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"NEW POTENTIAL IN RIGID POTTING COMPOUNDS"

By

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Materials Engineering

THE BENDIX CORPORATION

Kansas City Division

For Presentation

At

Society of Plastics Engineers

RETEC

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INTRODUCTION

This paper concerns several rigid epoxy potting compounds used for potting large structural-type electrical packages. Two of the compounds have been used in production at Bendix for at least five years and the third is a new experimental compound which offers promise of superior performance. The paper contains formulations, manufacturing procedures and a discussion of production difficulties.

In spite of the fact that there is an infinite number of potting compounds available, a few of these compounds have been standardized for potting the large (1 to 15 pounds of potting compound) electrical packages. Where weight consideration is of prime importance, packages are potted with rigid urethane foam; however, foam potting will not be covered by this paper. The two standard compounds that will be considered are based on general-purpose liquid epoxy resin highly filled with powdered mica or glass microballoons, and catalyzed with diethanolamine. These compounds are used in a multitude of different sizes and shapes of packages with a variety of performance requirements, generally based around standard military environmental requirements. These requirements include temperature cycles (-65°F to 165°F), mechanical vibration and shock, and the various humidity cycles.

In large packages, the requirement which causes the most trouble is thermal shock or thermal cycle. The difference between shock and cycle is the rate of temperature change, which varies between an immediate change (shock) to a gradual change over a period of hours. The more rapid the change the more severe the test. Failure of a part to pass the thermal shock requirement is normally identified by a surface

crack in the potting compound. Thermal shock test may also cause internal cracks or loss of adhesion between the potting box and potting compound. These latter types of failures may be detected by X-ray analysis or the sectioning of typical parts. The number of thermal shock cycles to which a part is subjected is also important, as failure sometimes does not occur until the fourth or fifth cycle. Thermal cycle tests are sometimes classified as destructive tests, since repeated applications of stress cause fatigue failure in the part.

DESIRABLE ATTRIBUTES

To compare relative merits of plastic compounds which are suitable for potting large electronic packages, the following factors should be considered:

1. Low shrinkage
2. Low peak exotherm
3. Good electrical properties
4. A matching thermal coefficient of expansion (with items being potted)
5. Good thermal conductivity
6. High mechanical strength
7. Good moisture resistance
8. A good seal at electrical or mechanical outlets
9. Good processability
10. Thermal shock resistance.

DISCUSSION OF PARAMETERS

To discuss each of these parameters briefly, we would say that the needs of each are:

1. Low shrinkage -- to control dimension of the part, minimize the stress on the potted components and reduce the tendency to have mold line voids.

2. The exotherm peak temperature should be low, to avoid overheating of the potted electrical components during the curing of the compound and to permit fabrication of large castings without degrading the center of the potted mass.
3. Good electrical properties -- to allow designers to place components and conductors in close proximity to each other.
4. A matching thermal coefficient, to minimize the stress relationship which is built up between the potted components and the potting compound during temperature changes.
5. Thermal conductivity enters into the picture when a potted component generates its own heat and this heat must be dissipated. It is also important to reduce the temperature gradient from the outside of the unit to the inside as the potted part is subjected to temperature change.
6. Mechanical strength -- to resist inevitable thermal shock stress and to rigidly support each component during any vibration or mechanical shock, thus reducing relative motion between the various parts of the circuit. Because these potting compounds do have mechanical integrity, some designers have found it convenient to mount the assembly through the potting compound to eliminate a potting container or mounting bracket.
7. The moisture resistance requirement is essential, as moisture will interfere with the performance of any electrical circuit.
8. A good moisture seal where electrical or mechanical connections are made to the potted unit, with the circuit designed to allow the potting compound to bond to these areas to form a moisture seal.
9. Last but not least, the compound must lend itself to efficient manufacturing processes.

ADVANTAGES OF FILLERS

Rigid epoxy compounds come out second to none in approaching these requirements. Rigid epoxies, however, will not do the job without carefully selected fillers and a catalyst which is compatible with the job at hand. The addition of a filler offers the following advantages:

1. Lowers shrinkage
2. Lowers peak exotherm during curing
3. Extends pot life
4. Lowers thermal coefficient of expansion
5. Increases thermal conductivity (Except microballoons)
6. Allows larger castings to be made
7. Lowers cost (usually)
8. Increases thermal shock resistance.

The disadvantage of adding fillers is:

Increased viscosities cause processing difficulties.

STANDARDIZED COMPOUNDS

As a general rule, adding more filler improves the potting compound, if the casting has a minimum of air entrapment. Standardized compounds contain the maximum filler which can be processed into a part with minimum air entrapment, according to the process available. To get the most filler into a system, elevated temperature processing has been utilized and an evacuation cycle has been made a part of the standard process.

The first compound which we standardized contained 100 parts epoxy resin, 100 parts mica, and 12 parts diethanolamine (DEA). This mixture was a good balance between processability and good resultant properties. Later, as requirements for a potting compound with better thermal shock resistance became desirable, a new compound was

developed. This compound was based on epoxy resin with glass microballoons as the filler. The formula by weight was 100 parts epoxy resin, 43 parts glass microballoons (GMB), and 12 parts diethanolamine. This compound also had the advantage of lower specific gravity (.87 against the mica formulation of 1.64). It also had less shrinkage during cure. The epoxy-mica compounds passed the standard thermal shock test, consisting of the potting of a standard 1/2-inch nut and bolt, which was then submitted to thermal cycling between -65°F and 165°F (Figure 1). The microballoons required the development of a more severe test; this new test, now the standard, consists of a steel sleeve test specimen which is potted and then cycled (Figure 2). The microballoons easily pass this test, but the epoxy-mica compound does not. Table I compares the important properties between these two compounds; for comparison purposes, an unfilled epoxy system is also given in the table.

Note that as the fillers (all powders) are added, the mechanical strength falls off. As the filler volume increases, the thermal coefficient of expansion decreases. As previously mentioned, the density of the GMB formulation is much less than the other formulas; therefore, the dielectric constant is lower as expected. The volume resistance and the dielectric strength of all the compounds are fairly good. The cost comparison indicates that the microballoon compound is a premium material, being two to three times more expensive for the basic ingredients than the other compounds.

PARTS MANUFACTURE

The process used for manufacturing parts from these compounds is outlined in Table II. The processes outlined are relatively successful using these compounds, but a few problems have been observed. One problem is shrinkage voids that sometimes occur in the top corners of the parts. Another problem is small voids that come to the surface before gel and are missed by the spray operator. Occasional voids occur in areas that are difficult to evacuate; for example, under printed circuit

boards or connectors. The larger parts (containing 5 to 10 pounds of potting compound) that are potted in metal molds sometimes have voids in the mold parting lines. Theoretically, all of these problems do have corrective measures; however, they still re-occur from time to time in production. The high viscosity and the shrinkage of the compound can be blamed for most of these manufacturing difficulties.

SYNTHESIS OF A NEW COMPOUND

Recently, we had an occasion to desire a potting compound that would more closely resemble the thermal coefficient of expansion of iron than either of our standard compounds. The engineer needed this property to relieve pressure on a sensitive iron transformer core. We knew from experience that we could not increase the filler content of either of the standard compounds to any appreciable extent, because the viscosity was already as high as could be put into production; therefore, we considered other potential fillers that either would more efficiently lower the thermal coefficient or would allow greater volumetric loading. Our survey revealed a filler called Zerifac, which is lithium aluminum silicate manufactured by Foote Mineral Company, Exton, Pennsylvania. Reportedly, this material had a negative thermal coefficient of expansion.

The first compound investigated was 100 epoxy resin, 185 Zerifac R, and 12 diethanolamine. We tested this compound, made some parts from it and learned some rather interesting facts. First, it was equally processable to the glass microballoon compound; later in test, the strength of the compound was found to be much greater than expected; and third, thermal coefficient of expansion was much lower than expected. Work on parts revealed that the mold shrinkage was much lower; in fact, it indicated that mold draft is required in order to remove a part from the mold. The experiments on parts also illustrated that manufacturing problems such as shrinkage in top corners and the mold line voids were reduced considerably or eliminated. There was also an indication that the Zerifac compound had thermal shock resistance

superior to the glass microballoon compound, which up to that time possessed the best thermal shock properties we knew of. (See Table I for a property-by-property comparison.)

However, all the problems were still not solved. The Zerifac compound failed to match the thermal coefficient of expansion of iron. To remedy the situation, higher loadings were tried. To induce the resin to wet higher loadings of fillers, higher processing temperatures were used. In order to have sufficient pot life at these high processing temperatures, diaminodiphenylsulfone was used as a catalyst instead of diethanolamine. The highest loading we were able to process was 100 epoxy, 560 Zerifac and 25 diaminodiphenylsulfone (DDS). The thermal coefficient of the compound was measured and found to closely resemble that of iron; this thermal coefficient data is illustrated in Graph I. Incidentally, the material did the job that the design engineer theorized it would. You will notice on the graph that Shell's Z Hardener was used for loadings up to 70% filler and above that DDS was used. You will also note a conflict between the thermal coefficient data given in Table I and this graph. This variation is caused by the use of different catalysts. The high temperature (165°F to RT) thermal coefficient for the DEA cured system is much higher, because the heat distortion temperature of DEA-cured system is about 165°F and that of the Shell Z Hardener cured system is much higher (approximately 280°F).

The processing of these highly loaded epoxies required some changes from the processing standards which we had been following for years. A more intensive mixer such as Sigma-Blade mixer had to be employed. Higher temperatures of mixing and preheating had to be used. The catalyst had to be added during the filler-resin mixing, to get sufficient volume of liquid present to allow complete wetting of the filler. The cure temperature was also raised to be compatible with the catalyst. The interesting thing was that it was processable even at these very high loadings. Table III shows a process comparison between a standard and a highly-loaded Zerifac compound.

The uses of a material with the characteristics of a highly-loaded compound would be many. It will allow the electrical design engineer a new freedom in reducing stress on components in rigid potting systems. As a tooling compound, it would be of use where temperature changes ordinarily cause dimensional trouble. The adhesive engineer has the opportunity to match thermal coefficient with the material he is bonding.

The application of this material in the potting area will result in fewer voids caused by shrinkage, lower peak exotherms, and will relieve thermal cracking problems.

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SUPPLIERS OF MATERIAL

Epoxy Resin	- Shell's Epon 828, Jones Dabney Epi-Rex 510
Mica	- Mineralite Sales Co. 4X
Diethanolamine	- Union Carbide Corp, Jefferson Chemical Co.
Glass Microballoons	- Emerson and Cummings Eccospheres R
Zerifac R	- Foote Mineral Co.
Curing Agent Z	- Shell Chemical Co.
Diaminodiphenylsulfone	- Polychemical Laboratories

TABLE I

Property	100 Epoxy 12 DEA	100 Epoxy 100 Mica 12 DEA	100 Epoxy 43 GMB 12 DEA	100 Epoxy 186 Zerifac R 12 DEA
Tensile Strength, psi	11,600	7,700	4,660	11,400
Tensile Modulus, psi	$.41 \times 10^6$	1.19×10^6	$.44 \times 10^6$	1.54×10^6
Flexural Strength	17,300	12,300	5,850	12,200
Flexural Modulus	$.47 \times 10^6$	1.21×10^6	$.43 \times 10^6$	1.66×10^6
Steel Sleeve Thermal Shock 3 cycles +160°F, -65°F	Fails	Fails	Passes	Passes
Thermal Coefficient of Expansion, in./in./°C				
-65°F to RT	53×10^{-6}	37×10^{-6}	34×10^{-6}	23×10^{-6}
RT to 165°F	82×10^{-6}	48×10^{-6}	42×10^{-6}	33×10^{-6}
-65°F to 165°F	65×10^{-6}	41×10^{-6}	37×10^{-6}	28×10^{-6}
Specific Gravity, Cured	1.2	1.64	.87	1.75
Dielectric Constant 10 ⁶ cps	3.69	4.52	2.92	4.7
Dissipation Factor 10 ⁶ cps	0.0178	.0357		.024
Volume Resistivity RT ohm/cm	6.7×10^{15}	1.1×10^{15}	2.05×10^{14}	1.9×10^{14}
Dielectric Strength Short Time V/Mil	513	545	465	445
\$/ Cubic Inch	.0294	.0221	.0657	.0244

TABLE II

<u>Process Operations</u>	<u>100 Epoxy 100 Mica 12 DEA</u>	<u>100 Epoxy 43 GMB 12 DEA</u>
Time for Mixing Filler into Resin	15 minutes	10 minutes
Temperature of Ingredients at Start of Mix	RT	RT
Preheat Compound Prior to Adding Catalyst	160°F	150°F
Mixing Time with Catalyst	3 minutes	3 minutes
Evacuated Immediately to	1/2 - 3 mm Hg	1/2 - 3 mm Hg
Time after Foam Collapse	3 minutes	3 minutes
Temperature at Pouring	170 - 180°F	185 - 190°F
Sufficient Material is Poured into Part to Cover Components and Evacuated to	1/2 - 3 mm Hg	1/2 - 3 mm Hg
Vacuum is Held for	3 minutes	3 minutes
Vacuum is released slowly and part is topped off with evacuated resin and cured	15 hours	24 hours
Cure Temperature	160 $\pm$ 5°F	150 $\pm$ 5°F

- Notes: 1. Before the gel takes place in the oven, the parts are sprayed with a surface active agent such as isopropyl alcohol several times, to break any bubbles that rise to the top surface. These bubbles are caused by the lack of an evacuation cycle after the topping operation.
2. All mixing performed in a Hobart mixer.

TABLE III

Process	100 Epoxy 186 Zerifac 12 DEA	100 Epoxy 566 Zerifac 25 DDS
Time for mixing filler and resin	15 minutes	30 minutes* (incl catalyst)
Temperature of Ingredients	RT	200°F
Preheat compound prior to adding catalyst	165°F	*Added with filler.
Mixing time with catalyst	3 minutes	*Initial time shown.
Reheat of material prior to evacuation	No reheat	280°F
Evacuation of material	1/2 - 3 mm Hg	1/2 - 3 mm Hg
After foam collapses	3 minutes	1 minute
Stabilize temperature for pouring	185 - 190°F	Immediate pouring
Enough material is poured into part to cover components and evacuate to:	1/2 - 3 mm Hg	1/2 - 3 mm Hg
Vacuum held for:	3 minutes	1 minute
Vacuum released slowly, part is topped off with evacuated resin and cured for:	15 hours	3 hours
At a temperature of:	160 $\pm$ 5°F	300 $\pm$ 5°F

*Mixing of ingredients accomplished on Sigma Blade Mixer.

Test Procedure:

- a. Place the specimens in a refrigerated cold box maintained at a temperature of $-80^{\circ} \pm 2^{\circ}\text{F}$ for at least 4 hours. Specimens shall be spaced at least $1\frac{1}{2}$ inch apart.
- b. Remove specimens from cold box. Place a bench top at room temperature, spacing specimens at least $1\frac{1}{2}$ inch apart. Allow specimens to come to equilibrium at room temperature (at least 4 hours).
- c. Repeat steps (a) and (b) for 2 more complete cycles.
- d. After the specimens have warmed to room temperature, they shall be visually examined for any evidence of cracking or other indication of failure in the casting.

1. Cross support
2. Copper wire AWG 18, 1 piece, $1\frac{1}{2}$ turns around bolt.
3. Steel cap screw $1\frac{1}{2}$ -13 x $1\frac{1}{2}$
4. Steel nut $1\frac{1}{2}$ -13
5. Hot drink cup, 6-oz Capacity, No. 2336 Unwaxed, Dixie Cup Division of American Can Co
6. Material to be tested
7. All dimensions are in inches

Figure 1. Thermal Shock Specimen

Figure 2. Sleeve Thermal Shock Test

Graph 1