Ormet Circuits, Inc.

Shelf- and Work-Life

Task Summary: Determine the effects of temperature, out time, re-use, viscosity recovery by solvent addition, and time between process stages on the performance and viability of Ormet 7001 paste.

Background: Ormet 7001 paste was designed to be used under specific process conditions and lifespan parameters. The acceptable use parameters have been set rather narrowly based on controlled laboratory conditions. Performance of this task will broaden the scope and limits of the use parameters to reflect the environment and process flow of a production manufacturing operation.

Purpose: The purpose of this task is to identify how the Ormet 7001 paste would be expected to perform in a production environment.

Scope: The activities in this task were designed to identify how the Ormet 7001 would react to different shop floor conditions including the temperature environment and issues such as significant lag times between manufacturing steps. Additionally, the activities were designed to determine if the increase in viscosity observed with extended out-times has an adverse effect on performance of the Ormet 7001 paste and whether adjusting the viscosity with solvent to compensate for the viscosity increase was a viable practice. In all activities, resistance of serpentine traces processed in a lamination press was used as the primary indicator of paste performance. Differential Scanning Calorimetry (DSC) analysis was used as a supplemental indicator where appropriate.

Activities:

The viscosity (kcps) of the Ormet paste was recorded as a function of temperature in accordance with the Brookfield rheometer, and the thixatropic index (viscosity @ 1rpm divided by viscosity @ 10rpm) was calculated. The thixatropic index ranged from 1.64 at 20ºC and 1.95 at 30ºC.

Resistance (Ώ/10,000 squares) measurements were taken over a period of 125 days. The average resistance varied +/- 9.2%.

The effect of room temperature out-time on Ormet paste samples that had been deposited and dried was also measured. Ormet's standard DN410 resistance test card (FR4 with 8, 10, 15 and 20 mil width serpentine trenches photo defined in a dielectric topcoat) was used as the test vehicle. Ormet 7001 was doctor-bladed into the serpentine trenches of ten DN410 test cards and the test cards were dried at 95° for 30 minutes. All of the test cards were then placed on a cart in the corner of the lab and left to age without any special protection. Samples were removed from the cart on a periodic basis, processed in a lamination press at 190°C for 15 minutes @ 300 psi, and the resistance across the serpentine traces was measured.

The total duration of the experiment was 125 days. The average resistance varied +/- 6.5%.

To measure shelf life at room temperature, a jar of Ormet 7001 was left in the laboratory area for 57 days. DSC analysis was performed on a small sample from the jar at the beginning of the aging study to provide a control for the extent of the sintering reaction. Periodically the sample jar was opened, a viscosity measurement was made and a DN410 card with recessed serpentines was filled and processed (95°C/30 minutes followed by 190°C/15 minute/300psi lamination press). Remainder material from the filling process was recaptured and returned to the jar. 45 days into the experiment, the viscosity of the Ormet 7001 in the jar had become unworkable. The viscosity was reduced to the original specification with solvent. DSC analysis was performed before and after the viscosity reduction.

Periodic viscosity and resistance tests were made and recorded as a function of time:

1. Upon removal from the freezer.
2. After 45 days at room temperature, with a viscosity of over 4 million centipoise (5 rpm).
3. After thinning with solvent to the original viscosity specification.

All three samples were reacted in the DSC in hermetic pans with a 10°C/minute ramp rate from 25°C to 190°C with a 10 minute hold at 190°C. Ormet materials are typically analyzed with these parameters to provide controlled conditions by which extent of reaction can be compared. Once the initial reaction has been completed, the samples are heated in the DSC from 25°C to 300°C at a 10°C/minute ramp rate. This second thermal excursion is analyzed to determine to what extent the original alloy was consumed in the reaction with the copper. The extent of reaction is determined by integrating the endotherm peak located at the approximate melt temperature of the original alloy. To meet manufacturing specifications, the Ormet 7001 must demonstrate a melt energy of less than 1 joule/gram. The fresh and thinned samples are both within this specification.

The high viscosity sample was outside the specification, but still had a very low melt energy. The melt energy of the alloy in uncured Ormet 7001 paste is in the low 20 joules/gram, but tends to be masked by the exothermic reaction with copper. Analysis of these ‘second scans’ of the paste has proven to be a very reliable predictor of quality in Ormet pastes. The second peak integrated on the DSC scans represents the alloy composition that is created when the tin reacts with the copper. This new alloy forms the ‘bridges’ between the alloy encased copper particles. There is no specification associated with this peak, but it is an interesting artifact.

Conclusion: Ormet 7001 paste is very robust and well suited to a manufacturing environment. In conditions ranging from 20-30°C (68-86°F) the paste maintains a usable viscosity and desirable shear thinning characteristics.

The second set of experiments demonstrate that Ormet 7001 paste, once deposited and dried, can be stored in normal shop conditions for up to 4 months with little or no deterioration in performance after lamination.

The third set of experiments indicate that freezer storage of the Ormet 7001 paste is necessary to maintain viscosity stability. Significant changes in viscosity of the paste were observed after only a few days at shop temperature. However, even with an increase in viscosity of over ten-fold, the performance of the Ormet paste was apparently unchanged according to the resistance measurements and only slightly changed according to the DSC analysis. Further, addition of solvent to restore the material to its original viscosity seemed to have only a minor effect on resistance and completely reversed the changes observed in the DSC analysis. One possible explanation for this phenomenon is that the increase in viscosity reduces the effectiveness of the flux by restricting its mobility.

Overall, cold storage of the Ormet 7001 paste seems to be more significant for maintaining a consistent viscosity for repeatable deposition than for preserving the performance characteristics of the material. Further, it appears that changes in viscosity that might occur during an out-period for a particular container of paste can be remedied on the shop floor without detriment to performance.