

SENSING WHEN THE MOLDING CYCLE IS OVER

THE KEY TO PRODUCTIVITY AND PRODUCT CONSISTENCY

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Ashland Performance Materials

Abstract

Breakthroughs in dielectric sensor design have resulted in the development of durable in-mold sensors that can operate on the production floor. Thermoset molders can now “see” changes in flow and cure inside their production tools, allowing automatic “real-time” adjustments for process variation and enabling significant gains in productivity and quality. A laboratory system has also been added to compliment production improvements.

Dielectric cure monitoring has been used in thermoset laboratories for decades to characterize materials. Historically, attempts to take the technology to the production floor where the benefits can be maximized in production tools have failed due to shortcomings in sensor durability and system reliability. Significant advances have been made to the basic impedance monitoring system including the addition of analysis software and a modified spiral mold tool that provides better correlation between the lab and production environments.

Production dielectric cure control uses in-mold sensors to measure the electrical impedance across the mold cavity during curing of the thermoset materials. The dielectric properties of thermosets vary dramatically during cure due to the changing ability of dipolar molecules to oscillate in the applied electrical field. An impedance “signature” is created for the material during the cure, which is correlated to adequate cure state by a computer control system.

Benefits to compression and injection molders include:

- 10-25% reductions in cycle time due to the elimination of safety factors built into the process to accommodate for process variation
- Improvements in quality because you are curing to a fixed cure state rather than a fixed cure time. Fixed cure states mean consistent finished part properties and the elimination of cure related scrap
- Better understanding of flow and cure rates inside the mold. This improved understanding will shorten development times, aid continuous improvement initiatives and increase process control.

Background

Combining advances in dielectric cure monitoring with advanced computer programming resulted in SmartTrac impedance sensing technology. This new technology provides an important method for thermoset molders to improve cure process productivity and quality. Similar to dielectric cure monitoring, impedance technology uses the changing electrical properties of the thermoset as it cures to determine the optimum time to end the cure. This paper reviews the implementation of impedance sensing technology in SMC and phenolic and presents results from several production and lab applications.

The results described in this paper will show that SmartTrac technology:

- Is robust and repeatable in a production environment, with many years of operation on a high volume automotive SMC part.
- Can deliver cure time savings up to 38%.
- Automatically adjusts cure times to compensate for mold temperature fluctuations while simultaneously manufacturing consistent parts.
- Shows the impact of charge placement on cure rates across a mold when multiple sensors are installed.
- Identifies SMC flow anomalies and provides feedback to efficiently change process variables to improve the molding process.
- Identifies material variations and provides a mechanism to continually improve the SMC formulation.
- Provides repeatable data and strong correlation to the cure process for mineral filled phenolic.

To date, SMC molders using the SmartTrac impedance sensing system have realized a return on investment in less than one year based on cure time savings alone.

SmartTrac impedance sensing technology uses in-mold sensors to measure the electrical impedance across the mold cavity during cure. The sensors are designed for harsh production environments and are proven through thousands of cycles at high temperatures and pressures. A low level AC voltage is applied to the sensor, creating a capacitor field coupled to the opposite side of the mold cavity. The strength of the capacitor is driven by the dielectric properties of the material between the sensor and the other side of the mold. Figure 1 below is a schematic of the sensor installation in the mold. Figure 2 is a picture representative of the sensor design and physical layout. The white area on the sensor face is ceramic, which serves as a wear-resistant surface and isolates sensing elements from the material.

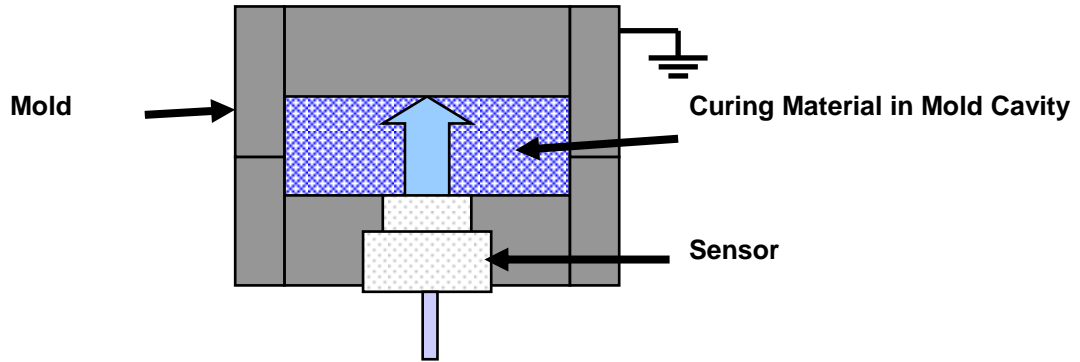


Figure 1: Capacitor formed by mold/sensor arrangement



Figure 2: Impedance sensor

The dielectric properties of SMC and other thermoset plastics vary during cure, due to the changing ability of dipolar molecules to oscillate in the applied electrical field. Ashland systems use this attribute of SMC and other dielectric materials to monitor and control curing in a production environment. An impedance “signature” is created for the material during the cure, which is correlated to the adequate cure state by the SmartTrac impedance sensing technology control system.

Figure 3 below shows a typical SMC (polyester, styrene monomers) impedance signature with time in seconds shown on the x-axis and the relative conductance shown on the y-axis.

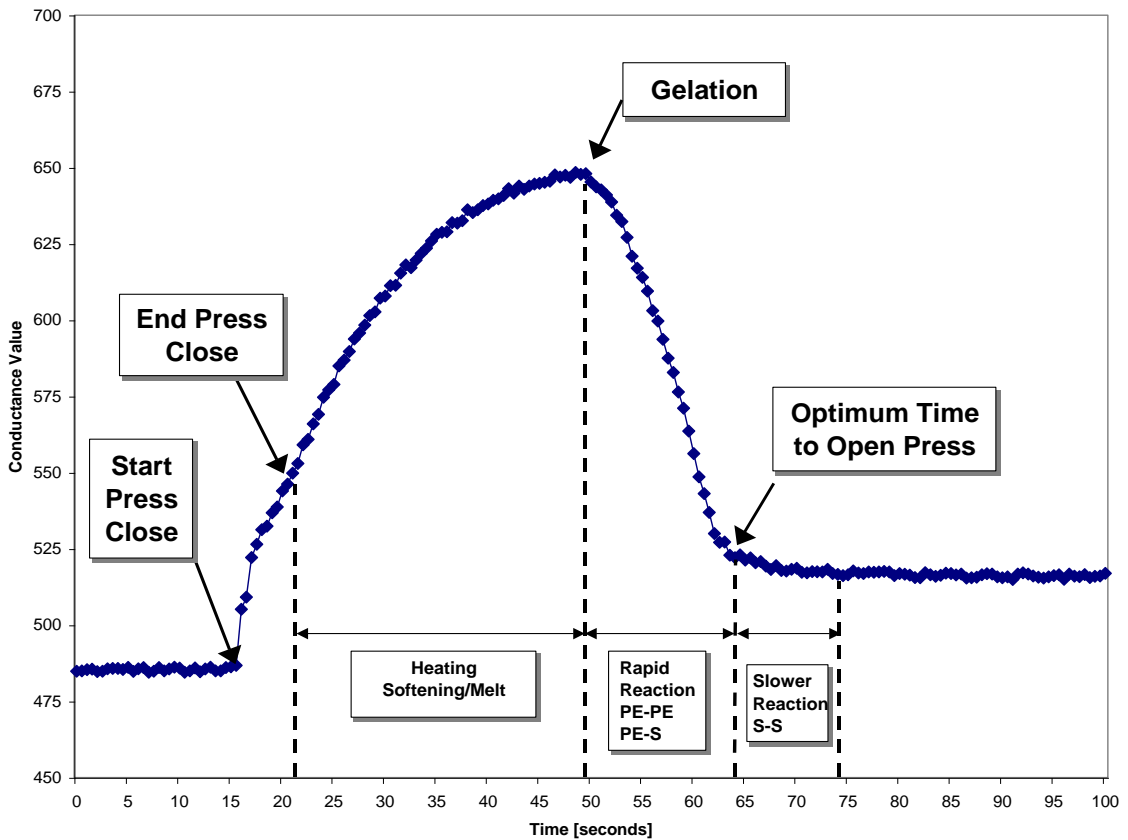


Figure 3: Typical SMC Impedance Signature

Figure 3 shows that the signature initially rises as the press closes, the SMC comes into contact with the sensor, and the sensor couples with the opposing ground plane. The signature continues to rise as the compound begins to soften and ionic and molecular entities are more capable of moving within the sensor’s electric field. The signature “peaks” as the compound reaches the point of gelation. After the peak, the impedance rapidly decays as the polyester and styrene react and cross-linking restricts the motion of ionic and molecular entities within the sensor’s electric field. The signature then “tails” to a flat-line condition as the remaining styrene-styrene reaction takes place.

SmartTrac impedance sensing technology uses a real-time algorithm called a rule base to adjust the cure time based on the impedance sensor's data signature. Referring to Figure 3 above, the rule base first identifies the impedance signature's peak, which correlates to the gelation zone. Once it identifies this point, the rule base then identifies a slope value near the transition to a flat. The proper slope to end the cure is determined empirically by measurement or observation of an applicable part property. Blistering before or after post-bake is often used for SMC parts to identify the point of adequate cure.

Case Study Results

Case 1: 18% Cure Time Reduction for SMC Instrument Panel

A SmartTrac impedance sensing technology was installed on a press running an SMC production part. The part is an instrument panel for a light truck. The part typically runs more than 700 cycles-per-day on three shifts, seven days a week. Figure 4 below shows typical impedance signatures for this part with times expressed as a percent of normal.

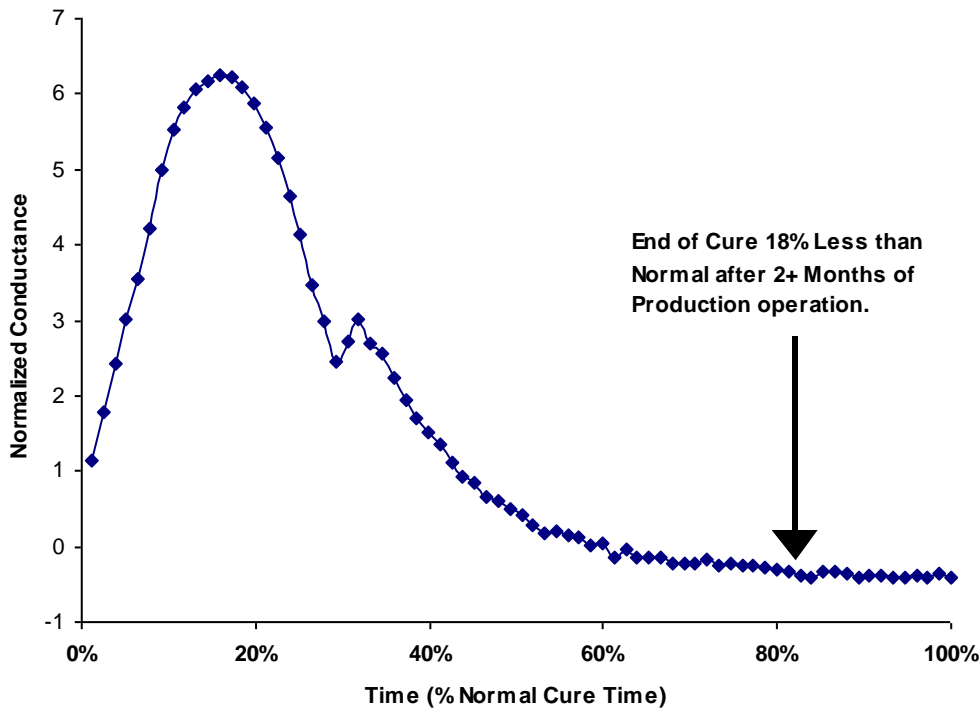


Figure 4: Impedance Signature – SMC Automotive Instrument Panel

The impedance signature in Figure 4 is slightly different from the signature in Figure 3 due to a change in the press clamp pressure about 30% of the way through the cycle. During SmartTrac impedance sensing technology startup on this application, the proper slope to end the cure was determined empirically by identifying the time when blisters started to occur. This setting was incorporated into the SmartTrac impedance sensing technology rule base to maximize production yield while simultaneously assuring adequate cure. Figure 4 shows the point used by the rule base to open the press. For those using the SmartTrac impedance sensing technology, this setting has reduced the cure time by 18% on average.

Cure time safety margins are a required and standard practice for plastics molders due to inherent variability in the process and material. Without real-time feedback from the mold cavity, the manufacturer must add safety margins to prevent increased scrap and production upsets. SmartTrac impedance sensing technology provides the feedback necessary to eliminate much of the cure time safety margins, allowing for increased productivity with no increased risk of under-cured parts.

Case 2-1: Temperature Variation and Cure Time Reduction for an SMC Body Panel

A production evaluation of SmartTrac impedance sensing technology was conducted at a major SMC automotive manufacturer. The evaluation was conducted on a production press running an automotive body panel made with polyester SMC (styrene monomers). In this application, the normal cure temperature was 300 degrees F for the lower mold and 310 degrees F for the upper mold. The normal cure time for this application was 105 seconds.

To determine the impact on impedance signatures caused by temperature variation, temperatures were intentionally changed ± 15 degrees F from nominal. Impedance data was collected for numerous cures at each temperature. Figure 5 below shows typical impedance signatures from cures at the nominal temperature, at nominal minus 15 degrees F, and at nominal plus 15 degrees F.

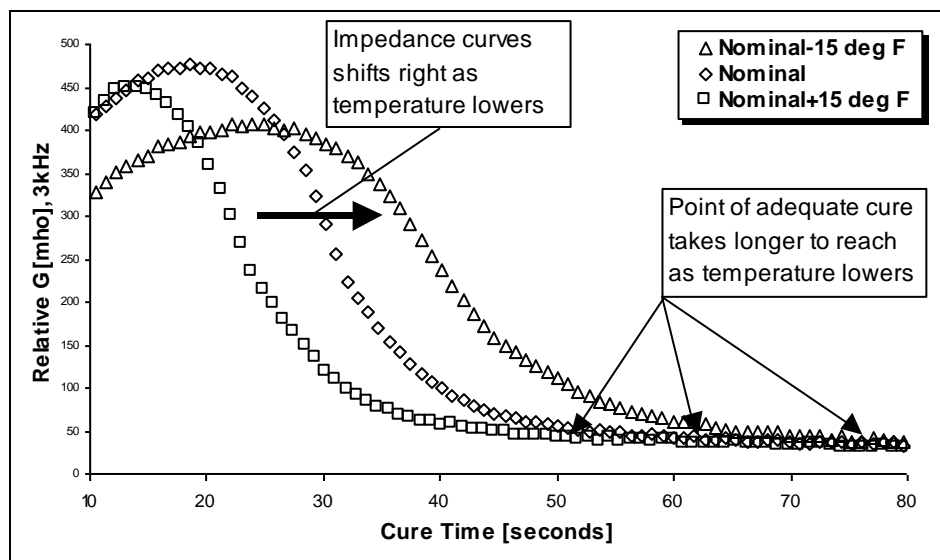


Figure 5: Impedance Data Automatically Detects Temperature Variation

Evaluation of Figure 5 shows that adequate cure under nominal conditions was reached at approximately 65 seconds. This represents a potential cure time reduction of 38%.

Figure 5 also shows that the impedance signature shifts to the right as the temperature is lowered. This shift reflects a slower melt and reaction rate as expected when the temperature is lowered.

In this application, the SmartTrac impedance sensing technology rule base automatically detected the expected cure time based on the changing mold temperature. Using this algorithm, SmartTrac impedance sensing technology determined that the time to reach adequate cure is 64 seconds at the nominal temperature. At nominal minus 15 degrees, adequate cure is reached in approximately 77 seconds. At nominal plus 15 degrees, adequate cure is reached in approximately 53 seconds.

SmartTrac impedance sensing technology's in-mold sensors showed the change in cure rate as the mold temperature changed, and automatically determined the impact of temperature variation on adequate cure time. This capability allows manufacturers using SmartTrac impedance sensing technology to reduce the safety margins most manufacturers build into cure times to account for temperature and other variations. SmartTrac impedance sensing technology's real-time feedback is also useful in establishing optimum temperature conditions and understanding the impact temperature settings have on reaction rates.

Case 2-2: Using Multiple Sensors to Detect Charge Pattern Effects on SMC Flow

Three sensors were installed in the body panel mold for a trial. Figure 6 below illustrates the relative locations of the sensors and charge pattern.

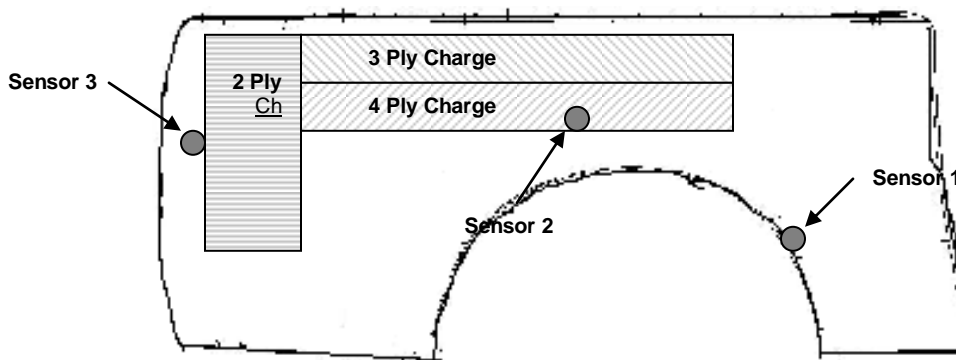


Figure 6: Relative Locations of Sensors and Charge Pattern

Numerous impedance signatures were collected using the standard charge placement. Figure 7 below shows typical impedance signatures using the normal pattern.

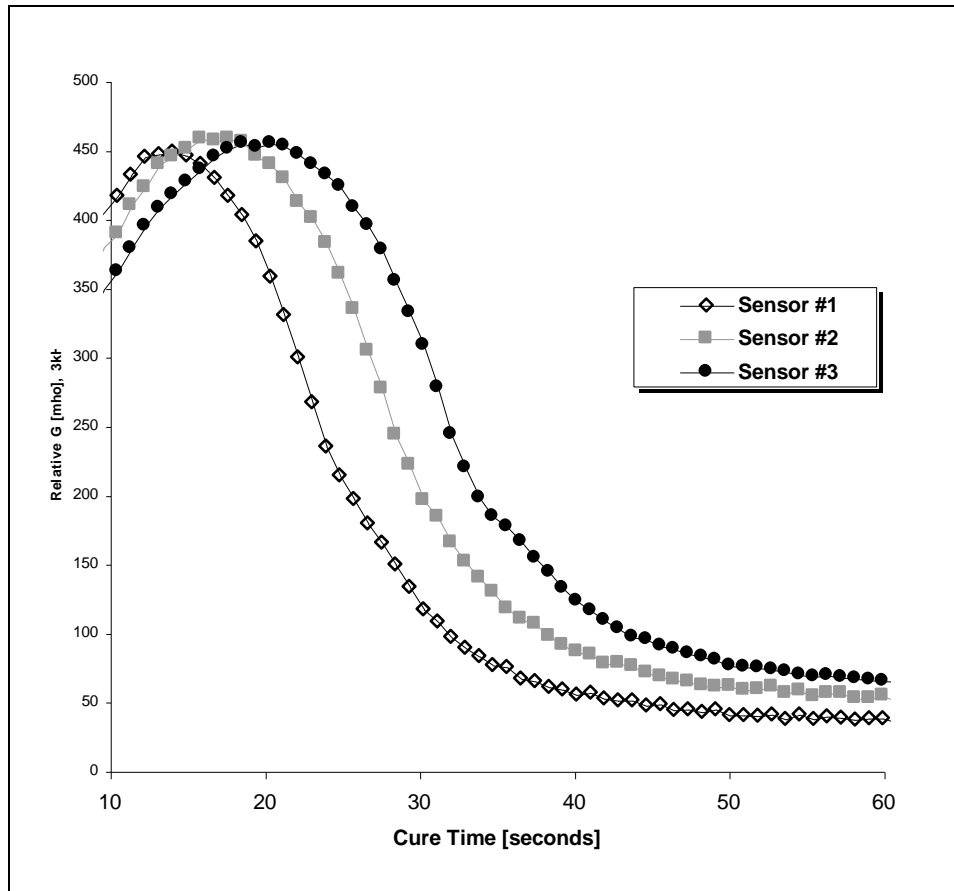


Figure 7: Impedance signatures show mismatch in cure rates

Evaluation of this impedance data indicated the SMC cured more rapidly near Sensor 1. This was confirmed during another portion of the test, when parts were cured at a temperature 15 degrees F above normal. At the elevated temperature, the mold was non-filled near Sensor 1 due to the SMC curing too rapidly. Based on this evidence, the charges were moved closer to Sensor 1 by approximately 6 inches, to see if this would equalize the cure rates of the SMC in the different mold locations.

Figure 8 below show typical impedance signatures from cures after the charge pattern was shifted closer to sensor 1.

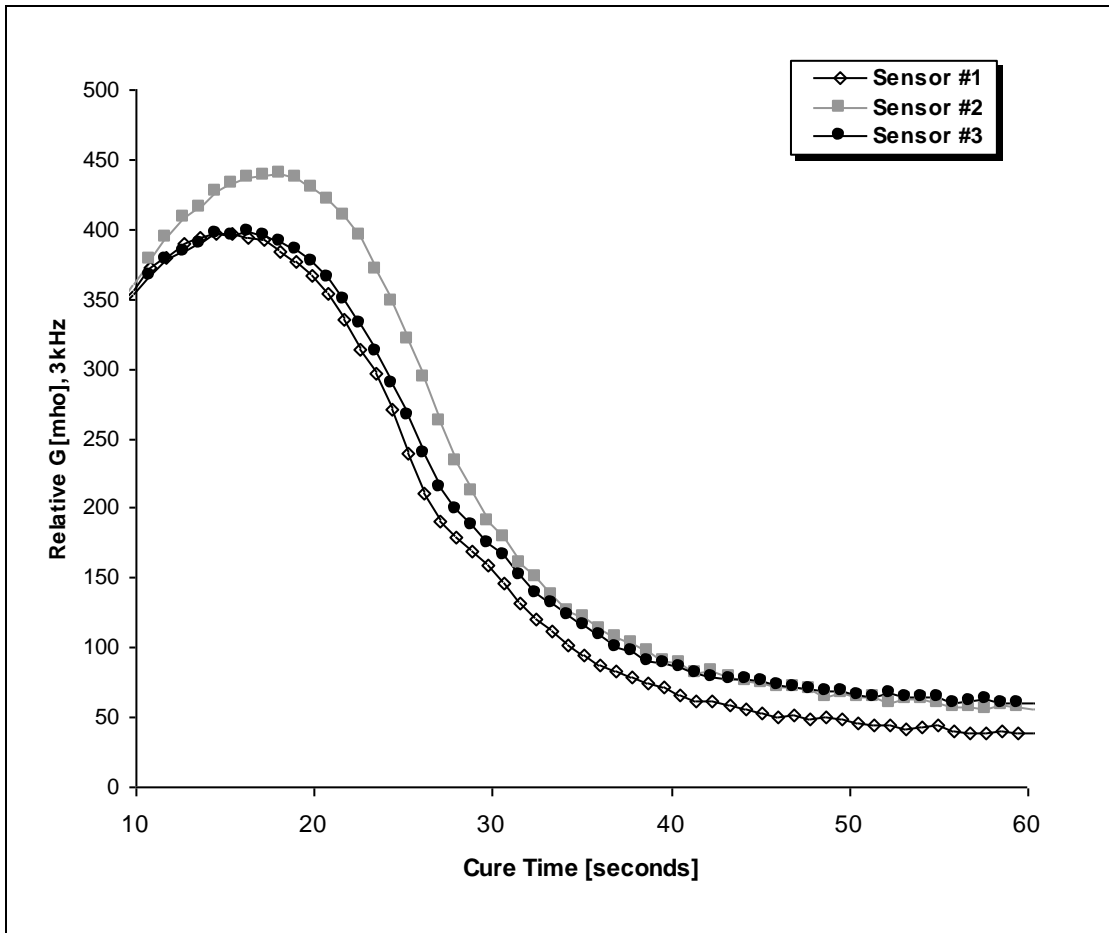


Figure 8: Impedance signatures matched after charge pattern shifted

The starting point and gel points of the curves are closer together for the three sensors. The impedance signatures are also aligned more closely near the flat, indicating the shift in the charge pattern normalized the cure rates at the three sensor locations.

By placing sensors in multiple locations, the SMC molder was able to understand the flow of SMC through the mold and its impact on cure rate at different mold locations. The charge placement was successfully modified to more closely match the cure rates in different sections of the mold using feedback from the impedance sensors.

Case 3-1: Detecting SMC Flow Anomalies in Production Molding

A production trial of SmartTrac technology was conducted on a large mold requiring a high quality surface finish. Figure 9 below shows impedance signatures from an initial cycle of this trial.

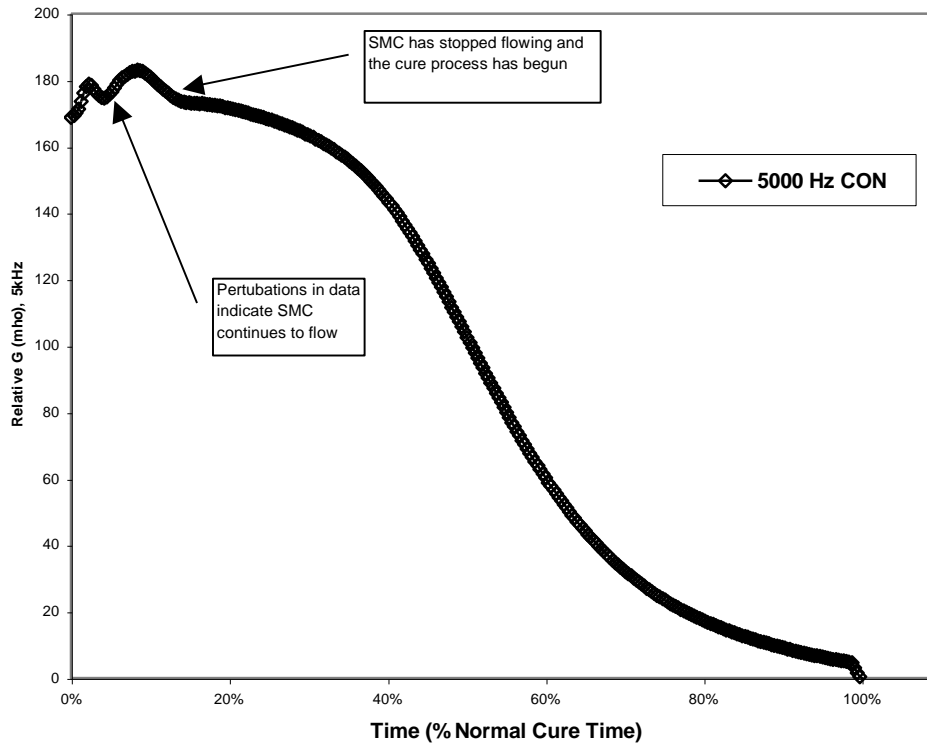


Figure 9: Impedance Signature Showing Poor SMC Flow

Referring to Figure 9, there are some irregularities seen within the first seconds of reaching full tonnage. The impedance data is erratic and indicates the material continues to flow after full tonnage has been reached. This observed behavior is unexpected since full tonnage normally indicates the SMC has stopped flowing and the gel period has begun. In addition, the data indicates that once the material stopped flowing, it immediately began cross-linking (evidenced by the impedance data falling). There is normally a period where the SMC is allowed to completely melt to ensure the mold is filled before cross-linking starts. It is evident the cure reaction has started before material flow is complete.

Based upon indications that the SMC was not flowing well before full tonnage was reached, the press tonnage was increased and the press closure rate was slowed. Figure 10 below illustrates the impedance signature collected after this process change.

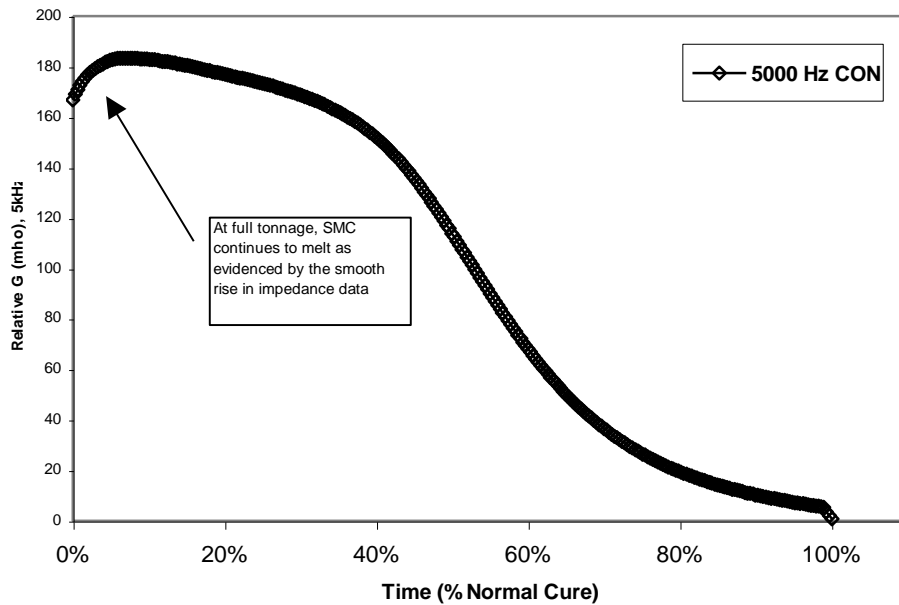


Figure 10: Impedance Signature with Improved SMC Flow

Examining Figure 10, it is clear the material is flowing better with more tonnage and a lower closure rate. The impedance data is rising and smooth at full tonnage, indicating material is no longer flowing, and has not reached the gel region.

In this production application, the impedance data provided a useful tool to understand the flow properties of the SMC in the production mold. This information was used to adjust the press settings for improved processing.

Case 3-2: Detecting SMC Compound Variation in Production Molding

During the January 2004 production trial described in the previous section, impedance data was collected while several formulations of SMC were molded. Two of these formulations are compared in this report and identified as Compound 1 and 2. Impedance data presented in the previous section of this report was from Compound 1.

Figure 11 below illustrates the impedance data collected while molding Compound 2.

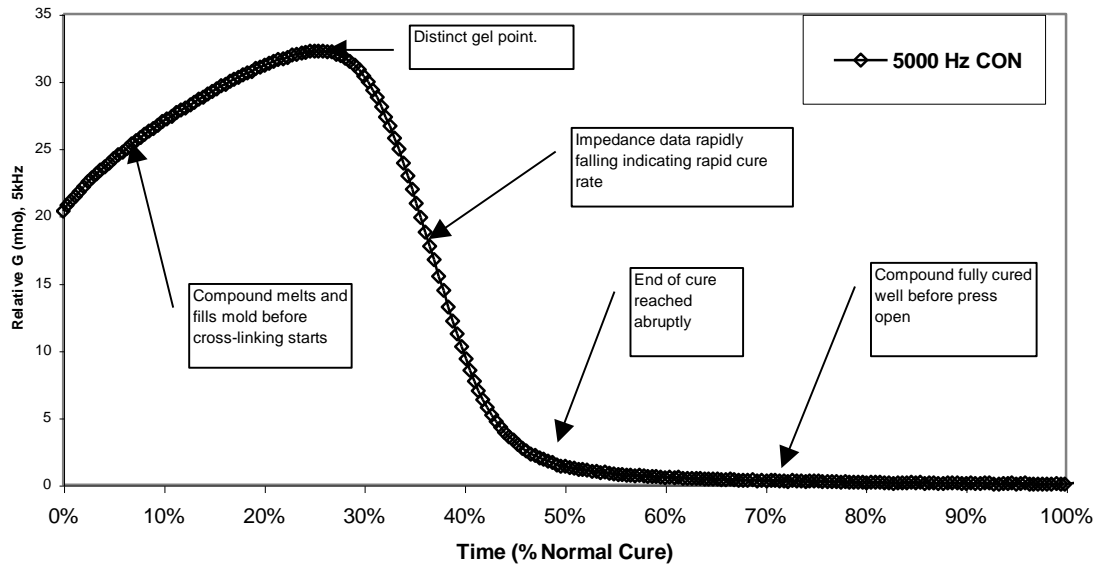


Figure 11: Compound 2 Impedance Signatures

Examining Compound 2's impedance signatures in Figure 11, the sensor indicates the material flowed well (no erratic jumps in the data) and melted approximately 25% into the normal cure time. The rapid drop in the impedance signatures after the peak indicates the material quickly transitioned from gel to a fully cured part. The part was completely cured after 70% of the normal cure time. A post inspection of the part revealed no defects.

It is useful to contrast the impedance data from Compound 1 and 2 to understand how each cures in a production environment. To accurately compare the two SMC materials it is important to minimize the effects of other process variables. To achieve that, impedance signatures were collected from each compound within 30 minutes of each other utilizing identical press settings (tonnage, closure rates, temperature settings, etc.). Figure 12 below contrasts Compound 1 and 2 impedance signatures.

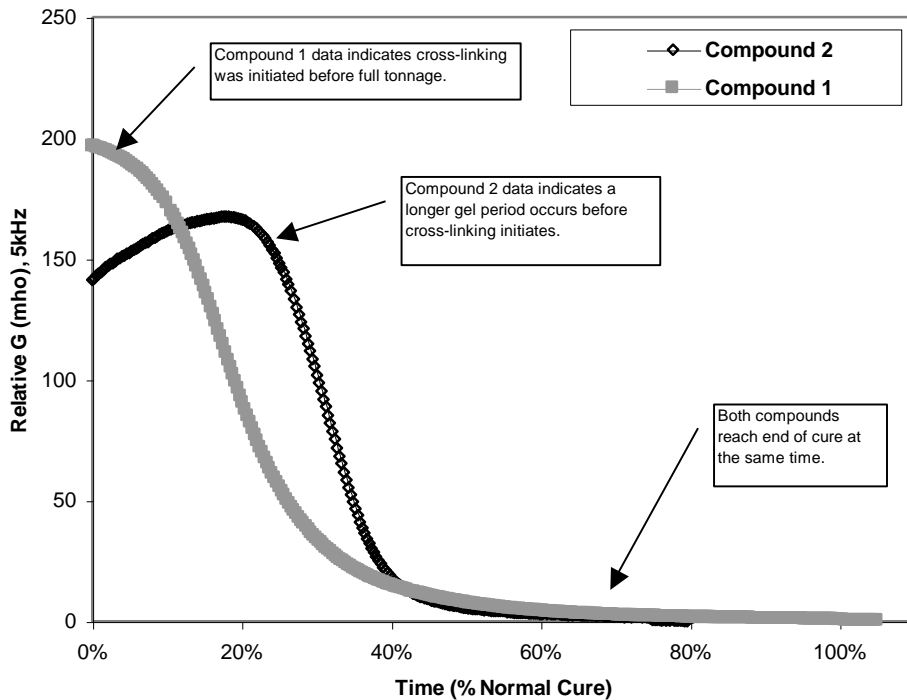


Figure 12: Comparison of Compound 1 and 2 Impedance Data

Figure 12 illustrates the different gel points of the two compounds. Compound 2 does not reach a gel point until well after full tonnage, while Compound 1 has already passed the gel point and is cross-linking at full tonnage. Ideally, the SMC should gel after full tonnage to allow ample time for the material to completely fill the mold. By not reaching a gel point until after full tonnage, the material tends to cure too quickly and not fill the mold.

Examining the cross-linking portion of both materials (the down slope of the impedance data), Compound 2 initiates and ends the cross-linking crisply. At the beginning of the cycle, Compound 1 slowly transitions to the cross-linking phase, while the end of the cycle shows the data slowly tapering to a plateau. The end of cure for both materials is at the same time.

Although both materials reach the end of cure at identical times, Compound 2 transitioned to a gel point smoothly (i.e. the material melted and was allowed to fill the mold completely) then rapidly initiated cross-linking. Compound 1 skipped the gel period and transitioned into the entry and exit of the cross-linking phase slowly. Without a clear gel period, it is doubtful the material would consistently fill the mold every cycle without creating non-fill failures. In fact, to create more consistent parts with Compound 1 (reduce the scrap rate associated with non-fills and under-cure) requires a lower mold temperature with an extended cure time. The lower temperature would delay the cross-linking to ensure the material gels and fills the mold. The cure time would be extended to ensure the material was fully cured at this lower mold temperature before de-molding.

SmartTrac impedance sensing technology's ability to measure different SMC formulations in a production setting provides several benefits to the SMC molder. During production startup, different SMC formulations can be evaluated to determine the best formulation for the mold design. The molder can also more thoroughly understand the effects process settings (mold temperature, close rate, press tonnage) have on the material flow and cure rates. In addition, the molder can use impedance signatures to identify the cause of material related processing problems during normal production.

Case 3-3: 32% Cure Time Reduction in Production Molding

During the last phase of a production trial, SmartTrac impedance sensing technology was setup to automatically detect the end of cure for Compound 2 and open the press at the optimum time. A post inspection of the 50 parts cured by SmartTrac technology verified there were no defects. Figure 13 illustrates the typical impedance data collected by SmartTrac technology during these cycles.

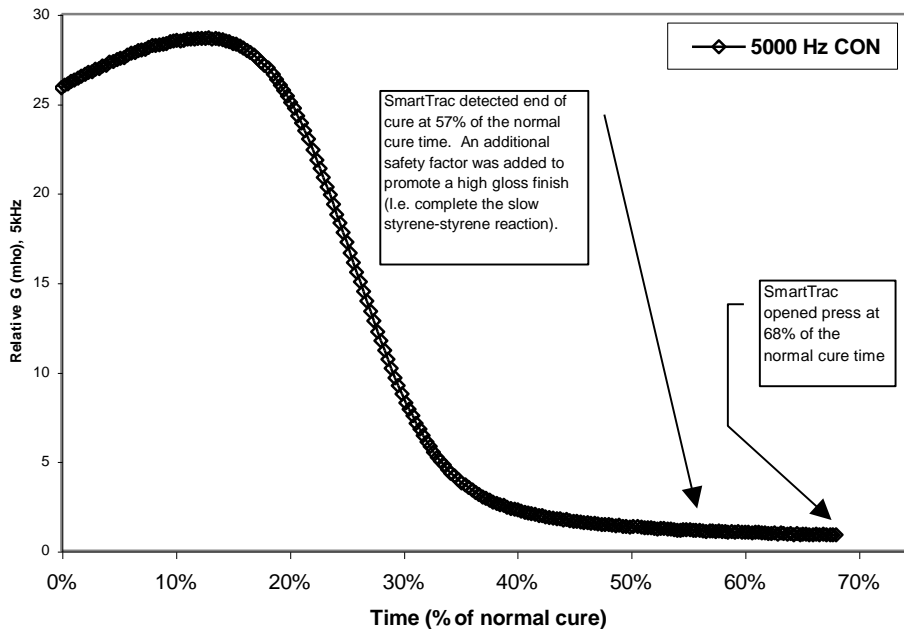


Figure 13: 32% Reduction in Cure Time

Figure 13 shows SmartTrac impedance sensing technology detected the end of cure at 57% of the normal cure time. An additional time factor was added to ensure the Styrene-Styrene reaction was complete and the part was de-molded with a high gloss finish. Even with this added safety factor, the average cure time reduction was 32%.

Case 4: Lab Trial on Mineral Filled Phenolic

A lab experiment utilizing SmartTrac technology was conducted on a brake piston mold curing a mineral filled phenolic resin. The parts were compression molded with an impedance sensor installed in the cavity side of the mold. The phenolic material was weighed, compressed, and pre-heated before placing the charge in the mold. The experiment was designed to vary the mold and pre-form temperature while collecting impedance data, to determine the correlation between impedance and rate of cure. Cure times were varied at each condition to fabricate both acceptably cured and unacceptably cured parts at each process condition. Producing acceptable and unacceptable parts at each condition was performed to correlate the impedance data to the state of cure of the finished part.

Figure 14 below shows a typical impedance curve from this experiment.

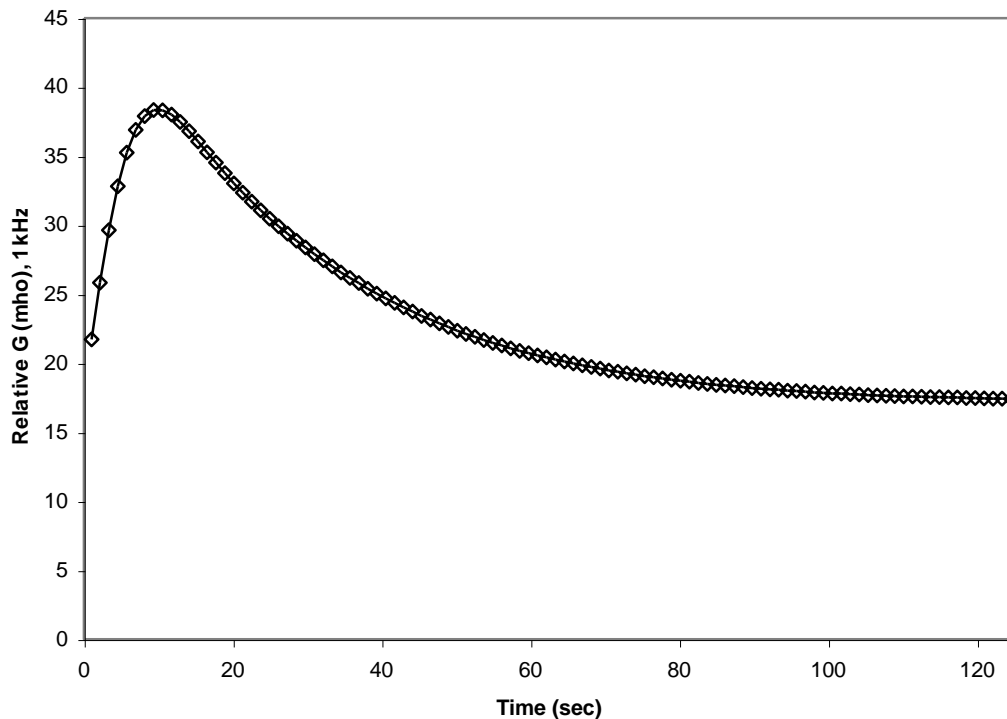


Figure 14: Typical Phenolic Impedance Signature

Figure 15 below shows the phenolic impedance data overlaid with typical SMC impedance data.

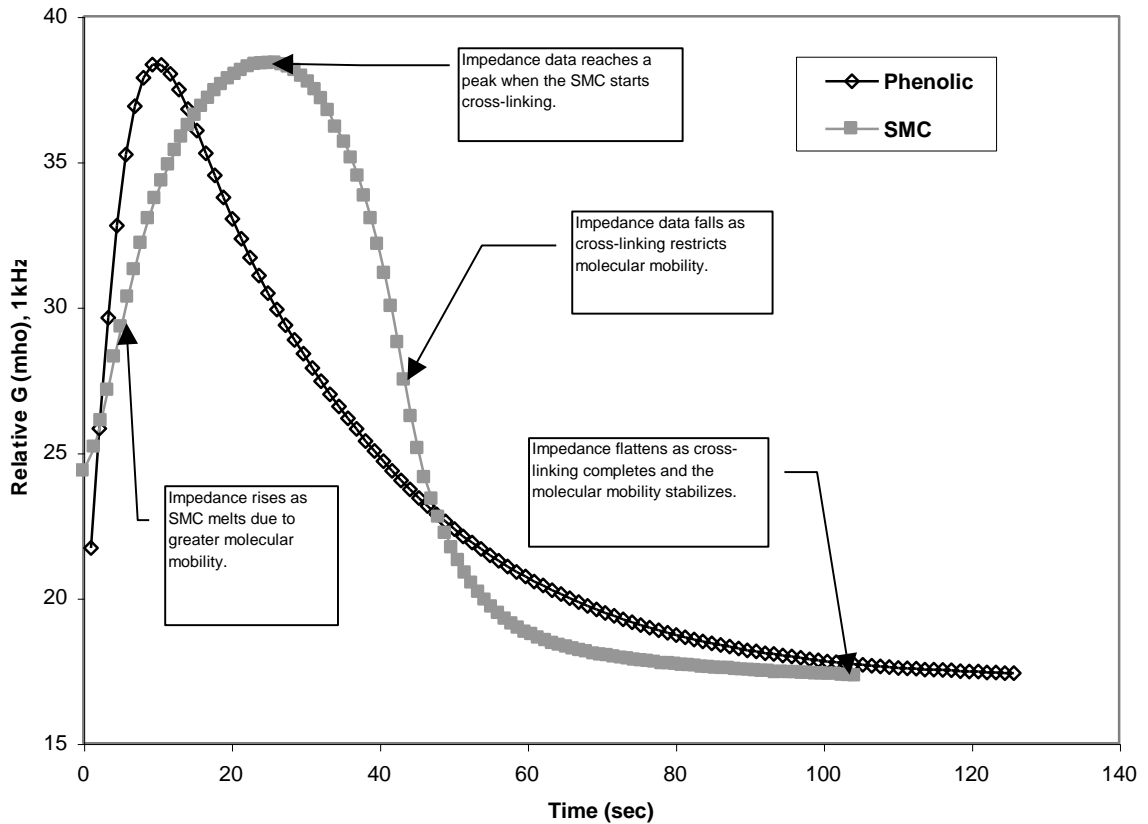


Figure 15: Comparison of Phenolic and SMC Signatures

As discussed in Figure 3, the correlation of impedance to SMC's physical and cure properties is well established. Figure 15 illustrates that the phenolic impedance signature is closely matched in shape to that of SMC. Both signatures initially rise, which is related to increasing molecular mobility during melt for SMC. The phenolic and SMC curves reach a peak then fall to a flat. In SMC, the peak and fall have been shown to relate to the onset and continuation of the curing reaction. The flat in the SMC signature has been proven to correlate to the end of the cure process.

Figure 16 below shows the effect mold temperature changes had on the phenolic impedance signature.

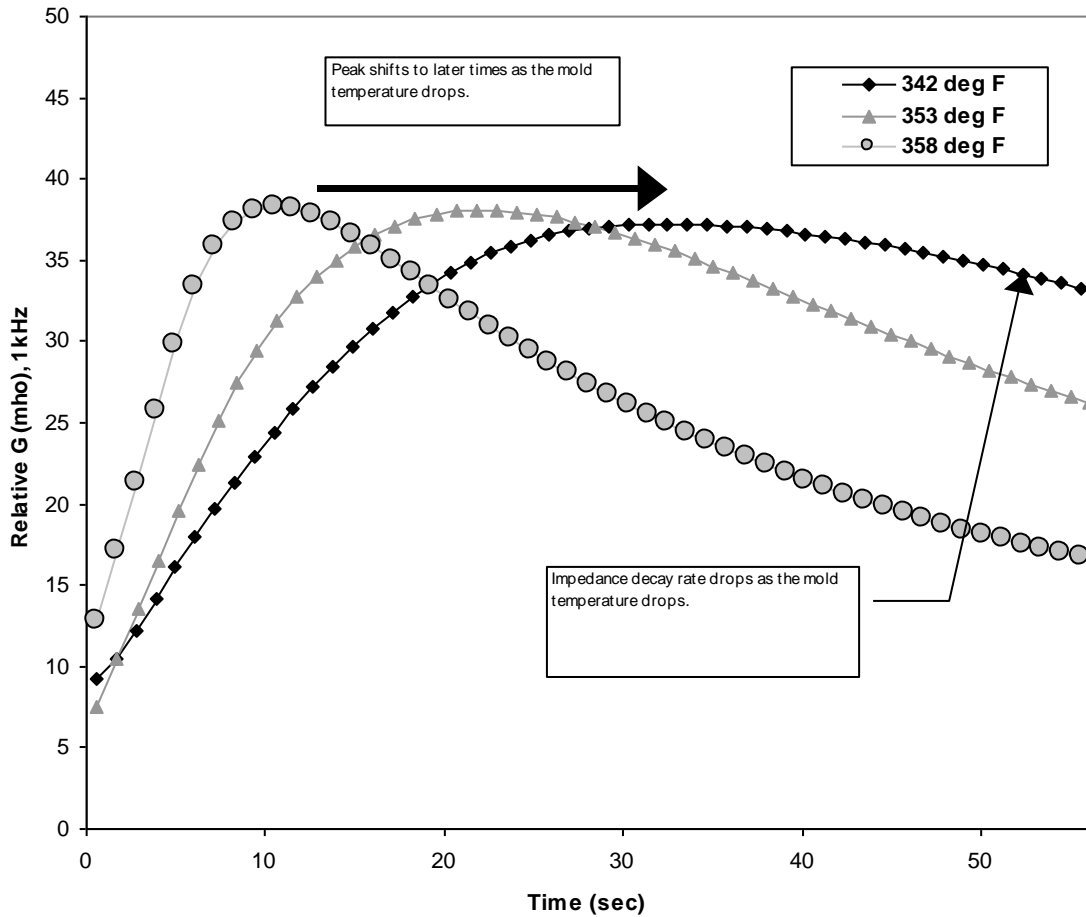


Figure 16: Change in Phenolic Impedance with Mold Temperature

Lowering the mold temperature slows the melt and reaction rates. Figure 16 shows that this causes the impedance curves to shift to the right, with peaks and flats occurring later in time. The changes in the phenolic impedance curves with temperature change are similar to the changes that occur in SMC impedance curves (see Figure 5).

Figure 17 below shows the effect pre-form temperature changes had on the phenolic impedance signature.

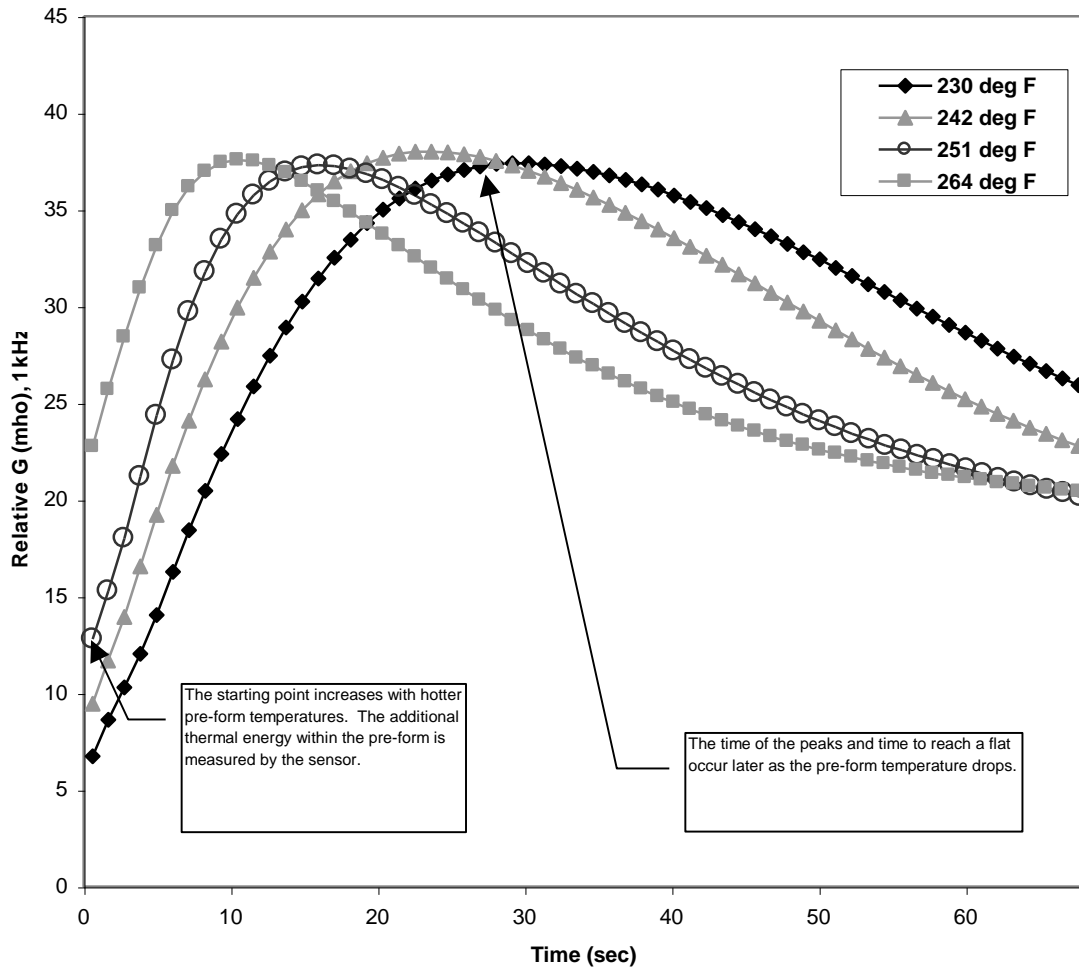


Figure 17: Change in Phenolic Impedance with Pre-Form Temperature

As the pre-form temperature rose, the starting point of the impedance signature rose, indicating the sensor was detecting the additional thermal energy and increased molecular mobility in the material. The time of the peaks shifted to later times as the pre-form temperature was lowered. It also took longer for the impedance signature to reach a flat as the temperature was lowered.

Figure 18 below compares the impedance signatures of representative parts cured at different conditions with different cure properties at the time the press opened.

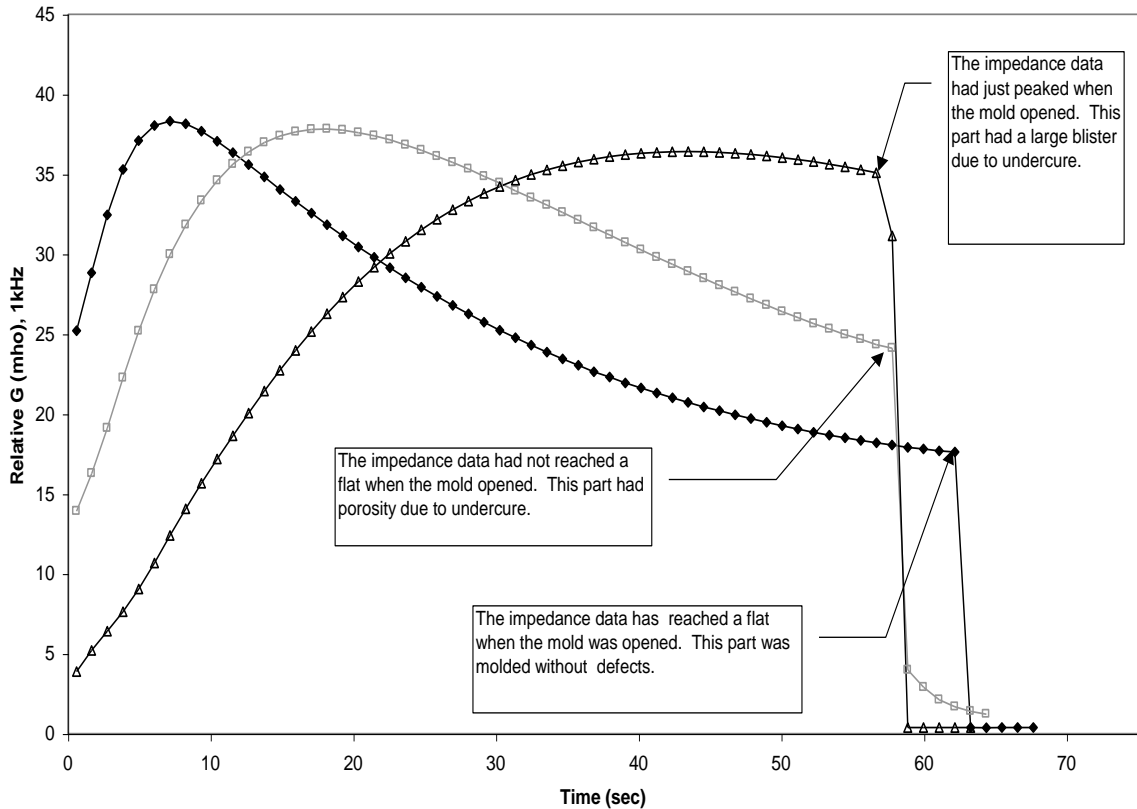


Figure 18: Relationship between Impedance and Cure State for Phenolic

The part produced when the press opened just after reaching an impedance peak had a large blister due to under cure. A part produced when the press opened about forty seconds after the peak but before the curve reached a flat had porosity due to under cure. The part produced when the press opened at the time the impedance had reached a flat showed no cure-related defects. As with SMC, the impedance data in figure 16 indicates the flat portion of the impedance curve correlates to adequate cure to prevent porosity and blisters in the part.

Examination of Figures 16, 17 and 18 shows that SmartTrac impedance sensing technology's impedance data has a strong correlation to the rate and state of cure for a mineral filled phenolic resins.

Flow Analysis Cure Time System (FACTS™)

Ashland Performance Materials has developed a new industry standard for laboratory characterization of thermoset compounds. The Flow Analysis Cure Time System (FACTS™) is a complete system that enables lab technicians to test for variation in production compound using impedance monitoring and a spiral flow tool specially designed to accommodate long fiber materials such as sheet and bulk molding compounds (SMC/BMC). The system incorporates the patented SmartLab™ real-time impedance monitoring system and special analysis software for performing statistical characterizations.

A new spiral flow tool has also been developed to further enhance this technology that incorporates a 2" wide channel that is 48" long for a relative flow measurement. It is a compression mold with shear edges and can mold plaques up to 0.75" thick. Molding pressure can be varied to control flow in the part.

A SmartTrac impedance sensing technology dielectric cure sensor is utilized along with temperature and pressure sensors in the spiral flow tool. With this system the user has the ability to evaluate the effects of catalyst, inhibitor, temperature, pressure, reinforcement loading, reinforcement type and part thickness on the cure behavior. The temperature and pressure sensors located at 0" and 30" yield additional data on the differences in cure under the charge pattern and after flowing 30" in a hot mold.

The system may be used independently or in combination with SmartTrac impedance sensing technology system to test for variation in cure reaction from batch to batch or compound to compound to ensure optimal processing.

Material checks using the FACTS™ System can ensure consistent sheet quality, correlate directly to in-mold behavior, compensate for maturation/viscosity differences, screen out-of-spec material and perform off-line troubleshooting.

Study

A study was made with a 40 percent glass, production SMC to find correlation with Differential Scanning Calorimeter (DSC) and FACTS on completeness of cure. The cure time for the SMC of 45 seconds was established using the FACTS systems. FACTS panels were molded at the FACTS cure time, 10 seconds less than the FACTS cure time and 20 seconds less than the FACTS cure time. A panel made at each cure time and at 400 PSI was submitted to Ashland's Analytical Services for DSC analysis and no indication of residual cure was found in any of the samples. Additional panels were molded at the above cure times and quenched in ice water immediately after de-molding. These panels also showed no indication residual cure from the DSC analysis. A panel made at each cure time and 1000 PSI was submitted to analytical for DSC analysis and the amount of residual cure was found to be minimal at 45 seconds and increasing proportionally as the cure time was reduced.

The use of the impedance sensor and newly developed software package to make the data more accessible to evaluate changes makes the FACTS system a useful tool for both development and quality control. The ease of analyzing the effects that changes in components and processes have on gel, cure and flow behavior will allow the comparison of identical formulations from one run to the next and help determine if "off-spec" material will flow enough to be used in production.

Conclusion

SmartTrac impedance sensing technology provides a robust, proven technology to improve thermoset molding efficiency and quality. SmartTrac technology's impedance sensors have been proven through thousands of cycles in high volume SMC production. The three production case studies and one lab case study presented in this report demonstrate:

- SmartTrac impedance sensing technology is robust and repeatable in a high volume production environment.
- SmartTrac impedance sensing technology reduced cure times by 32% on a large SMC part with no sacrifice in product quality.
- SmartTrac impedance sensing technology reduced cure times by 18% in a high volume production environment over a two month period.
- SmartTrac impedance sensing technology demonstrated potential cure time savings of 38% on an SMC automotive body panel.
- SmartTrac impedance sensing technology automatically adjusted cure times to compensate for mold temperature fluctuations.
- SmartTrac impedance sensing technology identified SMC flow property variations in a production setting and enabled an efficient change in process variables to prevent non-filled parts.
- SmartTrac impedance sensing technology identified material variations in a production setting and provided a mechanism to continually improve the SMC formulation.
- SmartTrac impedance sensing technology's impedance signatures showed a strong correlation to phenolic cure rate and state.
- The FACTS system has proved to be valuable for both quality control and development of thermosetting compounds.