Insight — Technical Overview 3.03

Equipment for Dielectric Cure Monitoring

Dielectric cure monitors

Dielectric cure monitors measure the dielectric properties of thermoset or composite. The use of a range of frequencies selectively reveals the response of ion motion and dipole rotation, allowing the study of material state through the entire cure.

Dielectric/conductivity sensors

Dielectric cure monitors measure the resistance (*R*) and capacitance (*C*) of material between a pair of electrodes, which can be modeled as a resistance in parallel with a capacitance, as shown in Figure 3-1.

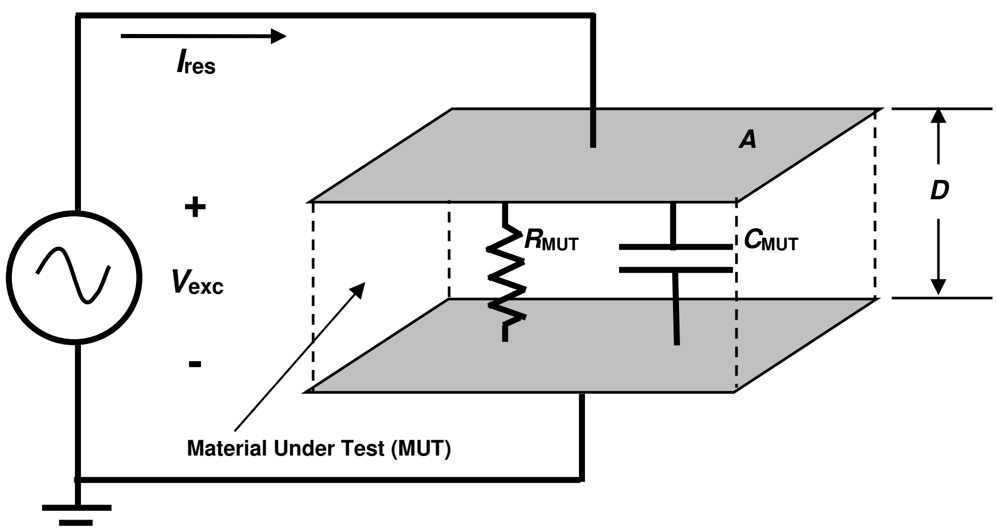


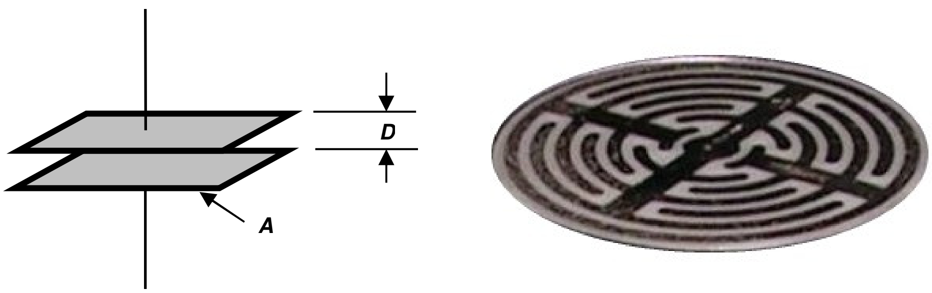
Figure 3-1
Dielectric model of a Material Under Test

Simple parallel plate electrodes, shown in Figure 3-2, are often used for this purpose. The ratio of electrode area *A* and the distance *D* between them—the *A*/*D* ratio—is a figure of merit. A larger *A*/*D* ratio corresponds to greater sensor sensitivity. The *A*/*D* ratio is also the scaling factor for calculating resistivity (ρ) from resistance, and permittivity (ε') from capacitance. Unfortunately, distance

D can change with pressure, or with expansion and contraction of the material, causing inaccurate results.

An alternative is the interdigitated electrode, also shown in Figure 3-2. A rigid substrate supports the electrodes and resulting the planar structure does not change with pressure, or expansion and contraction of the material under test (MUT). While the parallel plate sensor makes a bulk measurement, an interdigitated sensor makes a surface measurement.

As a rule of thumb, interdigitated electrodes with the same width and separation measure to a depth approximately equal to the electrode width. The parameter of *A*/*D* ratio also applies to interdigitated electrodes as a figure of merit and is the scaling factor for calculating resistivity and permittivity.



Parallel plates—
ρ = R • (*A*/*D*)

Interdigitated electrodes—
ρ = R • (*A*/*D*)

Figure 3-2
Comparison of parallel plate and interdigitated electrodes

Figure 3-3 shows a Varicon[1] disposable dielectric/conductivity sensor with interdigitated electrodes 100 microns wide. Constructed as a Kapton® flex circuit, this sensor is thin enough to be inserted between the plys of a laminate and may be discarded after use. The narrow electrodes, too small to be visible in the photograph, result in a large A/D = 160, with correspondingly great sensitivity. The trade-off is the measurement of dielectric properties only within 100 microns of the surface.

Figure 3-4 shows a Ceramicomb-1″[2] reusable dielectric/conductivity sensor embedded in a platen for a small press. This sensor is constructed with interdigitated electrodes embedded in ceramic and has *A*/*D* = 10. When mounted as shown, it is possible to place a sample in the press, then heat and compress it and simultaneously make dielectric measurements to monitor cure. Afterwards the sample can be removed from the sensor and the process can be repeated.

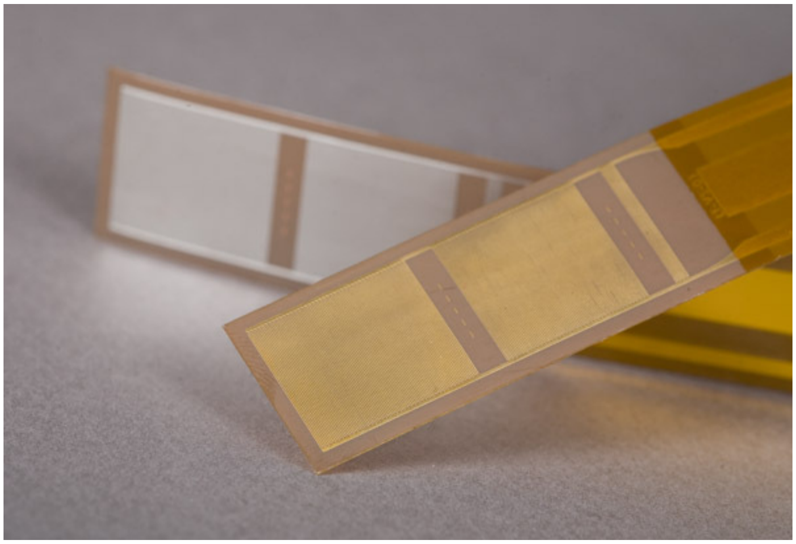


Figure 3-3
Disposable Varicon[1] dielectric/conductivity sensor on polyimide flex circuit

Reusable sensors are convenient for applications such as Quality Assurance/Quality Control (QA/QC), which involve repetitive testing. Note the wider electrodes, visible in Figure 3-4. This sensor is able to measure more deeply into the material, with the trade-off of decreased sensitivity because of the smaller *A*/*D* ratio.

Figure 3-4
Reusable Ceramicomb-1″[2] dielectric/conductivity sensor embedded in press platen

AC vs. DC measurements

Interestingly, it is often *not* useful to measure frequency independent resistivity (ρ_{DC}) using DC signals. A phenomenon called *electrode polarization* can create a blocking layer across sensor electrodes during early cure, when material is most conductive. This blocking layer acts like a capacitor and prevents the passage of DC current. In the presence of electrode polarization, dielectric data from very low excitation frequencies are distorted, as shown in Figures 3-5 and 3-6 for the cure of a "five-minute" epoxy.

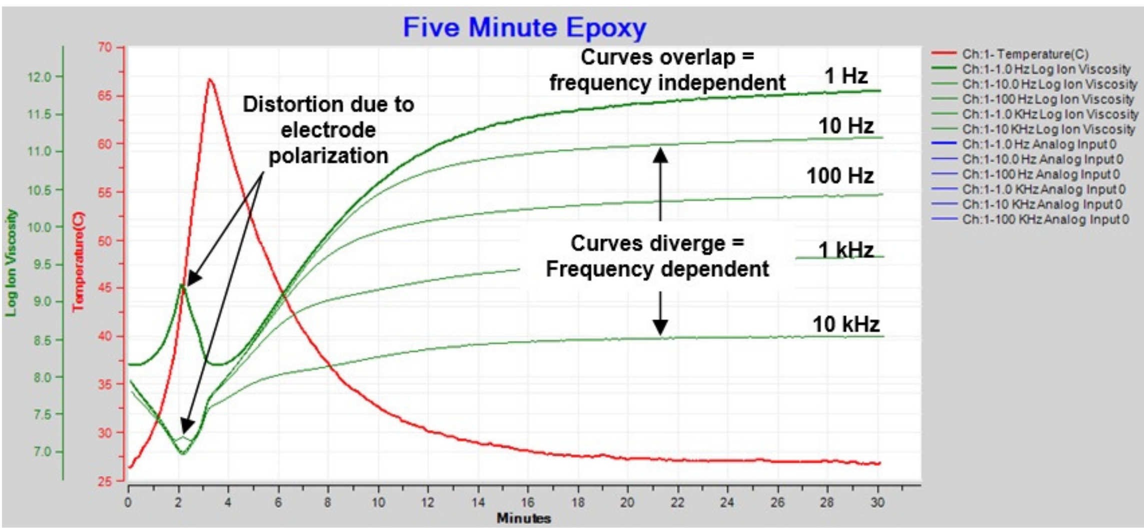


Figure 3-5
Distortion in ion viscosity from electrode polarization

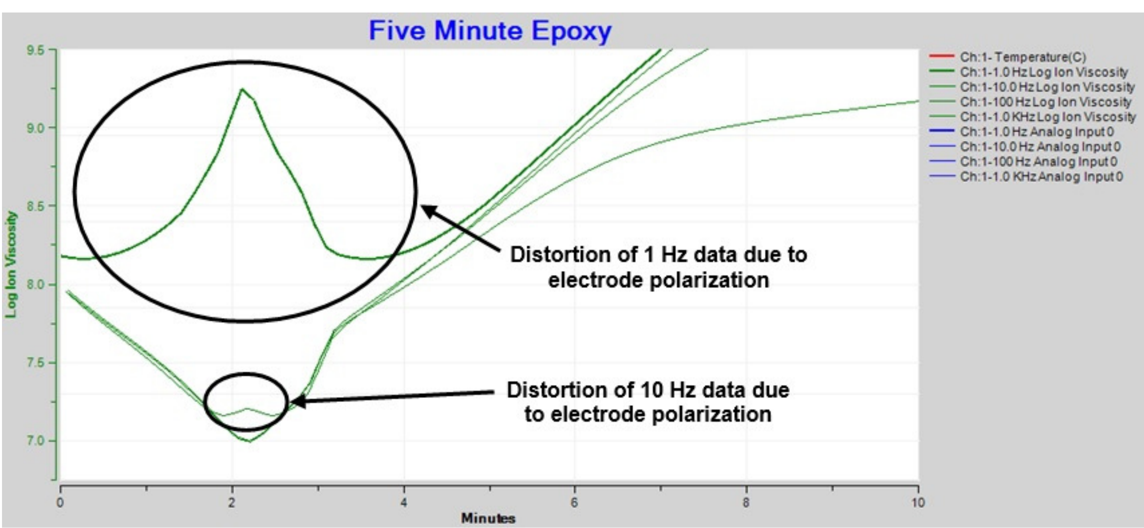


Figure 3-6
Distortion in ion viscosity from electrode polarization (detail)

Figures 3-5 and 3-6 show plots of resistivity against an axis labeled *ion viscosity*. For convenience, these data may collectively be called ion viscosity. At the beginning of cure, measurements at the higher excitation frequencies—1 kHz to 10 kHz—show no distortion and correctly identify the ion viscosity minimum.

Electrode polarization causes a small amount of distortion in the 10 Hz data, visible in the expanded plot of Figure 3-6. This distortion changes the expected single minimum in resistivity/ion viscosity to a peak with *two* local minima.

Data from 1 Hz measurements display much greater distortion because the boundary layer effect increases with decreasing frequency. This distortion becomes worse at lower frequencies, and with DC signals a conductive material can even appear *non-conductive*.

Besides avoiding distorted data from blocking layers, AC signals can also make measurements through a release film, which is a very thin layer of plastic used to prevent material from adhering to a mold or platen.

AC signals have significant advantages over DC signals. For dielectric cure monitoring, the optimum range of frequencies depends on the material and application. Extremely low frequency data require long acquisition times and are subject to distortion from electrode polarization. For most thermosets 0.1 Hz to 10 Hz is a reasonable lower limit. High frequency data tends to be dominated by dipolar rotation, which masks ion viscosity at the end of cure as shown in Figure 3-5. For most thermosets 10 kHz to 100 kHz is a good upper frequency limit for measurements of ion viscosity.

Temperature measurements

Temperature measurement, usually with a thermocouple, is an essential function for dielectric cure monitoring because ion viscosity depends on both cure state and temperature. At a given cure state, ion viscosity decreases as temperature increases and increases as temperature decreases. Knowing temperature provides additional insight into the nature of the cure and can avoid misinterpretation of data. Figure 3-7 shows the isothermal cure of an epoxy, in which the minimum ion viscosity occurs at time $t = 0$ and ion viscosity increases monotonically during cure.

In contrast, Figure 3-8 shows the cure of a “five-minute” epoxy, which produces an exotherm. Here temperature initially increases as curing begins, liberating heat and resulting in a decrease in ion viscosity.

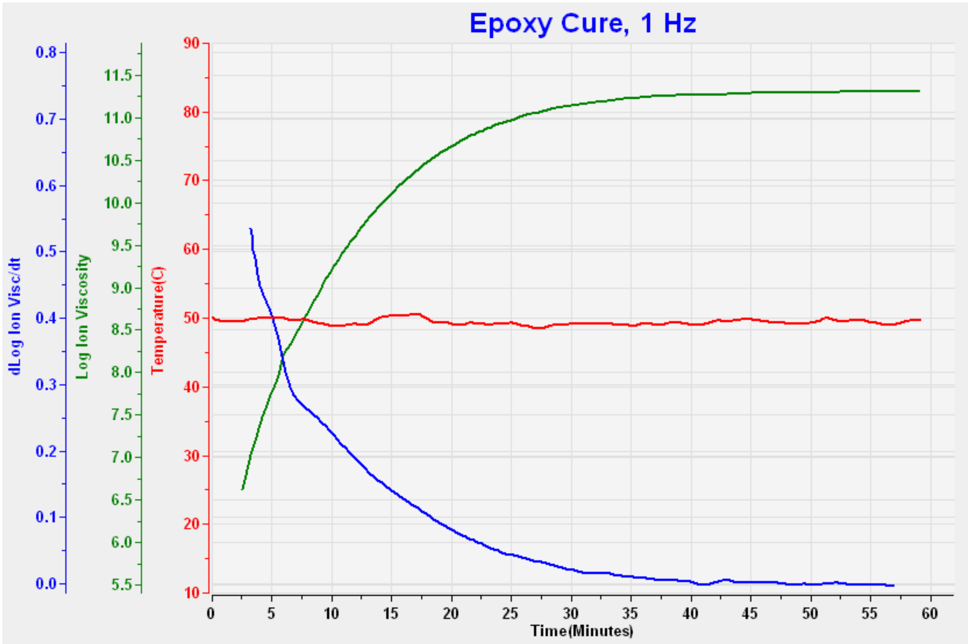


Figure 3-7
Isothermal epoxy cure at 50 °C

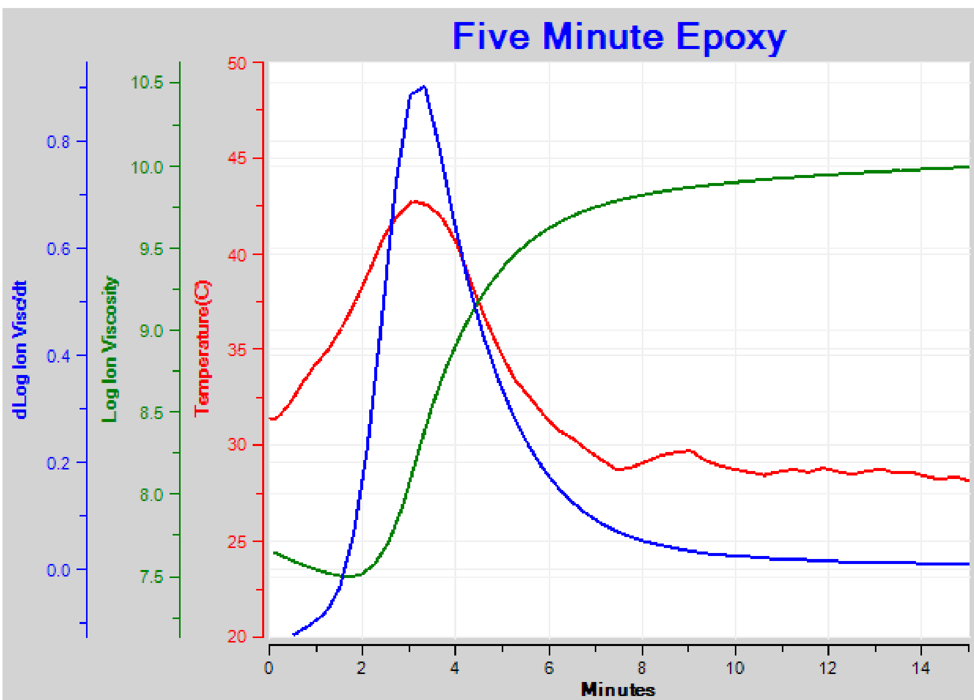


Figure 3-8
Five-minute epoxy cure with exotherm

Eventually the reaction dominates, ion viscosity goes through a minimum then increases. Notice also how the peak exotherm occurs at the same time as the maximum slope of ion viscosity—identifying the point of maximum reaction rate. Temperature data is valuable to understanding how a material cures, and is necessary when developing a process or formulation.

A dielectric cure monitoring system

Figure 3-9 shows the essential elements of a dielectric cure monitoring system. Making contact with the thermoset under test, dielectric/conductivity sensors have two general configurations, parallel plate or interdigitated electrodes. The selection of a sensor depends on the desired type of measurement, either surface or bulk, and the desired sensitivity as indicated by the *A*/*D* ratio.

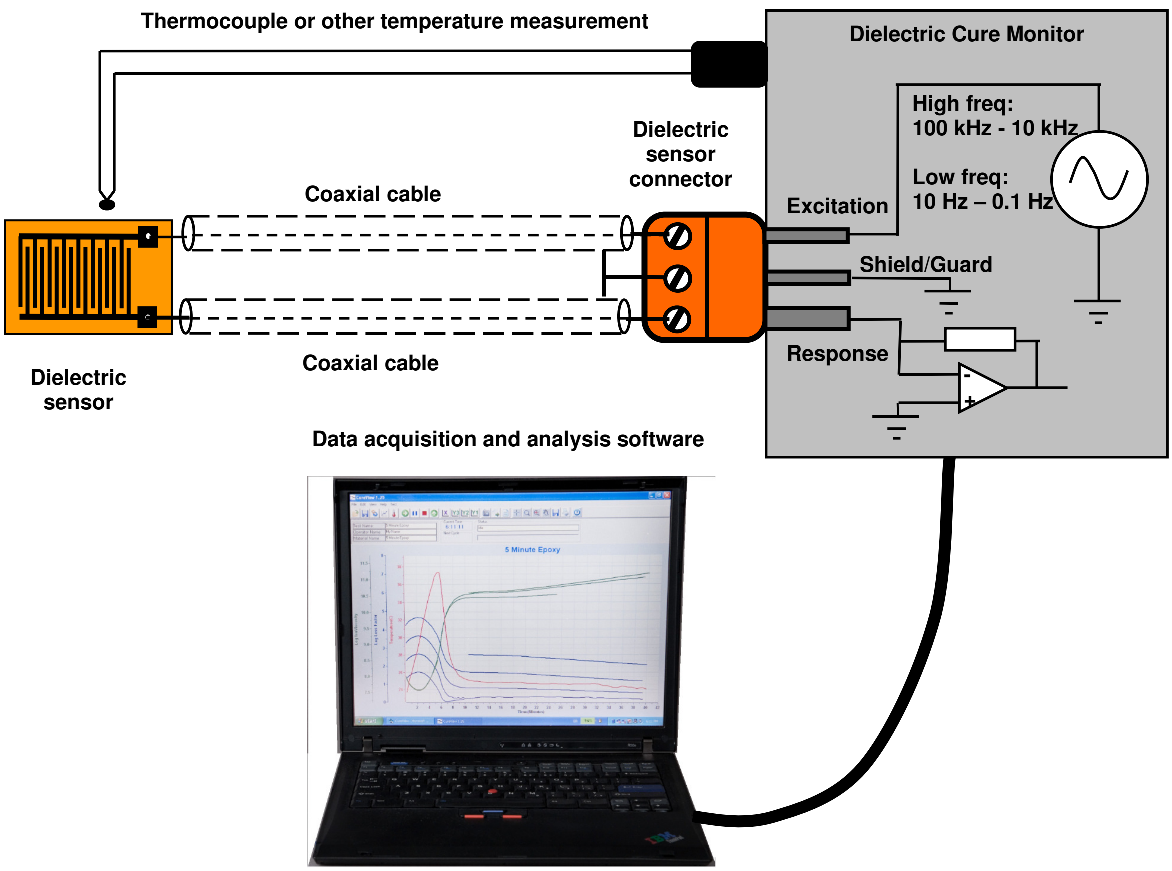


Figure 3-9
Essential elements of a dielectric cure monitoring system

Sensors connect to an instrument. The most versatile instruments use AC signals for measurement. A wide range of excitation frequencies allow selection of an optimal frequency for observing ion viscosity, and multiple frequencies enable studies of the dipolar response. Temperature measurement is important for understanding cure, especially under non-isothermal conditions.

Finally, software controls the instrument, usually through a computer connected to the dielectric cure monitor. Cure state cannot be determined from a single point measurement, but must be extracted from the change of ion viscosity and the shape of the curve over time. So software, which makes repetitive measurements, and stores and analyzes data, is crucial to the performance of the system.

References:

1. Varicon sensor, manufactured by Lambient Technologies, Cambridge, MA USA. https://lambient.com

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

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