Insight — Application Note 2.25

Cure Monitoring of Near-UV Cured Gel Nail Polish

Introduction

Lambient Technologies tested the base gel for a nail polish system that requires near-ultraviolet light to cure. Normal use of gel nail polish consists of application of a base gel, color gel then top gel to the fingernail. As recommended by the manufacturer, each coat requires exposure to blue-violet LED illumination for 45 seconds to cure before application of the next layer. The base gel was brushed onto Varicon sensors and tested with three lamps: "Normal," "Wave" and "Gradual" to determine whether the cure times change under different illumination schedules.

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*.

Electrical conductivity (σ) has both frequency independent (σ_{DC}) and frequency dependent (σ_{AC}) components. In an oscillating electric field, σ_{DC} 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 are added together as expressed below:

(eq. 25.1) $$\sigma = \sigma_{DC} + \sigma_{AC} \qquad (\text{ohm}^{-1} - \text{cm}^{-1})$$

Resistivity (ρ) is the inverse of conductivity and is defined as:

(eq. 25.2) $$\rho = 1/\sigma \qquad (\text{ohm-cm})$$

From its relationship to conductivity, resistivity also has both frequency independent (ρ_{DC}) and frequency dependent (ρ_{AC}) components. Crosslink density, which is a measure of cure state, affects both mechanical viscosity and the movement of ions, and therefore influences ρ_{DC}. As a result, the term *Ion Viscosity* was coined to emphasize the relationship between mechanical viscosity and ρ_{DC}. Ion viscosity (*IV*) is defined as:

(eq. 25.3) $$IV = \rho_{DC} \qquad \text{(ohm-cm)}$$

Procedure

A thin layer of base gel was applied to the Varicon sensor with a nail polish brush. Then the sensor was inserted beneath the selected lamp and data acquisition began. An LTF-631 High Speed Dielectric Cure Monitor measured cure with a 1 kHz excitation at a rate of 0.1 sec/data point. The LTF-631 has a maximum measurement speed of about 50 ms/data point and was designed for rapidly reacting materials such as UV cured adhesives and thermosets with fast gelation times.

To observe whether cure occurs before light exposure, the lamp was turned on approximately 30 seconds after starting data acquisition. Although the manufacturer's recommended exposure time was 45 seconds, data acquisition continued with continuous near-UV exposure for several minutes. Lambient Technology's CureView software acquired and stored the data, and performed post-analysis and presentation of the results.

Results

Figures 25-1 a, b and c are plots of temperature, log(*IV*) and slope for cure of the base coat with the different lamps. Because the log(*IV*) trace is flat and does not change before the lamps turn on at 0.5 minutes, it is clear that no cure occurs during this time. Cure does start immediately after the lamps turn on, as indicated by the increase of log(*IV*) at 0.5 minutes. Cure ends when log(*IV*) becomes level a short time later.

The slope rises from zero at the start of cure to a peak at the point of maximum cure rate. Then slope decreases, returning to zero at the end of cure.

Temperature for all tests was 20 °C, approximately the temperature of human fingers.

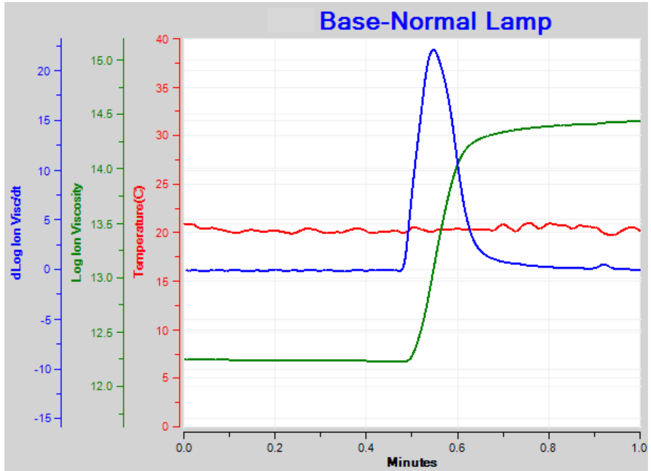

Figure 25-1 a

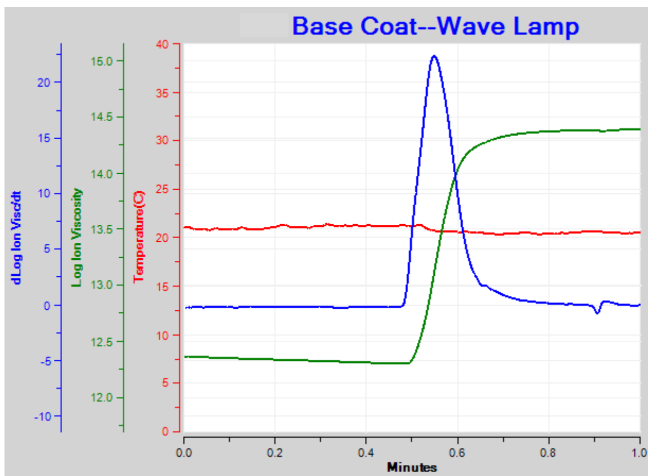

Figure 25-1 b

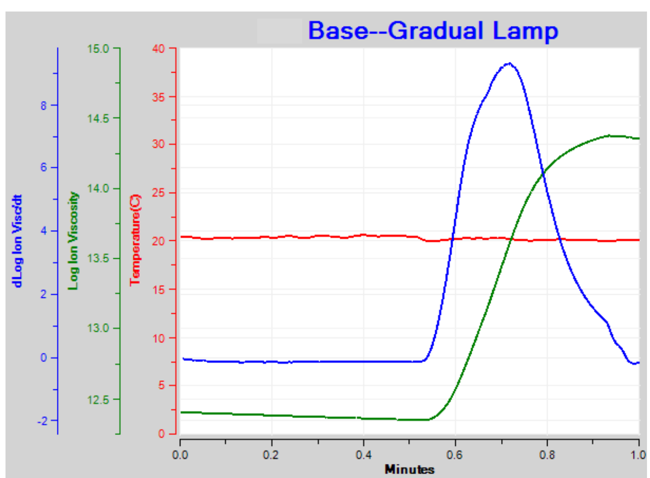

Figure 25-1 c

The cure curve can be characterized by four *Critical Points*, which identify specific events during cure. Critical Point 1 (CP(1)) indicates the onset of flow caused by softening of the material. The gel base coat does not soften and flow after cure begins, so Critical Point 1 is not relevant. Critical Point 2 (CP(2)) is the viscosity minimum, but the base gel viscosity is at its minimum at the beginning of cure and so does not yield useful information.

The point of maximum slope is called Critical Point 3 (CP(3)) and identifies when the curing reaction is fastest. The value of this slope indicates the relative speed of reaction. Both the "Normal" and "Wave" lamps produce a value of 25 for the maximum slope. In contrast, the "Gradual" lamp produces a value of only 9.5 for maximum slope, which is a measure of slower reaction when driven by this lamp.

End of cure occurs when the slope reaches a user selected level called Critical Point 4 (CP(4)). The value of CP(4) depends on the requirements of the application, and for this report is defined as a slope of 0.1. Total cure time is the difference between time to end of cure and time of start of cure. The "Normal" lamp cures the base coat in 24.6 seconds. The "Wave" lamp cures the base coat in 19.8 seconds. The "Gradual" lamp produces the longest cure time of 38.4 seconds, which is a result of the slower reaction, as indicated by the lower value of CP(3) for this case. Table 25-1 summarizes the Critical Points for cure of the base gel under the different lamps.

Table 25-1
Critical Points from base gel cure monitoring

Lamp	Lamp turn-on time		CP(2) Min. Visc.		CP(3) Max Slope		CP(4) Crit. Slope	
		Start of cure	Value	Time after start	Value	Time after start	Value	Time after start
Normal	---	28.8 s	12.25	28.8 s	25.0	8.0 s	0.1	24.6 s
Wave	---	29.4 s	12.25	29.4 s	25.0	8.0 s	0.1	19.8 s
Gradual	---	31.2 s	12.35	31.2 s	9.5	10.8 s	0.1	38.4 s

Characteristics of a thermoset Cure

Ion viscosity is defined as the frequency independent resistivity, ρ_{DC}. In many cases ion viscosity is proportional to mechanical viscosity during the early portion of cure, and indicates cure state in the latter portion of cure.

Ion viscosity derived from data at a single frequency produces a curve that characterizes the progress of cure. In simplified form, Figures 25-2 and 25-3 show the behavior of a typical thermoset with one temperature ramp step and one temperature hold step.

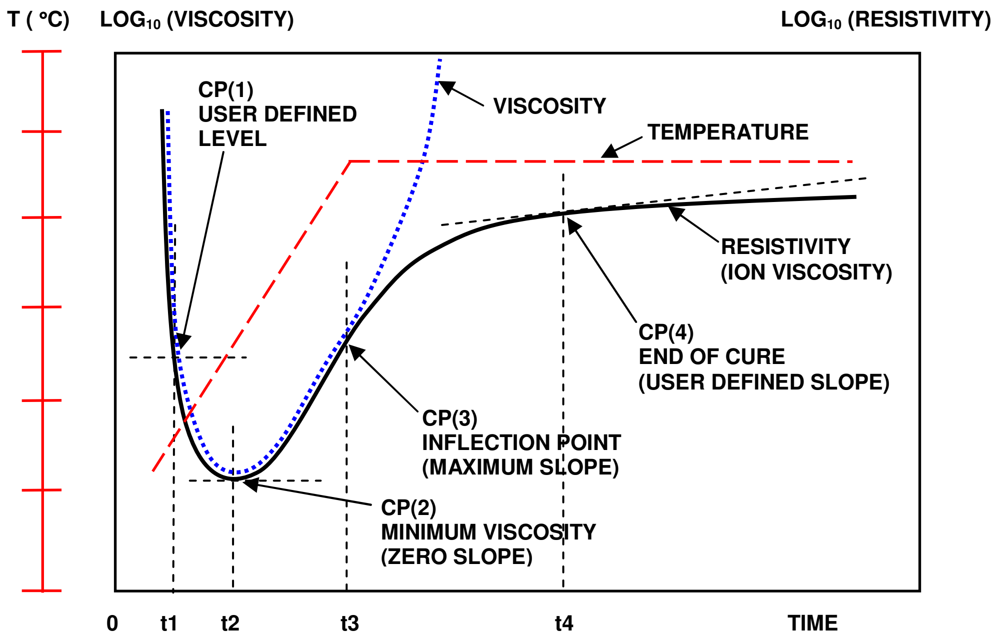

Figure 25-2
Typical ion viscosity behavior of a curing thermoset

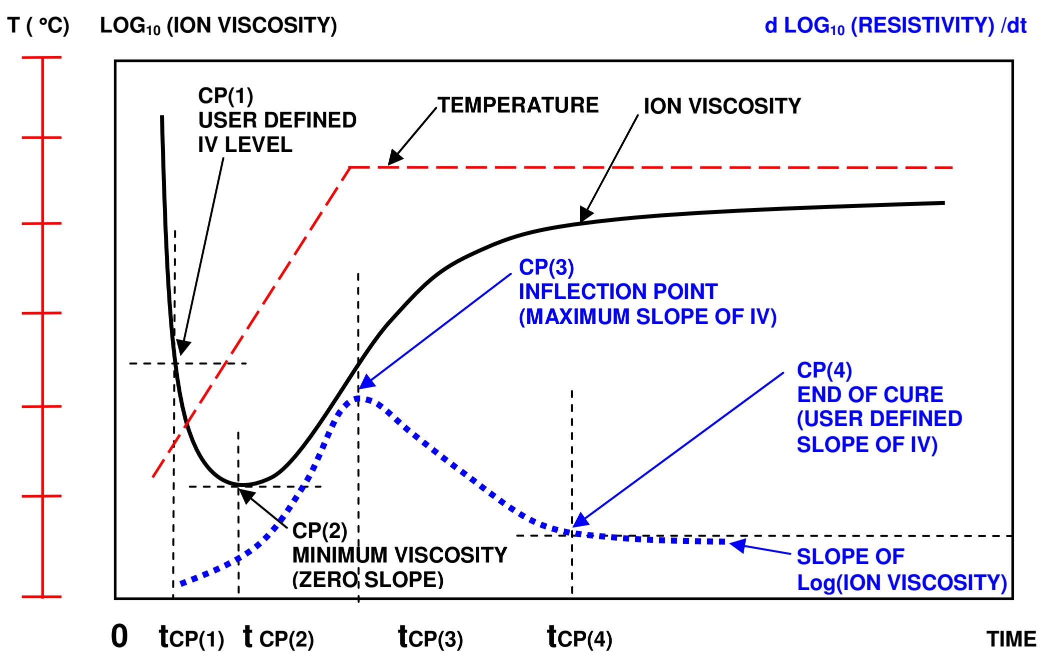

Figure 25-3
Ion viscosity curve and slope of ion viscosity of a curing thermoset

At first, as temperature increases, ion viscosity decreases because the thermoset is melting, becoming more fluid and therefore less resistive. The reaction rate increases as the material becomes hotter. At some time the increase in ion viscosity due to crosslinking overcomes the decrease in ion viscosity due to increasing temperature. This point is the ion viscosity minimum, which also occurs at the time of minimum mechanical viscosity.

After the minimum point, ion viscosity increases continuously until the concentration of unreacted monomers diminishes and the reaction rate decreases; consequently the slope of ion viscosity also decreases and eventually ion viscosity will have zero slope when cure has stopped completely.

Four Critical Points characterize the dielectric cure curve:

- CP(1)—A user defined level of ion viscosity that is typically used to identify the onset of material flow at the beginning of cure.
- CP(2)—Ion viscosity minimum, which typically also corresponds to the physical viscosity minimum. This Critical Point indicates the time when the crosslinking reaction and resulting increasing viscosity begins to dominate the decreasing viscosity due to melting.
- CP(3)—Inflection point, which identifies the time when the crosslinking reaction begins to slow. CP(3) is often used as a signpost that can be associated with gelation.
- CP(4)—A user defined slope that can define the end of cure. The decreasing slope corresponds to the decreasing reaction rate. Note that dielectric cure monitoring continues to reveal changes in the evolving material past the point when mechanical measurement of viscosity is not possible.

Conclusion

Dielectric cure monitoring is effective for observing the entire cure of a gel polish, which requires near-UV to harden. The normal process involves subjecting the base coat to blue-violet LED illumination for 45 seconds. For the "Normal" and "Wave" lamps, however, only 25 seconds are required for full cure. For these lamps the illumination time can be reduced to 30 seconds—*a reduction of 15 seconds*, including some margin—ensuring full cure while at the same time minimizing the customer's exposure to near-UV radiation.

The "Gradual" lamp requires 39 seconds to cure, and in this case the 45 second exposure time is reasonable to ensure full cure.

The ability to observe gel polish cure in real time is valuable for quickly measuring the effects of different formulations and illumination intensities, as well as determining optimum lamp exposure time.

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