

AN EXAMINATION OF EPOXY SYSTEMS USEFUL IN PACKAGING "HIGH G" RADIO TELEMETERS

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ABSTRACT

The electrical components used in "high g" (500,000 g) telemetry packages will survive gun launchings only if potted in suitable materials. The materials considered for this application were: polyesters, epoxies, silicones, phenolics, and urethanes. The epoxy materials appeared to fulfill most of the requirements for this application. This report describes epoxy materials, their curing, methods of potting, and the tests performed to select an epoxy material for embedding telemetry packages launched from hypervelocity guns in aeroballistic ranges.

Contrails





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1.0 INTRODUCTION

The performance of aeroballistic range telemeters described in Refs. 1 and 2 was largely a function of the encapsulating material used to secure the electrical components during "high g" launchings. Polyesters, epoxies, silicones, phenolics, and urethanes were all considered, but the epoxy resins seem best suited to protect the electrical components.

The disadvantages of the different materials are as follows: (1) Polyesters have a high rate of shrinkage which causes many cracking problems when parts are embedded in the resin. (2) Acid catalyst and water vapor produced during curing of phenolics can produce severe corrosive action. (3) Silicones and urethanes do not have the rigidity or strength required. (4) Bubble-free castings of filled epoxy resins are difficult to achieve. The difficulty in achieving void-free castings of fill epoxy resins was overshadowed by the excellent mechanical and electrical properties, low shrinkage, and the low temperatures required for curing. Several methods of potting epoxy resins have been devised to almost entirely eliminate air bubbles in the end product.

In general, unfilled epoxy resins are not suitable for embedding electronic components for "high g" applications. Low density fillers can be added to the resin with, at worst, only a slight decrease in compressive yield strength*, an important property of the resin. The low density of the resin is probably the most important property, however, because it determines the external mechanical loading which the potted components receive for a given launching acceleration.

Since large volumes of filler were incorporated into the resin, peak exotherm would be reduced and almost any room temperature curing agent could be used. The literature available generally considers only exothermic heat and heat distortion temperature in the selection of a curing agent.

The curing agent giving maximum compressive yield strength with a room temperature cure would be most desirable. Information on this aspect is still being sought. The fact that a different activator will affect

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^{*}Compressive yield strength for the purposes of this work, is defined as the stress at which the material starts yielding and not the usual 0.2 percent offset.



compressive yield strength has been suggested, but the activator which would give optimum results has not been determined.

The compressive yield strength and density of the epoxy resin systems were determined before the resin systems were used in potting telemeters. The tests devised are described in this report. From the test firings it was shown that a low density epoxy was more suited for potting telemeters than a heavier material with a higher strength because mechanical loading would apply pressure to components of the telemeter. The best material would be weightless and have an infinite compressive yield strength. Emerson and Cuming's Stycast 1090 appears to approach this ideal material more nearly than any of the materials tested.

2.0 EPOXY RESIN SYSTEMS

2.1 RESINS

Epoxy resins are thermosetting plastics that become hard, infusible solids when converted by a curing agent. The systems may be visualized as three-dimensional cross-linked networks. The relative toughness of epoxy resins, as compared to other thermoplastic resins, is attributed to the short distance between cross-linking points and the presence of integral aliphatic chains (Ref. 3).

Epoxy resins differ from many thermosetting materials in that they give off no by-product during cure. Cure is by direct addition, and shrinkage of unfilled epoxy resins is ordinarily less than two percent. Adding fillers to the resin will lower this value still further, and large metallic inserts can be embedded without cracking the resin during curing.

Epoxy resins are generally produced by reacting epichlorohydrin and bisphenol A in the presence of a caustic (NaOH). The difference in molecular weights of epoxy resins is determined by the different amounts of caustic used. The general formula for an epoxy resin made by this reaction may be written as follows:

$$\begin{array}{c} cH_{2}^{0} - cH_{2} + c$$



Several important characteristics of epoxy resins which may be used as guides to their use are:

- 1. viscosity
- 2. epoxide equivalent
- 3. hydroxyl equivalent and
- 4. average molecular weight and molecular weight distribution.

The viscosity of the resin is generally a guide to the amount of filler that can be incorporated in the resin system. In some cases, however, even if the resin has a very low viscosity, it may not be possible to incorporate certain fillers and obtain a homogeneous mixture. The viscosity is also a good index to the molecular weight of the resin except in cases where the resin contains a diluent or filler. The lower molecular weight resins have the lowest viscosities.

The epoxide equivalent and hydroxyl equivalent are quantities representing the weight of resin in grams containing one gram chemical equivalent of epoxy and one equivalent weight of the hydroxyl groups, respectively. These properties of the resin are needed to determine the amount of activator required for curing the resin.

The molecular weight presented in commercial resins is usually an average value. The resin actually consists of many molecules having different molecular weights with molecules of one particular weight being predominant. "The properties of the cured resin (i. e., tensile strength, heat-distortion temperature, etc.) would be affected by the actual percentages present." (Ref. 3)

2.2 FILLER MATERIALS AND MODIFIERS

An extremely wide range of end properties can be obtained in a cured resin by the choice of resin and curing-agent combinations. An even wider range of properties is available when fillers are added to the resin. Different fillers affect a given resin property in varying degrees, but the nature of the effect is often similar regardless of the filler employed. It is believed that the primary function of any filler is simply a bulk effect (Ref. 4).

Fillers greatly improve the thermal properties of epoxy resins. The thermal conductivity of the resin generally increases and the thermal expansion coefficient decreases when fillers are added to epoxy resins. However, some low density fillers decrease thermal conductivity. More



important, fillers reduce shrinkage and exotherm and also increase the pot life of the system. The latter properties are very important when embedding electrical components if crack-free packages are desired.

There are two types of fillers used in epoxy resins, reinforcing and non-reinforcing. Non-reinforcing fillers are sometimes referred to as bulk fillers...."Fillers generally improve compressive yield strength but decrease ultimate compressive strength...." (Ref. 5). This phenomenon is illustrated further in this paper. Reinforcing or fibrous fillers increase impact strength and tensile strength, whereas bulk fillers have an opposite effect.

The effect of fillers on electrical properties is not as pronounced as the effect on mechanical properties. The dielectric strength of the cured system is not normally improved by filler addition. The use of low-density fillers can, however, improve electrical properties somewhat.

After all water vapor is removed from the filler, it can generally be mixed into a resin by simple mechanical mixing. The maximum filler concentration is a function of the viscosity of the resin and the filler used. The maximum concentration of filler can be increased by increasing the resin temperature if pot life of the mixture is not a critical factor.

Not all fillers can be easily mixed into a resin. Heavier fillers have a tendency to settle, whereas lighter fillers float near the surface. If there is not a high adherent force between the filler and resin because of improper filler wetting, the separation effect becomes even more pronounced. If reduced viscosities are desired to incorporate more filler or improve handling properties, diluents can be added to a resin with some sacrifice to mechanical properties. Diluents may hinder the wetting of certain fillers, for example, Eccospheres*, and increase the separation problem. Additional curing agents must be added when reactive diluents are used. Reference 6 is a comprehensive report on the use of reactive diluents to lower the viscosity of epoxy resins.

2.3 CURING EPOXY RESINS

There are five terms used to describe materials which will react with and solidify epoxy resins: curing agent, converter, hardener,

^{*}Trade name of Emerson and Cuming, Inc. for hollow glass microballon filler material.



activator, and catalyst. All the terms, excluding the last, can be used interchangeably, and all describe materials which become part of the end product. The term catalyst should be used only when describing a material which causes epoxy units to react with one another. Some curing agents have molecular structures containing atomic groupings which take active part in the reaction and also groups which would be considered catalytic in nature, for example, Diethylamino Propylamine.

An epoxy resin system can be cured by three reactions*: (1) direct linkage between epoxy groups, (2) linkage of epoxy groups with aromatic or aliphatic hydroxyls, and (3) cross-linkage with curing agents through various radicals. The different types of curing agents which can be used with epoxy resins can be broadly divided into three classes: (1) organic nitrogen compounds, usually amines, (2) organic acids or acid anhydrides, (3) certain other resins, such as polysulfides and polyamides. The amine group, however, is by far the most important group for electronic packaging. Acid anhydrides generally require curing temperatures far too high for survival of electronic components; polysulfides and polyamides produce flexible systems when used as curing agents. Flexible systems are undesirable for "high g" telemetry encapsulating materials because they allow intra-package component movement during launching (Ref. 2).

Amine curing agents can be classed, according to structure, as aliphatic or aromatic. Aliphatic describes an organic chemical not using the benzene ring as a building block. Curing agents also are classified under sub-headings as primary, secondary, or tertiary. The sub-terms deal with the number and location of the nitrogen or amine $(-N H_n)$ where n equals the number of hydrogen atoms) groups in the molecule of amine curing agents.

Commercially available, aromatic amines are undesirable for electronic packaging because they generally require high temperature post-cures to achieve thorough curing. A number of modified compounds based on commercial amines are available for curing epoxies. These materials have better handling characteristics (e.g., more convenient stoichiometric ratios, extremely fast room temperature cures, reduced irritation potential, etc.), but the cured systems have somewhat reduced mechanical and chemical properties.

The aliphatic polyamine curing agents are the most desirable for curing epoxy resins used to embed electrical components of "high g"

^{*}One or more of the different reactions can be taking place during the cure cycle of an epoxy system.



telemeters. Their advantages are good room temperature cures, high strength, and high versatility as systems. Table 1 lists the more common, polyfunctional, primary aliphatic amines with some comments about their usage.

The percentage of activator necessary to cure an epoxy can be calculated for cross-linking curing agents. Generally one epoxy group must be allowed for each active hydrogen atom of the reagent. The stoichiometric ratio would then be calculated by dividing the epoxy equivalent of the resin by the molecular weight per active hydrogen of the activator. The calculated amount should result in optimum properties in the cured system, and variations from it should not be greater than 10 to 20 percent. Excessive amine curing agent will tend to stop chain building at low molecular weights, thereby embrittling the resin. Too little curing agent will fail to provide adequate cure.

The amount of curing agent cannot be calculated when using catalytic curing agent or curing agents containing unblocked tertiary amine groups. The amount of a particular catalytic curing agent to employ is not considered critical and, in most cases, can be varied by as much as 50 percent from the experimentally determined amount without seriously affecting the properties of the cured system.

Not only is it necessary to employ the proper amount of curing agent to achieve thorough cross-linking, but also it is necessary to have information about the curing time and cure temperature. In many cases, low temperature gelling appears to promote linear polymerization; the long-chain molecules so formed, later, under the influence of thermal agitation induced by high temperature post-curing, continue to react and provide a higher degree of cure. Long post-cures may be necessary to complete the reaction if the initial gelling temperature cycle is not correct. Similarly, in other cases, low-temperature gelling appears to create molecules with steric hindrances or a degree of unsymmetry such that even high temperature post-cures do not improve the degree of cure (Ref. 7). Cure cycles are generally best determined for each application.

In general, the desired castings properties can be affected in several ways. Ultimate compressive strength can be moderately improved with higher temperature cures, whereas compressive yield strength will show slight improvement with low temperature cures. During the early stages of curing several epoxy resins go through a stage where they are hard and brittle and easy to break in rapid impact yet are very flexible and rubbery when handled slowly. This is definitely an undercured stage which cannot be visually revealed. Resins in this



state are capable of giving unusually high tensile strengths but are neither usable nor physically stable (Ref. 8).

3.0 POTTING TECHNIQUES

3.1 POURING

The simplest method of potting telemetry systems consists of placing the telemeter into an outer shell and pouring the resin around the electrical components. Telemeters potted in this manner have often contained very large voids which resulted in failure of the telemeter and sometimes failure of the entire package during a "high g" launching. The voids were caused by the small passages through which the potting materials were required to flow; the voids are a direct result of the telemeter component density. Because the viscosity of the resin could not be sufficiently reduced to eliminate void formations, this method of potting was abandoned.

3.2 VACUUM POTTING

A detailed outline of this method of potting can be found in Ref. 1. A summary of the technique follows: Small holes were drilled in the closed end of the body shell. The telemeter was inserted with the battery leads protruding from the open end, and a potting fixture was placed on each end of the model and clamped in place (Fig. 1). A vacuum line connected to the top fixture pulled the epoxy through the package and evacuated the epoxy resin. When the cup and reservoir were filled, the bottom tube was clamped shut. The vacuum was maintained until just before solidification; then about 55-psi air pressure compressed the epoxy for the remainder of the cure cycle. This method of potting produced void-free epoxy castings, but it was very time-consuming and required molds which had to be machine-cleaned regularly.

3.3 VIBRATION VACUUM POURING

This technique appears to produce adequate castings with the least effort. It may be summarized as follows: The epoxy is mixed and evacuated in a vacuum chamber prior to pouring. The epoxy is poured into the outer shell of the telemeter package mounted on a vibration table. After sufficient filling, the telemeter is vibrated into the epoxy; then the completed unit is placed in a vacuum chamber. Just before the



epoxy hardens, the chamber is pressurized until the final cure is completed. This process can be used for medium viscous or thixotropic epoxy systems.

The vibration table used is a rectangular plywood platform mounted on springs at each of the four corners. To the bottom of the board is mounted a motor with an unbalanced shaft. The frequency of vibration can be varied by the speed of the motor, and the amplitude can be varied by adjusting the spring tension. The vibration frequency is generally adjusted to 1800 cycles/sec and the amplitude, approximately 0.03 inches; however, it should be noted that the frequency and amplitude required may vary for different epoxies and should be adjusted to fit the situation.

4.0 MATERIALS TESTED

4.1 STYCAST 1090, STYCAST 4030 CM, AND ECCOMOLD L-28

Stycast 4030, Stycast 1090, and Eccomold L-28 are manufactured by Emerson and Cuming, Inc. Stycast 4030 CM was undesirable because of its high specific gravity (1.55) and low yield strength. Eccomold L-28 is a general purpose epoxide laminating resin. Stycast 1090 is a low weight (specific gravity about 0.8) epoxide casting resin for electronic embedments. This material has a high viscosity and should be heated to a temperature near 140°F before mixing. The low density was obtained by the addition of Eccosphere filler by the manufacturer.

4.2 EPON 828, EPON 815, ARMSTRONG C-7, AND ARALDITE 502

Epon 828 (Shell Chemical Company) and Armstrong C-7 (Armstrong Products Company) are pourable, unfilled, epoxy resins at room temperature and are widely used for embedding electrical equipment. Epon 815 and Araldite 502 (Ciba Products Corporation) are very similar to Epon 828; Araldite 502, however, contains a plasticizer, and Epon 815 contains a diluent.

4.3 MARASET 121E

Maraset 121E (The Marblette Corporation) is a general purpose resin filled with silica. The density of the cured rigid system was 1.53 gram/cc. This material was tested in gun-launched projectiles



containing telemeters and proved undesirable before static testing was initiated. It is believed that the high density of this material excessively loaded the electrical components.

4.4 RP 1710

RP 1710 (Ren Plastics, Inc.) is a slightly filled laminating epoxy. It was tested using the company's recommended safety hardener, which is believed to be an amine-resin adduct. A different activator would probably increase the mechanical properties of this material.

4.5 ECCOSPHERES FILLER

Eccospheres are hollow glass microballoons with diameters ranging from 30 to 300 microns and a wall thickness on the order of a few microns. Reference 9 describes Eccospheres in detail, and Ref. 10 describes some of the problems of using Eccospheres as a filler for epoxy resins.

5.0 MATERIAL TESTS

5.1 STATIC COMPRESSION TESTS

The purpose of this test was to obtain static stress-strain curves of all the materials which were being considered for potting "high g" telemeters. The epoxy slugs used for this test were nominally 1.125 inches in diameter by 1.5 inches in length. All slugs were cured at room temperature with no post-cure. At least two weeks elapsed between pouring of slugs and time of testing. After the density of each slug had been recorded, four SR-4 type A-7 strain gages were mounted. and placed at 90-deg intervals around the cylindrical slug. The strain gages were attached in a four-component bridge and connected to a carrier amplifier. The strain, represented by the output voltage of the amplifier, was then fed to the "X" axis of an oscilloscope. The load, measured by a Baldwin SR-4 type U-1 load cell, was similarly recorded on the "Y" axis of the same oscilloscope. The assembled apparatus is shown in Fig. 2. Stress-strain curves obtained from this apparatus were used for comparison purposes only, and data were not considered reliable beyond the yield point of the material. The data presented in Fig. 3 were obtained using a commercial testing machine and can be considered comparable with other published data of this sort. The percentage and type of activator was as recommended by the manufacturer.



5.2 DYNAMIC TESTS

The materials having the highest compressive yield strengths and the material having the lowest density were tested in the cold gas gun range as described in Ref. 1. Simple r-f oscillators were potted in Scotchply cups (Fig. 4) using each material and were launched from the cold gas gun. The in-flight frequency shift of the oscillator and the condition of the potting after recovery were indications of the performance of the potting material.

6.0 RESULTS

Partial results of the static and dynamic tests are summarized in Tables 2 and 3, respectively. Additional static test data have been tabulated in Figs. 3 and 5.

The compressive yield strength of epoxy resin systems was not adversely affected by the addition of Eccospheres filler. The ultimate compressive strength, however, was significantly decreased by the addition of this material. The results of these tests were in agreement with those of Ref. 3 but show little resemblance to those obtained by Kenneth Cressy (Ref. 11). The results obtained by Mr. Cressy indicate that the ultimate compressive strength, yield strength, and compressive modulus decrease with the addition of low density fillers. It was not stated in the report (Ref. 11) which fillers or epoxy systems were used; thus, a direct comparison cannot be made. It is possible that Mr. Cressy used mica or microballoons made of a material with a lower compressive yield and modulus than the epoxy.

The first four slugs presented in Table 2 were presumably made from the same material. Slug 1 was poured from the first supply obtained and had a density comparable to the value quoted by the manufacturer. This material was used for all dynamic tests obtained for material comparison. The latter three slugs were made from a later shipment and show a considerably greater density than quoted. No dynamic tests of this material have been made for comparison purposes.

Slugs 2, 3, and 4 were used to determine if the percentage activator could affect the compressive yield strength of the epoxy. The yield strength appeared to increase with decreasing phr of activator. Not enough data have been taken to determine at which phr activator would give the peak strength. The superior performance with the lower activator percentage might arise from a lower temperature cure resulting



from a lower peak exotherm and may not appear when smaller quantities, such as used to embed telemeter packages, are poured.

The epoxy formulations to be dynamically tested were selected on the basis of high strength or low density. From the results, it appears that the lower the density, the better the performance of the telemeter. The compressive yield strength will eventually become more important as higher launching accelerations are sought, but at present, the strength of the Stycast 1090 has proven adequate. It should be noted that when the materials were tested dynamically, the epoxy was contained in a very strong fiberglass cup; thus radial expansion of the epoxy material which enclosed the electrical components was restricted. Also, the loading was applied and removed in a very short time. Loading of this type generally increases greatly the yield strength of materials having stress-strain curves similar to Stycast 1090 (Refs. 12 and 13).

7.0 CONCLUSION

Epoxy resins have been generally accepted as electrical embedding materials. In "high g" electronic packaging, it is not only required that the epoxy resins have high strength, but it is also required that they have very low density. Stycast 1090 has been temporarily selected, but it is believed that a lower density formulation can be made if Eccospheres with a density of 0.3 gr/cc can be acquired. The Eccospheres used previously had a density of 0.5 gr/cc, near the value reported in Ref. 10. The density reported by the manufacturer was 0.3 gr/cc.

The addition of Eccospheres to epoxy resins was shown not to effect the yield strength of the resin until the resin formulation became too viscous to be used for electronic embedding. It has also been shown that, for Stycast 1090, the maximum yield strength is a function of the phr of activator used. It was shown that the compressive yield strength increased with decreasing phr of activator as low as 8 phr. The optimum percentage activator has not been established.

A new type of Eccospheres has recently been marketed (Eccospheres S1). It is believed that this new type will prove superior to those tested previously and will be tested in Armstrong C-7, because this resin has the highest yield strength of all the resins tested. If the density of the new Eccospheres approaches the quoted value, it should be possible to formulate a very low density, high strength system. If the density is significantly reduced, all future tests will be directed toward increasing the yield strength by varying the percentage activator



and the type of activator used. As the state of the art allows higher temperature curing, epoxy resins can be formulated with higher yield strengths, and the maximum launching capabilities of telemetry systems can thus be increased.

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TABLE 1

TYPICAL, POLYFUNCTIONAL, PRIMARY ALIPHATIC AMINE CURING AGENTS

Curing Agent	Phr*	Gel Time**	Curing Schedule	Remarks
Diethylene Triamene (DETA)	8-12	30 min	overnight at room temperature, several days for full cure***	good general purpose room temperature curing agent but fairly toxic
Triethylene Tetramine (TETA)	10-14	30 min	overnight at room temperature, several days for full cure	usually preferred over DETA
Diethylamino Propylamine (DEAPA)	4-8	3-5 hr	several days at room temperature	requires heat curing at moderate temperatures (185-200°F)
Dimethylamino Propylamine (DIMAPA)	!	!		

*Parts by weight per 100 parts of Epon 828 (Shell) or equivalent

^{**}For a 1-1b batch at an ambient temperature of 77°F

^{***}Tensile yield strength (0.2% offset) increased for 30 days then decreased slightly (Shell Technical Bulletin SP-23-A)



TABLE 2
STATIC COMPRESSION TEST RESULTS

No.	Ероху	Activator	Phr(1) Activator	Approximate Specific Gravity	Phr(2) Eccospheres	Compressive (3) Yield Strength (psi)	Compressive Yield at 0.2% Offset (psi)
1	Stycast 1090	Cat #9(4)	10	0.80		7,000	11,000
2	Stycast 1090	Cat #9 ⁽⁴⁾	8	0.92		8, 000	11,400
3	Stycast 1090	Cat #9 ⁽⁴⁾	9	0.92		7, 700	10,400
4	Stycast 1090	Cat #9(4)	10	0.92		7,400	10,400
5	Epon 815	TETA	12	1.20	None	9,000	12,000
6	Epon 815	TETA	11		22	7,800	10,500
7	Epon 828	TETA	10	1. 20	None	7,500	12,000
8	Epon 828	TETA	14	1. 20	None	5,000	7,000
Э	C-7	DETA	8	1. 26	None	12,000	>14,000
10	C-7	DETA	8	0.86	20	9, 000 ⁽⁵⁾	9,000
11	C-7	DEAPA(6)	7	1, 18	None	7,000	11,500
12	Araldite 502	TETA	10	1.15	None	7,000	
13	Stycast 4030cm	Cat #7	1	1.55	None	3,000	10,000
14	Marasett 121E	Hardener #6	5	1.53	None	+==	

⁽¹⁾ Parts by weight of activator per 100 parts of resin.

⁽²⁾ Parts by weight Eccospheres per 100 parts of resin.

⁽³⁾ The stress at which the stress-strain curve was no longer linear and not the usual 0.2% offset stress.

⁽⁴⁾ Catalyst #9 is a primary amine and not a catalyst as defined in this paper. Its percentage should not be varied by more than ±10% from the recommended 9 phr. The Stycast 1090 (2, 3, 4) used for determining the correct percentage of activator for optimum compressive yield strength were taken from a recent shipment and have not been dynamically tested. The specific gravity was not as quoted by the manufacturer (S, G. = .78).

⁽⁵⁾ The yield occurred at the ultimate because of the excessive amount of Eccospheres used. This material could not have been used for potting telemeters because of the difficulty in removing air voids.

⁽⁶⁾ The yield strength might have been increased if the slugs had been post-cured. All the slugs tested were cured at room temperature because they were to be used for potting telemeters with temperature sensitive components.

TABLE 3

TYPICAL DYNAMIC TEST RESULTS

Comments	potting cracked after recovery	potting cracked after recovery	working normally after recovery	missed catcher box and destroyed*	
Frequency Shift	-150 kc	no output	50 kc	5 kc	
Potting	C-7 Act. E	C-7 Act. A	C-7 Act. A Eccospheres	Stycast 1090	
Acceleration	285,000	274,000	540,000	532,000	
Telemeter No.	295	297	323	325	

*Telemeters potted with this material which were caught in the catcher box showed no ill effects from launching or recovery.



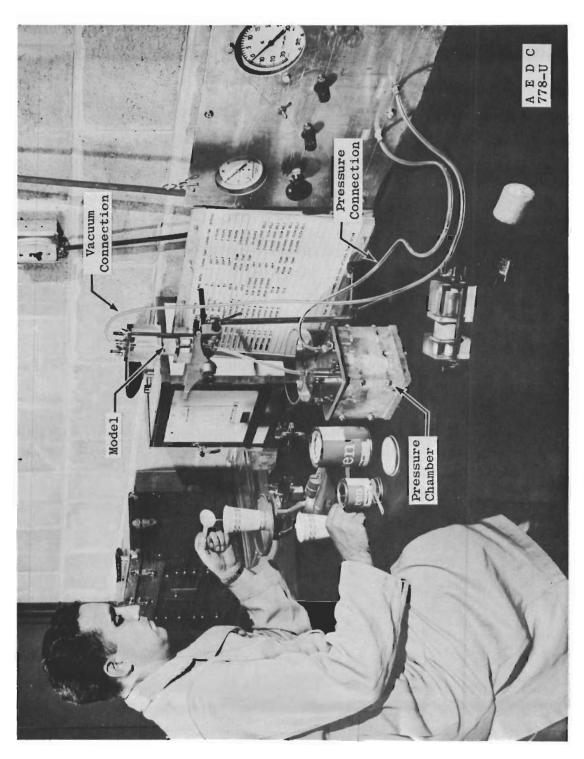


Fig. 1 Vacuum Potting Apparatus

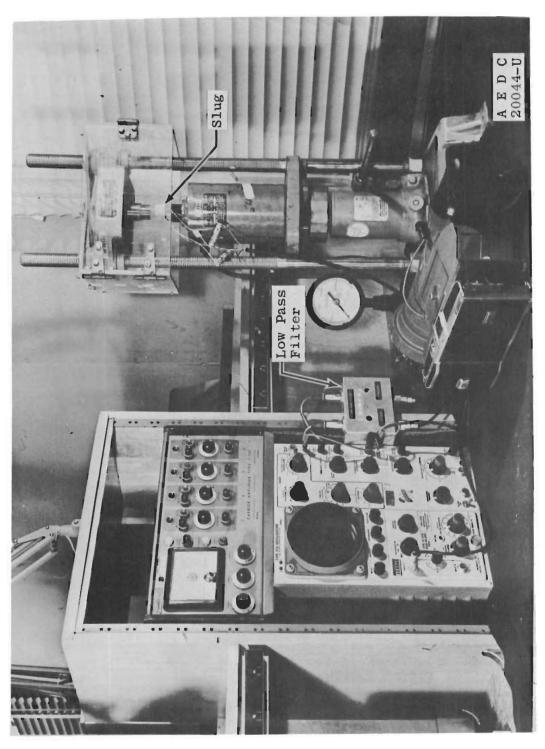


Fig. 2 Compression Test Apparatus

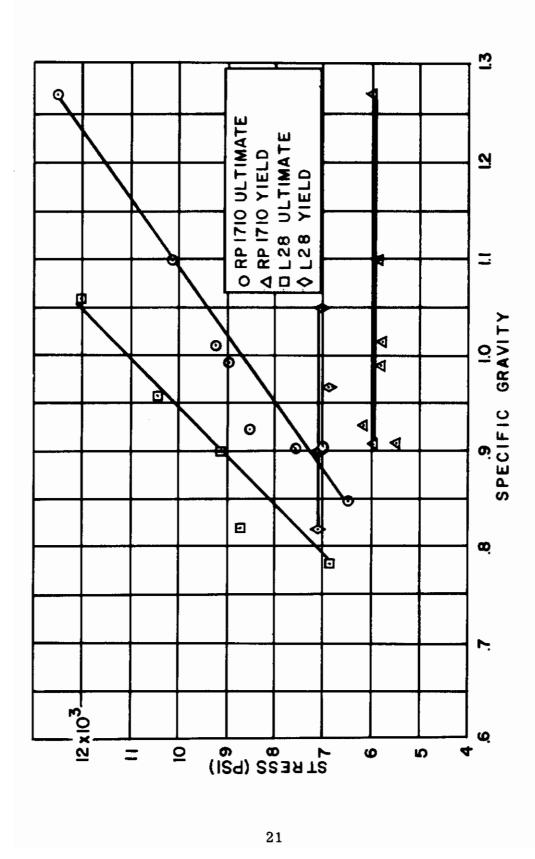
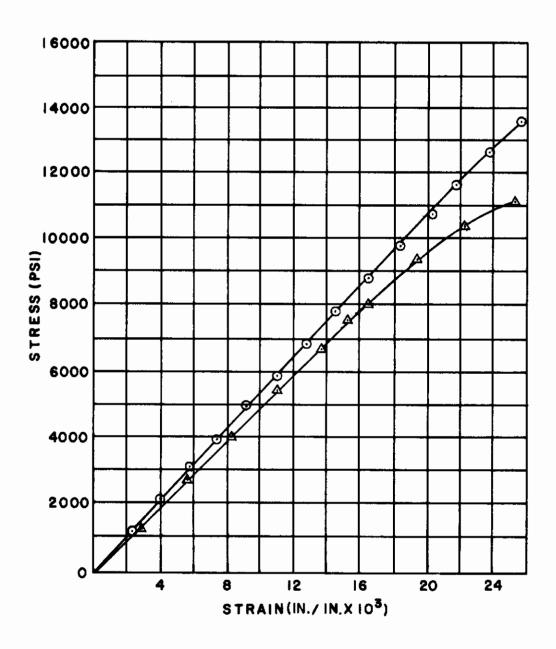


Fig. 3 Strength-Density Characteristics of Epoxy-Eccosphere Formulations



Fig. 4 R-F Oscillator and Scotchply Cup





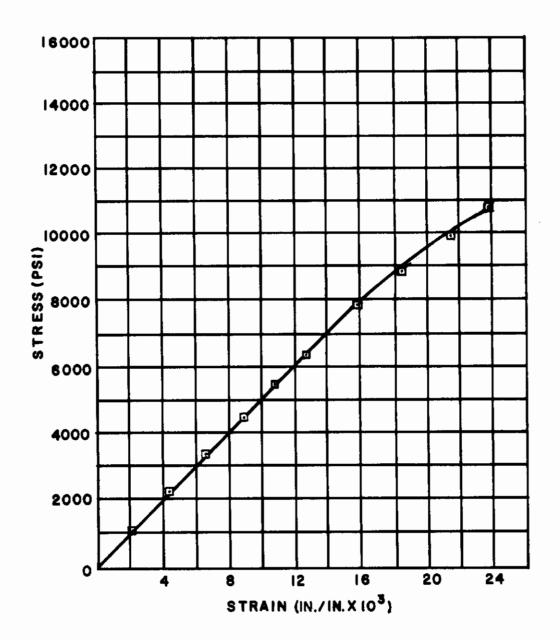
O ARMSTRONG C7-8phr. DETA

A ARMSTRONG C7-7phr. DEAPA

a. Armstrong C-7

Fig. 5 Stress Strain Diagrams





B STYCAST 1090

b. Stycast 1090

Fig. 5 Concluded