Insight — Application Note 3.36

Cure Monitoring of Carbon Fiber Composites with Carbon+ Coated Sensors

Introduction

The increasing use of carbon fiber composites in high-volume production is driving the need to know cure state in real time. Dielectric cure monitoring (also called *Dielectric Analysis*, or DEA) is the only method that can probe material properties during manufacturing, and a past study had demonstrated its ability to reduce average SMC press cycle times by detecting end of cure. Compared to a timer set at 60 seconds, the 10 second reduction in SMC molding time, attributed to DEA, was estimated to save $70,000/year/press in labor costs alone.[1]

Dielectric sensors normally require filters to block conductive fibers and prevent short circuiting of the electrodes. Filters, however, must be replaced manually after each test and add time, effort and cost, so it is necessary to avoid them in rapid, repetitive operations.

For cure monitoring without filters, Carbon+Unitrode sensors from Lambient Technologies have a rugged, insulating coating that allows direct contact with carbon fiber composites. As a result, Carbon+Unitrode sensors can be used in manufacturing to observe the entire cure, and can detect end of cure for opening a press or mold.

Figure 36-1
Carbon+Unitrode-1″ coated, reusable sensor

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*.

Dielectric cure monitoring measures the conductance (G_{MUT}) and capacitance (C_{MUT}) of the Material Under Test between the electrodes of a sensor. These two quantities are then used to calculate the material properties of conductivity, resistivity and relative permittivity as they change with time. Conductivity (σ_{MUT}) is simply the conductance scaled by a sensor's cell constant, or *A*/*D* ratio:

(Eq. 36-1) $$\sigma_{MUT} = G_{MUT} / (A/D) \qquad (\text{ohm}^{-1}\text{-cm}^{-1})$$

Conductivity has both frequency independent (σ_{FI}) and frequency dependent (σ_{AC}) components. In an oscillating electric field, σ_{FI} 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 add together to produce the material conductivity:

(Eq. 36-2) $$\sigma_{MUT} = \sigma_{FI} + \sigma_{AC} \qquad (\text{ohm}^{-1} – \text{cm}^{-1})$$

The resistivity of the Material Under Test (ρ_{MUT}) is the inverse of conductivity:

(Eq. 36-3) $$\rho_{MUT} = 1 / \sigma_{MUT} \qquad (\text{ohm-cm})$$

Resistivity is the inverse of conductivity and is the bulk resistance ($R_{MUT} = 1/G_{MUT}$) scaled by the *A*/*D* ratio:

(Eq. 36-4) $$\rho_{MUT} = R_{MUT} (A/D) \qquad (\text{ohm-cm})$$

Like conductivity, resistivity also has both *frequency independent* (ρ_{FI}) and *frequency dependent* (ρ_{AC}) components. Frequency independent resistivity has particular importance for cure monitoring. Before gelation, the amount of polymerization affects both mechanical viscosity and the movement of ions, and therefore influences ρ_{FI}. For many materials the change in ρ_{FI} is proportional to the change in mechanical viscosity. To emphasize this relationship, the term *ion* *viscosity* (*IV*) or *AC ion viscosity* (IV_{AC}) was coined as a synonym for frequency independent resistivity and is defined as:

(Eq. 36-5) $$IV = \rho_{FI} = 1 / \sigma_{FI} \qquad \text{(ohm-cm)}$$

Cure monitoring with the Carbon+Unitrode-1″ coated sensor

Carbon fiber reinforced prepreg (CFRP) and carbon fiber sheet molding compound (CF-SMC) take advantage of the high strength and low weight of carbon fibers. However, direct contact of conductive fillers with dielectric sensors can short circuit electrodes and interfere with the measurement.

Deposition of an insulating layer on electrodes, to prevent fiber contact, was reported by McIlagger[2] in 2000, and this technique has been used by others since then to study the cure of carbon fiber composites. Similarly, the reusable Unitrode-1″ dielectric sensor of Figure 36-2, with its Carbon+ coating, enables cure monitoring of CFRP and CF-SMC.

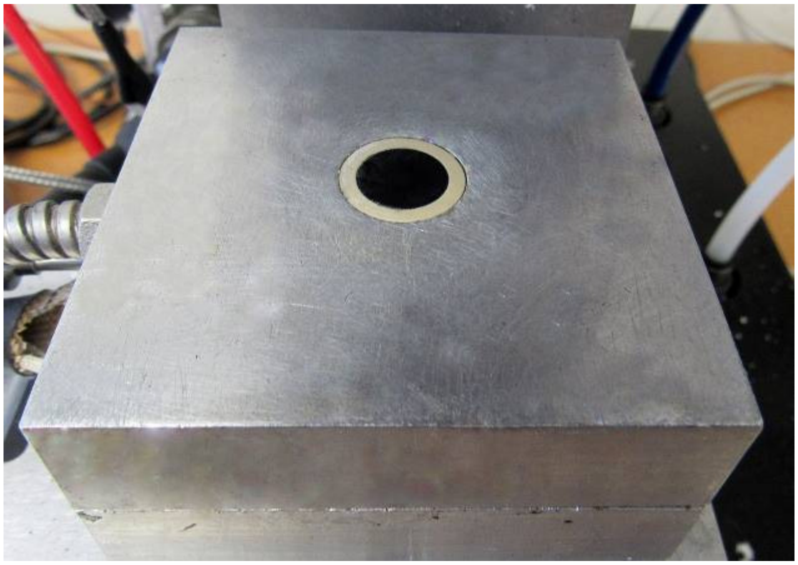

Figure 36-2
Carbon+Unitrode-1″ coated dielectric sensor in press platen

Figure 36-3 depicts a lay-up with the Carbon+Unitrode sensor. A non-porous insulating layer is permanently bonded to the electrode, preventing contact with conductive fibers. As a result, a composite can be placed directly on the sensor without a filter. Figure 36-4 is a cross-section of this lay-up, showing how the electric field from the electrode passes through the insulating layer into the composite.

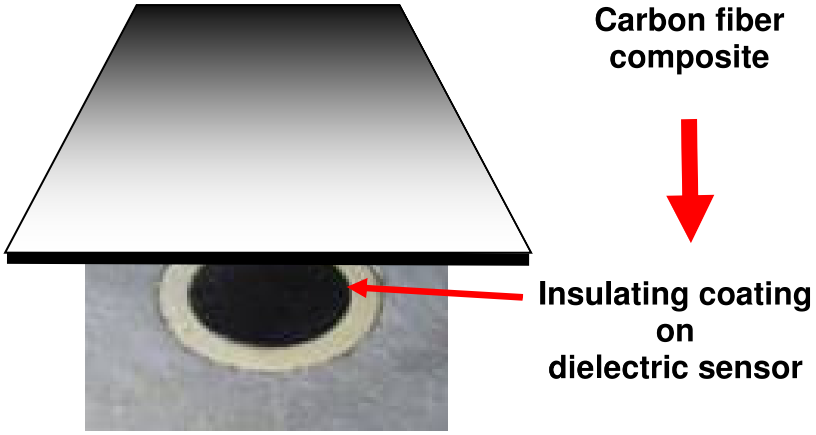

Figure 36-3
Typical lay-up with a Carbon+Unitrode sensor

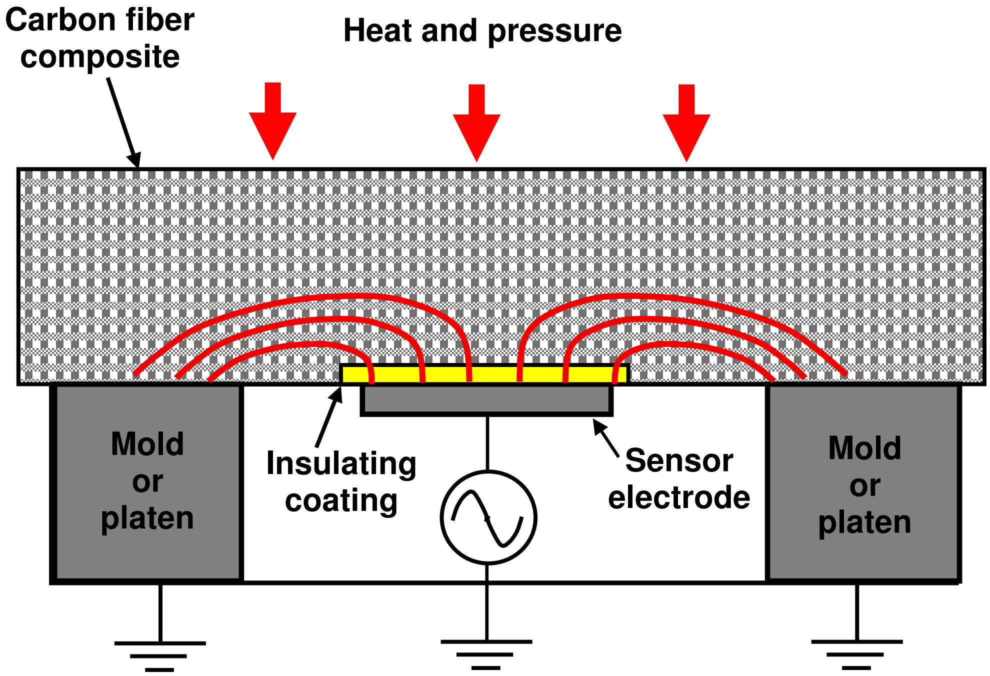

Figure 36-4
Cross section of lay-up with coated sensor, showing electric field (red)

Figure 36-5 plots log(*IV*) and slope during the two-stage cure of a carbon fiber reinforced prepreg on a Carbon+Unitrode sensor. A heated press applied pressure to the CFRP, which is in direct contact with the sensor, for the first stage at 120 °C. During the second stage, at 190 °C, the dielectric response follows the typical behavior of thermosets. With the sudden increase in temperature, the resin's mechanical viscosity and ion viscosity both quickly decrease. For a moment the material is at minimum mechanical and ion viscosity, until the curing reaction dominates the response—in this case at about 70 minutes—and mechanical and ion viscosity increase.

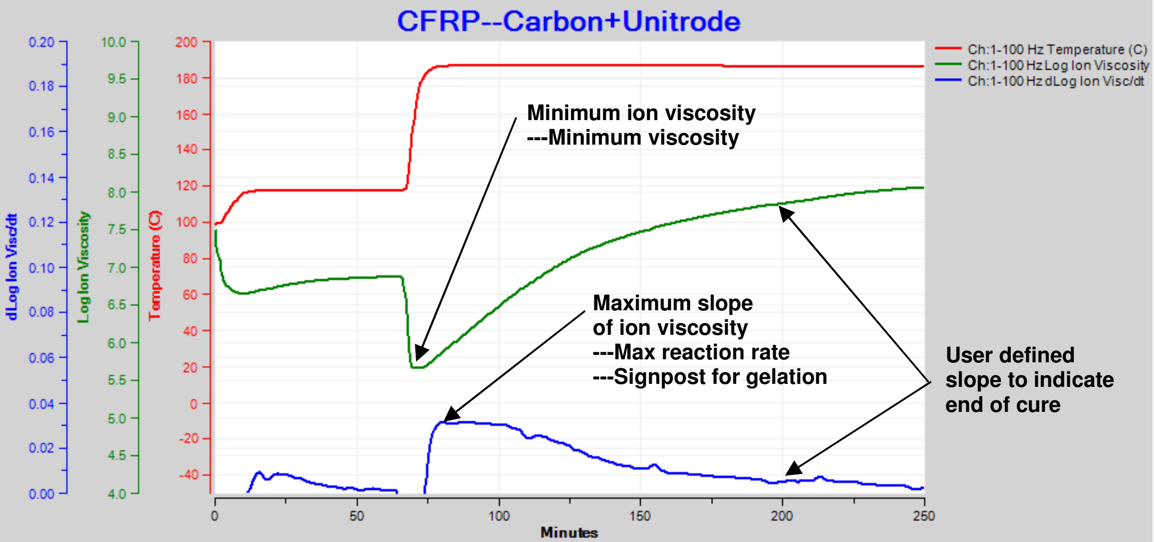

Figure 36-5
Cure of CFRP in direct contact with Carbon+Unitrode-1” sensor

At gelation, mechanical viscosity increases rapidly until it becomes unmeasurable. Because gelation is a mechanical—not an electrical—event, no dielectric feature indicates the gel point, although the time of maximum slope can be used as a signpost that can correlate with gelation.

As the reaction ends, the ion viscosity curve flattens and its slope approaches zero. In manufacturing, end of cure is a user defined slope that depends on the requirements of the application. Once end of cure is identified, dielectric cure monitoring equipment can issue a signal to automatically open a press or mold.

Cure monitoring with a filtered Unitrode-1” sensor

Figure 36-6 shows a Unitrode-1” reusable sensor in a platen. By allowing accurate observation of cure state, filters to block fibers and pass resin to dielectric sensors have been the preferred method of dealing with carbon fiber composites. A filter may be porous paper, fiberglass felt or other suitable material. The carbon composite sample is placed on the filter as illustrated in Figure 36-7.

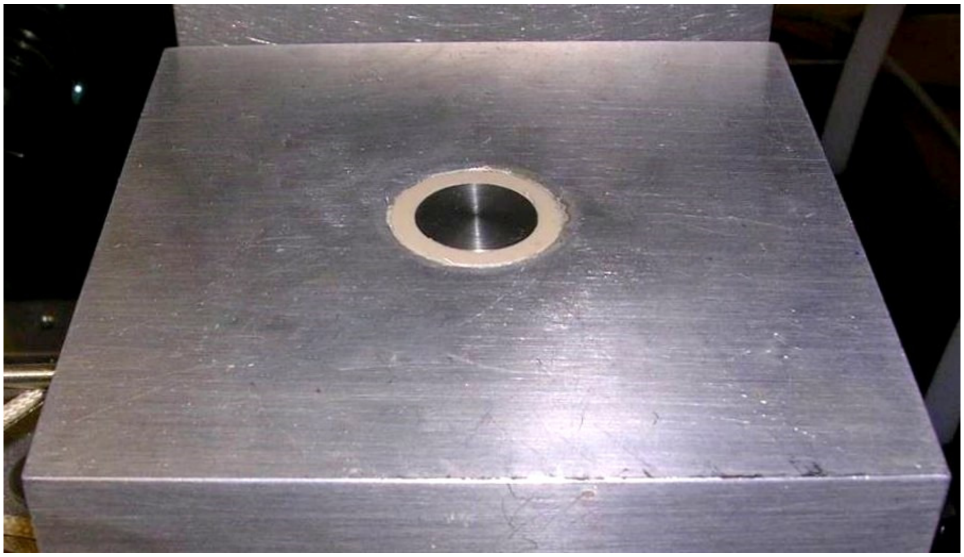

Figure 36-6
Unitrode-1" dielectric sensor in press platen

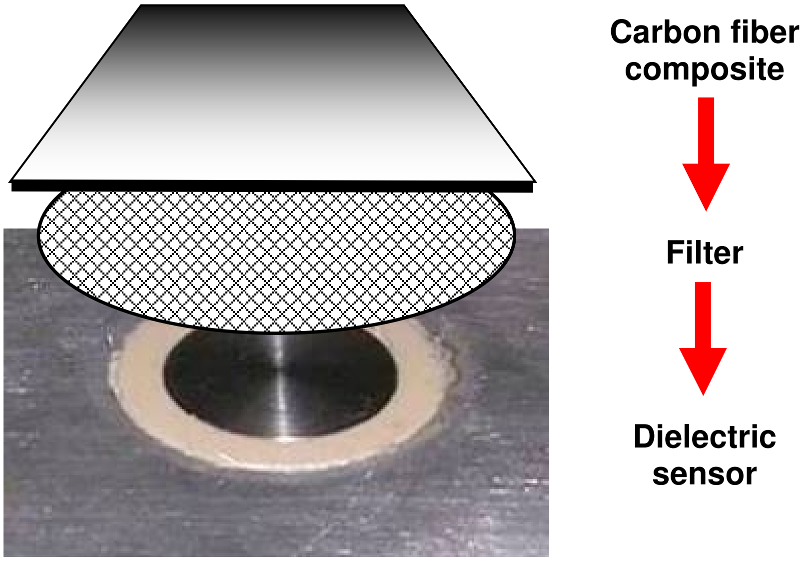

Figure 36-7
Typical lay-up with filter on sensor

Upon heating and compression, resin flows from the sample into the filter. Figure 36-8 is a cross-section of the lay-up, showing how the electric field from the electrodes penetrates the filter, which is non-conductive and has little effect on the electrical response. Consequently, dielectric measurements with a filter primarily detect only the resin and accurately indicate the state of the material.

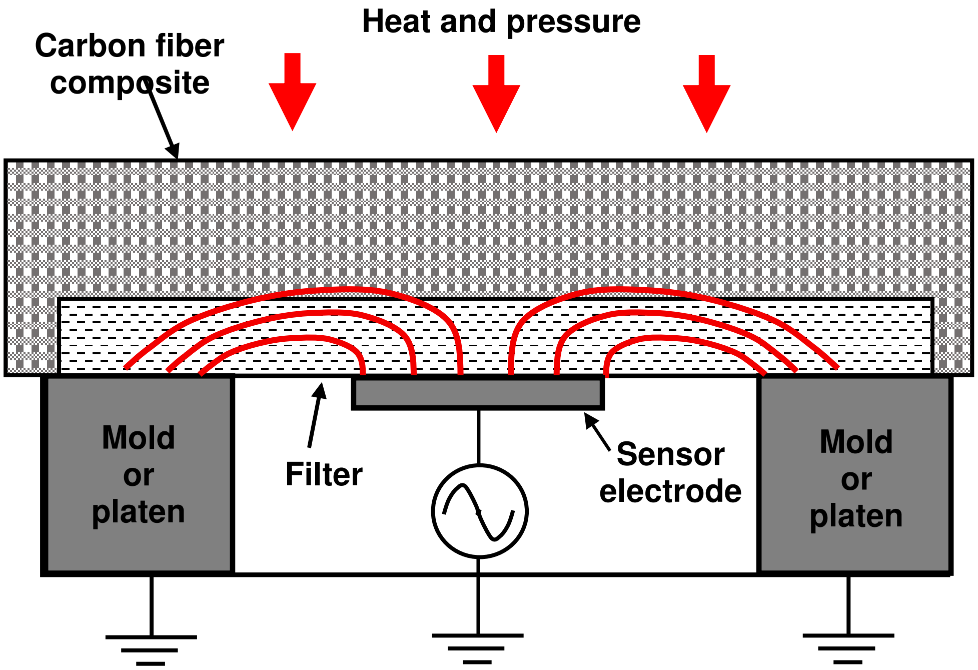

Figure 36-8
Cross section of lay-up with filtered Unitrode sensor, showing electric field (red)

Figure 36-9 compares ion viscosity and slope of CFRP measured with the filtered and coated Unitrode.

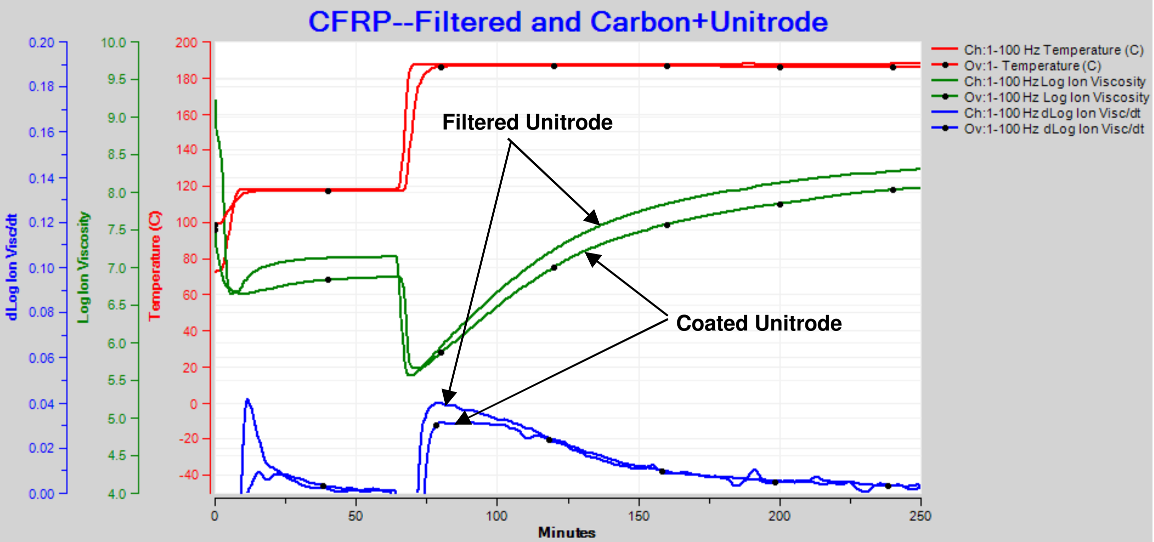

Figure 36-9
Comparison of CFRP data from filtered and coated Unitrode sensors

Except for a minor difference in level, the ion viscosity curves are essentially the same for the two sensors. This difference may be caused by the added conductivity of the carbon fibers, which would decrease the ion viscosity measured with an unfiltered, coated sensor. The slope curves, however, largely overlap and indicate the coated Carbon+Unitrode can measure the cure state of CFRP—minimum viscosity, maximum reaction rate and a user defined slope for end of cure—as well as a filtered sensor.

Electrical models of filtered and coated sensors

Figure 36-10 shows the electrical model of the resin-electrode system for a filtered sensor. Ion viscosity, which is electrical resistivity, is derived from G_{MUT}, correlates with cure state and is the property most useful for dielectric cure monitoring.

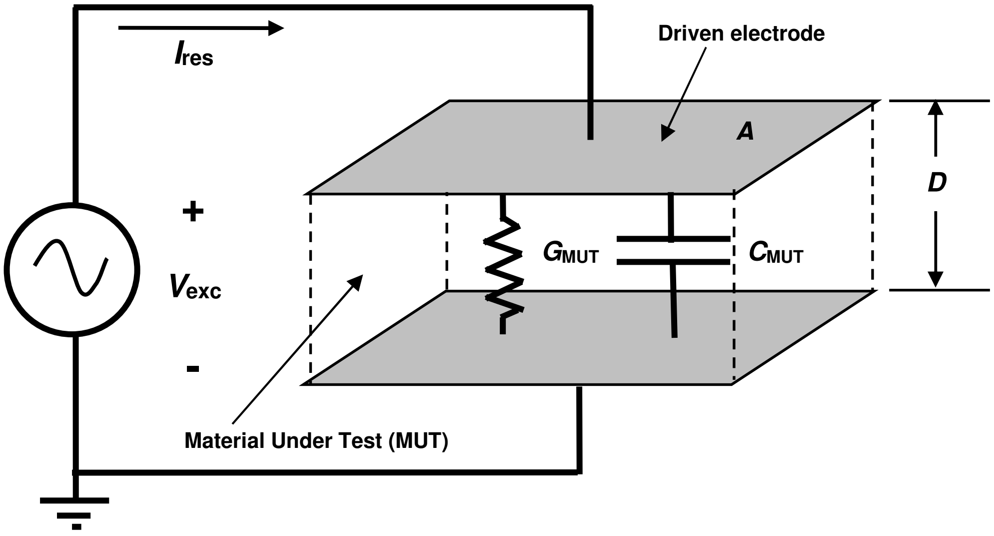

Figure 36-10
Electrical model of resin on filtered sensor

In contrast, Figure 36-11 is the electrical model of the composite-insulator-electrode system for a coated sensor. The insulator introduces a capacitor between the electrode and the material under test. This capacitor acts as a boundary layer, an extra element not included in Figure 36-10. Because capacitors pass only AC signals, cure monitoring with coated sensors is not possible with DC methods.

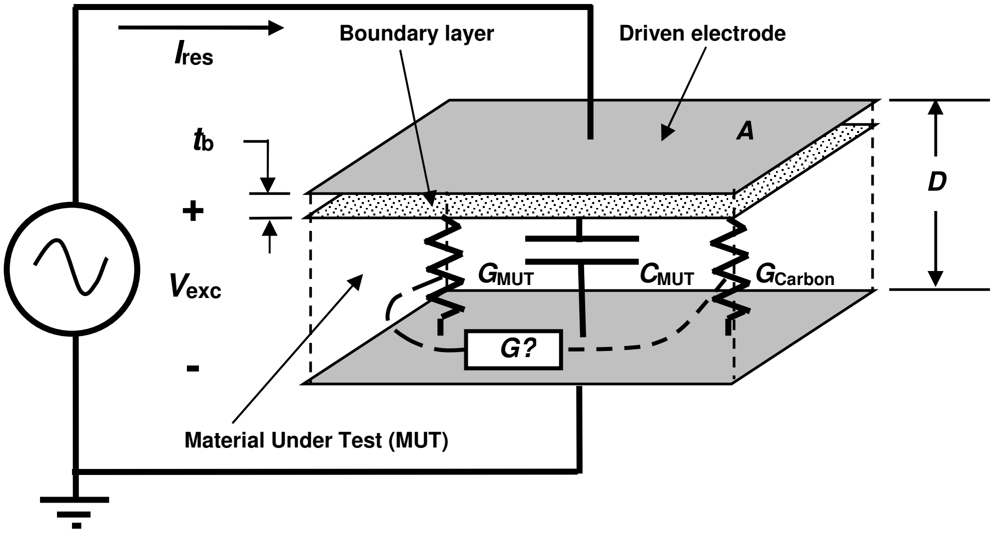

Figure 36-11
Electrical model of carbon fiber composite on a coated sensor

With a carbon fiber composite placed directly on the electrode, dielectric measurements combine the contributions of:

- The boundary layer capacitance
- The fixed conductance of the carbon fibers: G_{Carbon}
- The time varying conductance of the resin: G_{MUT}
- The unknown and time varying conductance between the carbon fibers and the resin: *G?*

For slowly curing materials like CFRP, the additional elements, G_{Carbon} and *G?*, have little effect—and the coated Carbon+Unitrode yields essentially the same data as the filtered sensor, as plotted in Figure 36-9. However, in rapidly reacting materials like CF-SMC, which cures in two minutes, the carbon fibers can distort ion viscosity measured with coated sensors. The result may be delayed times of minimum log(*IV*) and maximum slope, like the data of Figure 36-12 for CF-SMC.

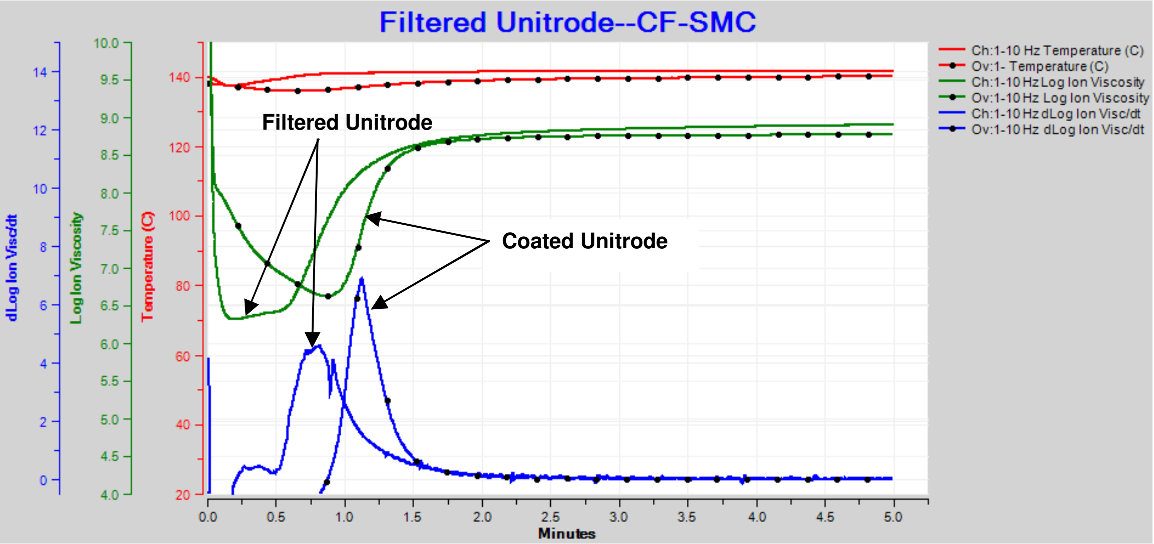

Figure 36-12
Comparison of CF-SMC data from filtered and coated Unitrode sensors

Figure 36-13 plots ion viscosity during the 140 °C cure of four samples of CF-SMC on the Carbon+Unitrode, demonstrating the repeatability of measurements with a coated sensor, even with distorted data. Variations of ion viscosity at end of cure are common with carbon filled composites, and are probably caused by sample-to-sample differences in the mix of fiber and resin. The slope curves, however, do not depend on ion viscosity level and have the consistency required for detecting end of cure during manufacturing, when identifying the time for opening a press or mold is the main concern.

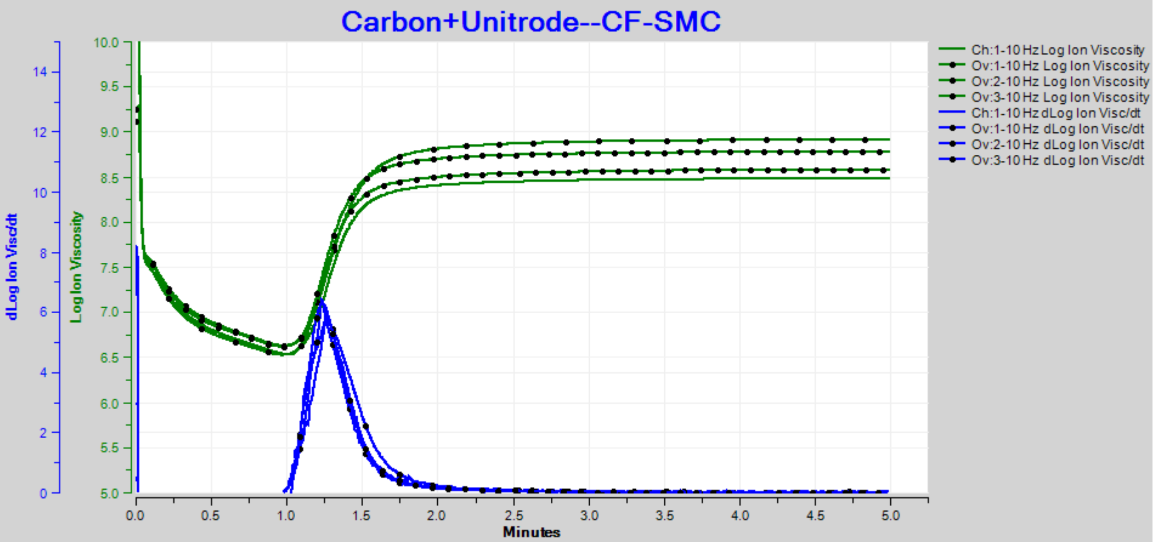

Figure 36-13
Four runs of CF-SMC with the Carbon+Unitrode-1” sensor, @ 140 °C

Conclusion

- The Carbon+Unitrode makes measurements that correspond well with the true cure state of slower reacting composites like CFRP, and can replace dielectric sensors that require filters
- For rapidly curing materials, ion viscosity and slope data from a Carbon+Unitrode may be distorted during early through mid-cure
    - A coated sensor can still provide useful correlations with the material state, and determine end of cure based on a chosen slope

References:

1. Day, D.R. and Lee, H.L., “Analysis and Control of SMC Part to Part Variations,” Session 13-C of *Proceedings of the 17th Annual Conference, Composites Institute*, *the Society of the Plastics Industry, Inc., Feb 3-6, 1992.*

2. A. McIlhagger, D. Brown, B. Hill, “Development of a dielectric system for the on-line monitoring of the resin transfer moulding process,” Composites Part A Applied Science and Manufacturing, 31(12): 1373-1381, December 2000

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