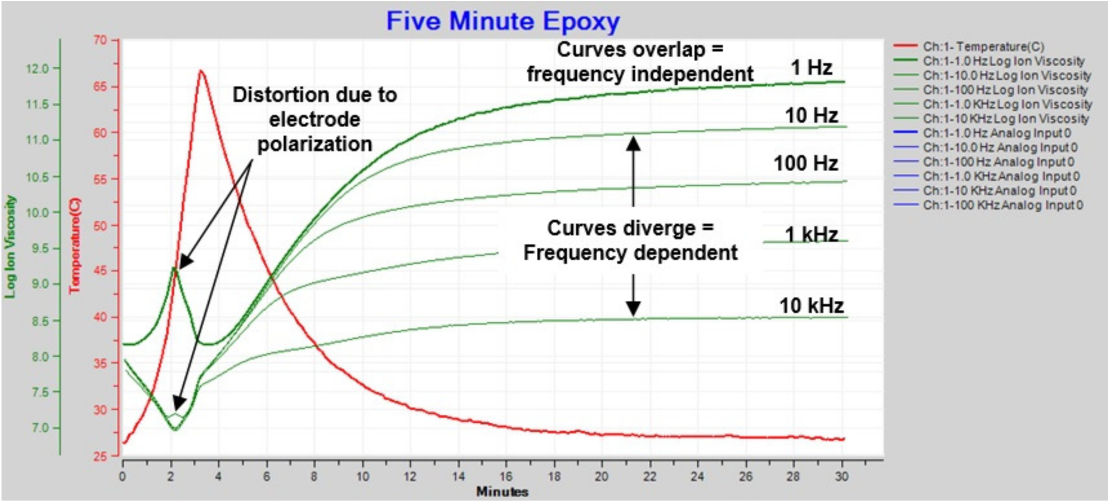

Figure 4-1
Ion viscosity / resistivity during cure of 5-minute epoxy

Three features are apparent in Figure 4-1:

- 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 causes considerable distortion in the 1 Hz data, shown in the expanded plot of Figure 4-2. This distortion changes the expected single minimum in resistivity/ion viscosity to a peak with *two* local minima.

Data from 10 Hz measurements 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.

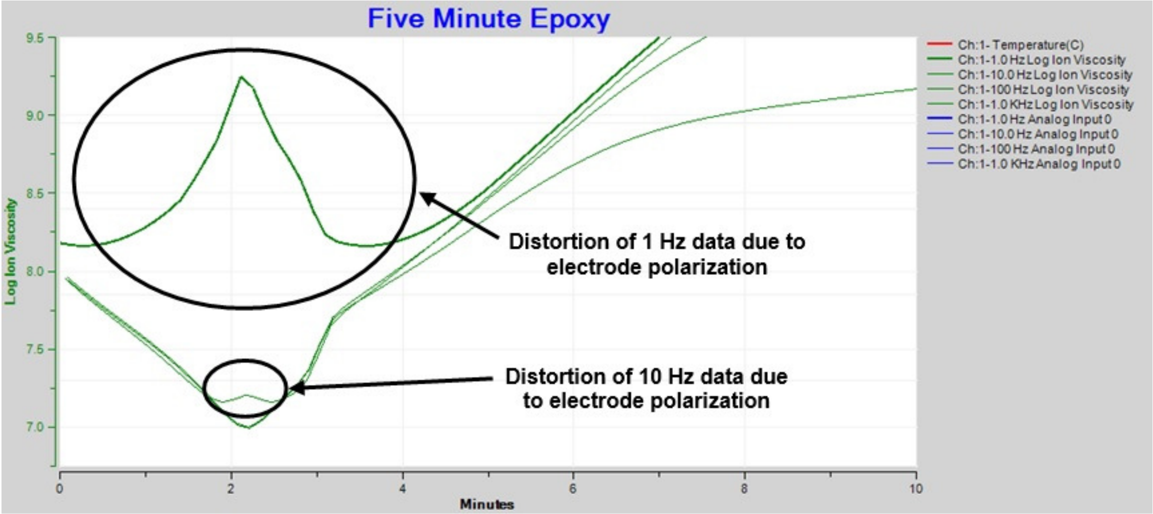

Figure 4-2
Expanded ion viscosity / resistivity around time of minimum viscosity

In many cases it is possible to mathematically restore information about cure.[1,2] Figure 4-3 shows how boundary layer correction—also called *electrode polarization* (EP) correction—recovers affected data. After EP correction, 1 Hz and 10 Hz ion viscosity show a proper minimum and are now consistent with the higher frequency data.

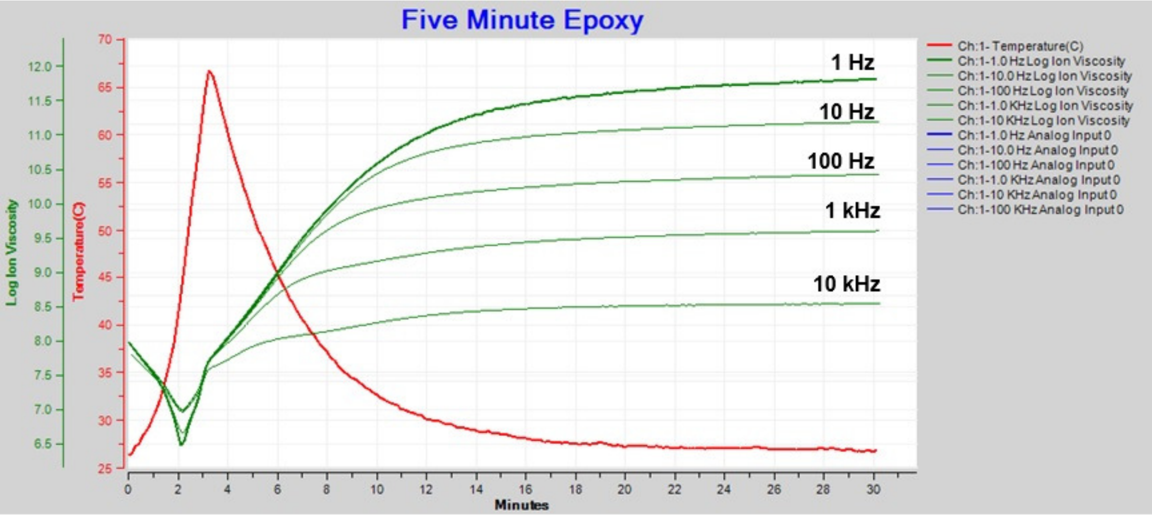

Figure 4-3
Resistivity / ion viscosity with boundary layer (EP) correction

After applying boundary layer correction, it is possible to use only 1 Hz ion viscosity to follow the entire five-minute epoxy cure, as shown in Figure 4-4.

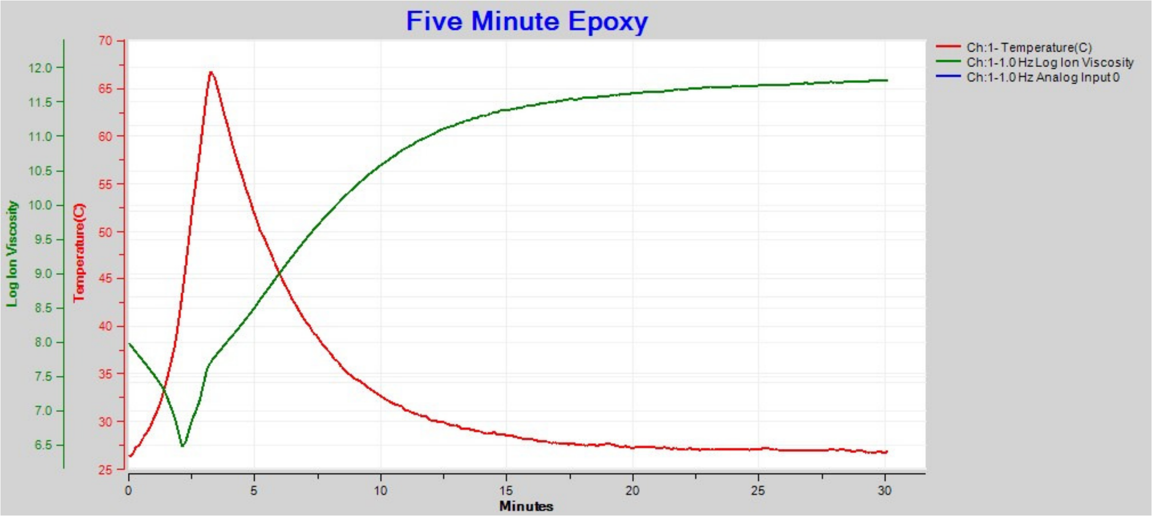

Figure 4-4
1 Hz ion viscosity with boundary layer (EP) correction

Release films with AC and DC measurements

Figure 4-5 illustrates a sensor in a mold with release film. Because release films are usually made from PTFE or other electrically insulating material, they can pass only AC signals, making cure monitoring impossible with DC methods. A suitably designed dielectric sensor, however, allows AC cure monitoring through a release film.

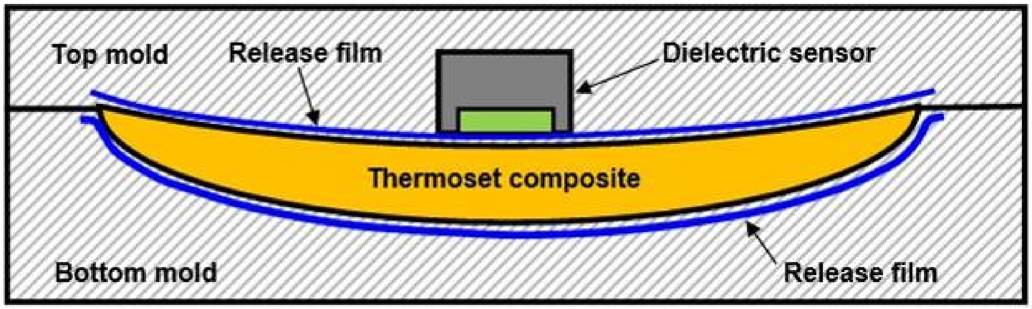

Figure 4-5
Dielectric sensor with release film

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

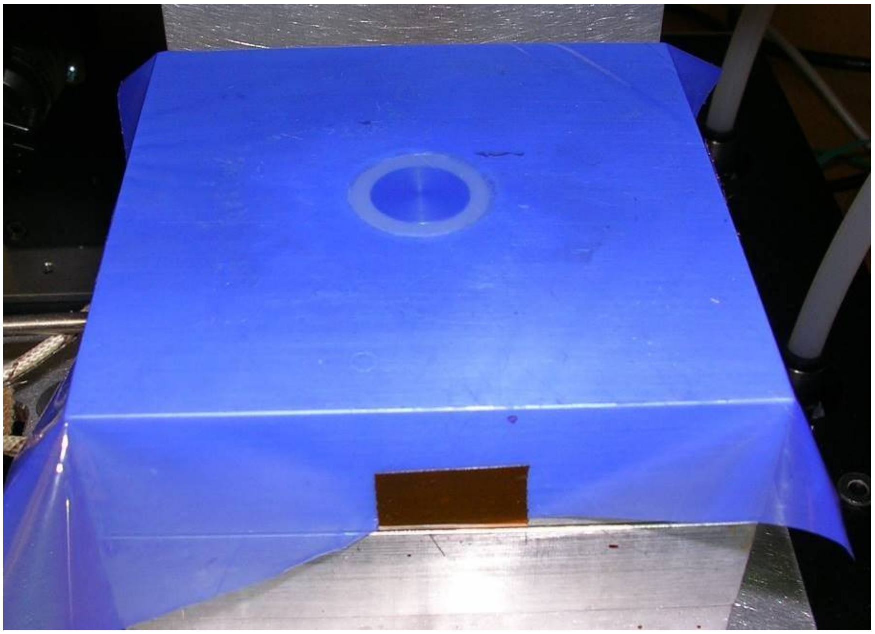

Figure 4-6
1” Single Electrode Sensor in press platen with HTF-621 release film

Figure 4-7 compares 100 Hz ion viscosity measured with and without the HTF-621 release film during cure of bulk molding compound (BMC). The curves are substantially the same except around the time of minimum ion viscosity, when the boundary layer effect distorts measurements through the film.

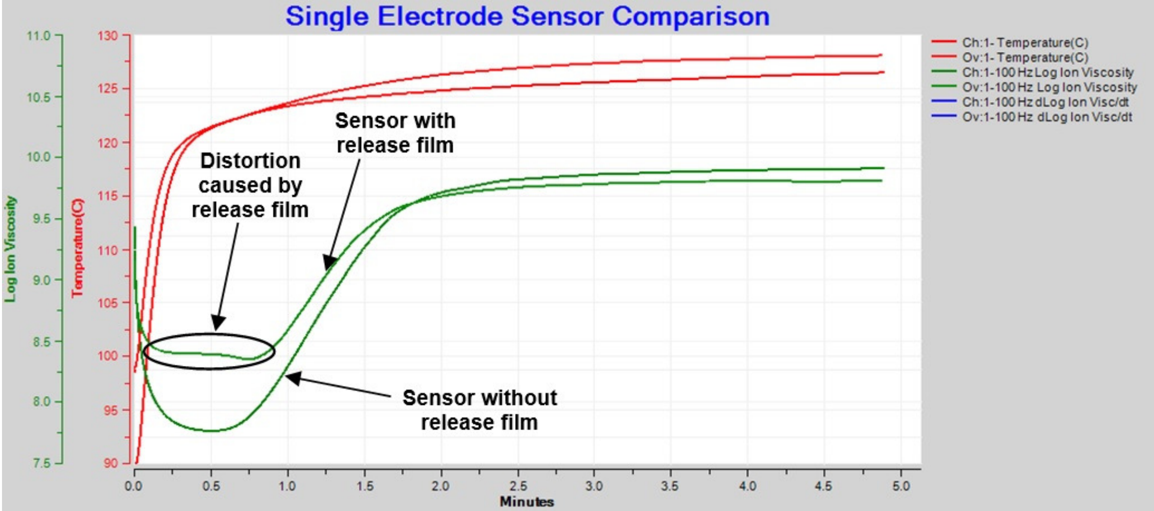

Figure 4-7
Comparison of BMC cure with and without release film,
100 Hz AC measurement

In many cases it is possible to mathematically correct this distortion and restore information about the cure.[1,2,3] Figure 4-8 shows how boundary layer correction recovers affected data. After correction, ion viscosity measured with the release film correctly follows ion viscosity measured without the release film. Note that for these two tests the minor differences between curves are largely due to differences in process temperatures.

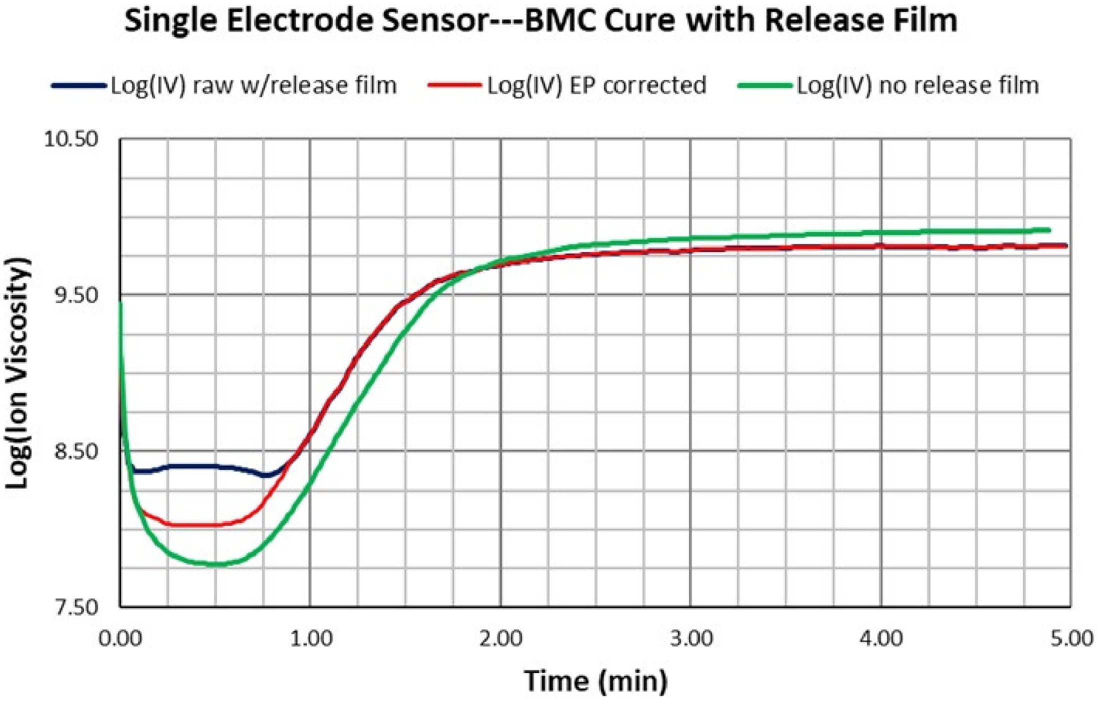

Figure 4-8
Comparison of raw ion viscosity and ion viscosity with EP (boundary layer) correction

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

- Possible distortion of data due to electrode polarization
- Inability to measure cure state through release films or vacuum bags

Figure 4-9 compares DC measurements of BMC using the 1-inch Single Electrode Sensor with and without the HTF-621 release film. For reference, data from the 100 Hz AC measurements are also plotted.

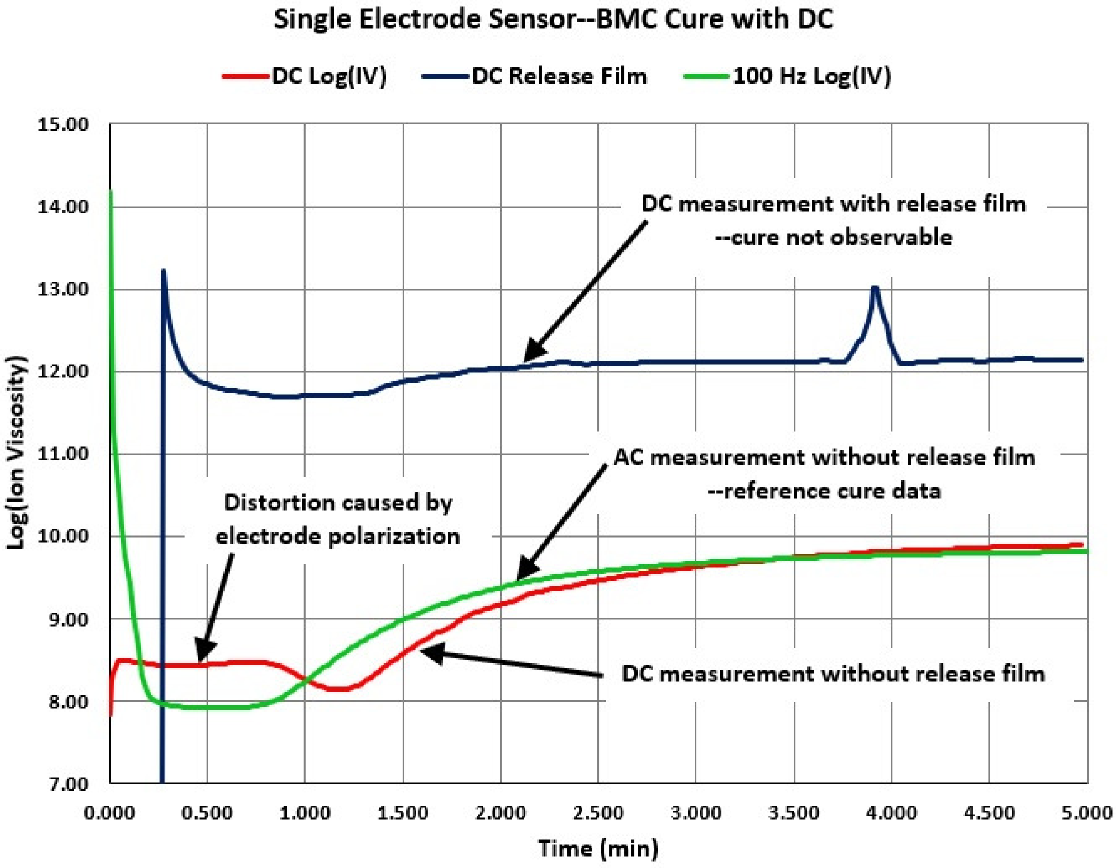

Figure 4-9
Comparison of AC and DC measurements of ion viscosity

DC measurements, even with no release film, show significant distortion around the time of ion viscosity minimum. While it is possible to correct distortion of AC data, it is not possible to correct DC data. Furthermore, DC measurements through the release film are not possible at all, indicated by the high ion viscosity at the measurement limit of the instrument.

Conclusion

While DC measurements of resistance are simple to make, they have disadvantages compared to AC measurements of dielectric properties. For thermoset cure monitoring, electrode polarization may distort DC data and cause misinterpretation of cure state. Although electrode polarization can also affect low frequency AC measurements, the additional information gained from dielectric properties allows correction of the distorted data.

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

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

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

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