Sandia Corporation

REPRINT

TRANSPARENT COLD-SHOCK-RESISTANT

EPOXY CASTING RESIN

by

Ben Carroll

and

John Smatana

APRIL 1960

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ABSTRACT

This paper discusses the development of a transparent
cold-shock-resistant epoxy casting resin; physical and electrical
properties are presented. In addition, a simple inexpensive test
method for determining cold-shock-resistance is described.

LIST OF ILLUSTRATIONS

TRANSPARENT COLD-SHOCK-RESISTANT EPOXY CASTING RESIN

The need for an epoxy formulation which would be resistant to the effects of severe thermal cycling has long been recognized in the electronics industry. Considerable work has been done on the subject, but little has been published of a basic nature. Probably the dearth of related publications can be ascribed in part to policies wherein useful proprietary systems are frequently made "trade-secrets" of the company sponsoring the research. This paper discusses the development of a cold-shock-resistant transparent epoxy casting resin that is intended to fulfill the existing need.

How does one define "cold-shock resistance"? In general, this term refers to the inherent ability of a cured resinous system to survive repeated exposure to varying temperature environments. For test purposes, a metallic insert of an appropriate configuration and size is usually embedded in the casting in order to maximize the encountered stresses. In most instances a rigid resin will crack when it is restrained from shrinking, and shrinking occurs as a result of curing and the subsequent cooling of the resin. A more serious cause of cracking is the apparent inability of most resins to survive the effects of differential expansion which occur when a chilled casting containing a metallic insert is exposed to elevated temperatures. Cracking under these conditions is ascribed to the difference in coefficients of expansion between the insert and the surrounding resin. In either circumstance, there are apparently only three methods which can be utilized to prevent such ruptures: first, inorganic powdered fillers can be added to the resin in order to lower the coefficient of expansion so that it will more nearly approach that of the metal inserts; second, fibrous fillers can be added to the resin in order to increase the cohesive character of the mass; third, the resin itself can be internally modified to lower its elastic modulus, thus the system can preferentially stress-relieve itself or "cold-flow" around the inserts rather than cleave at the points of greatest stress. Very frequently, any two, or all three approaches can be combined to produce a shock-resistant casting system.

At the Sandia Corporation, the task of formulating a shock-resistant resin was made somewhat more complex by the addition of three specific requirements. The first requirement stipulated that the system must be optically clear so that encapsulated units could be

visually inspected; the second requirement was that the system must be curable at temperatures not exceeding 165°F in order to protect temperature sensitive components from thermal destruction; the third requirement was that the electrical properties must be of the same high order encountered in conventional rigid systems.

It was believed that such a casting resin would be welcomed by production, control and quality assurance groups. It could also be of great utility to design engineers who have always had to contend with the deleterious effects of severe pressures on many pressure-sensitive electronic devices.

From the start of the investigation it was obvious that the resin itself would have to be modified, since fillers and fibers rendered the system opaque. To this end, several flexibilizing systems were studied empirically in an effort to eliminate the least desirable types. Slide 1 briefly illustrates the results of the screening; from these observations, it was decided to concentrate on polyglycol ethers through a moderate range of molecular weights and functionalities to achieve the desired modifications.

It is desirable at this point to review those particular epoxy curing mechanisms which are pertinent to the choice of glycol ethers as flexibilizers. Slide 2 illustrates the basic structure of a bisphenol-epichlorohydrin resin. This discussion is concerned entirely with liquid resins wherein the repeating unit, designated "n," has a value of approximately 0.1 to 0.2. Slide 3 illustrates the three principle reactions occurring between a typical anhydride and the selected epoxy resin. In step (a) on the slide, the anhydride reacts with a hydroxyl group to yield a reactive acid group. In step (b), the acid then condenses with the epoxide to yield the hydroxy-ester. It is here noted that a hydroxyl group has been regenerated and thereby permits the recurring reaction shown in step (a). Step (c) illustrates the random condensation which occurs between any epoxy with any hydroxyl group to form a hydroxy-ether. All three reactions are known to occur in the uncatalyzed system;[1] however, in the base-catalyzed reaction, evidently only reactions (a) and (b) occur.[2] It is also worthy of note that a base-catalyzed system can be carried to completion at temperatures far lower than those required for uncatalyzed systems.[2]

The mechanism of the anhydride cure has been shown to occur through an initial reaction between anhydride and hydroxyl. Where desirable, the hydroxyl may be deliberately introduced from numerous sources other than the epoxy resin; the structure to which the hydroxyl has been originally attached then becomes an integral part of the resinous system and reflects its own character in the final properties of the cured resin.

Flexibilizer	Color	Volume Resistivity (200°F)	Thermal Stability (220°F)	Pot-Life	Resistance to Thermal Shock
Polysulfide	Very dark	Low	Poor	Short	Excellent
Polyamide	Very dark	Low	Fair	Very short	Extremely poor
Reactive Polyester	Dark amber	Excellent	Excellent	Moderate	Excellent
Glycol-Ether	Light amber	Excellent	Excellent	Moderate	Excellent

Slide 1. PROPERTIES OF SYSTEMS MODIFIED WITH SELECTED FLEXIBILIZERS
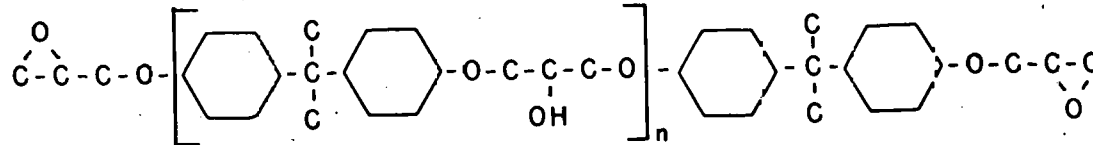
Slide 2. BISPHENOL-TYPE EPOXY RESIN
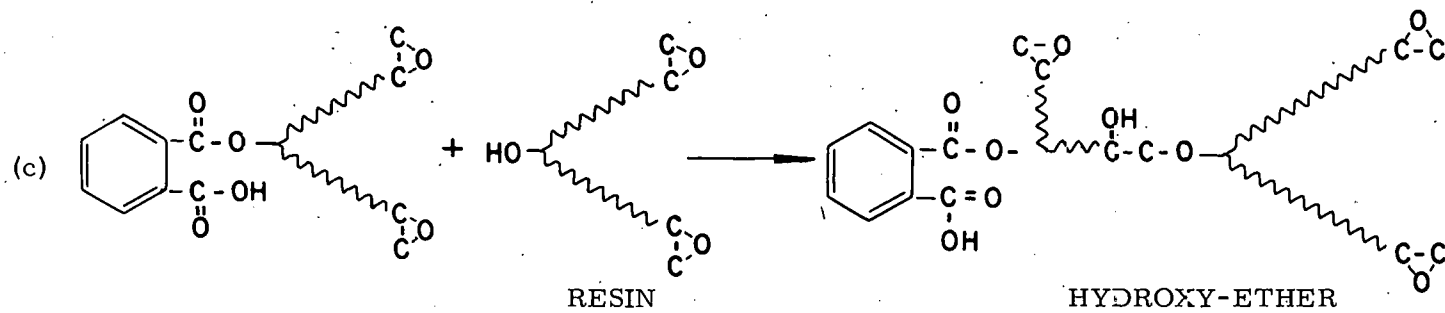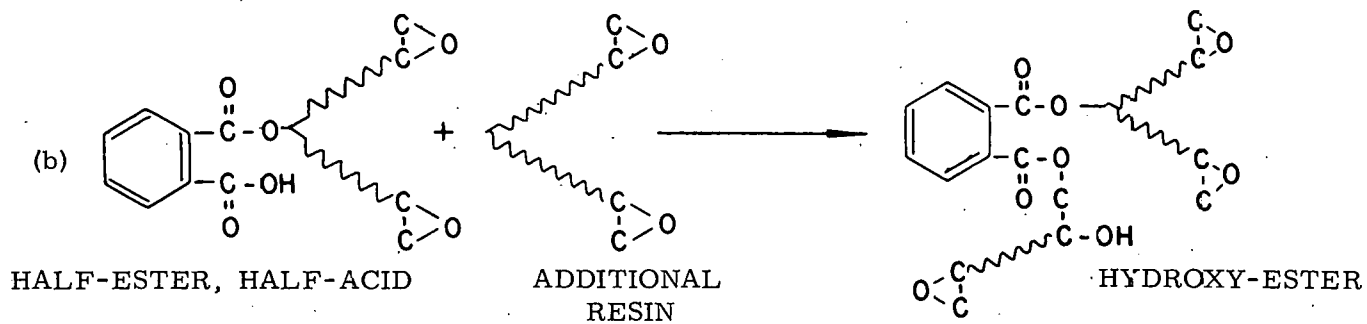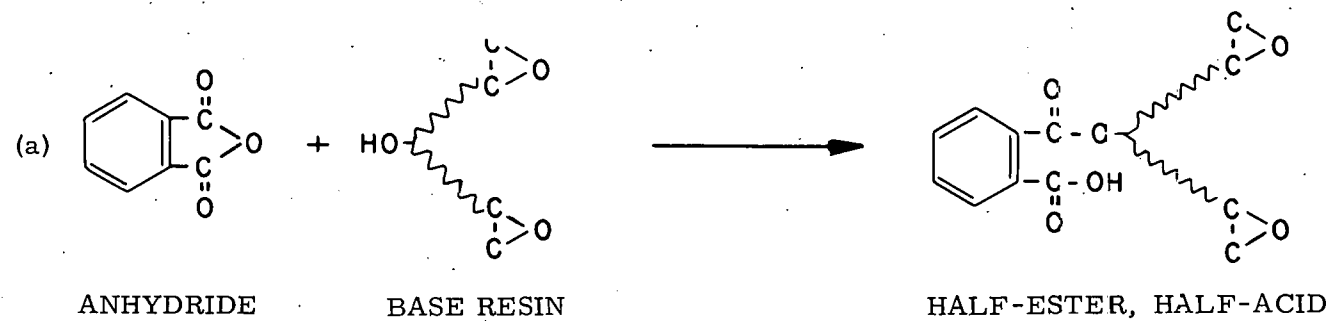
Slide 3. ANHYDRIDE REACTIONS

Slide 4 illustrates the reaction which occurs initially when a long-chain glycol is introduced into an epoxy-anhydride system. Two mols of anhydride and 1 mol of glycol combine to form a long-chain dibasic acid. Slide 5 shows the reaction between this dibasic acid and the liquid epoxy resin. For simplification the center groups have not been illustrated. In this illustration it is now observed that the dibasic acid and liquid epoxy resin have effectively combined "in situ" to form an elongated structure that still retains reactive epoxy groups on the terminal positions. It is this elongated structure which contributes materially to the flexibility of the system.

A series of experiments was planned in order to utilize the best of the available polyols and anhydrides. Two glycols and two triols were selected as modifiers, and three anhydrides as curing agents. Fortunately, all of the selected polyols were low viscosity liquids. Slide 6 illustrates the approximate structures of the selected polyols. The molecular weights were restricted to the approximate range of 500 to 1000, on the basis of the results obtained from preliminary screening. Slide 7 illustrates the structures of the selected anhydrides, all of which are liquid at, or slightly above, room temperature. These anhydrides were selected because they were representative of typical liquid anhydride structures presently available on the commercial market. A fourth compound (Methyl Nadic Anhydride) was omitted from these studies because it did not cure well at the maximum allowable temperature of 165°F. The preliminary screening also indicated that benzyldimethyl amine (BDMA) would be the preferred promoter. Practically any tertiary amine can be used to promote the cure of anhydride-epoxy systems;[2] however, BDMA produced a cured product having the lightest color.

In order to limit practically the number of formulations to be studied, it was imperative to fix certain ratios of ingredients while others were being varied. Fortunately, sufficient experimental evidence had been produced in the field and at the Sandia Laboratories to indicate that the mol ratio of anhydride to epoxy could be properly established between 0.85: 1.0 and 1:1. The arbitrary reduction from unity is sufficient in most cases to accommodate several competing reactions, one of which has been previously shown (Slide 3, Step (c)). The ratio of the promoter to epoxy resin was then established at 2% by weight. Experiments performed during the screening proved that the concentration of promoter affected the rate but not the sequence of reactions. The polyols were varied in several proportions while the rest of the formulation remained fixed. A final and important restriction was the cure which, through prior specification, was not permitted to exceed 165°F.
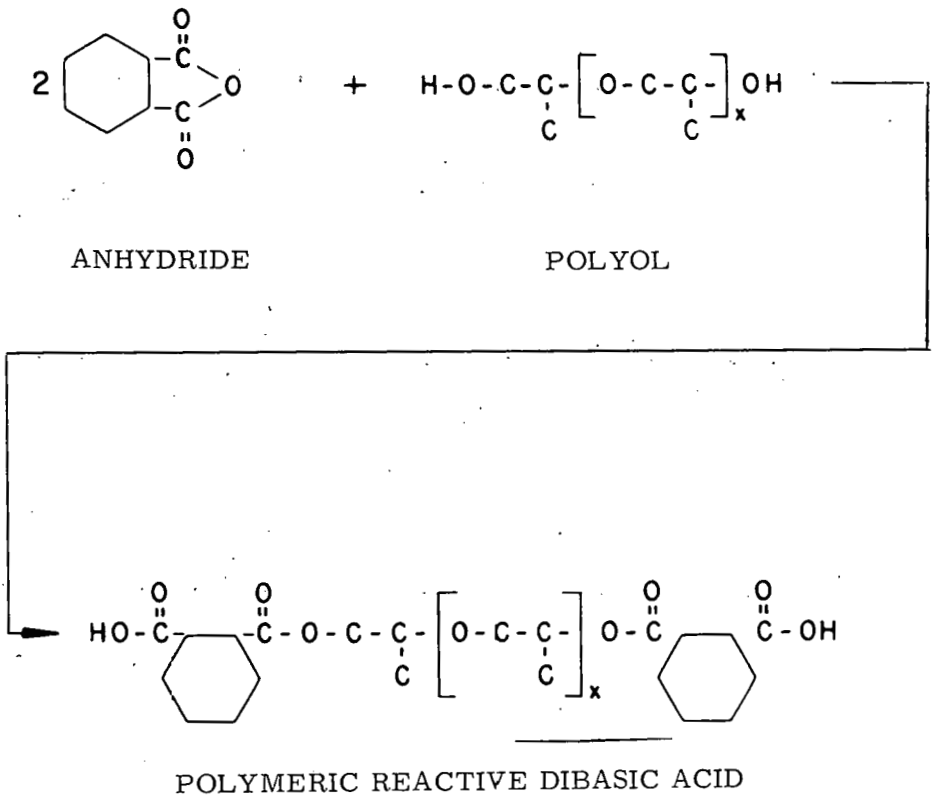
Slide 4. FORMATION OF LONG-CHAIN DIBASIC ACIDS

EPOXY RESIN

DIBASIC ACID

base catalyzed

EPOXY TERMINATED FLEXIBLE INTERMEDIATE
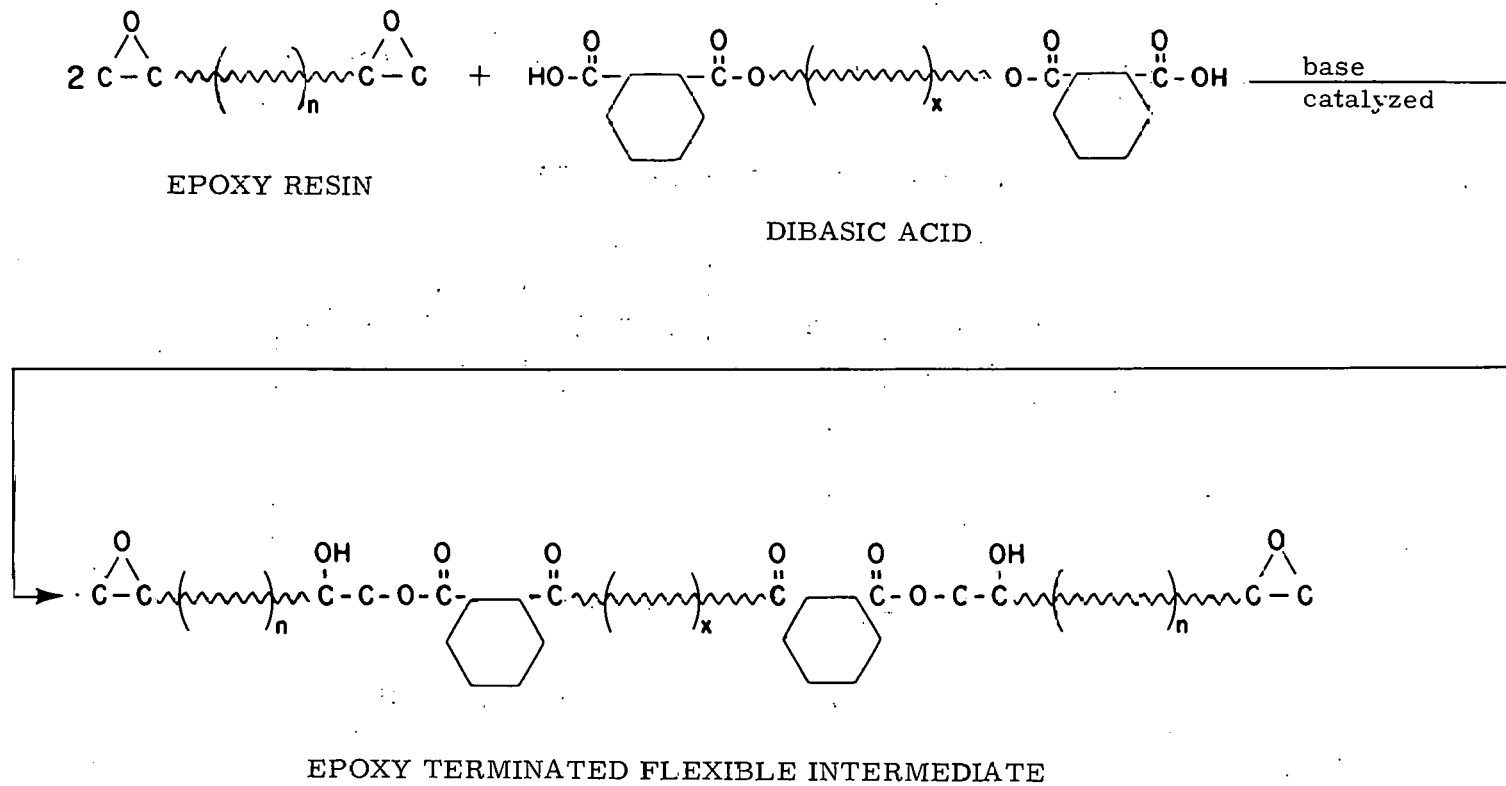
Slide 5. REACTION OF EPOXY RESIN WITH LONG CHAIN DIBASIC ACID

	Identity	Type	Av Mol Wt	Value of X	Structure
(a)	P 750	Diol	750	12.5	
(b)	P 1025	Diol	1025	17.4	
(c)	P 11-80	Triol	720	3.6	
(d)	P 11-100	Triol	1050	5.5	

Slide 6. COMPARISON OF SELECTED POLYOLS

Identity	Mol Wt.	M. P.	Average Structure
(a) Nonenyl succinic anhydride (NSA)	224	<12°C	
(b) Hexahydrophthalic anhydride (HHPA)	154	35°C	
(c) Butenyldimethyl tetrahydrophthalic anhydride (Beta-S)	238	-10°C	

Slide 7. COMPARISON OF SELECTED ANHYDRIDES

The basic formula that was followed now appears as illustrated on Slide 8. As can be seen, the ratios of resin, anhydride, and promoter were fixed, while the choice of polyol and quantities to be evaluated were left open. Once again, preliminary screening suggested that each polyol should be evaluated in each basic formulation in varying amounts, specifically 50, 60, and 70 parts per hundred of resin. Formulations containing less than 50 PHR (parts per hundred of resin) were too rigid and easily cleaved during cold cycling; formulations containing greater than 70 PHR were too soft to be considered for military applications. Since each of the four polyols was to be employed in three different quantities in each of three different basic formulations, it can be seen readily that a total of 36 different chemical formulations would be the result. If six test samples were to be made of each formulation, a total of 216 specimens would have to be prepared for testing. In order to evaluate these specimens properly, it was imperative that a test method be adopted which would be simple, rapid, effective, and inexpensive. Fortunately, the Sandia Laboratories had already developed a suitable test method.

Two factors predominate in the selection of a suitable thermal shock test: first, the test must be severe enough to eliminate all marginal or mediocre resins; second, the test must not be so severe that it will fail the best resins. A suitable procedure presently in use is the Navy hex-bar test per MIL-I-16923C. In this test, a hexagonal insert is embedded in a removal mold and shocked between $130^{\circ}C$ and $-55^{\circ}C$ for 10 cycles or until the specimen cracks. This sytem has gained some acceptance in the field. However, preparation of the specimens and molds is costly, and the process of reclaiming the inserts following testing is time-consuming. Shell Development Company is credited with a test based on the encapsulation of a short length of steel pipe with milled-out feet.[3] In the latter system, the specimen is cooled down gradually, with periodic inspection for cracking. Finally, 3M has developed a test based on the encapsulation of 1/8" and 1/4" thick washers in a shallow aluminum dish. These castings are then rapidly cycled between $105^{\circ}C$ and $-55^{\circ}C$. All of these systems are good. Nevertheless, the preparation of inserts and the reclamation of the inserts following the testing is tedious and frequently expensive.

At the Sandia Laboratories the system in use involves the encapsulation of a nut-and-bolt assembly in a 6 oz. paper cup. The cured casting is exposed to the selected cold temperatures and alternately permitted to thaw at room temperature. Slide 9 shows a sketch of the entire assembly. It will be observed that the insert assembly possesses several horizontal planes, all of which tend to induce cleavage in the severe thermal cycling process. In addition, the threads have sharp edges which tend to induce clevage. All of these destructive tendencies are most desirable since encapsulated devices usually have multitudes of flat planes and sharp edges.

Mol Eq.	Identity	Formula	Formula	Formula
0.53	Epoxy Resin	100 gs.	100 gs.	100 gs.
0.49	NSA	110 gs.		
0.49	HHPA		76 gs.	
0.49	Beta-S			116 gs.
-	Polyols	open	open	open
-	BDMA	2 gs.	2 gs.	2 gs.

Cure: $\dfrac{15 \text{ hours}}{130°F} + \dfrac{24 \text{ hours}}{165°F}$

Polyols $\begin{cases} P & 750 & (50, 60, 70 \text{ PHR}) \\ P & 1025 & (50, 60, 70 \text{ PHR}) \\ P & 11\text{-}80 & (50, 60, 70 \text{ PHR}) \\ P & 11\text{-}100 & (50, 60, 70 \text{ PHR}) \end{cases}$

NSA = Nonenyl Succinic Anhydride
HHPA = Hexahydrophthalic Anhydride

Beta-S = Butenyldimethyltetrahydrophthalic anhydride
BDMA = Benzyldimethylamine
Epoxy Resin = SHELL EPON 828

Slide 8. MATERIALS CHART
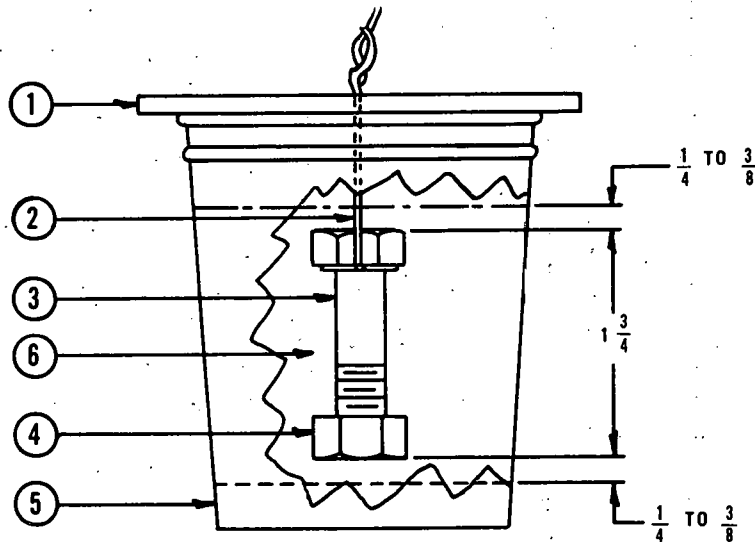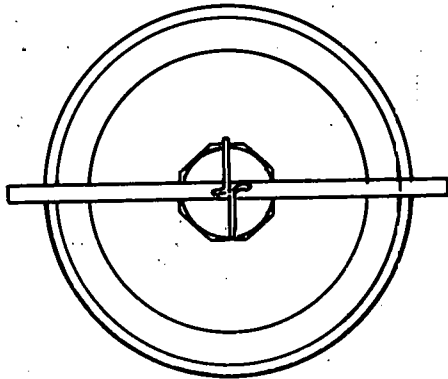
1. Cross Support

2. Copper wire AWG, 1 piece, 1-1/2 turns around bolt

3. Steel cap screw 1/2-13 x 1-1/2

4. Steel nut 1/2 - 13

5. Hot drink paper cup, 6 oz. capacity, unwaxed

6. Material to be tested

A. Apply release agent DC-4 or other Silicone grease to insides of cup, remove excess.

B. Clean steel cap screw and nut (no plating) with toluene and clean paper towel. Air dry.

C. Suspend nut and bolt assembly with copper wire per print.

D. Pour in Catalyzed test compound to height shown (1/4 to 3/8") over bolt top.

E. Cure for time and temperature requirements of compound.

F. Remove paper cup from casting.

Slide 9. THERMAL SHOCK TEST SPECIMEN

The thermal cycling equipment required for these tests is a forced-convection cold box capable of maintaining temperatures as low as -90°F. This system is preferred to immersion in a dry ice-alcohol bath, which is considered to be so severe as to be unrealistic. The series of thermal cycles finally selected proved to be a most severe one; it is presented in Slide 10. On the poorest resins, cracking usually occurs while the sample is being chilled. Improved systems which do not crack during chilling may cleave or actually shatter when suddenly exposed to a room temperature environment. Two pictures of cracked test specimens are shown in Slide 11 along with an uncracked specimen. More frequently than not, it has been observed that failure of one specimen at a given low temperature cycle indicates that, during continued cycling within the same temperature limits, the other specimens of that set will fail. Often, all six specimens of one formulation would fail simultaneously. Qualification was based on five samples out of six of any one formulation surviving the entire series of cold cycles. Most groups of samples either completely survived or completely failed during cycling over the selected range.

The results of the testing program were gratifying in view of the direct nature of the evidence. Slides 12, 13, and 14 illustrate in detail the exact formulations used and the results obtained from the thermal cycling procedures. Slide 12 specifically presents data based on the use of NSA with each of the selected polyols. In a like manner Slide 13 presents data based on HHPA, and Slide 14 presents data based on the use of Beta-S.

A review of the data reveals several interesting facts, all of which correlate well with theory. Neither NSA or Beta-S perform as well with the various polyols as does HHPA. A review of their structures as shown on Slide 7 reveals the presence of inert hydrocarbon side-chains. These side-chains increase the fluidity of the uncured system measurably and also decrease the elastic modulus to the point of flexibility. Unfortunately, however, they also tend to lessen the cohesive character of the cured structure by failing to contribute to the cross-linking network. On the other hand, HHPA does not have any side-chains, and practically its entire bulk is utilized to contribute to the cross-linking density of the resin.

Comparison of the results obtained with the various polyols (Slide 6) will also allow for some correlation of structures with physical properties. The higher molecular weight materials, though used in the same percentages by weight as those having low molecular weights, evidently do not contribute as well to the desired toughness at low temperatures. It is a fact that on a gram-for-gram basis, the low molecular weight polyols contain more mols of compound than the high molecular weight materials and will form more terpolymer with epoxy and anhydride. Conversely, the high molecular weight materials will contain less

 * I (a) Samples exposed at -65°F for four or more hours.
    (b) Samples exposed to room temperature for two or more hours.
    (c) Repeat steps (a) and (b) for a total of five cycles.

  II (a) Samples exposed at -80°F for four or more hours.
    (b) Samples exposed to room temperature for two or more hours.
    (c) Repeat steps (a) and (b) for a total of five cycles.

  III (a) Samples exposed at -90°F for four or more hours.
    (b) Samples exposed to room temperature for two or more hours.
    (c) Repeat steps (a) and (b) for a total of five cycles.

 * All samples to be stabilized at room temperature previous to start of test.

Slide 10. COLD SHOCK SCHEDULE
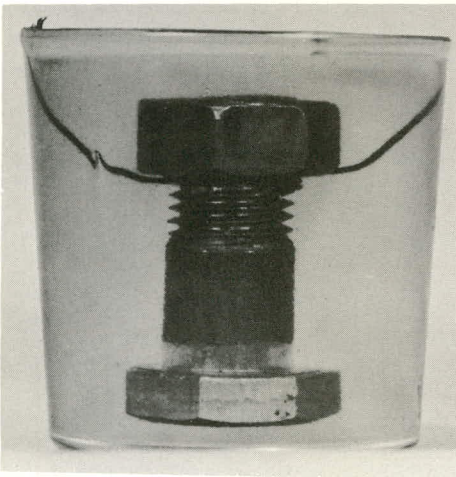
Slide 11. TEST SPECIMENS FOLLOWING THERMAL SHOCK

	①	②	③	④	⑤	⑥	⑦	⑧	⑨	⑩	⑪	⑫
Epoxy Resin	100	100	100	100	100	100	100	100	100	100	100	100
NSA	110	110	110	110	110	110	110	110	110	110	110	110
P 750	50	60	70									
P 1025				50	60	70						
P 11-80							50	60	70			
P 11-100										50	60	70
BDMA	2	2	2	2	2	2	2	2	2	2	2	2
-65°F shock	P	P	P	F	F	F	P	P	P	F	F	F
-80°F shock	F	F	F	-	-	-	F	F	P	-	-	-
-90°F shock	-	-	-	-	-	-	-	-	F	-	-	-

P = Passed
F = Failed

Slide 12. FORMULATIONS EMPLOYING NSA WITH SELECTED POLYOLS

	⑬	⑭	⑮	⑯	⑰	⑱	⑲	⑳	㉑	㉒	㉓	㉔
Epoxy Resin	100	100	100	100	100	100	100	100	100	100	100	100
HHPA	76	76	76	76	76	76	76	76	76	76	76	76
P 750	50	60	70									
P 1025				50	60	70						
P 11-80							50	60	70			
P 11-100										50	60	70
BDMA	2	2	2	2	2	2	2	2	2	2	2	2
-65°F shock	P	P	P	P	P	P	P	P	P	P	P	P
-80°F shock	P	P	P	F	F	F	P	P	P	F	F	F
-90°F shock	F	F	F	-	-	-	F	P	P	-	-	-

P = Passed
F = Failed

Slide 13. FORMULATIONS EMPLOYING HHPA WITH SELECTED POLYOLS

	㉕	㉖	㉗	㉘	㉙	㉚	㉛	㉜	㉝	㉞	㉟	㊱
Epoxy Resin	100	100	100	100	100	100	100	100	100	100	100	100
Beta-S	116	116	116	116	116	116	116	116	116	116	116	116
P 750	50	60	70									
P 1025				50	60	70						
P 11-80							50	60	70			
P 11-100										50	60	70
BDMA	2	2	2	2	2	2	2	2	2	2	2	2
-65°F shock	P	P	P	P	P	P	P	P	P	P	P	P
-80°F shock	F	F	P	F	F	F	F	P	P	F	F	F
-90°F shock	-	-	F	-	-	-	-	F	F	-	-	-

Slide 14. FORMULATIONS EMPLOYING BETA-S WITH SELECTED POLYOLS

mols per unit weight, forming less terpolymer with epoxy and anhydride, thereby permitting more copolymer formation between epoxy and anhydride. It is the terpolymer which contributes to flexibility and elongation at the lower temperatures; it is the epoxy copolymer which contributes to the rigidity, and it is the rigid portions of the polymeric matrix which tend to induce failure during occasions of severe thermal stress.

In addition to the advantages apparently inherent in the use of low molecular weight flexibilizers, it is also notable that the triol, P 11-80, was noticeably superior to the diol, P 750. Although the molecular weights of these compounds are almost identical, the triol nevertheless proved to be the best at the lower testing temperatures. Evidently, the triol contributes to a greater cross-linking density in cured structures than the diol which can only contribute linearity.

The outstanding systems are No.'s 20 and 21, on Slide 13, each of which include HHPA and P 11-80. Since No. 21 was somewhat soft at elevated temperatures, it was decided to compromise between the two. Slide 15, part 1, shows the basic formula finally selected. This combination was somewhat awkward to use, however, and to facilitate more effective and dependable field usage, the chosen formulation was so arranged as to form a two-part kit (Slide 15, part 2). The new arrangement can be used far more efficiently and with less basic control on the part of supervision, since it lends itself well to both hand and machine mixing. It would be well to emphasize here, however, that this semi-rigid inspection resin (SRIR) is hardly proposed as a "universal casting resin." It was designed for a specific type of function at the Sandia Laboratories, i.e., to be used in the manufacture of inspectable castings which would survive thermal shock. In practical operations, the material is generally used over the temperature range of -80°F through 180°F. At higher temperatures, the material softens excessively, and devices so encapsulated may not meet vibration or acceleration requirements. However, there is no appreciable embrittlement in cured casting which have been aged through 220°F for periods up to 90 days.

A list of physical properties is presented on Slide 16, and they are not far different from what might be expected in a flexibilized system. Tensile, compression, and flexural strengths are all below that which would be obtained with rigid materials, and impact strength is considerably higher than that of rigids. Electrical properties are presented on Slide 17. Of particular interest are the high values obtained for volume resistivity over the range of temperatures. Semi-rigid epoxy systems have usually been observed to have low volume resistivity at elevated temperatures, and it is gratifying to note the improvement

			Part A	Part B
Epoxy Resin	100	Epoxy Resin	100	--
HHPA	76	HHPA	---	76
P 11-80	66	P 11-80	22	44
BDMA	2	BDMA	---	2
			122	122

Slide 15.   (1) BASIC SEMI-RIGID FORMULA*   (2) KIT FORMULA*

*Parts by weight

Tensile strength (ASTM D 638), psi   3,900
Elongation (%)   115
Modulus (psi x 10^6)   0.09

Compressive strength (ASTM D 695), psi   5,600
Compression at yield (%)   5.4
Modulus (psi x 10^6)   0.19

Impact strength (ASTM D 256-56), ft lbs/in. notch   0.66

Coef. of expansion (ASTM D-696), in/in/°C
    -65°F (-54°C) to 72°F (22°C) 67×10^{-6}
    72°F (22°C) to 165°F (74°C) 142×10^{-6}

Specific gravity (D 792)   1.18

Slide 16. PHYSICAL PROPERTIES CHART

Dielectric Constant (ASTM D 150)

10^3 cps	3.67
10^6 cps	3.46
10^7 cps	3.29

Dissipation Factor (ASTM D 150)

10^3 cps	0.0210
10^6 cps	0.0330
10^7 cps	0.0300

Volume Resistivity (ASTM D 257)

72°F , ohm cm	2.0×10^{14}
100°F , ohm cm	1.7×10^{14}
150°F , ohm cm	2.0×10^{13}
200°F , ohm cm	9.3×10^{10}
250°F , ohm cm	2.4×10^{9}
300°F , ohm cm	3.7×10^{3}

Slide 17. ELECTRICAL PROPERTIES CHART

in this respect. A particularly undesirable feature of the semi-rigid system is the high coefficient of expansion. Since steel and aluminum have far lower values (10×10^{-6} and 25×10^{-6} in/in/$^{\circ}$C respectively), the semi-rigid resin can not be successfully packaged within integral metal containers. At low temperatures, the resin will shrink excessively and tend to cleave from the walls of the metal container, usually leaving a path for moisture penetration and resultant electrical failure. Because of this feature, the Sandia Laboratories usually package their embedments within molded plastic shells selected for high strength and impact resistance. The plastic shells have coefficients of expansion considerably higher than steel or aluminum, and, at low temperatures, cleavage from the walls has been the exception rather than the rule. Plastic shells can also be molded to include desirable mounting fixtures, and, because of the high strength-to-weight ratio of selected molding compounds, the substitution of plastic for metal shells usually produces superior results. Where embedments are required which are fully inspectable from all directions, castings will have to be made in removable molds. As in all cases, it is strongly recommended that preliminary models be made in order to contend with the usual difficulties prior to production runs.

A particularly revealing photograph can be noted on Slide 18. Here we see 6-inch slugs of aluminum and steel packaged in the semi-rigid resin and contained within an integral molded plastic shell. This package has survived the same series of cold shocks which were used on the nut-and-bolt test samples and does not show any signs of cleavage from the walls.

A complete process for using the semi-rigid resin will be found at the end of this paper. Interested packaging engineers will find that they now have another useful material to include in their ever growing list of embedment resins.
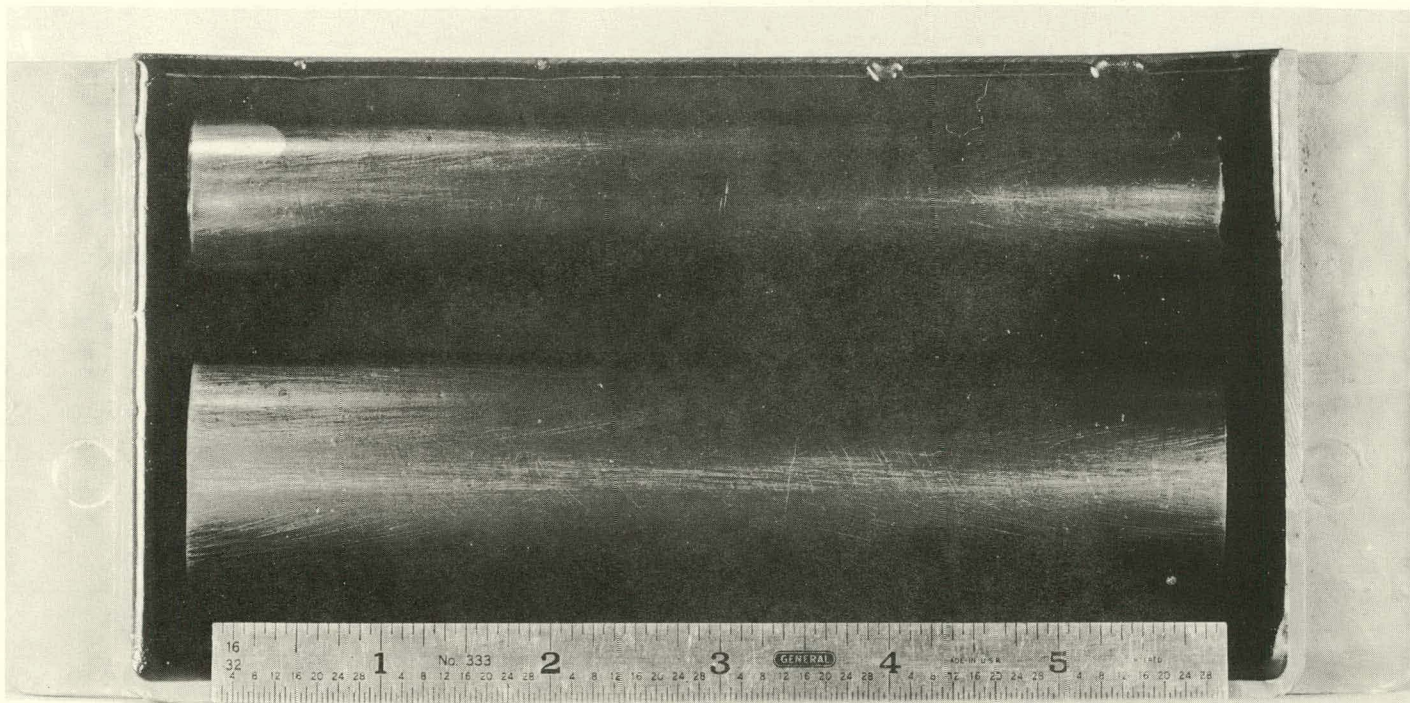
Slide 18. SLUGS OF STEEL AND ALUMINUM CAST IN SEMI-
RIGID RESIN WITHIN MOLDED PLASTIC SHELL

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APPENDIX A

PROCESS SPECIFICATION FOR SEMI-RIGID INSPECTABLE RESIN

APPENDIX A

PROCESS SPECIFICATION FOR SEMI-RIGID INSPECTABLE RESIN

1. SCOPE

1.1 This specification designates the requirements for embedding electrical components in an unfilled, transparent, shock-resistant epoxy resin.

2. APPLICABLE DOCUMENTS

2.1 There are no other applicable document.

3. REQUIREMENTS

3.1 Formulation - The embedding compound shall be formulated from the materials listed below, taken in the requisite proportions on a weight basis. The tolerance on weights of materials shall be plus or minus one per cent.

Part A	100 parts by weight
Part B	100 parts by weight

3.2 Mold Preparation - The mold and electrical components shall be preheated to a temperature of $130° \pm 5°$F at time of pouring of the embedding compound. When the mold is not an integral part of the component, the mold shall be clean and coated with a thin, even film of a suitable mold-release agent and allowed to dry, (see Section 6.3.2).

3.3 Mixing of Encapsulating Compound - For best results during the subsequent deaeration step, it is recommended that a container whose diameter is approximately equal to its height and whose volume is at least four times the volume of the mixed compound should be used for mixing. The requisite amounts of each component shall be combined in accordance with the following procedure.

(a) Each component of the embedding compound shall be thoroughly mixed in its shipping container before use. If crystallization has occurred, the material shall first be warmed to $130°$F.

(b) Both the Part A and Part B components shall be prewarmed to $130° \pm 5°$F before combining. Caution: The shelf life of the B component at $130°$F is twenty-four hours. Therefore, only that quantity of B component which is required for the day's production should be heated at one time.

(c) Combine the two components of the embedding compound and mix thoroughly until the resulting mixture is homogeneous.

3.4 <u>Deaeration of Embedding Compound</u> - As soon as mixing is complete the warm mixture shall be placed in a vacuum chamber and evacuated to a pressure of 1 to 3 mm Hg absolute. This reduced pressure shall be maintained for at least two minutes after the foam collapses. The deaerated resin mix shall be used in the subsequent steps as soon as possible.

3.5 <u>Pouring of Embedding Compound</u> - At the time of pouring, the embedding compound shall be at a temperature of $130^o \pm 5^oF$. If necessary, the activated resin mix shall be reheated immediately prior to pouring. The electrical components shall be encapsulated in accordance with one of the procedures outlined below. In no instance shall the pouring operation be completed later than one hour after the two resin components have been combined (Step 3.3 (c)).

3.5.1 <u>Vacuum Filling Process</u> - The preheated mold and components shall be placed in a vacuum chamber and evacuated to a pressure of 1 to 3 mm Hg absolute. Maintain this reduced pressure for at least two minutes to degas the components. After the degassing is complete, and while under this reduced pressure, the warm deaerated resin shall be introduced into the mold. The mold shall be filled to the height indicated on the product drawing. During the filling operation, the absolute pressure shall be no greater than 10 mm Hg. After the mold has been filled the absolute pressure shall be allowed to return to atmospheric. Any bubbles which appear at the surface of the filled mold prior to gelation of the embedding compound shall be broken by spraying the surface of the compound with either toluol or methyl ethyl ketone, using an atomizer-type sprayer. (NOTE: Alcohols must not be used.)

3.5.2 <u>Atmospheric Pressure Process</u> - The preheated components and mold shall be partially filled with the warm deaerated embedding compound. Degas the mold and components by placing them in a vacuum chamber and evacuating to a pressure of 1 to 3 mm Hg absolute, maintaining the pressure for at least five minutes. Allow pressure to return to atmospheric. For some units it may be necessary to add another portion of embedding compound and repeat the evacuation step. Add additional embedding compound to bring level up to height indicated on the product drawing. No further evacuation is necessary after this point. Any bubbles which appear at the surface of the filled mold prior to gelation of the embedding compound shall be broken by spraying the surface of the compound with either toluol or methyl ethyl ketone, using an atomizer-type sprayer. (NOTE: Alcohols must not be used.)

3.6 <u>Cure</u> - The filled mold shall be cured for a minimum of fifteen hours in a forced convection oven at a temperature of $130^o \pm 5^oF$ plus an additional cure of at least twenty-four hours at a temperature of $160^o \pm 5^oF$. If a separate mold is used, the casting may be removed when it has cooled to room temperature.

3.6.1 The following alternate curing schedule may be used only when specifically called out on the product drawing:

Cure for a minimum of fifteen hours in a forced convection oven at a temperature of $130^o \pm 5^oF$, plus an additional cure of at least eight hours at a temperature of $180^o \pm 5^oF$.

3.7 <u>Finishing</u> - Sharp corners or edges of the cured compound shall be rounded or chamfered.

4. SAMPLING, INSPECTION, AND TEST PROCEDURES

4.1 Not applicable

5. PREPARATION FOR DELIVERY

5.1 Not applicable

6. NOTES

6.1 Intended Use - This encapsulation process is intended for the embedment of electrical and electronic components or other applicable units where it is desirable to have a transparent embedment system wherein the components may be visually inspected after encapsulation, and where the encapsulating material must remain semi-rigid at subzero temperature. Normally the total weight of embedding compound poured is less than ten pounds per casting or embedment.

6.2 Shelf Life - The shelf life of each component in its unopened shipping container is approximately six months at room temperature (90°F maximum). Since both components are moisture sensitive, they should be kept in tightly sealed containers. When opened for removal of contents, containers should be resealed. If either component is clouded by a precipitate and the material becomes clear upon heating to $130° \pm 5°F$ then that component is still usable. If the cloudiness does not disappear after heating, then the entire container of material shall be discarded.

6.3 Sources of Supply

6.3.1 Mold Release - A mold release agent which has been found satisfactory when casting in a separate mold is:

> Garan 225   Ram Chemical Company
> Gardena, California

6.3.2 When specifically allowed on the product drawing, the following silicone-type mold release agents may be used:

> DC 7   Dow Corning Corporation
> Midland, Michigan
>
> DC 20   Dow Corning Corporation
>
> SR-53   General Electric Company
> Silicone Products Department
> Waterford, New York

6.4 Caution: The hardener component (Part B) of this embedding compound is known to be toxic. Hence, adequate ventilation should be provided in the handling of these components to prevent undue exposure to vapors. Ingestion or skin contact with these materials shall be avoided. If accidental skin contact should occur, the exposed areas should be washed immediately with soap and water.

APPENDIX B

MATERIALS SOURCES

APPENDIX B

MATERIALS SOURCES

Polypropylene Diols

| P | 750 | Dow Chemical Company |
| P | 1025 | Union Carbide Chemicals Company |

Polypropylene Triols

| P | 11-80 | Dow Chemical Company |
| P | 11-100 | Dow Chemical Company |

Anhydrides

Nonenyl Succinic Anhydride   Humphrey-Wilkinson Corporation

Hexahydrophthalic Anhydride   National Aniline Division
Allied Chemical and Dye Corp.

Beta-S   Heyden Newport Chemical Corp.