A LOW-DENSITY POTTING COMPOUND

A. J. Quant
Sandia Corporation, Albuquerque, New Mexico

INTRODUCTION

The development of certain electronic packages for missile and airborne applications has created a need for a lightweight potting compound that offers greater environmental protection than that provided by the available polyurethane foams. Preliminary efforts indicated that this objective could be best achieved by incorporating low-density fillers into conventional epoxy resins.

Several such fillers* were investigated during the course of this effort, but none possessed processing characteristics and end properties equal to those of glass microballoons. An exhaustive review of possible resin systems was required as this type filler proved more difficult to handle than conventional solid fillers. It is not interchangeable with other fillers and cannot be freely substituted into existing formulations since it must be processed over rather limited ranges of time, temperature, and viscosity. Failure to maintain these conditions yields nonhomogeneous castings or mixes which are too viscous to process.

DEVELOPMENT

It should be borne in mind that the formulation developed is intended for use in the encapsulation of complex, expensive, electronic circuitry designed to withstand extremely severe environments. Therefore, a difficult-to-process material is not objectionable when it yields an end product of the high confidence level required.

The system finally selected parallels a previous Sandia system using the standard filler, mica, in that diethanolamine-cured Epon 828 was utilized for many of the same reasons that made it attractive earlier. Several of these reasons are:

1. good pot life
2. fluidity at the processing temperature
3. low exotherm
4. almost complete absence of toxicity.

A processing study of the microballoon-filled mix showed that other resins of a similar epoxide equivalent are not all interchangeable since differences in viscosity and reactivity result in less homogeneous castings.

Other variables, in addition to the choice of resin and hardener, were necessarily fixed to ensure homogeneous mixes. The concentration of filler was fixed at its maximum processable limit; anything less results in thinner mixes which permit flotation of the hollow spheres. The cure temperature was firmly fixed at 150°F; lower temperatures enhance flotation because the mix is in a fluid state for a longer time. Increased temperatures also enhance flotation since the viscosity of the mix is sharply reduced. Batch size was also restricted as

*Urea-formaldehyde, phenolic, and silica microballoons as well as polystyrene and aluminum silicate hollow spheres of a much larger particle size were investigated.
small batches lose heat readily and become too viscous to process. Large batches exhibit unusually high exotherms because of the thermal insulation imparted by the hollow spheres; this results in a reduced pot life. Timing is very important if the mix is to be successfully evacuated during periods of optimum viscosity. The type of mixer and time of mixing must also be defined to avoid excessive crushing of the fragile microballoons.

These investigations of the properties of the several raw materials and their interaction within a mix resulted in the recommendation of a single formulation and a tightly defined process. This single formula is the one which yielded the maximum advantages of the glass microballoon filler consistent with a minimum of processing limitations.

From the foregoing, it can be seen that the processing of a microballoon-filled epoxy resin is critical. If such a critical process is to remain manageable, reasonable control must be exercised over the individual constituents of the formulation. Fortunately, the resin and hardener give little difficulty. However, the glass microballoons sometimes exhibit serious batch-to-batch variations that lead to processing difficulties or a reduction in end properties of the cured mix.

When these batch variations became evident, a program was set up to make several properties measurements on the batches on hand and to continue the measurements on all future batches.

The property of greatest importance is average particle density. Since the formulation is based on parts by weight, any change in particle density is reflected by an adverse change in viscosity due to the variation in the volume of filler incorporated. Particle size distribution is also important since it controls the packing of individual particles within the resin matrix. Moisture content, the last requirement considered mandatory, is controlled because excessive moisture results in undesired agglomeration of the filler particles. Additional properties presently being determined on an "information only" basis are alkalinities, water solubility, and flotation ratio.

Cold-shock tests are being run by Sandia on all lots of microballoons received. This test is not part of the microballoon specification, since it must be evaluated from the final mix rather than from the microballoons alone. The tests are primarily intended to ensure that a processable mix which will pass thermal and mechanical shock testing can be guaranteed. A continuing investigation is under way to determine what properties and what limits of these microballoon properties best define this objective.

The study of the properties of early batches of glass microballoons led to the issuance of a tentative specification. From the data listed in Table I, it can be seen that the more recent batches generally meet the limits established in the specification and yield processable mixes that pass thermal shock testing.

An observer studying Table I may question the broad limits on the sieve analysis. However, all attempts to narrow this range and to include information on further sieve sizes have resulted in a mass of confused data.

The data for average particle density have proven to be more valuable. It can be seen that Batch 21-S, dated December, 1959, exhibited a particle density of 0.35 g/cc. Because of this low density, the formulation which is based on parts by weight was too viscous to pour, since it contained an abnormally large volume.

* See Appendix 1.
† See Appendix 2.