Insight — Application Note 3.37

Sensors for Cure Monitoring Of Carbon Fiber Composites

Introduction

Testing pure resins or resin-fiberglass composites is generally simple and only requires placing the material on the sensor. With carbon-fiber composites, however, a conductive fiber contacting the sensor will short circuit its electrodes and cause bad, unusable measurements. Despite this problem, cure monitoring of carbon fiber reinforced prepreg (CFRP), carbon fiber sheet molding compound (CF-SMC) and similar composites is possible with filtered or coated sensors.

Dielectric and DC resistance cure monitoring

The LT-440 Dielectric Channel[1] is the only instrument that can make AC and DC measurements for studying thermosets and composites. Both methods can probe cure state, but it is important to understand their differences, and their different effects on sensors, when deciding which to use.

Figure 37-1
LT-440 Dielectric Channel with AC/DC cure monitoring capability

Dielectric cure monitoring, also called *Dielectric Analysis* (DEA) is an AC technique that excites a sensor with a sinusoidal signal of chosen frequency and amplitude, and a DC bias of zero volts. In contrast, DC resistance cure monitoring uses a constant bias voltage for the excitation. Figure 37-2 illustrates these two types of signals.

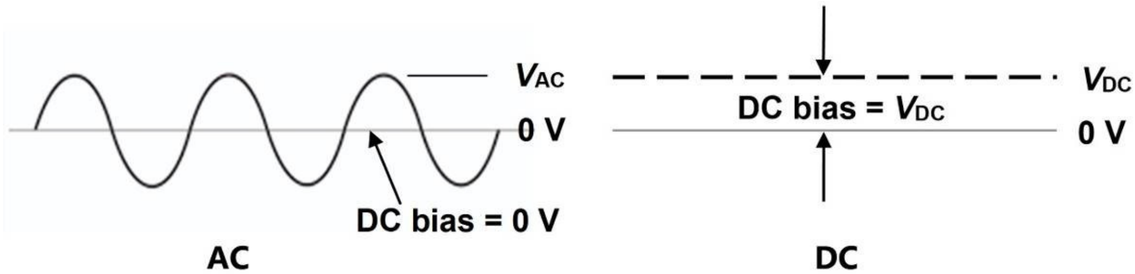

Figure 37-2
AC and DC excitations used for cure monitoring

Dielectric cure monitoring measures the AC electrical resistance and capacitance of a material, which change as the material cures. These measurements are then scaled by a sensor's cell constant and converted to the material properties of resistivity and permittivity. Resistivity has *frequency independent* (ρ_{FI}) and *frequency dependent* (ρ_{AC}) components, but the frequency independent portion 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. 37-1) IV or $IV_{AC} = \rho_{FI}$ (ohm-cm)

DC resistance cure monitoring measures the DC resistance of a material as it cures, and this resistance can be converted to DC resistivity (ρ_{DC}) by scaling with a sensor's cell constant. Just as AC ion viscosity is a synonym for frequency independent resistivity, DC ion viscosity (IV_{DC}) may be used in place of DC resistivity:

(Eq. 37-2) $IV_{DC} = \rho_{DC}$ (ohm-cm)

This application note presents 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 user defined end of cure. For brevity, log(ion viscosity) will be called log(*IV*) and *slope of* log(ion viscosity) will simply be called *slope*.

Filtered sensors for cure monitoring

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 sensors can short circuit electrodes and interfere with the measurement.

The use of filters, illustrated in Figure 37-3, is the easiest way to deal with this problem. When placed on a sensor, the filter blocks conductive fibers while allowing resin to flow to the electrodes. Recommended filters are made of glass microfiber without binders, such as Whatman 1827-047 or equivalent.

Glass microfiber filters are soft, and occasionally, upon application of pressure, carbon fibers may still penetrate the filter and contact the sensor. To further reduce the likelihood of short circuits, a second filter may be placed on top of the glass microfiber filter. This second filter is usually a coarse fiberglass cloth that is more porous but presents additional resistance to the infiltration of carbon fibers.

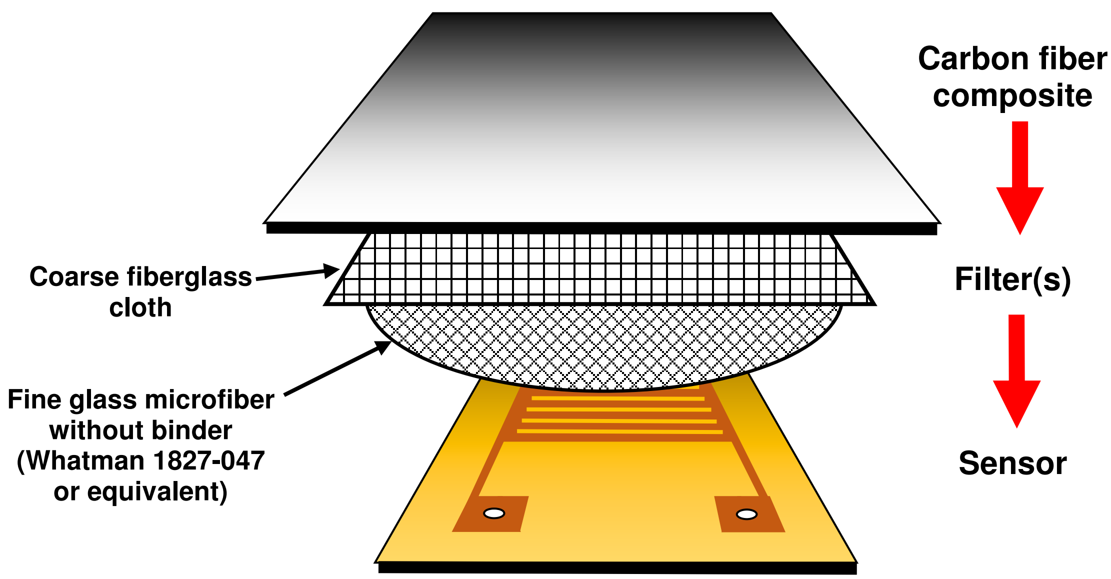

Figure 37-3
Typical lay-up with filter(s) on a disposable, flexible sensor

Filters work equally well with AC or DC cure monitoring and may be used with flexible, disposable sensors as shown in Figure 37-3, or with rigid, reusable sensors installed in a platen or mold, such as the Ceramicomb-1"[2] of Figure 37-4.

Figure 37-4
Reusable Ceramicomb-1" sensor installed in laboratory press platen

Figure 37-5 shows log(*IV*) and slope from three consecutive tests of a carbon fiber sheet molding compound with the filtered Ceramicomb-1" sensor of Figure 37-4. The repeatability for this particular CF-SMC is due to the low viscosity of the resin, which results in consistent, 100% filter penetration.

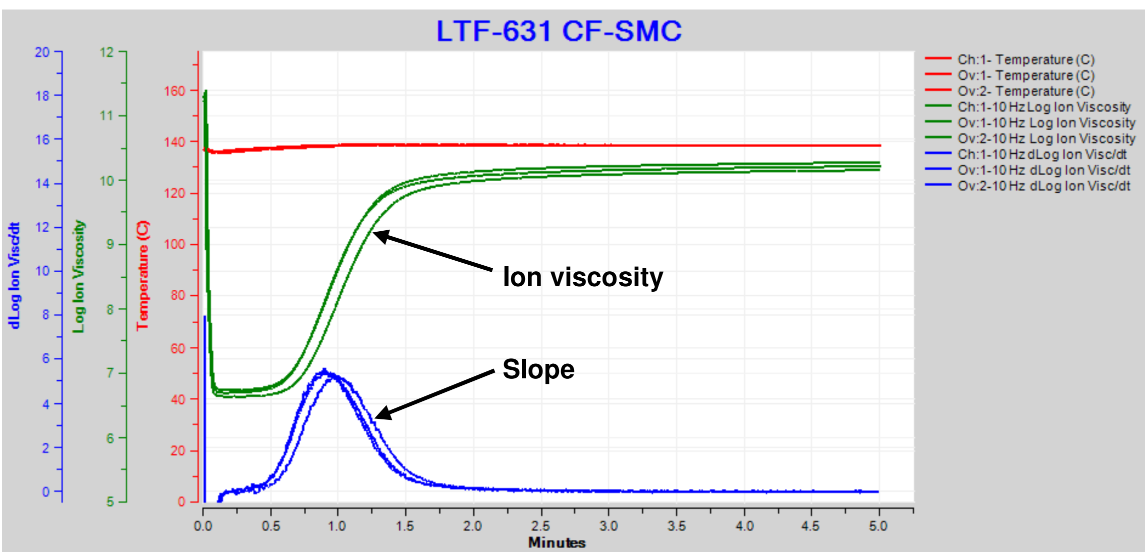

Figure 37-5
CF-SMC data during cure with filtered Ceramicomb-1" sensor, three consecutive tests

However, more viscous resins may soak through a filter by varying amounts. Figure 37-6 shows lesser degrees of filter penetration cause the ion viscosity curves to shift to higher levels. Smaller amounts of resin in contact with the sensor result in higher measured resistance and therefore higher *apparent* ion viscosity.

Regardless of the difference in resin penetration, the shapes of the ion viscosity curves are remain the same, as indicated by the close overlap of slope data. So identification of characteristic features of the cure—minimum ion viscosity, maximum slope/reaction rate and the user defined slope for end of cure—are unaffected, even with large variances in the amount of resin that reaches the sensor. *For more information, see application note AN 3.05, "Linear vs. Logarithmic Scales."*

100 % filter penetration **~50% filter penetration** **~10% filter penetration**

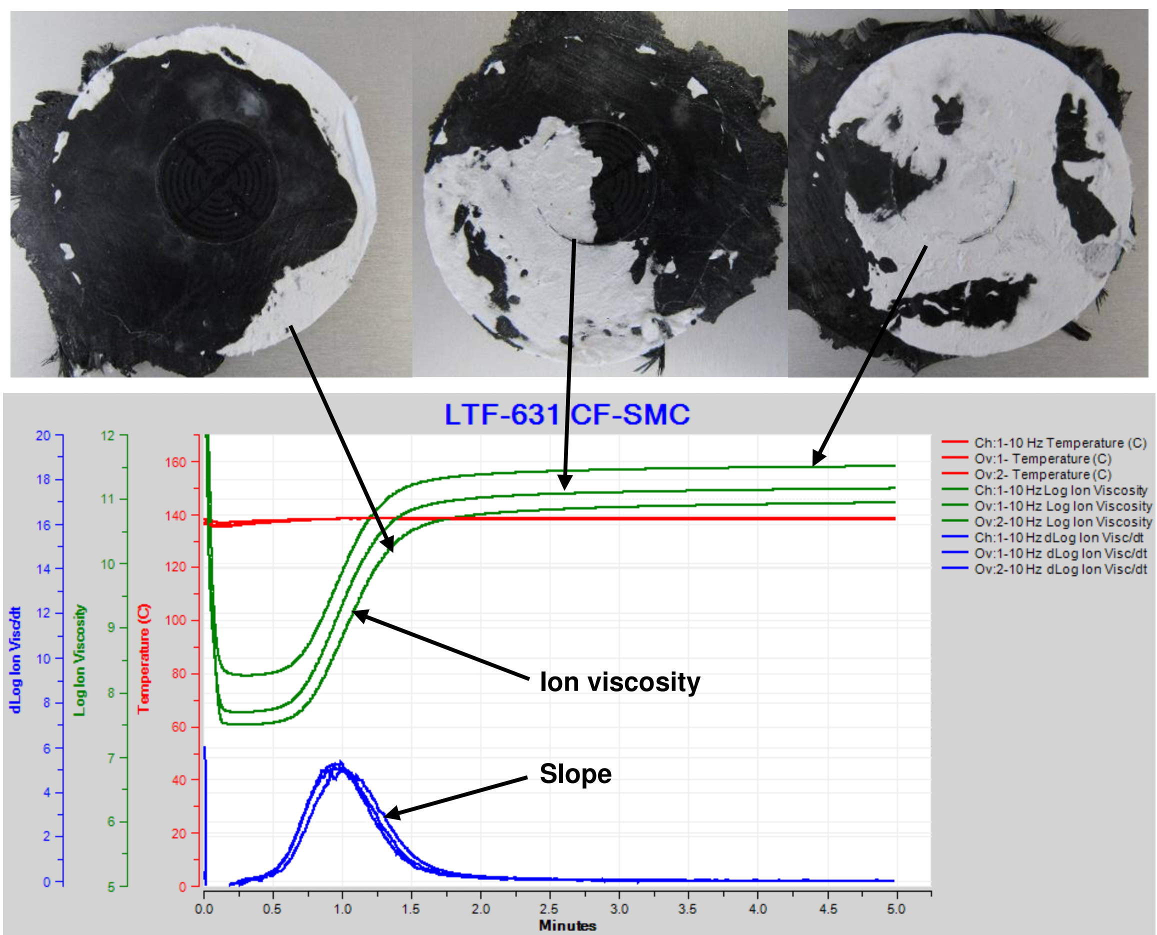

Figure 37-6
CF-SMC data during cure, three tests with different filter penetration

Cure monitoring of carbon fiber composites in manufacturing

The increasing use of carbon fiber composites in high-volume production is driving the need to know cure state in real time. AC dielectric cure monitoring (DEA) and DC resistance cure monitoring are the only methods that can probe material properties during manufacturing. A past study had demonstrated the ability of DEA 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 dielectric cure monitoring, was estimated to save $70,000/year/press in labor costs alone.[3]

Dielectric sensors 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 and use sensors with direct contact for manufacturing or rapid, repetitive operations.

Shaped sensor for direct contact in RTM or VARTM

For resin transfer molding (RTM) or vacuum assisted resin transfer molding (VARTM), a sensor with a specially shaped surface can take advantage of the lateral forces in the infusion process. Figure 37-7 illustrates such a sensor, which has a shallow cavity on its face.

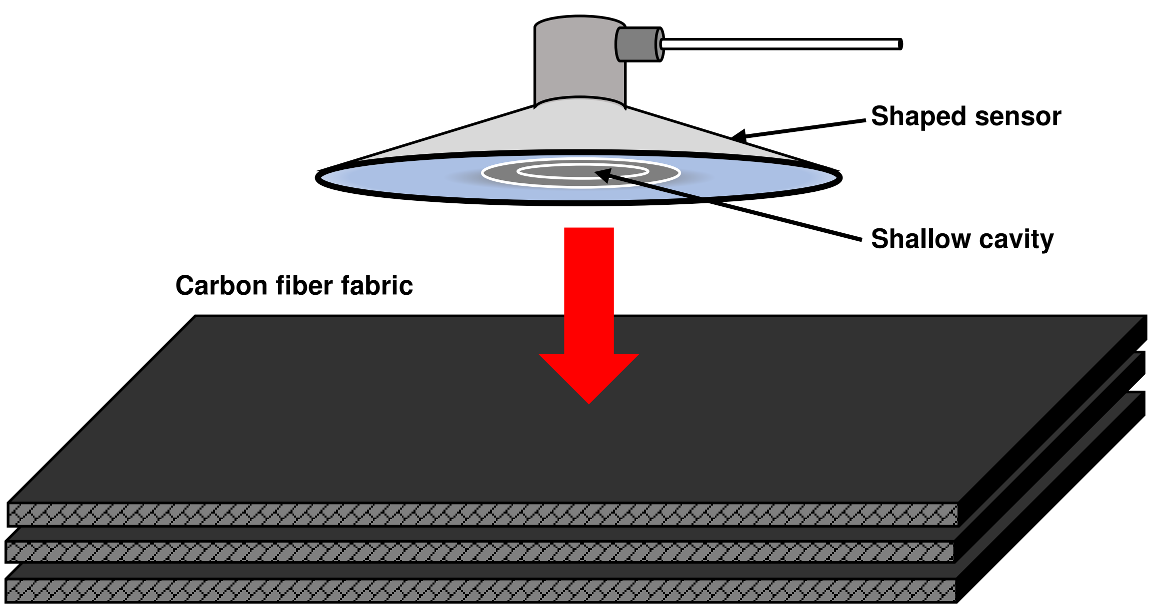

Figure 37-7
Shaped sensor for direct contact in RTM or VARTM applications

As shown in the cross-section of Figure 37-8, the shaped sensor is placed on the carbon fiber fabric and must protrude through a hole in the vacuum bag, which is sealed around the sensor with a gasket. The sensing electrode, located on the floor of the cavity, does not contact the fabric during infusion, when resin flows along the layers of material into the cavity. Because the forces are mainly parallel to the sensor, the fabric is stiff enough to prevent deformation of carbon fibers against the electrode, enabling cure monitoring without a short circuit.

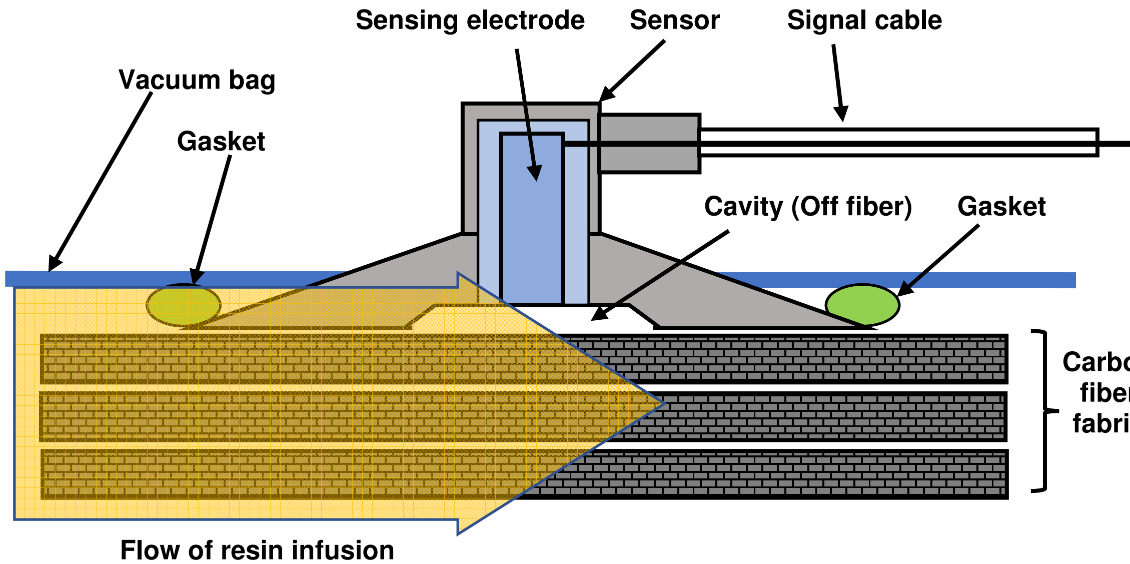

Figure 37-8
Cross-section of shaped sensor in vacuum bag during RTM or VARTM resin infusion

While this shaped sensor is useful for RTM or VARTM, it is not suitable for compression molding, where the forces are perpendicular to the sensor and can push carbon fibers against the electrode.

Coated sensor for direct contact with carbon fiber composites

Deposition of a non-conductive layer on electrodes, to prevent short circuits by carbon fibers, was reported by McIlagger[4] in 2000, and this technique has been used by others since then. Similarly, the reusable Carbon+Unitrode[5] sensor of Figures 37-9 and 37-10, has a rugged, insulating coating that allows direct contact with carbon fiber composites in applications like compression molding, as well as RTM and VARTM.

Figure 37-9
Carbon+Unitrode coated, reusable sensor

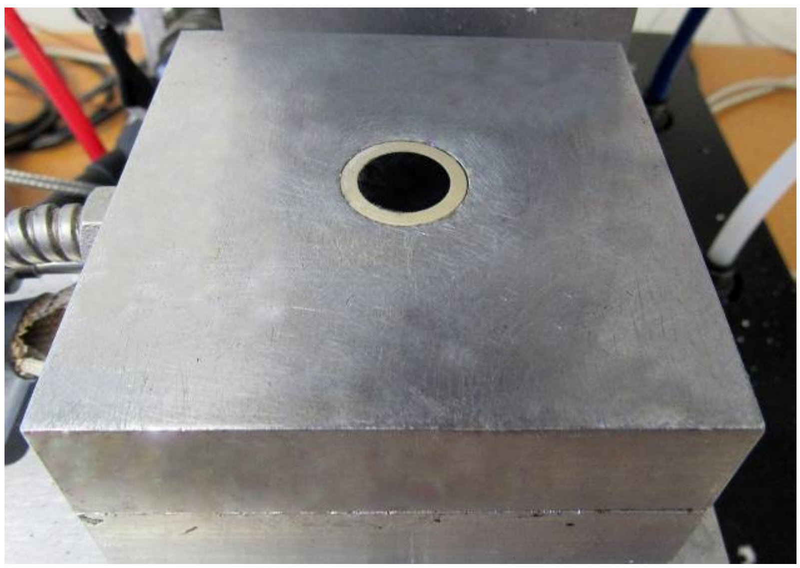

Figure 37-10
Carbon+Unitrode coated, reusable sensor in press platen

Figure 37-11 depicts a lay-up with the Carbon+Unitrode and a composite placed directly on it. The insulating coating introduces a capacitor, or blocking layer, between the electrode and the material under test. Because capacitors pass only AC signals, cure monitoring with coated sensors is not possible with DC methods.

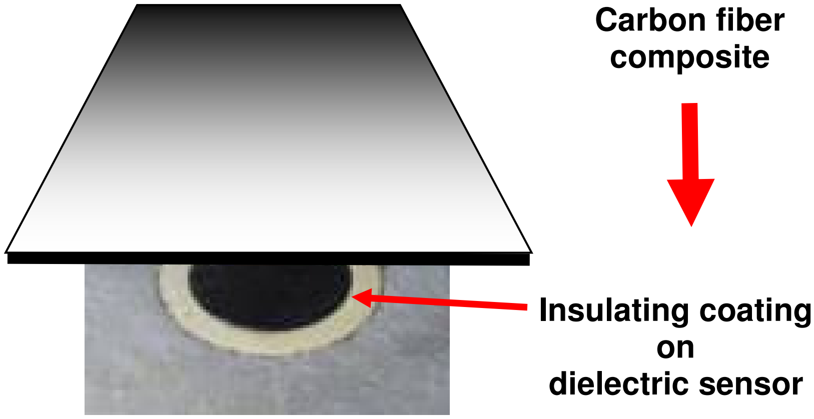

Figure 37-11
Typical lay-up with a Carbon+Unitrode sensor

Figure 37-12 plots log(*IV*) and slope during the two-stage cure of a carbon fiber reinforced prepreg on a Carbon+Unitrode, and compares the data with results from a similar, but uncoated, filtered sensor. Except for a minor difference in level, the ion viscosity curves are essentially the same, indicating the coated Carbon+Unitrode can measure the cure of CFRP and other carbon fiber composites as well as the filtered sensor.

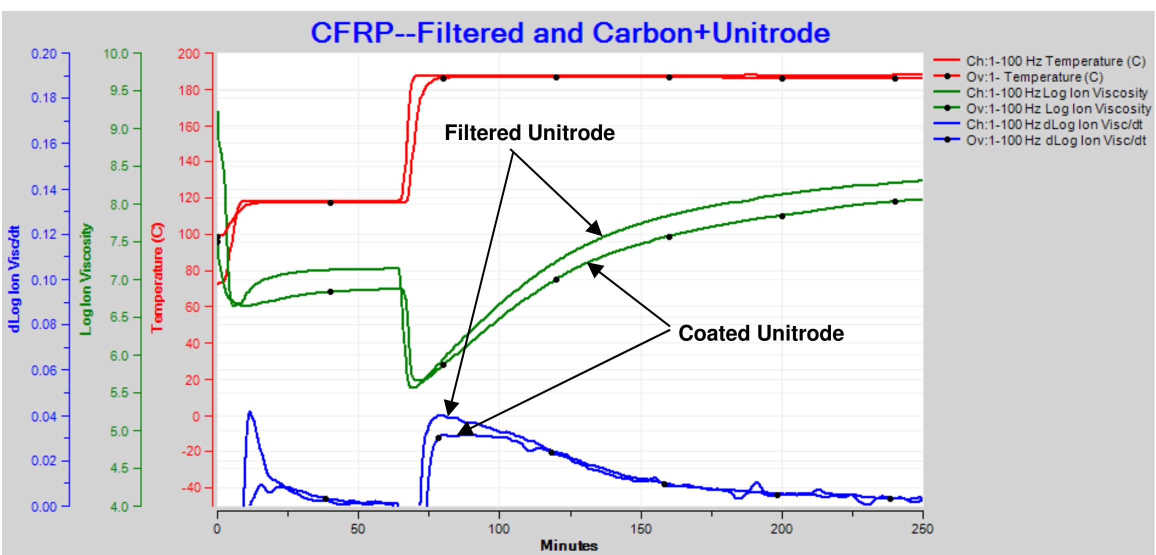

Figure 37-12
Comparison of CFRP ion viscosity (green) and slope (blue) from filtered and coated Unitrode sensors

For both tests, a heated press applied pressure to the CFRP 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 flows and its ion viscosity quickly decreases, indicating a corresponding decrease in mechanical viscosity. For a moment the material is at minimum ion viscosity, until the curing reaction becomes dominant—in this case at about 70 minutes—and then ionic and mechanical viscosity both increase.

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 toward the end of cure, which is a user defined slope that depends on the requirements of the application. To reduce cycle times and increase throughput, determining end of cure is one of the primary goals in manufacturing. Coated sensors like the Carbon+Unitrode, by allowing direct contact with carbon fiber composites, can measure material state without a filter or the extra handling a filter requires. Once end of cure is identified, dielectric cure monitoring equipment can issue a signal to automatically open the press or mold.

Conclusion

A number of sensors are suitable for cure monitoring of carbon fiber composites, but they must be either filtered or coated to prevent conductive fibers from short circuiting the electrodes. For resin transfer molding or vacuum-assisted resin transfer molding, a specialized, shaped sensor will also work, but its application beyond RTM and VARTM is limited. Once installed in a mold or platen, coated sensors are especially useful in general applications, including compression molding as well as RTM and VARTM. To aid in selection, Table 37-1 summarizes the characteristics of these various sensors.

Table 37-1
Sensors for cure monitoring of carbon fiber composites

Sensor Type	AC	DC	General use	For fast, repetitive operations	For manufacturing	Notes
Filtered disposable	•	•	•	**X**	**X**	May also be placed between the plies of a laminate
Filtered reusable	•	•	•	**X**	**X**	Useful for slower, repetitive QA/QC testing
Shaped reusable	•	•	**X**	**X**	See note	Specialized for RTM or VARTM; not suitable for compression molding
Coated reusable	•	**X**	•	•	•	Useful for QA/QC testing, RTM or VARTM and compression molding

References:

1. LT-440 Dielectric Channel, manufactured by Lambient Technologies, Cambridge, MA USA. https://lambient.com

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

3. 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.*

4. 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

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

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