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RESIN PROCESS CONTROLS

By D. E. Shepherd

Published October 1978

Final Report

Prepared for the United States Department of Energy
Under Contract Number EY-76-C-04-0613.

**Kansas City
Division**

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By D. E. Shepherd

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Final Report
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An automated encapsulation system is used for potting transformers with mica-filled epoxy. Process controls on the system would assure the material is dispensed accurately. Three methods for monitoring the mixed material were investigated, but none of them were suitable for use on the automated encapsulation system. This study did show that a successful measurement can be made in the laboratory under a static condition; however, different techniques are needed for production environments.

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SUMMARY

An automated encapsulation system is used to encapsulate magnetic devices with a mica-filled epoxy. This system is calibrated prior to filling molds. With changing motor speeds, incorrect operating valves, or stoppages in material lines, variations in material will not be observed until the epoxy has been cured. A monitor system on the automated encapsulation system could provide feedback to the machine to make adjustments as material is dispensed.

Three methods for monitoring the mixed material were investigated. The methods were by chemical titration, exotherm experiments of the material, and dielectric analysis. Dielectric analysis was used to set a baseline for tests on the automated encapsulation system. The first two methods were unsuccessful for use on the system.

Laboratory results by dielectric analysis were encouraging as constant readings were observed. These laboratory tests were performed in a static situation. A dynamic situation exists on the automated encapsulation system as the material is flowing across the electrodes. Because of the material's dynamic condition in the automated encapsulation system, a static charge is developed across the electrodes and data is inconclusive. Capacitances were also measured; however, readings were not stable enough to reach a definite conclusion.

Further activity is beyond the scope of this study. This study does show that a successful measurement can be made in the laboratory under a static condition. Different techniques need to be evaluated to obtain successful results on the automated encapsulation system.

DISCUSSION

SCOPE AND PURPOSE

The purpose of this effort was to determine if a continuous electrical measurement can be made on a mica-filled epoxy as dispensed by the automated encapsulation system (AES) to show that the mixed ratio is correct. In the AES, the ratio can be affected by stoppages in the lines, incorrect operation of valves, and changes in pump speeds. Electrical parameters and physical characteristics of material can be altered with an incorrect material ratio. Resistance of the two-part epoxy can be measured because amines in the hardener are conductive in the uncured state. As the material cures, ions in the amines are captured by the resin material and resistance increases. If an electrical measurement can be made to determine proper ratio mix, this information can be fed back to an automatic control to alter pump speeds and valves to assure a continuous ratio.

ACTIVITY

An AES is used to dispense a two-part epoxy to encapsulate magnetic devices. This system is calibrated and material is then dispensed to encapsulate magnetic devices. Once the AES is calibrated, material is dispensed and no further measurements are made to check material calibration. If there are any changes in the material due to machine dispensing variations, these changes will not be detected.

The material dispensed by the automated encapsulation system is a mica-filled resin composed of diglycidyl ether of bisphenol-A with a hardener containing a eutectic of two aromatic diamines. The ratio of material is 60 parts by weight (pwb) resin to 12 pbw hardener and 40 pbw of mica. Material is dispensed for 15 seconds and is then shut off for another 15 seconds. It takes about two minutes for the material to reach the electrodes after the material has started dispensing.

Process

Prior to the dielectric analysis, chemical titration and exotherm experiments were run. Chemical titration of the unused catalyst was reacted with excess acid. This was then potentiometrically back titrated with a standard base. The exotherm of the mixed material was measured at different ratios.

Material resistance was measured on the automated encapsulation system using a digital electrometer. A constant current is generated by the digital electrometer and resistance is determined by measuring the voltage between two electrodes.

To establish a baseline, electrodes were set in a pint cup. The electrodes, made from a 1.6 mm aluminum sheet that was 38.10 mm square, were spaced 6.35 mm apart. The electrometer measured the resistance of the mixture against time. Temperature of the mixture was monitored during the test.

The resistance of the resin and hardener was measured as the temperature changed. Results are shown in Figures 1 and 2. Mica filler used in the epoxy system was not used in this test. Mica is chemically inert and increases the resistance of the resin. Figure 3 shows the resistance of the mixed resin and hardener. This figure has the different ratios of 100 pbw of resin to 15, 20, and 25 pbw of the hardener. Nominal mix is 100 parts resin to 20 parts hardener.

Prior to tests on the AES, a laboratory setup was used with a 1/2-inch stainless-steel pipe tee and a single stainless-steel rod, 1.6 mm diameter, as the electrode in the middle of the tee. The tee's shell was the other electrode. Figure 4 shows the laboratory results for the mixture of 100 pbw resin mixed with 15 and 25 pbw of hardener.

A 1/2-inch stainless-steel pipe tee was also used for a test in the laboratory. Two rectangular electrodes 6.35 mm wide by 19.1 mm long and spaced 5.08 mm apart were used. Again, three different mixed ratios were made with different hardener ratios of 15, 20, and 25 pbw. Figure 5 shows the measured results for the three mixes when mica was added.

For the automated encapsulation system, electrodes were placed in a 1/2-inch stainless-steel pipe tee. This pipe tee replaces an elbow following the static mixer on the AES. Electrodes were set in a threaded Teflon plug and the plug was screwed into the top of the tee. This places the electrodes in a fresh stream of dispensed material. On the AES, tests were performed with the rectangular electrodes. The electrodes were then changed to a 1.6-mm stainless steel rod in the center of the tee. The tee shell was the other electrode.

With a rod as an electrode in the center of the tee, capacitance, resistance, and dissipation factor were also measured by using an automatic RLC bridge. These measurements were made at frequencies of 120 and 1,000 Hz with the electrode in the pipe tee installed on the AES.

Analysis of Test Results

Poor repeatability caused the chemical titration method to be unsuccessful. The exotherm measurements showed that variations in curing agent to resin ratio can be seen by plotting the exotherm of the material. A disadvantage is the lack of sensitivity

Figure 1. Resistance Versus Temperature for Resin

Figure 2. Resistance Versus Temperature for Hardener

Figure 3. Resistance and Temperature Versus Time for Resin/Hardener

Figure 4. Resistance and Temperature Versus Time for Resin/Hardener and 1.58 mm Diameter Electrodes (1/2-inch SS Tee with 1.58 mm Diameter SS Wire for Center Electrode and Tee for Outside Electrode)

Figure 5. Resistance and Temperature Versus Time for Resin/Hardener and 40 pbw Mica

between different ratios. Time to achieve satisfactory separation is about 8 to 10 minutes after mixing. This is an excessive amount of time to exercise control of the automated encapsulation system.

Figures 1 through 5 are plots of the laboratory results as the material's resistance changes with time, temperature, or both. Figures 1 and 2 show that hardener is significantly more conductive than resin. At 70°C, the mixture of hardener and resin measured about 2.5 and 50 MΩ, respectively. Material in the automated encapsulation system is mixed and poured at 70°C. Data in Figures 1, 2, and 3 were taken with a pair of 38.10 mm square electrodes in a pint cup of mixture. Figure 3 shows the difference between various mixed ratios of 100 pbw of resin to 15, 20, and 25 pbw for hardener. The automated encapsulation system's mixed material is measured about two minutes after dispensing begins. Figure 3 shows good resolution between the three curves at two minutes. There is greater than 0.6 MΩ difference between a nominal mix of 100 pbw to 20 pbw to the other two mixes. Reading of the nominal mix at this point is 2.9 MΩ.

Using the 1.6 mm stainless-steel electrode and 1/2-inch stainless-steel pipe tee, readings of 8 and 50 MΩ were measured for mixtures of 100 pbw to 15 pbw and 100 pbw to 25 pbw of the resin and hardener mixtures, respectively. Using rectangular electrodes in a 1/2-inch stainless steel tee, resolution is decreased between the mixes. Differences of 0.2 and 0.9 MΩ from the nominal mix was measured for the low and high ratio mixes respectively. The nominal ratio material measured 4.3 MΩ.

With the same rectangular electrodes and 1/2-inch stainless steel tee, resistance increased when mica was added. A nominal material mix measured 11 MΩ. A 15 and a 9 MΩ reading was measured for the low and high mixed ratios.

Altering controls to increase or decrease the amount of hardener addition would not change the resistance readings of about 4,000 MΩ.

When the electrodes were changed to the 1.6 mm stainless steel rod in the center, resistance of the material initially measured 2 MΩ. Resistance of the mixture then started increasing. Increasing or decreasing hardener ratio would not alter the increasing resistance reading. The reading eventually reached 6 MΩ before the test was stopped.

The increasing resistance is because of the electrometer. A constant current is generated and the voltage is measured across the test points (electrodes). Material flowing across the electrodes increases static charge on the electrodes because of dielectric absorption. Dielectric absorption is the capacity of the material

to absorb and hold energy for an extended period of time. The static charge could not be removed by shorting the electrodes together.

Because of this unsuccessful measurement on the automated encapsulation system, capacitance and dissipation factor were measured at 1 kHz and 120 Hz. No successful measurement was made at 120 Hz. At 1 kHz, capacitance readings ranged from 20 to 23 pF regardless of the hardener setting. These readings varied throughout the range at random and was not associated with time. Measurement of dissipation factor was unsuccessful at 1 kHz.

ACCOMPLISHMENTS

The following conclusions were made by analyzing the results of this study.

- Chemical titration is unsuccessful because of poor repeatability.
- Exotherm measurements are unsuccessful due to lack of sensitivity and the time required for separation of exotherms.
- Laboratory tests are successful when resistance of the material is measured at a static condition. A center electrode in the center of a shell produces the best results.
- Because the material is flowing across the electrodes, resistance of the material cannot be monitored for controls.
- Capacitance of the material can be measured on the automated encapsulation system. These measurements, however, are not reliable to control material output.

FUTURE WORK

More tests on the automated encapsulation system are beyond the scope of this study. A vigorous and long-term effort would be required to determine methods of monitoring material ratio electronically.

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ELECTRICAL: Process Controls

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