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ADDENDUM

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EFFECTS OF TEMPERATURE ON FILLED EPOXY ENCAPSULATION MATERIALS

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INTRODUCTION

Voids annoy and are the concern of personnel using epoxy resin in encapsulating operations. Frequently, their varied locations in encapsulants provoke conflicting opinions regarding the conditions that cause them. Since some persons believe that faulty mold design allows air to seep across mold lands during material shrinkage, while others suggest that the exothermic temperature of cure releases dissolved air, and another idea that the duration of the deaeration period is responsible, brought a decision to attack the problem of their formation by a fresh viewpoint.

EXPERIMENTATION

Keeping in mind that mold design and void formation could be related, a resolution was made to encapsulate under air-tight conditions; and in order to see what occurs, a one-liter beaker, Griffin type without a spout, was chosen for the experiment.

A flat surface was honed on the lip so that a smooth glass plate could fit across the top and, when sealed properly, would not permit air to pass at the sealed surface. The beaker and glass plate were cleaned and dried, but were untreated with mold release. A mixture of epoxy (100 pbw), meta-phenylene diamine (14 pbw) and mica (100 pbw) was prepared, evacuated for four minutes, and then poured into the beaker to a depth that left a thin air space between the glass plate and the top of the material. The ground lip was coated thinly with the encapsulation mixture and the glass plate fitted. The unit was placed on the lowest shelf in a forced convection oven, whose air currents flowed vertically upward, to cure at an oven temperature of 135°F.

Frequent observations were made during the gelation period. While shrinkage developed at the surface of the material, the glass plate was slowly compressed inward and began to flake in concentric layers from its center towards its edge. Meantime, on the radius at the bottom of the solidifying mass, and about 60° apart, six voids formed inward for a short distance toward the center of mass of the material. Since the voids began to appear at the time the cover was flaking and before fracture of the beaker occurred, we had evidence that an air-tight mold produces its voids by shrinkage during the gelation period (Figure 1).

Following this finding, a large steel production mold associated with frequent void formations was selected for further study Normally the mold held a large thermally sensitive electronic assembly, but during much of our study castings were made without the assembly. About nine pounds of epoxy-mica-diethanolamine, in a 50-50-6 ratio respectively, was used to fill the mold. The mixture was evacuated at 1 - 3 mm mercury, absolute, for two minutes before pouring and again for 25 minutes, an unusually long deaeration period, after filling the mold in order to reduce the dissolved air. Then the filled mold was placed in a forced air oven in which air moved laterally, to cure at 130°F ambient temperature throughout the gelation period.

Several castings were made, but on each voids were found along the mold line and at bottom corners or on the radii between corners (Figure 2, Unshielded Unit). However, during cure of the castings a vertical air flow oven was used, and as we studied the voids afterward, the direction of flow of the oven air currents seemed to be related to the void characteristics.

Consequently, with further evidence needed, several small glass beakers were filled with a fresh mixture and placed in forced air ovens, one having vertical upward flow air currents and the other a horizontal movement of air. Examination of void characteristics of these clearly related them to the flow pattern of the oven air currents. Since experimentation with encapsulations unprotected from the cooling effects of oven air currents had produced voids, a series of shielded encapsulations for additional study was made.

In this study experimental units were placed inside larger glass beakers upon an insulator to form an air space about the experimental units. Each control consisted of an identical filled beaker placed nearby on the oven shelf and in a moving air temperature of 130°F. After curing the shielded encapsulations were found to be solidified into a smooth, uniform mass, in contrast to the controls which had developed voids peculiar to the type of oven air currents in which they were used (Figure 3 shows similar effects).

SHIELDING

Following this study, the steel mold was again used to determine whether similar results would be obtained if it were shielded. At first, the mold was covered with two layers of insulating glass mat, but voids developed as usual. Since the matting appeared to be an unsatisfactory shield against the cooling effects of the oven air currents, a shieldment was designed and made, large enough to protect the mold. The shieldment was a portable structure built with an outer aluminum shell and a middle layer of one-inch thick glass matting, laid upon an inner surface made of copper cloth (Figure 4).

Employing the shieldment, we found that a series of encapsulated units solidified voidless, while the controls formed voids. Controls were identical unshielded units cured in the currents of the oven, positioned beside the shielded experimental unit. Also, it was ascertained that shielded units developed smoother surfaces (Figures 2 and 3, Shielded Units).

MISCELLANEOUS OBSERVATIONS AND DISCUSSION

During gelation an inversion of an epoxy-mica mixture occurs in which top strata descends to lower levels, while lower strata solidifies near the upper surface. This phenomonen was easily seen in a 7 x 7 x 16 inches mold made with glass sides. Small amounts of different epoxy dyes were placed at top and bottom edges of the material with a pipette, followed by cure in a 130^{o}F oven without the shieldment. Dye movement was readily followed on the material surface until gelling was complete. The block was sectioned and the internal locations of the dyes determined. In general, the central core of material flows upward while the outer layer descends.

Also noted was the faster cure of material gelling in cylindrical molds at their upper level, effectively forming an air-tight mold below the cured surface. As the cure proceeds to lower portions of the mass, there is a shrinkage from the liquid phase at the periphery of the material inward toward the solidifying core. Voids are produced at those points in the cooler liquid mixture, especially at the edges and corners of the mass, where the ratio of mold surface area to material volume is higher than at the mold faces. Incidentally related perhaps are those voids which can be found by X-ray examination, that lie adjacent to the interior wall of a form that serves as a permanent mold.

Frequently, in large castings, a reservoir of material is poured above the finish height of the product in order to overcome void formation. In some instances the practice arises from a belief that during shrinkage the necessary material to prevent voids is pulled from the reservoir. Our study disclosed that a slump occurs at the corners of the material in the reservoir, but this happens for the same reason that voids occur at edges and corners at the base of the material. The coolest material solidifies last, whether it is at the top edges or corners of a reservoir, or at the lower mold extremities. One can see the characteristic at the corner of the unshielded block in Figure 2.

Of course, pouring the reservoir is costly because it entails machining the excess material after curing, and also appears unnecessary. If the mold surface temperature can be uniformly controlled, such as by a portable shieldment, mixture solidification is more uniform because the temperature difference at points between the central core and the peripheral material is lessened. In a sense, the mixture produces heat needed to partially cure itself initially, and while the shieldment is actually an effective barrier for loss of heat from the curing mass, it also serves to promote uniform temperature about an encapsulate. Similar results were obtained by placing experimental units at the highest levels in vertical air current ovens, though oven temperatures were kept at 130°F.

Lastly, where thermally sensitive components are to be encapsulated, the use of a shieldment serves to promote higher temperatures; therefore, caution must be exercised if the components cannot withstand the temperature produced.

SUMMARY

As a result of this study, we learned that voids form in vacuum conditions; that cooling effects differ on the surface of a mold in relation to the surface area of the mold and the volume of material producing heat; and that shielding a mold reduces heat losses and prevents voids, as well as improves the surface finish of filled epoxy castings.