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THE SYNTHESIS OF A
LOW-DENSITY RIGID
EPOXY FOAM

BDX-613-407

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Final Report

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ABSTRACT

The various formulation routes investigated in the course of developing a one-component rigid epoxy foam called Capoxyfoam I are discussed. As a back-up epoxy foam for Ablefoam 5, Capoxyfoam I is potentially useful as an encapsulant for the components of a new electronic assembly to be used in a missile. When compared to Ablefoam 5, Capoxyfoam I was found equal or superior to its counterpart in physical, electrical, and processing characteristics.

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SUMMARY

The purpose of this project was to develop a suitable back-up material for Ablefoam 5, a one-component rigid epoxy foam system used to encapsulate the electronic components of a missile subassembly. This development followed an evaluation of the properties of Ablefoam 5 by Sandia Laboratories, Albuquerque.

The evaluation of Ablefoam 5 at Bendix Kansas City began with a literature survey of the basic chemistry of epoxy resins and epoxy formulations. Basic solvency tests of the material were initiated in the laboratory and revealed ammonium carbonate to be the active blowing agent. Gel permeation chromatography (GPC) was used to correlate the molecular weights of Dow Epoxy Novolacs DEN 431 and DEN 438 which are used in Ablefoam 5. GPC was also used to identify meta-phenylenediamine and methylenedianiline as the aromatic amine curing agents.

In terms of the formulation work, it was found that Dow's DEN 431 and Shell's Epon 828 epoxy resins were suitable in a 50:50 ratio. The organic reactive diluent 1,2-epoxy-3-phenoxypropane was added to the resins to reduce their viscosity to a workable consistency.

The various formulations investigated were screened for a suitable curing agent, cell stabilizer, and gel agent. The desired back-up epoxy foam for Ablefoam 5 was eventually developed and given an identification as Capoxyfoam I.

Since the ammonium carbonate blowing agent was found to effect a curing response when mixed with the epoxy resin, a method was developed to halt the curing action by quick-freezing the Capoxyfoam I premix.

Comparative chemical and physical analyses of Capoxyfoam I and Ablefoam 5 indicated nearly identical compressive strengths as a function of density at room temperature. However, Capoxyfoam I appeared stronger at the 200°F test temperature. Thermalgravimetric analysis (TGA) revealed Capoxyfoam I to be thermally more stable than Ablefoam 5 since weight loss for Capoxyfoam I occurred at a higher temperature.

Capoxyfoam I again proved superior to Ablefoam 5 when subjected to heat aging at 400°F, with Capoxyfoam I retaining 93 percent of its compressive strength as compared to 35 percent for Ablefoam 5.

Both the dummy and production units molded from Capoxyfoam I have substantiated the foam system as an effective back-up material for Ablefoam 5.

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DISCUSSION

SCOPE AND PURPOSE

During the development of a new assembly, Sandia Laboratories found it feasible to encapsulate the electrical component system with Ablefoam 5 epoxy foam. Following a complete characterization of this single-source proprietary material, Sandia requested that Bendix develop a suitable back-up epoxy foam.¹

PRIOR WORK

Related prior work consists of the characterization of Ablefoam 5 at the Sandia Laboratories and a report by C. C. Thacker on the properties of Ablefoam 5.²

ACTIVITY

A literature survey of polymeric epoxy technology was conducted to provide background information on a polymeric epoxy resin-based foam system. The search encompassed subjects concerning the chemistry of basic epoxy resins, blowing agents, curing agents, cell stabilizers, and possible formulation compositions of epoxy foam systems. The information derived from the literature survey was used to establish a program of investigation which began with chemical and physical analyses of Ablefoam 5. Various epoxide reactions and formulations were then evaluated, and a polymeric epoxy resin-based foam system was developed.

Chemical and Physical Analyses of Ablefoam 5

Investigation of the solvency properties of uncured Ablefoam 5 indicated the presence of an insoluble solid identified as ammonium carbonate, the blowing agent. In addition to the ammonium carbonate precipitate, small black particles were discovered. These particles were insoluble in organic and inorganic solvents and acids and had a melting point greater than 300°C (572°F). Further analysis revealed the solid to be carbon black, an inert compound possibly intended to color (blacken) the foam.³

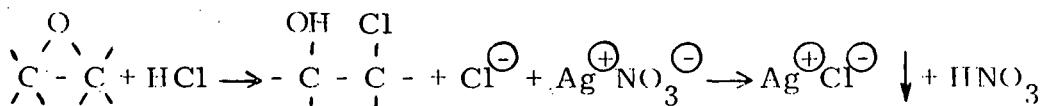
The preliminary examination of Ablefoam 5 in the Bendix Test Laboratory included subjecting the foam to gel permeation chromatography (GPC) in an effort to identify components through separation and molecular weight correlation. The standard molecular weight curves for the two commercial epoxy resins, Dow Epoxy Novalacs 431 and 438 (Dow Chemical Company) were determined. A comparison with the GPC molecular weight curve for Ablefoam 5 disclosed that this foam system contained a Novalac resin similar to DEN 431.

Also through the use of GPC, two aromatic amines, meta-phenylenediamine and methylenedianiline, were found to be present in the Ablefoam 5 system. These two amines are present in a cutectic blend of Epon Cure Agent Z.

commercially prepared by Shell for use in curing epoxy resins. To support these findings, comparative thin layer chromatography (TLC) was performed on the two amines and Ablefoam 5. The spot depositions of the two amines gave conclusive evidence that they are present in the Ablefoam 5 system. An aliphatic amine thought to be diethylenetriamine (DETA), an amine having high reactivity with epoxy groups, was also found to be present, presumably for the purpose of initiating the cure cycle.

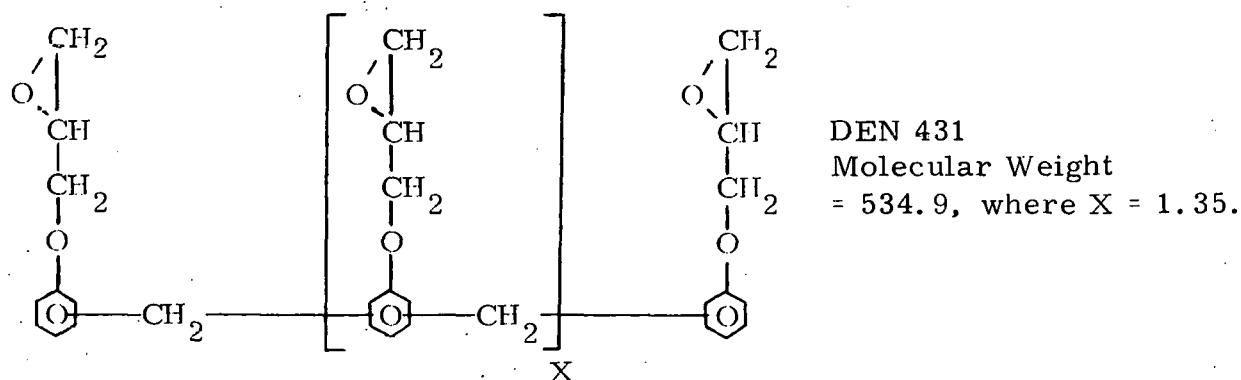
Ablefoam 5 was also analyzed by potentiometric titration, a technique adopted by the BKC Polymer Laboratory to establish the epoxide equivalent (content) of epoxy resins and to analyze the percent cure of the epoxy foams. This procedure determines the number of oxirane (epoxy) rings in the epoxy resin. It is then feasible to ascertain stoichiometric amounts of specific curing agents that are added to the resins. After the resin has been mixed with a curing catalyst, the reaction between the two can be monitored. Such monitoring consists of accounting for the number of existing epoxy sites reacted upon by the chemical compound and the number of epoxy sites left unreacted.

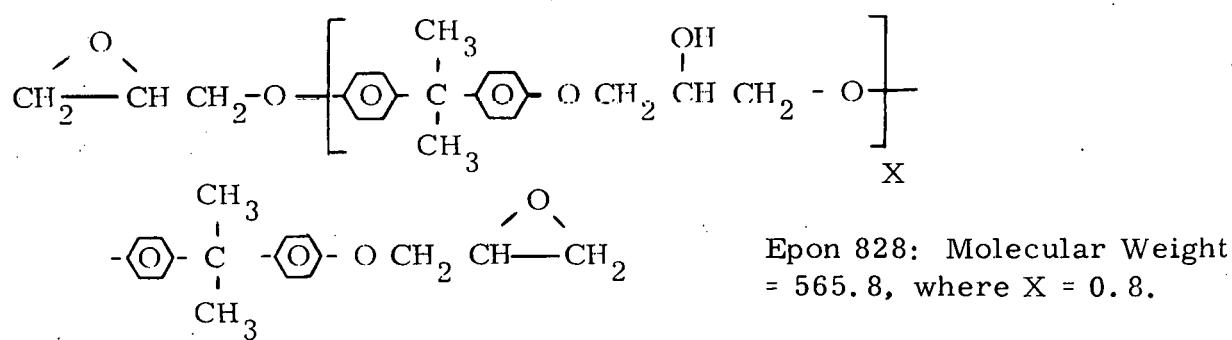
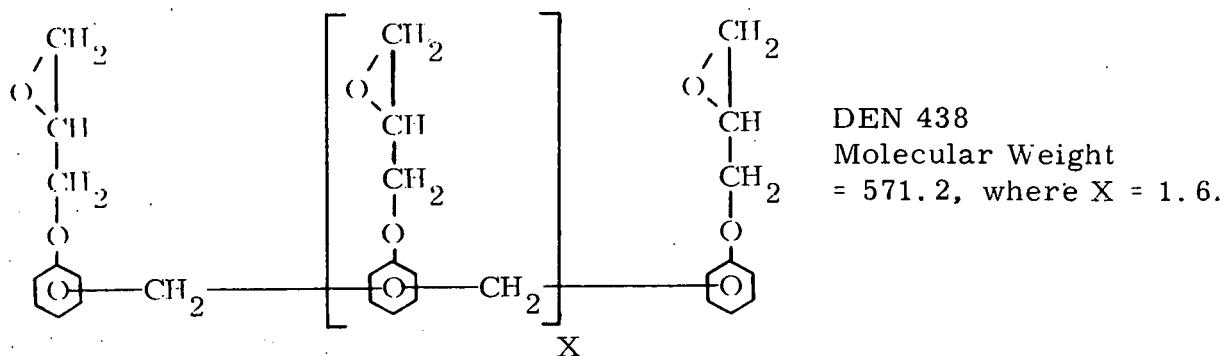
Potentiometric titration, in this instance, measures to a 300 millivolt endpoint the consumption of the remaining chloride ions from a 1.0 N HCl solution by the titratable silver ion from 1.0 N AgNO₃ through the following reaction.



The difference between the amount of acid (chloride ion) added and the amount unconsumed is a measure of the epoxy content.⁴ This procedure was used to analyze the various epoxy resins employed in the foam formulating process.

Based upon the GPC curves, the laboratory formulations were made up of DEN 431, an epoxy Novolac resin; DEN 438, its higher molecular weight complement; and a lower viscosity bisphenol-A type epoxy resin (Shell's Epon 828), which has a comparable molecular weight. Shown below are the chemical structures for these epoxy resins.



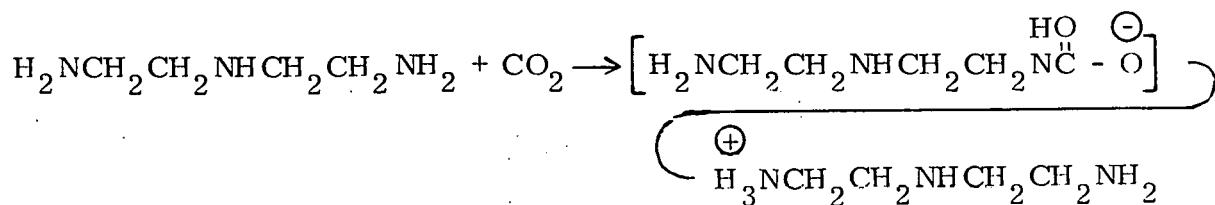


Evaluation of Epoxide Reactions and Formulations

Preliminary formulation work began with free-rise (unrestricted) and molded block foams. A comparison of the various resins and possible combinations revealed that a blend of DEN 431 and Epon 828 yielded stronger and more uniform foams.³ A study of the flow characteristics and compressive strength values of the foams indicated the optimum blend ratio of the two resins to be 50:50. Also blended with the two resins was the organic reactive diluent, 1, 2-epoxy-3-phenoxypropane, which was used to reduce the resin viscosity to a workable consistency.

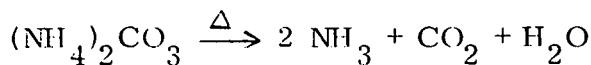
In the course of formulating the epoxy foam system, various blowing agents were examined. Acetone and the lower-boiling Freons were unsuitable because of their high volatility, since temperatures exceeding room temperature were required to cure the foam.^{5,6}

A laboratory-synthesized compound known as diethylenetriamine (DETA) carbamate was prepared by the reaction of DETA and carbon dioxide to form the following complex salt.⁷



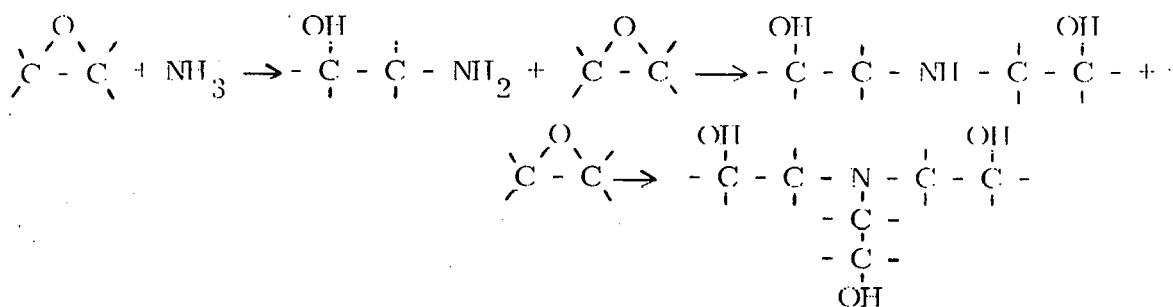
When heated, the amine carbamate decomposed into carbon dioxide (the active blowing agent) and DETA (an amine curing agent for epoxy resins). The dual character of this compound was not useful, however, since extremely large concentrations were required for minimal blowing and curing of the foam.

Ammonium carbonate, which was present as the blowing agent in Ablefoam 5, was found to be quite suitable. Ammonium carbonate decomposes at 58°C (136.4°F) through the following reaction.



Because all three decomposition products--ammonia, carbon dioxide, and water vapor--contribute to the foam system, this compound was practical as a blowing agent. With this compound, the blowing action is a controllable factor since the ambient temperature of the foam and mold must approach 58°C (136.4°F) before the foam will blow.

Results from potentiometric titration of the epoxy resins indicated that their epoxy content decreased when they were mixed with the ammonium carbonate. This fact suggests that, in addition to the blowing action, ammonium carbonate acts as a curing agent; therefore, that part of the manufacturing process where ammonium carbonate is added to the epoxy resin must be closely controlled to minimize lot-to-lot variation in the foam reactivity. The reaction shown below is the process by which the evolved ammonia gas reacts with the epoxy ring to effect an actual curing response.



Initial mixing of the foam formulations with a high-speed stirrer resulted in cured foams possessing resin sedimentation and numerous cell voids. To eliminate these problems and to blend the solid ammonium carbonate blowing agent with the epoxy resin and other foam constituents required the use of a laboratory-type three-roll mill. Before the ammonium carbonate was blended with the premix, it was passed through a Number 40 U.S. sieve. Photomicrographs of the mixture indicated that as the mill roll distances decreased, the solid-to-resin dispersement increased in conjunction with a breaking up of the larger ammonium carbonate crystals into a much smaller size.

Since, as previously mentioned, a reaction upon the epoxy resins occurs when ammonium carbonate is added, it was necessary to expedite the processing technique. A method was developed to collect the foam premix in plastic polyethylene cartridges as it flows off the mill. The cartridges are then sealed and immediately immersed in liquid nitrogen. Each cartridge is hand-held with forceps as it is lowered into the container of liquid nitrogen. This method halts any subsequent reaction between the chemical components of the foam system. When needed, the one-component frozen foam is allowed to thaw to an extrudable temperature; the foam premix is then poured into the desired mold.

With the refinement of the processing techniques, a contract was established with Midwest Research Institute for the screening of potential foam formulations by compression testing.⁸ The tests were performed on 1 inch by 1.129-inch diameter foam specimens cut from an 8 inch by 1.129-inch diameter foam cylinder. All specimens were tested edgewise (parallel to foam rise). Because of its physical similarity to the foam rise characteristics of the electronic assembly container, the 8-inch by 1.129-inch-diameter cylinder mold was subsequently used to optimize the formulation of a suitable back-up epoxy foam.

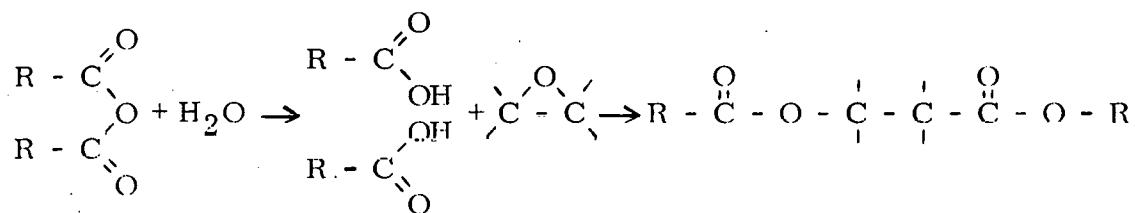
Development of the Polymeric Epoxy Resin-Based Foam System

The various foam formulations which were developed were evaluated for their potential applicability. Those samples were screened from further consideration which did not meet the requirements of SS282997 for density (20.0 lb/ft³ minimum) and compressive strength (1050 psi minimum at room temperature testing and 250 psi minimum at 200°F testing). All foam systems were cured for 4 hours at 74°C (165°F).

Several formulation studies were used to aid the development of the epoxy foam system. In one study, the effects of two aromatic amines, meta-phenylenediamine and methylenedianiline, were compared. However, the difficulty of processing the two amines with the epoxy resins rendered them unsuitable.⁹ Since these two amines are inherent to the composition of Cure Agent Z, the eutectic blend was selected as the aromatic amine curing agent.^{10, 11}

A cell-surfactant examination using Union Carbide's Silicone L-5320 indicated that its use at low concentrations produced high compressive strengths and uniform density gradients.¹² Much higher concentrations of other cell stabilizers were required for the same results. Consequently, the use of the other cell stabilizers was undesirable since with them it was possible that the silicone fluid would settle out of the foam at some time following storage.

The addition of methyl Nadic anhydride (MNA) to the foam system improved density uniformity and increased the compressive strengths. The anhydride appeared to function simultaneously as a gel agent and cure agent.¹³ The curing mechanism for the anhydride requires the formation of the diacid from the anhydride. The eventual reaction upon the epoxide ring is shown below.



Freshly distilled MNA was required to control more closely the reaction upon the epoxide groups. The diacid formation would then depend on the curing process. The formation of water from the decomposition of the ammonium carbonate is required for the diacid formation.

To ensure an effective anhydride cure, maleic anhydride (MA) was added. It has a low melting point (53°C, 127.4°F) and a high degree of reactivity as a curing agent. The foams containing this anhydride possessed a more uniform cell structure and a better surface finish.

After the various formulation factors had been considered, a back-up epoxy foam was finally adopted and named Capoxyfoam I. A typical example of the back-up formulation is presented in Table 1. Processing includes addition of the components in the order listed, use of the three-roll mill, and the quick-freezing of the foam premix.

Coincident with the development of Capoxyform I was the preparatory evaluation of a boron trifluoride-piperidine complex as an auxiliary curing agent. The crosslinking mechanism of this chemical complex substantially increased the compressive strengths of the foams to 1490 psi at room temperature testing and 890 psi at 200°F testing a density of 22lb/ft³. These values are comparable to those of Figure 1.

The secondary amine complex reacts with the epoxy ring in the following manner:

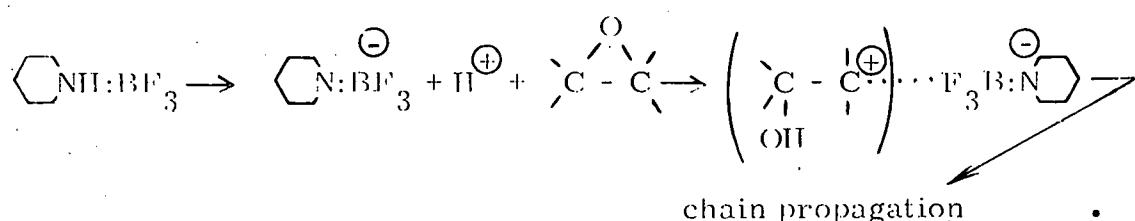


Table 1. Typical Formulations of Capoxyfoam I and Capoxyfoam II
(Chemical Components Listed in Order of Addition)

Capoxyfoam I	Quantity (g)	Proportion (phr)*
Shell's Epon 828 Bisphenol-A Epoxy Resin	100.0	--
Dow's DEN 431 Epoxy Novolac Resin	100.0	--
1,2-epoxy-3-phenoxypropane	2.0	1
Union Carbide's L-5320 Cell-Stabilizer	4.0	2
Shell's Epon Cure Agent Z	40.0	20
Ammonium Carbonate	12.0	6
Maleic Anhydride	2.0	1
Methyl Nadic Anhydride	8.0	4
Capoxyfoam II		
Shell's Epon 828 Bisphenol-A Epoxy Resin	100.0	--
Dow's DEN 431 Epoxy Novolac Resin	100.0	--
1,2-epoxy-3-phenoxypropane	2.0	1
Union Carbide's L-5320 Cell-Stabilizer	4.0	2
Shell's Epon Cure Agent Z	40.0	20
Ammonium Carbonate	12.0	6
Maleic Anhydride	2.0	1
Methyl Nadic Anhydride	8.0	4
Borontrifluoride-piperidine Complex	2.0	1

* phr--parts per hundred parts by weight of resin.

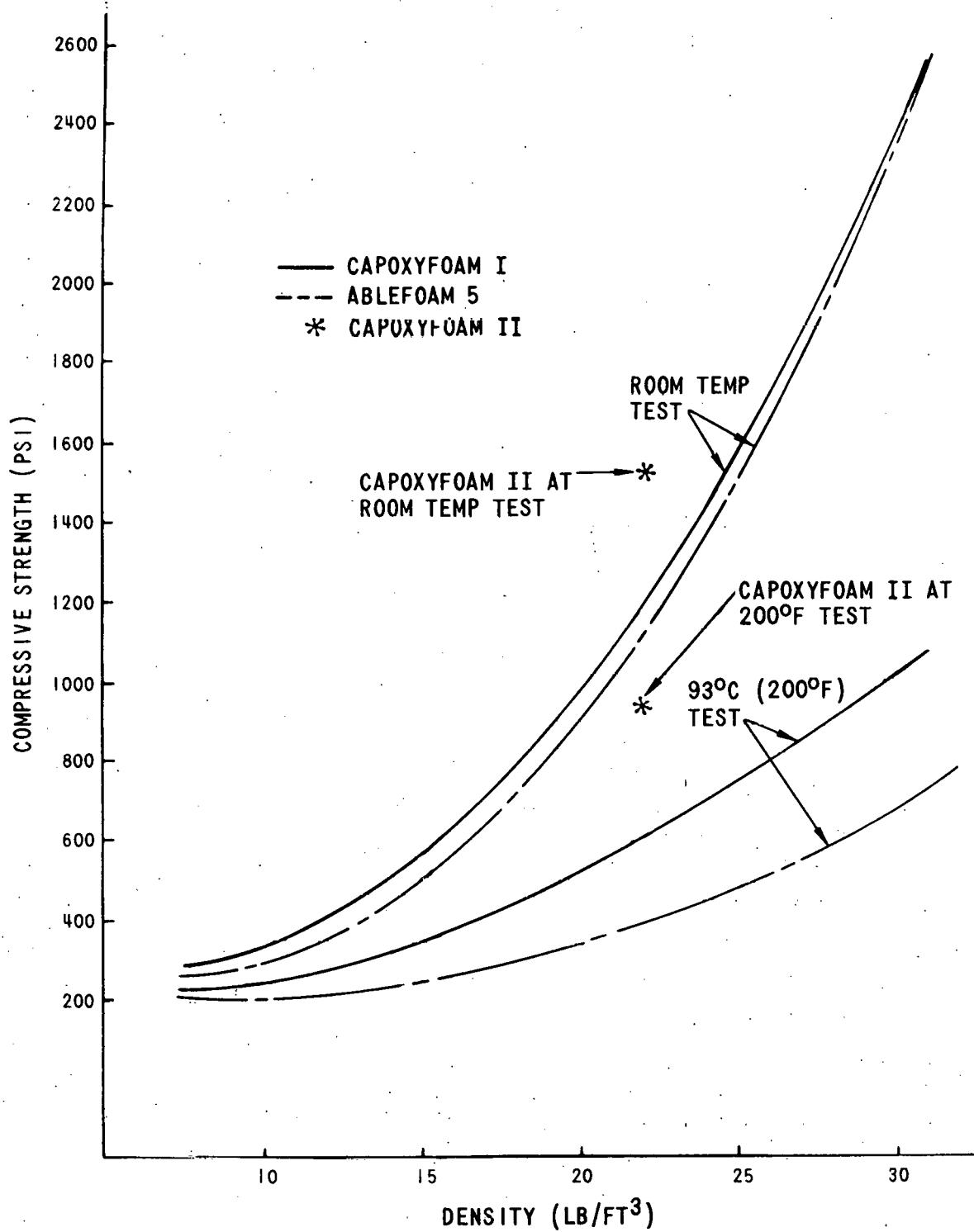


Figure 1. Density Versus Compressive Strength Comparison for Ablefoam 5, Capoxyfoam I, and Capoxyfoam II (Cured at 165°F for 4 Hours)

Because of the unique character of this foam premix, it was adopted as a second formulation and named Capoxyfoam II. Work on the Capoxyfoam II formulation was done in anticipation of future needs for a high-strength epoxy encapsulant. No additional testing of the material was performed during this investigation. Typical examples of the formulations for both Capoxyfoam I and Capoxyfoam II are given in Table 1.

Following the development of these polymeric epoxy resin-based foam systems, the property values of Capoxyfoam I and Capoxyfoam II were compared with those of Ablefoam 5. Figure 1 illustrates the compressive strength values as a function of density for Ablefoam 5 and Capoxyfoam I. Their strengths are nearly identical for the tests at room temperature, with a greater difference appearing at the 200°F test temperature.

The possibility that the silicone cell stabilizer would migrate out of the cured foam and eventually leak onto the electrical components was considered. A Soxhlet extraction experiment was performed to determine the amount of silicone, if any, that could be removed from the cured system. The organic solvent, methylethylketone, was used as the extracting medium. Although infrared results showed no silicone deposition with either Capoxyfoam I or Ablefoam 5, a partial constituent extraction had occurred since Capoxyfoam I exhibited an average weight loss of 0.97 percent and Ablefoam 5 exhibited a 1.01 percent weight loss.

To verify this weight loss, thermalgravimetric analysis (TGA) was performed on each foam. The weight loss per sample as a function of temperature occurred at the thermal degradation point of 375°C (707°F) for Capoxyfoam I and 309°C (588°F) for Ablefoam 5. The 66°C difference between the two foams clearly indicates the greater thermal stability of Capoxyfoam I.

Sandia Laboratories, Albuquerque, conducted a heat-aging study which included subjecting the foam samples to a 205°C (400°F) temperature environment as a function of time. While the compressive strength for Ablefoam 5 had decreased significantly with only a 35 percent retention of strength, Capoxyfoam I averaged nearly 93 percent in retention strength.

Aside from the mechanical and thermal variations, the open-to-closed cell content of the foams, compared below, was most noticeable.

	Capoxyfoam I (Percent)	Ablefoam 5 (Percent)
Open-Cell Void Content	5.03	62.00
Closed-Cell Void Content	67.03	11.40
Total Void Content	72.06	73.40

The cell structures of the two foams are visible in the scanning electron microscope (SEM) photographs in Figure 2.

Since no restrictions had been placed on the type of cell condition acceptable for a back-up epoxy foam system, no special efforts were made during the formulation of Capoxyfoam I to develop an open-cell material.

Both foams were compared to determine the effect of the open-to-closed cell content on the percent cure. Differential scanning calorimetry had indicated very little difference in the percent cure of the two foams. To substantiate this finding, percent cure analysis was performed with potentiometric titration and it was observed that both foams asymptotically approach an 85-percent maximum cure after 8 hours at 74°C (165°F).

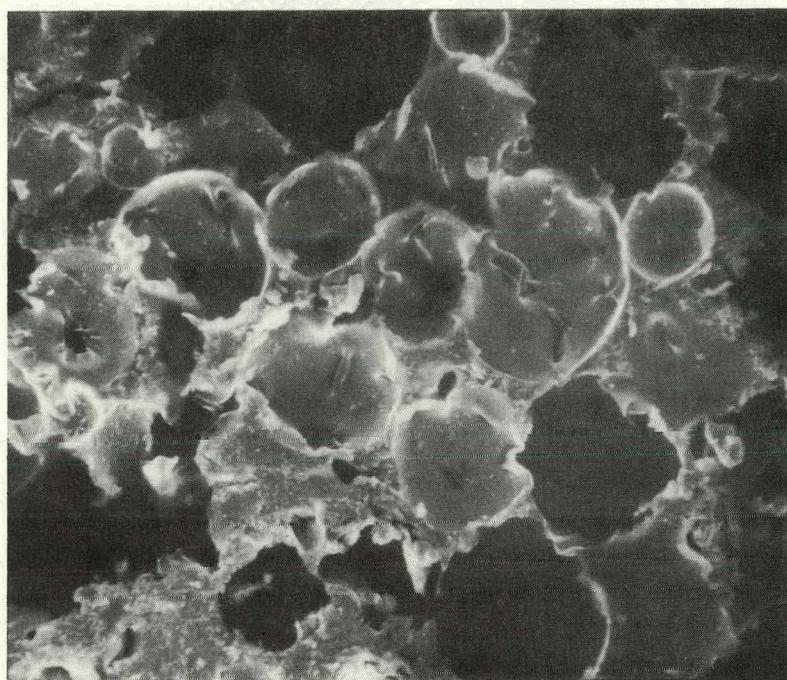
The electrical properties of the two foams were compared for the dielectric constant, dissipation factor, and volume resistivity. The foams were molded to 1.8 by 6 by 6 inches and tested at 22 lb/ft³. The dielectric constants of the two foams were 1.60 at 10⁶Hz for Ablefoam 5 and 1.703 at 10⁶Hz for Capoxyfoam I. The dissipation factor, which is a tangent of the loss angle, was 0.0018 for Capoxyfoam I and 0.025 for Ablefoam 5, both values falling within the literature-cited values for epoxy foams. The volume resistivity values for the two foams were 13.2 by 10¹³ ohms/cm for Ablefoam 5 and 6.27 by 10¹³ ohms/cm for Capoxyfoam I.

The compressive strengths, thermal degradation, and electrical properties for Ablefoam 5, Capoxyfoam I, and Capoxyfoam II are summarized in Table 2.

A notable feature of the Capoxyfoam system is its ability to be reproduced with consistency. A lot-to-lot examination of the epoxide values for Ablefoam 5 indicated a range of differing epoxide values with an average deviation of 2.43. By contrast, lot-to-lot examination of the epoxide values for Capoxyfoam I revealed a definite uniformity with an average deviation of 0.7. This data was recorded for seven lots each of the materials.

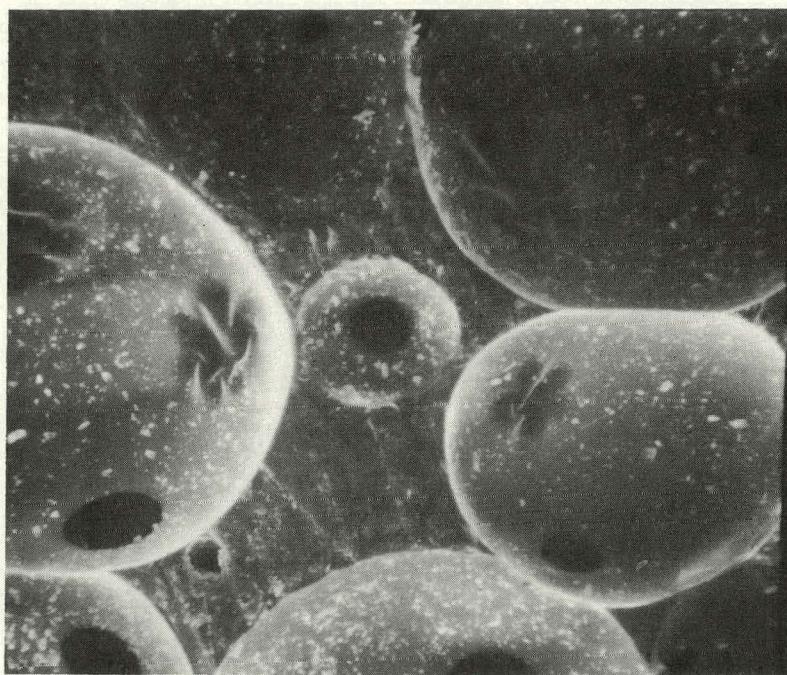
Numerous production A-B dummy units were processed with the Capoxyfoam I system. Mold fill characteristics and part surface finish were of excellent quality, and the density distribution of the various foam sections varied less than ± 0.5 lb/ft³.

The processing phases of molding dummy units proved barely satisfactory in the preliminary trials. Since Capoxyfoam I is more viscous than Ablefoam 5, extruding the thawed premix into the reservoir section of the dummy unit was difficult. Preheating the dummy unit to approximately 46°C (115°F) solved the problem. The thawed premix flowed into the reservoir without difficulty and well within the workable pot-life of the foam. The units shown in Figures 3 and 4 were visually acceptable. Figure 3 displays A-B dummy units molded from Ablefoam 5 and Capoxyfoam I. Figure 4 represents an actual production of the "C-deck" section of the assembly using Capoxyfoam I.



CAPOXYFOAM I

100X



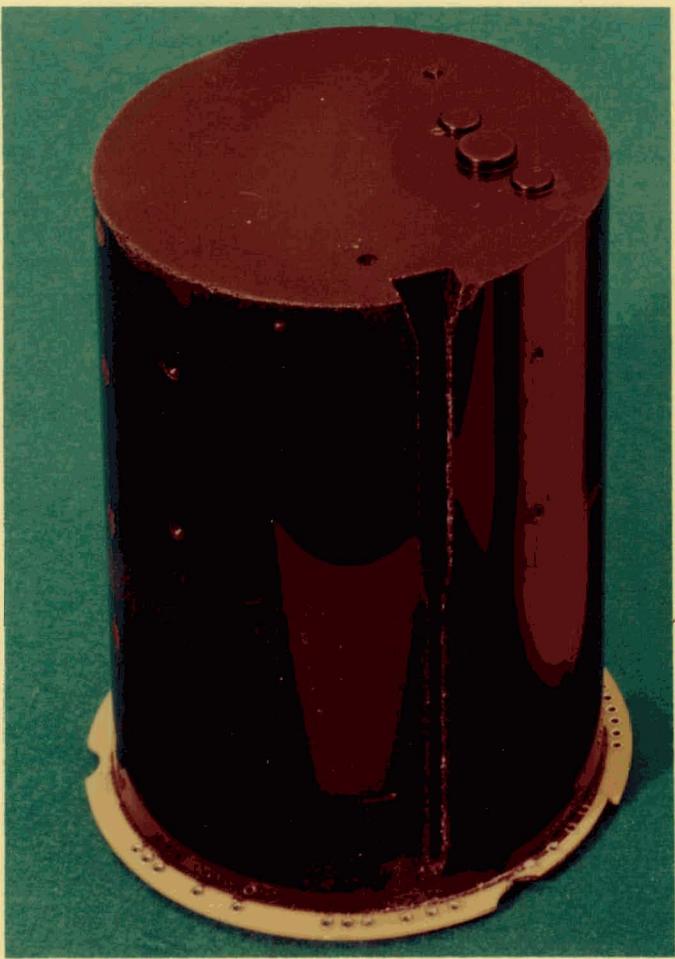
ABLEFOAM 5

100X

Figure 2. Scanning Electron Microscope (SEM) Photographs of Cell Structure Parallel to Foam Rise

Table 2. Property Comparison of Ablefoam 5, Capoxyfoam I, and Capoxyfoam II

Property Evaluated	Ablefoam 5	Capoxyfoam I	Capoxyfoam II
Compressive Strength (22 lb/ft ³)			
Room Temperature Test (psi)	1100	1200	1490
200°F Test (psi)	380	590	890
(Foam samples tested parallel to foam rise)			
Thermalgravimetric Analysis (TGA)			
Weight loss occurring at a thermal degradation point in °C	309	375	367
Electrical Properties			
1. Dielectric Constant	1.60×10^6 HZ	1.703×10^6 HZ	--
2. Dissipation Factor	0.025	0.0018	--
3. Volume Resistivity	13.2×10^{13} ohms/cm	6.27×10^{13} ohms/cm	--



ABLEFOAM 5



CAPOXYFOAM I

Figure 3. Comparison of A-B Dummy Unit
Employing Ablefoam 5 and Capoxyfoam I

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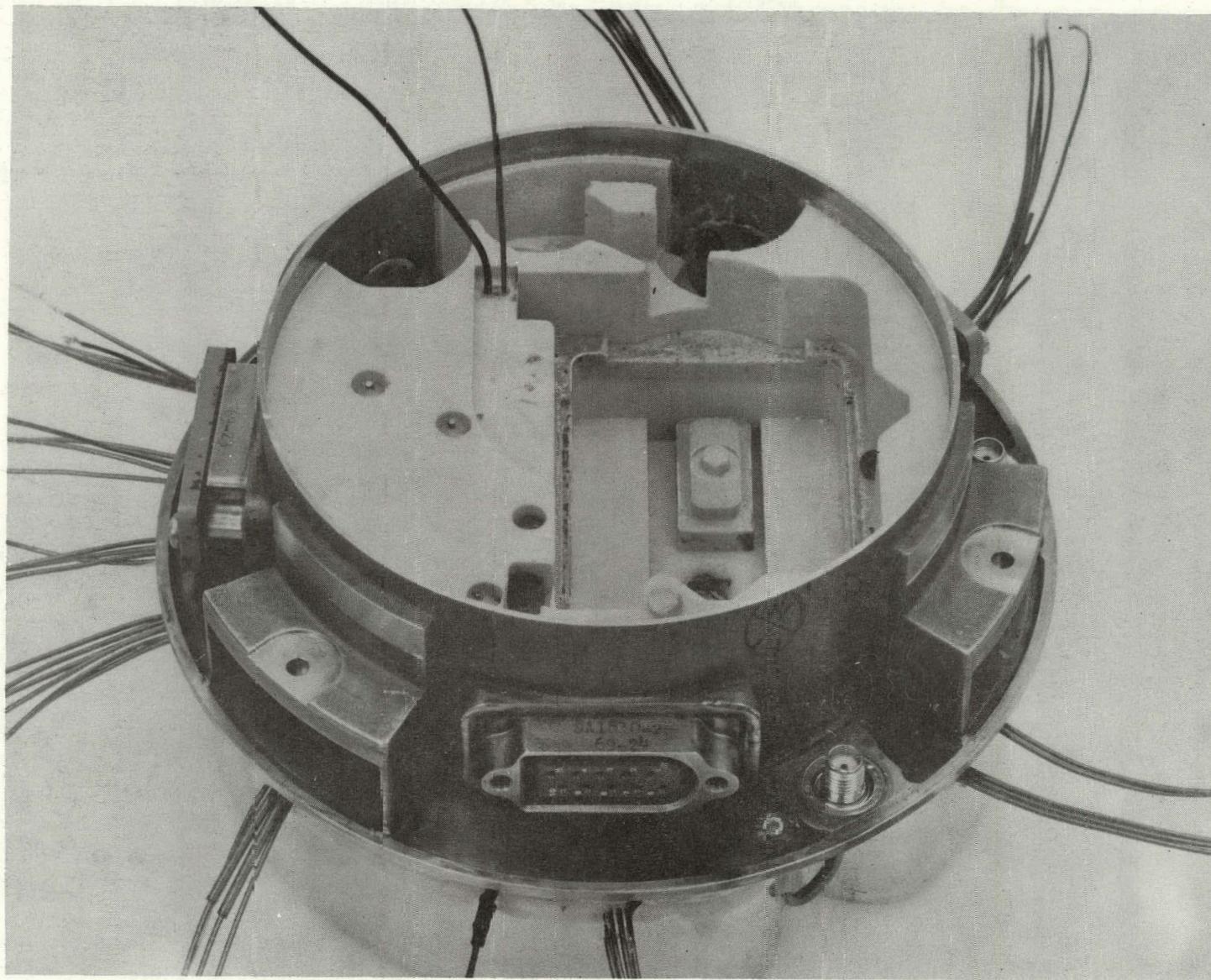


Figure 4. Production Model of "C-Deck" Section

ACCOMPLISHMENTS

Through the series of formulation routes described, a polymeric epoxy resin-based foam system identified as Capoxyfoam I was developed. The properties of Capoxyfoam I are similar to those of Ablefoam 5. A second formulation possessing higher compressive strengths was also developed and named Capoxyfoam II. This material was developed for future applications requiring a high-strength epoxy foam.

The epoxy foam premix is subjected to a quick-freezing process. When needed, it is thawed to an extrudable temperature and employed as a one-component foam system.

Capoxyfoam I was found to be satisfactory for use as an electronic encapsulant. The final products employing Capoxyfoam I are equal, if not superior, to Ablefoam 5 with respect to ease of processing and quality.

FUTURE WORK

At present no future work is planned for this project. At some future date, however, formulation of an epoxy foam having an open-cell content greater than Capoxyfoam I may be desirable. An open-cell foam facilitates more rapid diffusion of the blowing gases out of the foam matrix. Future work would include development of an epoxy foam employing an organic type blowing agent or one utilizing less ammonium carbonate than is present in the Capoxyfoam I system.

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¹² U. S. Patent 3,282,863 (November 1, 1966).

¹³ U. S. Patent 3,440,216 (April 22, 1969).