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Epoxy Foam Encapsulants: Processing and Dielectric Characterization

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Epoxy Foam Encapsulants: Processing and Dielectric Characterization

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Abstract

The dielectric performance of epoxy foams was investigated to determine if such materials might provide advantages over more standard polyurethane foams in the encapsulation of electronic assemblies. Comparisons of the dielectric characteristics of epoxy and urethane encapsulant foams found no significant differences between the two resin types and no significant difference between as-molded and machined foams.

Blown epoxy foams are an alternative to the more prevalent and versatile polyurethane foams used as DP firing set encapsulants and in a range of other commercial applications. Epoxy resins are not as readily foamed and processed as urethanes and have generally seen only limited use as encapsulants. Potential advantages for epoxy foams, however, might result from their dielectric properties and also elimination of toxic and sometimes sensitizing isocyanates used in urethane formulations. This study specifically evaluated the formulation and processing of epoxy foams using simple methylhydrosiloxanes as the blowing agent and compared the dielectric performance of those foams to urethane foams of similar density.

Epoxy foams with densities ranging from 0.25 to 0.90 grams/cc were prepared and the influence of various formulation parameters on those foams was established. The alkylhydrosiloxane blowing agents used generate hydrogen gas during the epoxy curing process, much as urethane foams generate carbon dioxide from water during the curing reaction.

Dielectric properties were measured on these epoxy foams and also urethane foams over a range of densities and with both machined and as-molded, skinned surfaces. No significant differences were found. Both as-molded foams with skinned surfaces and machined foams showed little dependence of volume resistivity on foam density except at very low densities. Urethane foams generally had higher volume resistivities than epoxy foams although all the samples, with and without skins, had resistivities above 10^{16} ohm-cm measured at one minute. Urethane foams were typically about an order of magnitude higher at 10^{17} ohm-cm.

Acknowledgements

The contributions of Paul Beeson, now retired from Org. 1812, were invaluable in carrying out the initial foam dielectric tests and in guiding the establishment of that test capability at Sandia, California. The bulk of the work in setting up the California test capability was carried out by Marion Hunter with additional assistance from Bill Even and Bob Crocker.

Table of Contents

	<u>Page</u>
Introduction	7
Background on Epoxy Foam Technology	8
Foam Formulations: Siloxane Blowing Agent	11
Foam Formulations: Curing Agents and Epoxy Resins	14
Foam Formulations: Surfactants	15
Processing Studies	15
Foam Density and Structure	16
Dielectric Testing Procedures	22
Dielectric Testing Results	23
Conclusions	36
References	37
APPENDIX A—Structure of Epoxy Formulation Ingredients	38
APPENDIX B—Surfactants Evaluated in Epoxy Foams	39
APPENDIX C—Foam SEM Pictures	40
APPENDIX D—Dielectric Testing Equipment	53
APPENDIX E—Dielectric Testing Data	54

Epoxy Foam Encapsulants: Processing and Dielectric Characterization

Introduction

Blown epoxy foams are an alternative to the more prevalent and versatile polyurethane foams used as firing set encapsulants and in a range of other DP and commercial applications (Ref. 1). Commercial epoxy foam applications have typically exploited the rigidity, adhesive strength, moisture resistance and toughness of epoxies, plus the lighter weight of a foam, in such structures as wind generator blades, windsurfing boards, automotive spoilers and in an aluminum honeycomb in the DC-10. These applications often include sandwich structures or fibers for reinforcement. Commercial epoxy adhesives also employ added foaming agents in some applications to insure gap filling.

Epoxy resins are not as readily foamed and processed as urethane foams and have generally seen limited use in encapsulant applications. Potential advantages of epoxy foam in encapsulant applications might lie in their dielectric properties and also the elimination of toxic or sensitizing isocyanates used in urethane formulations. DP programs have also cited the brittleness and dark color of the epoxy foams as advantages, either of which can be provided by appropriately formulated and filled urethane foams. The purpose of this study was to evaluate the formulation and processing of epoxy foams using simple alkylhydrosiloxanes as the blowing agent and, more importantly, to compare the dielectric performance of those foams to urethane foams of similar density.

Epoxy foams with densities ranging from 0.25 to 0.90 grams/cc were prepared and the influence of various formulation parameters on the foam density and processing was established. Alkylhydrosiloxane blowing agents were used to generate hydrogen gas during the epoxy curing process, much as urethane foams generate carbon dioxide from water during the curing reaction. This technology appeared appropriate for encapsulant applications due to the formulation flexibility and the gentleness of the foaming reaction.

Dielectric properties (volume resistivity, dielectric constant and dissipation factor) were measured on these epoxy foams and also urethane foams (CRETE formulations) over a range of densities and with both machined and as-molded, skinned surfaces. Dielectric breakdown properties have not been determined as yet.

Background on Epoxy Foam Technology

This section is meant to briefly review the various options available in creating epoxy foams and their relative advantages and disadvantages. The alkylhydro siloxanes selected as the blowing agent for this study is one of several could be used. A major consideration in this selection was the ease of formulation and processing and the similarity in behavior to current polyurethane foams.

Polyurethanes are readily foamed by the simple addition of various amounts of water that reacts with isocyanate groups to form the carbon dioxide blowing agent and amines. The resulting amines quickly react with remaining isocyanate groups to form urea linkages within the urethane matrix. Epoxies do not foam on the addition of water and have instead been foamed by the addition of various reactive and inert blowing agents, each of which provides advantages and disadvantages. Both resin types also utilize surfactants and catalysts as needed to modify the resin reactivity and foam morphology. (Note: Only blown foams are considered in this discussion. Syntactic epoxy foams, created by the addition of hollow spheres of glass, GMB, or other materials are a separate class of materials which generally cannot provide foam densities lower than about 0.7 g/cc)

Heat activated blowing agents have included azo and hydrazide compounds (nitrogen generation) and carbonate salts, all of which usually require milling equipment to insure a uniform distribution of the solid blowing agent and also refrigerated storage to prevent premature decomposition. High curing temperatures are often required to activate the blowing agents although this can be modified by added catalysts (Ref. 2). AbleFoam 5, a now discontinued commercial product used in the W76 firing set, used ammonium carbonate as the blowing agent (Ref. 3). This proprietary product was sold in Semco cartridges which required storage below about -50°F. The thawed cartridges could be injected into molds and had a potlife of about one hour. Curing at temperatures above 120°F, preferably at about 150°F, was required and heated molds appeared to be beneficial. Upon decomposition the salt would generate a mixture of ammonia and carbon dioxide. This product was discontinued by the manufacturer, Ablestick, due to the unavailability of the triglycidyl ether of glycerin used in the formulation. This component is a cancer suspect agent. A backup product developed at Bendix, KCP, was called Capoxyfoam and used the same blowing agent in a DEN 431/Epon 828/phenyl glycidyl ether/Shell Z formulation (Ref. 4). Proper dispersion of the blowing agent was critical in this formulation and it has not apparently been used in any weapon applications.

Many of the reactive blowing agents reported in the literature are integral components of the epoxy formulation and have included carbamate salts (Ref. 5), carbonate oligomers (Ref. 6), combinations of anhydride curing agents and tertiary amine catalysts (ref. 7), and trialkoxyboroxines (Ref. 8). Carbamate salts are an intermediate reaction product of carbon dioxide and aliphatic amine curing agents and require a difficult synthetic/purification process. Upon heating, the original amine curing agent plus carbon dioxide are generated. Formulation stability and processability are

challenges with this class of materials. Carbonate oligomers also generate carbon dioxide upon heating (about 140-180°C) and are specific epoxy resins containing multiple carbonate linkages that can be used in standard epoxy formulations. These oligomers, basically glycidyl-capped low molecular weight Lexan, do not appear to be commercially available. Tertiary amines and aliphatic anhydrides are standard ingredients in epoxy formulations and have been shown to react at elevated curing temperatures (at least 115°C) to generate carbon dioxide and foam the resulting epoxy matrix. A patent in this area suggests the use of added auxiliary blowing agents (Ref. 7). Note: This reaction of anhydrides and tertiary amine catalysts was noted previously at Sandia and AS/FM&T when a preblend containing such ingredients and used to cure a solid epoxy encapsulant system was found to slowly generate carbon dioxide and build up pressure during storage at room temperature. The chemistry of this reaction is not well defined but may involve loss of an allylic hydrogen and self-condensation (Ref. 9). Finally, trialkoxyboroxines have also been described as combination curing/blowing (generated methane gas) agents. They must be used at high levels or supplemented with other blowing agents. Pot life after mixing was extremely short and the exotherms appeared to be substantial.

Other reactive blowing agent additives, as opposed to simple heat activated agents, have included combinations of sodium borohydride and water (Ref. 10), and alkylhydro siloxanes (Ref. 11-13). Sodium borohydride reacts with advantageous and added water to generate hydrogen gas and sodium metaborate (NaBO_2). This blowing agent combination has also been used in thermoplastic foams. It was not considered in this study on encapsulant applications due to the expected detrimental effect of the sodium salt formed during the foaming process on dielectric properties.

Alkylhydro siloxanes are used as the blowing agent in a commercial foaming epoxy, RP-1774, from Ciba-Geigy. This specific formulation includes Bentonite clay and calcium carbonate fillers along with dibutyl phthalate as a diluent or flow modifier. As received, it would be too viscous for most encapsulant applications. Data sheets indicate both Bisphenol A and diluent epoxies, perhaps Epon 815, and a Versamid type curing agent. The third component, the blowing agent, is a methylhydrosiloxane homopolymer which reacts with the amine curing agent to generate hydrogen gas. Similar siloxane-blown systems were the subject of a German study on the fatigue behavior of fiber-reinforced epoxy foam laminates (Ref. 14). Russian work with this class of blowing agents has also been reported (Ref 15) including a patented formulation (Ref 16) using ethylhydrosiloxane as the blowing agent in a carbon black filled conductive foam.

Because of the formulation flexibility, the absence of any ozone-depleting agents, the absence of added or generated salts, and the processing similarity to urethane foams, the alkylhydro siloxanes were chosen as the blowing agents for this study.

Inert blowing agents include low-boiling solvents and Freons or injected gases and these have been used in numerous foaming applications with both thermosetting and thermoplastic resins. Freons were used in the late 70's by Thorn EMI Electronics Ltd.

in Great Britain to specifically formulate epoxy foams for electronic encapsulation (Ref. 11-13). The advantages cited were low toxicity (no isocyanates) and removability with acetone solvents. This series of products (Feldex F3-6) utilized a range of amine (F3/4) and polymercaptan (F5/6) and anhydride curing agents with the F5 foam being the most recommended although it had poor load bearing capability above 60°C. No performance advantages for these products over similar urethane foams were clearly demonstrated or claimed. Their ready softening in the presence of acetone was mentioned only briefly and no details such as ease of removal or possible correlation to poor mechanical properties were provided. Such ready dissolution of an epoxy by acetone usually suggests incomplete cure. All these products appear to have been discontinued or were never fully commercialized. In the 70's there was also work at Ciba-Geigy on special aliphatic epoxy resins, not apparently commercialized, which gave superior quality foams using inert blowing agents (Ref. 17).

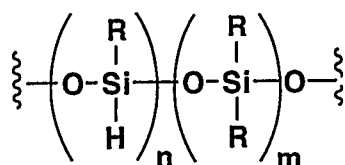
A program at Sandia/New Mexico has utilized an inert blowing agent, Fluorinert FC-72 (from 3M), as a blowing agent in a replacement material for the discontinued AbleFoam encapsulant and will be reported elsewhere.

Finally, an extensive study was carried out at Y-12 (Ref. 18) on mechanically frothed epoxies which contained no blowing agents but relied on surfactants to stabilize air whipped into the formulation by a high speed mixer. These Versamid cured materials showed a range of foam morphologies depending on the surfactant used. The most successful was a pre-reacted mixture of stearic acid and the Versamid curing agent. No advantage was found in the present study for such a surfactant over commercially available surfactants.

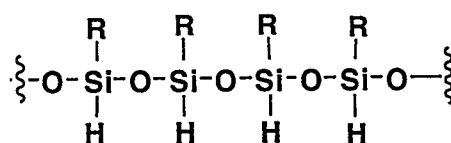
Foam Formulations: Siloxane Blowing Agent

The available literature did not define what range of alkylhydrosiloxane homopolymers and (alkylhydro)(dialkyl)siloxane copolymers might be useful in epoxy foam formulations. Both the level of siloxane additive and the type of homopolymer or copolymer might be used to adjust the foaming action and final density and the effects of these parameters were evaluated. The Ciba-Geigy data sheets for RP-1774 indicated the use of methylhydro homopolymers (confirmed by NMR) while the Russian work indicated the use of ethylhydro homopolymers. The use of copolymers was not discussed in any of the references.

(Methylhydro)(dimethyl)siloxane copolymer Alkylhydrosiloxane homopolymer



R = methyl



R = methyl or ethyl

Initial foaming trials with hand-mixed samples used a standard 60/40 Epon 828/Versamid 125 formulation and various types and levels of siloxane additives. The various siloxanes shown in Table 1 were all screened in both room temperature and oven cured (150°F) formulations at the 1 percent level and none showed significant foaming action other than the pure alkylhydrosiloxanes. Addition of a stannous octoate catalyst or water had no effect on the foam density and flame ionization analysis of the Ciba-Geigy RP-1774 siloxane blowing agent showed no added metallic compounds. The methylhydrosiloxanes gave better foaming and lower densities than ethylhydro-siloxane and both PS-120 and HMS 991 gave identical foaming behavior to the Ciba-Geigy additive. Based on these results, PS-120 was selected as the foaming agent for the remaining work.

Table 1. Siloxane Polymer Additives

Product	percent methylhydro	percent dimethyl	percent ethyl hydro	Molecular Wt.	Viscosity (cps)
PS-120	100			2270	30
PS-122.5	50-55	45-50		900-1000	10-15
PS-123	30-35	65-70		2000-2100	25-30
HMS-151	15-18	82-85			25-35
HMS-301	30-35	65-70			25-30
HMS-991	100			1500-1900	15-25
HES-992			100		75-125
RP-1774	100				

PS series from United Chemical Technologies, Inc.

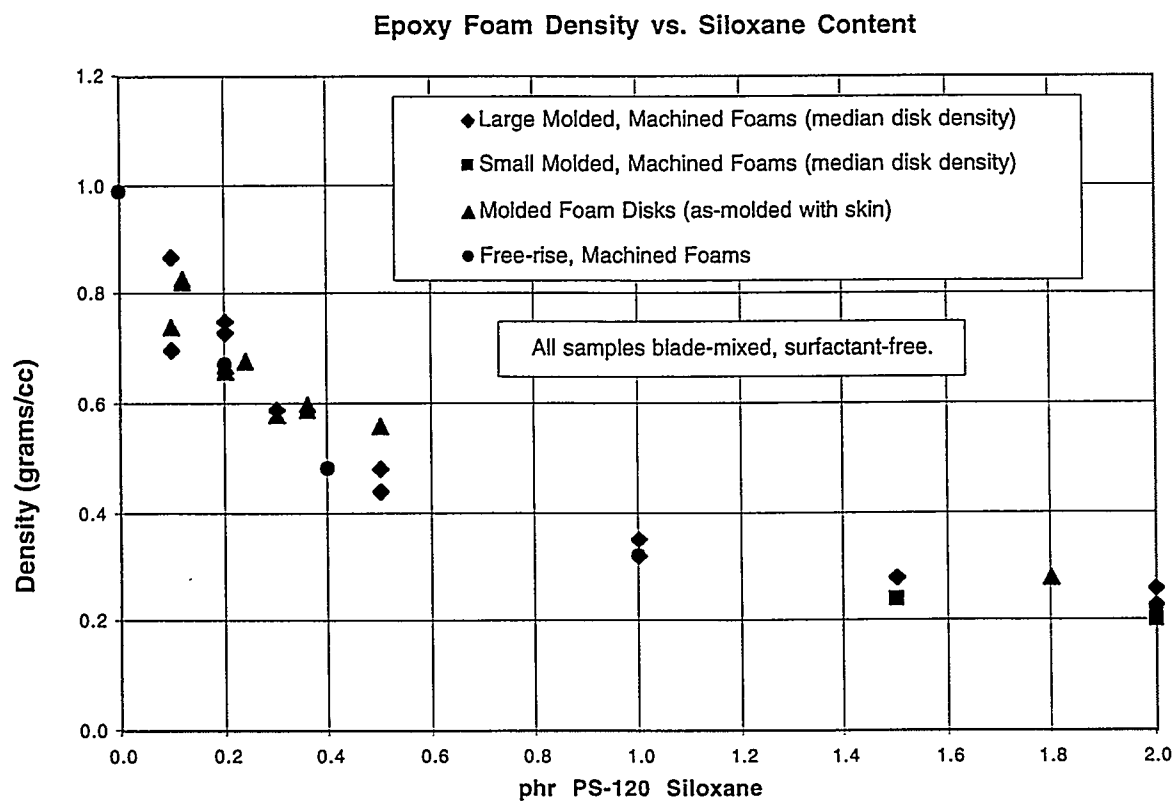
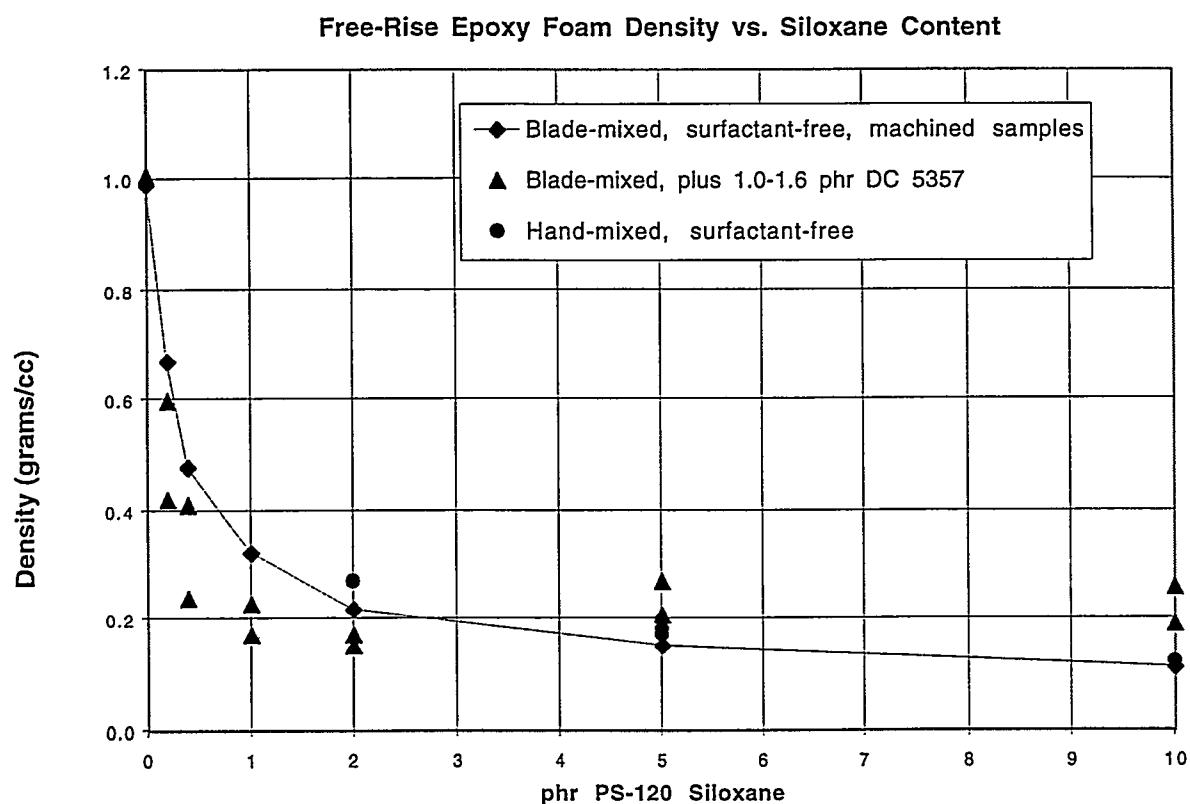
HMS/HES series from Gelest, Inc.

RP-1774 is the siloxane blowing agent supplied with Ciba-Geigy's RP-1774 3-part epoxy foam

The amount of added methylhydrosiloxane clearly had an effect on the level of foaming and further formulation tests varied the amount of PS-120 siloxane in order to obtain different foam densities. Figure 1 shows the results of early tests with the 60/40 Epon 828/Versamid 125 formulation. These included both hand-mixed and blade-mixed formulations as well as surfactant modified formulations. Use of a surfactant generally produced lower density foams although no further decreases in foam density were obtained above about 2 phr (parts per hundred resin including epoxy and curing agent).

Figure 2 shows the foam densities obtained from several series of samples including the initial free-rise foams shown in Fig. 1 (blade-mixed, surfactant-free), two series of foam disks cut from small and large diameter molded foams, and also a series of molded 7 mm thick disks which were not machined and therefore included a higher density skin near the foam surface. All four series showed similar correlations of density with the level of added siloxane. Only siloxane levels up to 2 phr were used in most cases.

Figures 1 and 2. Epoxy Foam Density vs. PS-120 Siloxane Content



Foam Formulations: Curing Agents and Epoxy Resins

Both Versamid cured systems, similar to RP-1774, and lower viscosity systems cured with Jeffamine D-230, Ancamine 2049 and PACM were investigated. The lower viscosity systems are similar to a solid epoxy encapsulant (Formula 459) now used in some DP applications. The epoxies and curing agents used are shown in Table 2.

Table 2. Epoxy Resins and Curing Agents (Structures shown in Appendix A)

Epoxy Resins:	Viscosity (cps)	Epoxy Equi. Wt.
Epon 826	6,500-9,500	178-186
Epon 828	11,000-15,000	185-192
Epon 830	17,000-22,500	190-198
Curing Agents:		NH Equi. Wt.
Versamid 125	6,500-9,500 @ 75°C	100
Versamid 140	8,000-12,000	125
Jeffamine D-230	9	60
Ancamine 2049	120	60
PACM	6	52.5

Changes in formulation viscosity can strongly effect the foaming behavior in both urethane and epoxy resin systems. Epon/Versamid formulations using Epon resins 826, 828, and 830 and Versamid curing agents V-125 and V-140 were evaluated and clearly showed improved epoxy foaming behavior as the formulation viscosity was increased by going to Epon 828 and 830 and by using Versamid 125 in place of 140.

Lower viscosity formulations cured with Jeffamine/Ancamine/PACM mixtures showed much poorer foaming behavior than the Versamid systems. Initial foaming tests with PS-120 in "Formula 459" (a solid epoxy encapsulant formulation containing 75 parts Epon 826 and 25 parts of a 1/1 Jeffamine D-230/Ancamine 2049 blend) resulted in the hydrogen gas simply bubbling out of the mixture with little or no foaming action. Addition of a surfactant, stearic acid or DC-193, and nucleating additives such as Cab-O-Sil and glass microballoons, gave some improvement, but the foam level and uniformity were still poor. Other alkylsiloxanes, as expected, showed no improvement. Improved foaming was observed when Epon 826 was replaced with the higher viscosity Epon 828 and 830 and when the Ancamine 2049 curing agent was replaced by the more reactive PACM (bis-(para-aminocyclohexyl)methane). PACM alone, without Jeffamine D-230, did not foam as well as the curing agent blend.

While some preliminary dielectric tests were carried out on the Jeffamine/PACM cured epoxy foams, the quality (cell size and uniformity) of these foams was generally much poorer than in the Versamid cured foams. Most of the dielectric testing on epoxy foams, therefore, used a 60/40 Epon 828 or 830 mixture with Versamid 125.

Foam Formulations: Surfactants

Surfactants are commonly used in urethane and other foams to control the foaming behavior. The effect of various commercial surfactants on the epoxy foam formulations was investigated with a standard 30/20/0.2/0.8 Epon 828/Versamid 125/PS-120/surfactant formulation. Over thirty mixes were evaluated for foam quality using a 150°F oven cure. The various surfactants used are shown in Appendix B. Of these, the most promising was DC-5357, a siloxane/glycol ether copolymer.

In addition to the above trials, a pre-reacted blend of stearic acid with a mixture of Versamid 125 and 140 was evaluated. This surfactant, presumably a stearic acid salt, was found to have superior foam stabilizing properties in a prior study of frothed epoxy foams (Ref. 18). A 140/47/2.2 gram mixture of Versamid 140, Versamid 125 and stearic acid was stirred and heated to dissolve the stearic acid. When this curing agent/surfactant blend was used in place of standard Versamid in a PS-120 foamed mixture, the level and uniformity of the foaming was actually decreased and no further evaluations were carried out. Use of stearic acid alone, with no pre-reaction, provided better foam behavior, and both were surpassed by the siloxane/glycol ether type surfactants.

While Versamid cured foams with surfactants showed slightly better uniformity in hand-mixed trials, blade-mixed samples showed only marginal differences with and without added surfactant. As noted earlier, the use of a surfactant generally gave slightly lower densities at the same PS-120 concentration, an effect that made adjustments of the foam density more difficult due to the smaller range of PS-120 used. The molded foams prepared for dielectric testing were formulated without added surfactant.

The lower viscosity Jeffamine/Ancamine 2049 cured formulations also showed lower densities and better uniformity when 1 phr of DC-193 surfactant was added. Higher surfactant levels gave no further improvement and other surfactants were not investigated with this formulation. This improvement was less noticeable when Ancamine 2049 was replaced with the faster curing PACM and those materials were also molded without surfactant for dielectric testing.

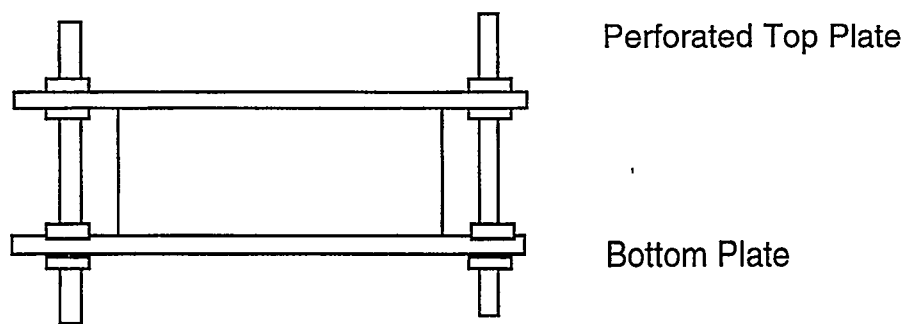
Processing Studies

Many of the initial screening evaluations was carried out on simple hand-mixed formulations. An overhead mixer with 2-3 inch Conn blades was used during later formulation trials to determine the density vs. siloxane blowing agent relationship and to prepare samples for dielectric testing. Blade mixing generally gave slightly lower densities due to air entrapment and also better uniformity.

Most of the epoxy foams, when cured at room temperature and then post-cured in an oven, showed some shrinkage of the foam. More stable and uniform foams were obtained with oven cures at 125-150°F with pre-heated molds. Larger samples

showed darkening in the foam interior, evidence of a significant exotherm, when cured at 150°F.

The molds used consisted of a bottom steel plate, a top steel plate perforated with small holes to allow gas and foam escape, and steel cylinders of various heights between the plates. Two series of molds were fabricated, an initial series with cylinders having a 5 inch diameter and a second series having a larger 6 inch diameter. The large diameter molds were required to provide samples for the larger electrodes fabricated for the dielectric testing. Cylinders of 1-3 inches in height were used to prepare foams that were later machined into 7 mm thick disks. Densities were measured on the individual test disks and did show some variance from the top to bottom of the molded sample in some of the larger molds. Very thin, 7 mm, steel cylinders were used to mold the individual dielectric samples tested with the molded skin



Steel Molds: Side View

Foam Density and Structure

Foam density in the epoxy foams was controlled by the level of added PS-120 methylhydro-siloxane up to levels of about 2 phr. Comparison polyurethane foams were made from a standard CRETE formulation (Ref. 19) based on varying levels of:

Isonate 143L (modified methylene diisocyanate from Dow Chemical, isocyanate equivalent weight, 144.5; functionality 2.1; viscosity 33 cps)

Voranol 490 (propylene oxide polyol from Dow Chemical formed by extension of a sucrose /glycerin base; functionality 4.3; avg. molecular wt. 460; viscosity 5572 cps)

Polycat 17 (tertiary amine catalyst from Air Products)

DC-193 (siloxane/glycol ether surfactant from Air Products)

water (generates carbon dioxide blowing agent)

This polyurethane foam is one of several which have been evaluated as replacement encapsulation foams for DP applications. The urethane foams were typically more exothermic and were cured several hours at room temperature before an overnight post-cure at 150°F. Foams were prepared over the same general range of densities as the epoxy foams.

Visual and SEM examinations of the epoxy and urethane foams showed the urethanes to have smaller and more uniform cell sizes. Figures 3 and 4 show low magnification and SEM (50x) crosscuts of both foam types over a range of densities. Figures 5 and 6 show similar side-by-side comparisons of three sets of foams at the same density. The difference in cell size at similar densities is most apparent in the side-by-side comparisons. At the lowest densities, both the epoxy and urethane foams were less uniform and showed the presence of large cells, particularly in the lower magnification pictures. Additional foam pictures are contained in Appendix C.

While it was anticipated that cell structure might have a strong influence on dielectric properties, the results as a whole did not show such trends for these particular samples. Earlier epoxy foam samples made with the Jeffamine D-230/PACM curing agent were significantly more non-uniform and did show poor performance in some tests that might be attributed to the poor foam morphology.

Density
(gr./cc)

0.23

0.30

0.44

0.51

0.53

0.61

0.69

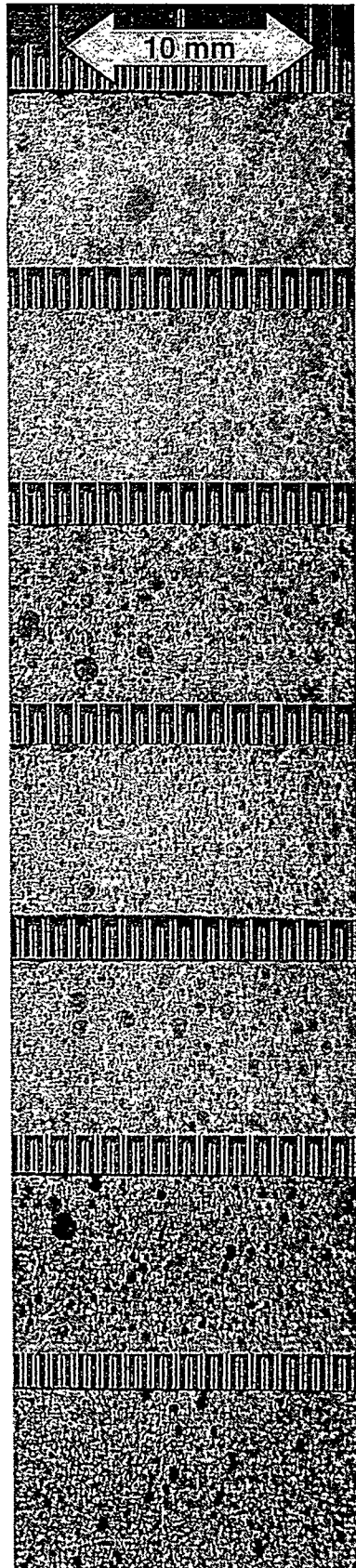


Fig. 3. Cross-Section Views of
Foams Used in Resistivity Tests



Epoxy Foams
(Versamid Cured)

Urethane Foams
(CRETE)



Density
(gr./cc)

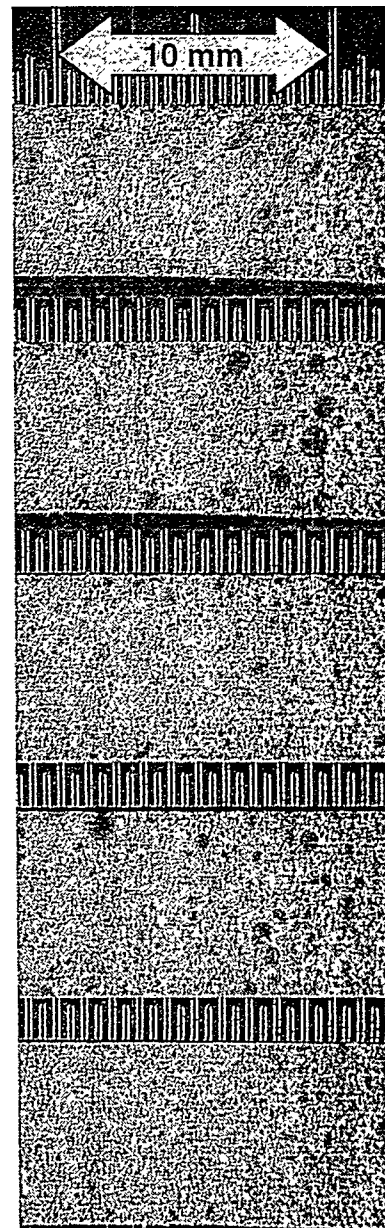
0.15

0.22

0.31

0.37

0.50



Density
(gr./cc)

0.23

0.30

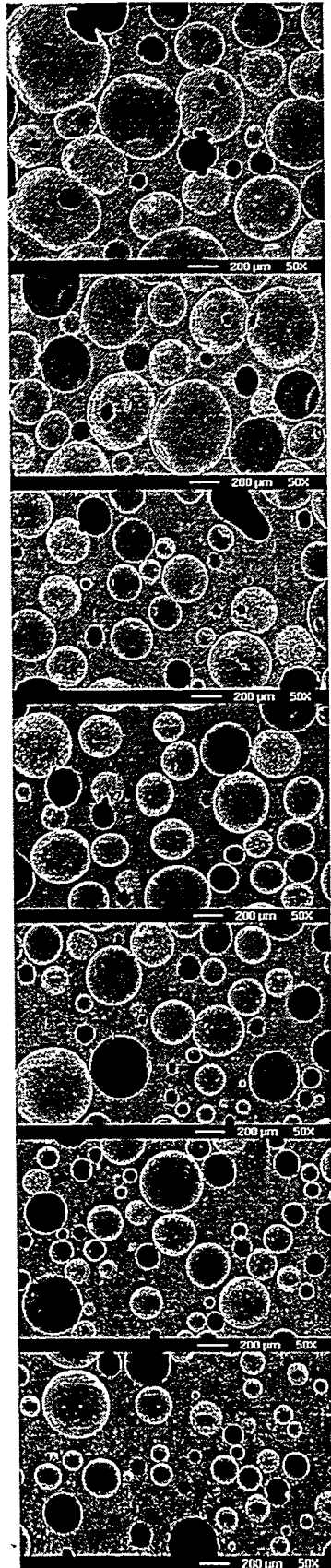
0.44

0.51

0.53

0.61

0.69



**Fig. 4. SEM Cross-Section Views
of Foams Used in Resistivity Tests
(all at 50X)**



Epoxy Foams
(Versamid Cured)



Urethane Foams
(CRETE)

Density
(gr./cc)

0.15

0.22

0.31

0.37

0.50

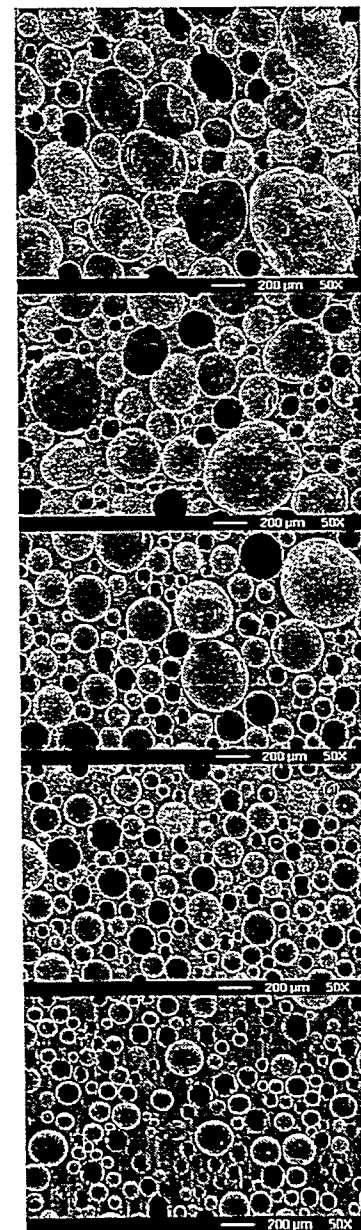


Fig. 5. Cross-Section Foam Comparisons: Epoxy vs. Urethane at Same Density (gr/cc)

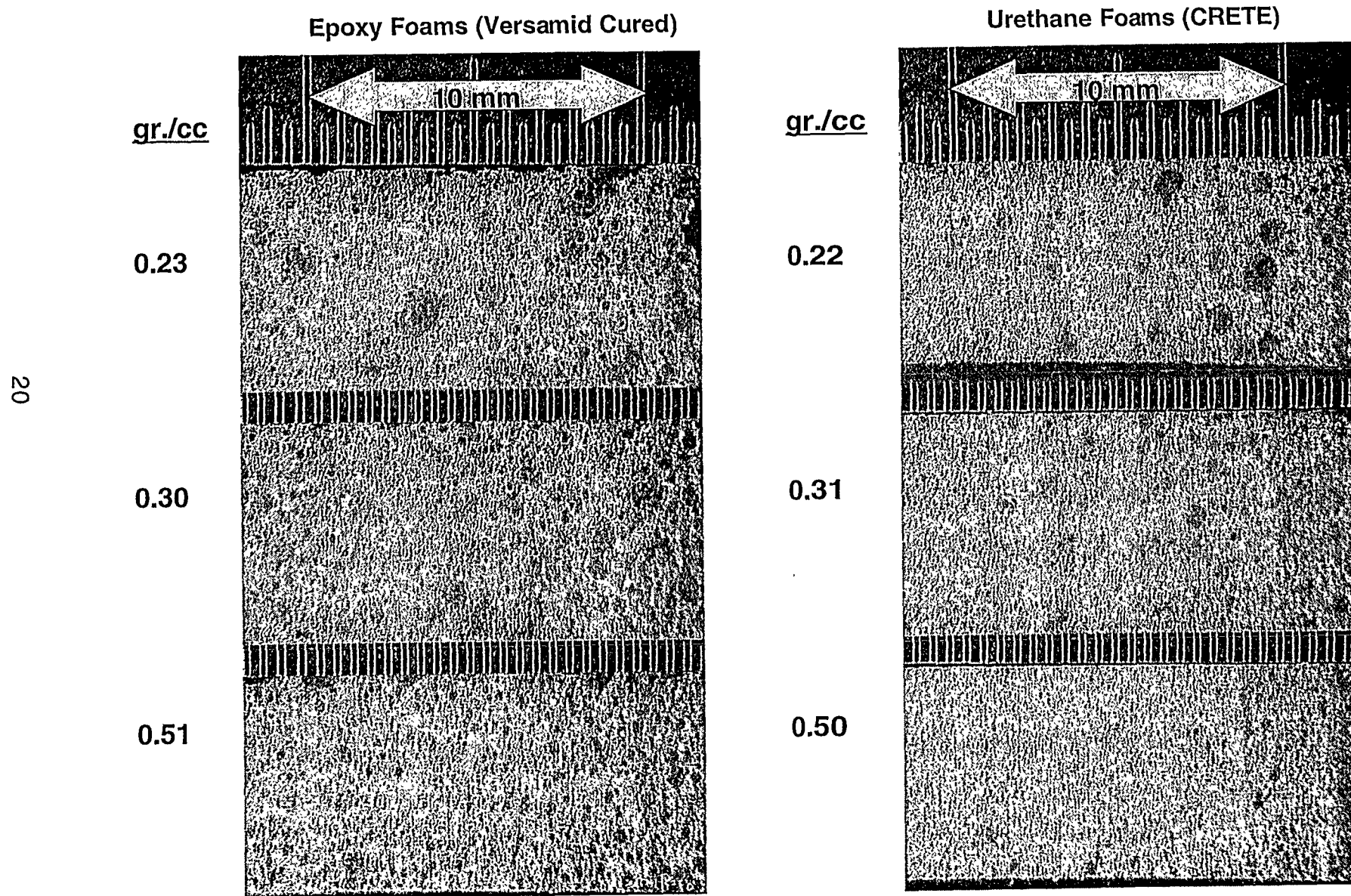
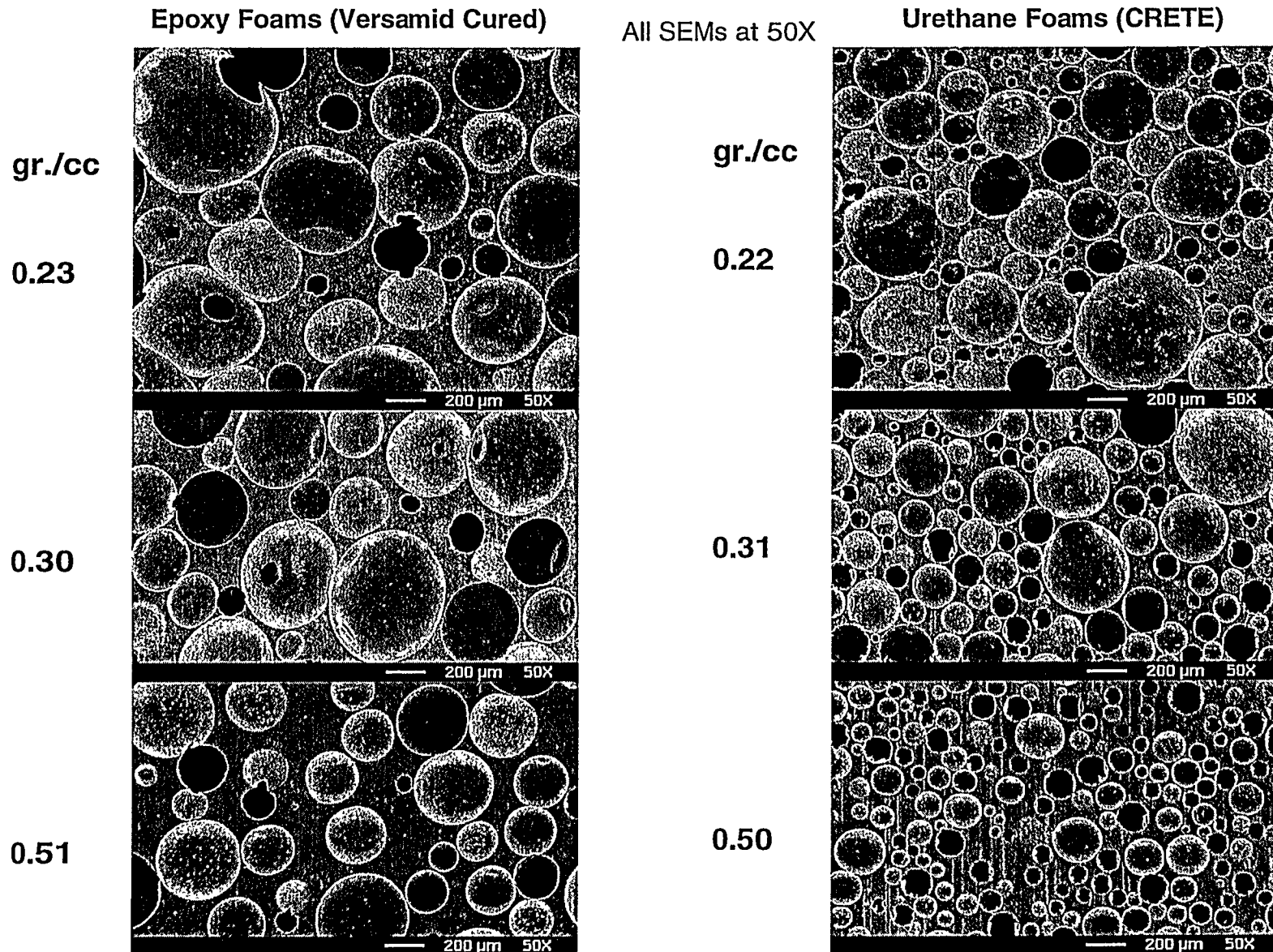
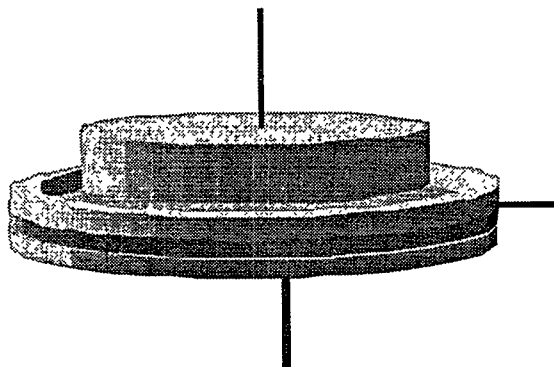


Fig. 6. SEM Cross-Section Foam Comparisons: Epoxy vs. Urethane at Same Density (gr/cc)



Dielectric Testing Procedures

Dielectric tests were carried out with cylindrical brass electrodes and a guard ring such as described in ASTM D150-94 and 257-93 and shown below. Foam samples were either machined disks cut from large molded billets or as-molded disks of the appropriate diameter and a thickness of about 7 mm. A detailed description of the test



equipment and procedures are given in Appendix D.

Polymers, i.e., plastics, have often been utilized because of their insulating capabilities. This capability is often characterized by dielectric constant, dissipation factor and volume resistivity.

Dielectric constant is the ratio of the capacitance of a capacitor, such as the test assembly above, containing the material of interest relative to the same system in which air (dielectric constant of 1) is the dielectric medium. It is measured with an alternating current. Values of 2-10 are typical of insulators and are generally sensitive to temperature and also the frequency. It is a dimensionless parameter and lower values are preferred for high frequency insulation.

Dissipation factor, also called the power factor in some cases, is the tangent of the dielectric loss angle. It describes the power lost, rather than stored, in an alternating current in which the material must rapidly change polarization. It is again a dimensionless and lower values are preferred.

Volume resistivity, measured in ohm-cm, directly measures the resistance in a non-alternating current of the material of interest. A high voltage, 1000 volts in most of the tests described here, is applied across the insulating material and the resulting current is measured. Most unfilled and unfoamed plastics have volume resistivities in the 10^{10} to 10^{15} range. A few plastics such as DAP (diallyl phthalate) and Teflon have values in the 10^{16} and 10^{17} range which was also observed for the more highly insulating foam samples studied here. Currents were measured in the pico (10^{-12}) amp range.

Dielectric Testing Results (Data in Appendix E)

Initial tests with 4 inch x 7 mm machined disks of two epoxy and two urethane foam formulations were carried out with the smaller electrodes in Sandia, NM. The epoxies were the Versamid cured formulations and the Jeffamine/PACM cured formulations while the urethanes were either the TDI-based foam now prevalent in the stockpile or the newer CRETE family of urethanes. The density range of these samples was limited and the quality of the foams was also variable. As shown in Fig. 7, however, there was a strong correlation of dielectric constant with foam density, independent of the resin type or the test frequency (1-1000 KHz). Dissipation factors, shown in Fig. 8, showed much greater scatter in the results with no clear correlation to resin type or density. The test frequency was clearly more dominant in this case although values measured at 1 KHz were considered too erratic to be reliable.

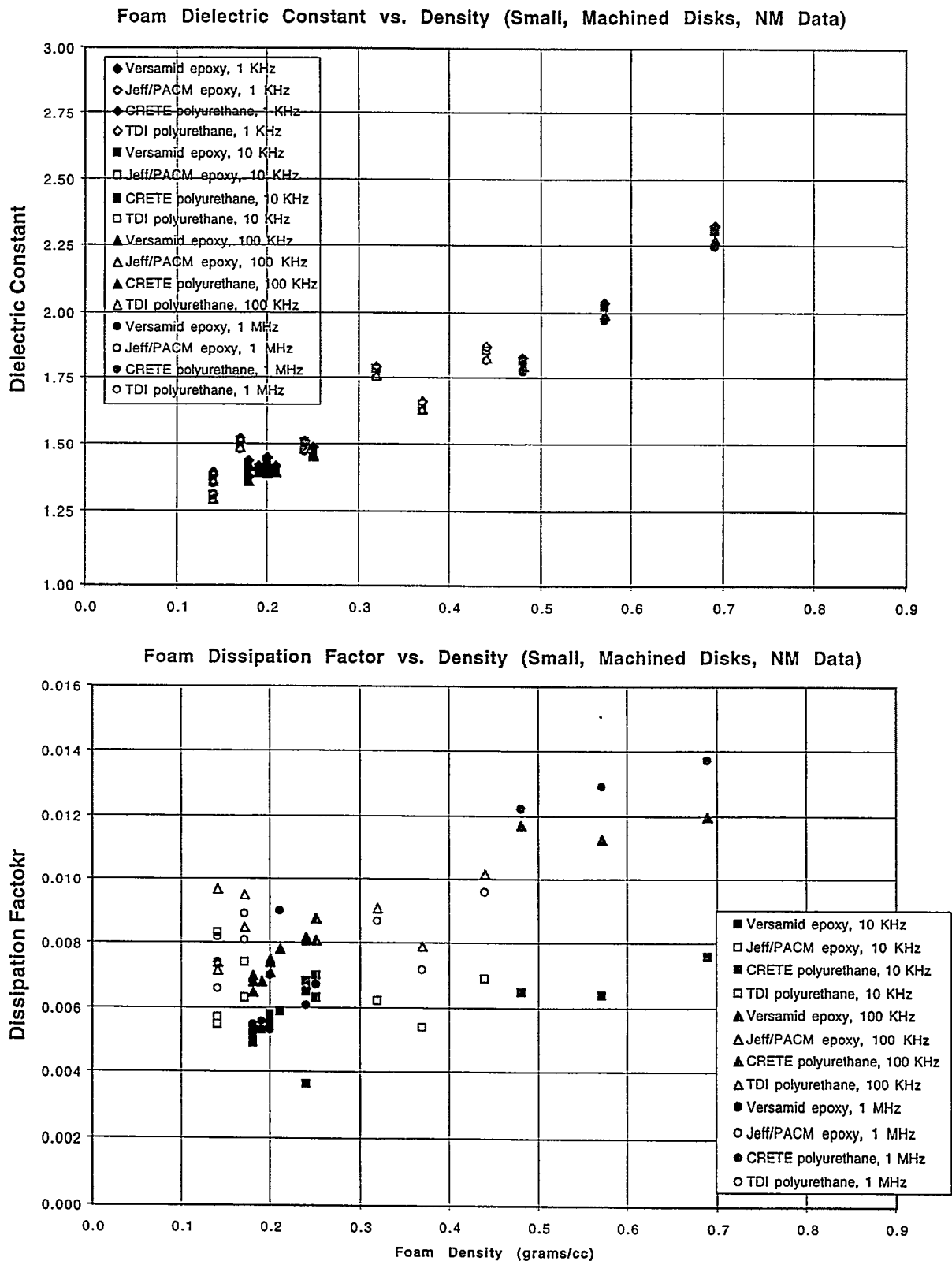
The initial volume resistivity tests were made at 3 kilovolts (KV) due to the sensitivity required and, as shown in Fig. 9, were largely in the 10^{16} ohm-cm range. Values were also measured at 1 KV on this series of samples but the data was erratic and in the same range as the noise. These initial results showed varying dependence of the resistivity on foam density. The poorer quality of the epoxy foams cured with Jeffamine/PACM appeared to result in lower resistivity for that group of samples. Overall, no clear conclusions could be drawn about the effects of resin type and foam density on volume resistivity from these tests.

It was because of the high sensitivity required that the electrodes fabricated for further testing in California were made with a larger surface area, roughly four times the area of the smaller electrodes. A second series of foams with the larger 5 inch diameter were fabricated and tested both in California using these larger electrodes and also in New Mexico. While the electrodes used in New Mexico were still the smaller size, the higher quality of the foam samples in this series did provide less erratic data and the agreement between the two test locations was, ultimately, very good. These larger foams were prepared from only the Versamid cured epoxies and the CRETE family of urethanes.

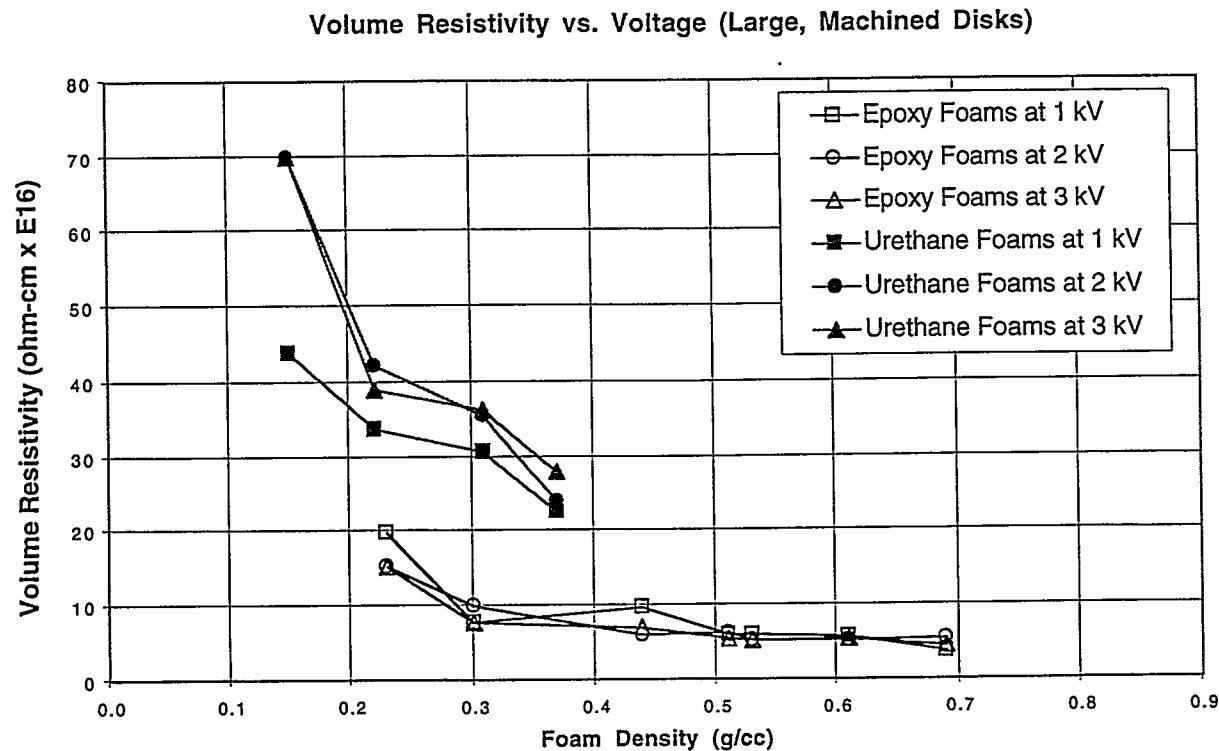
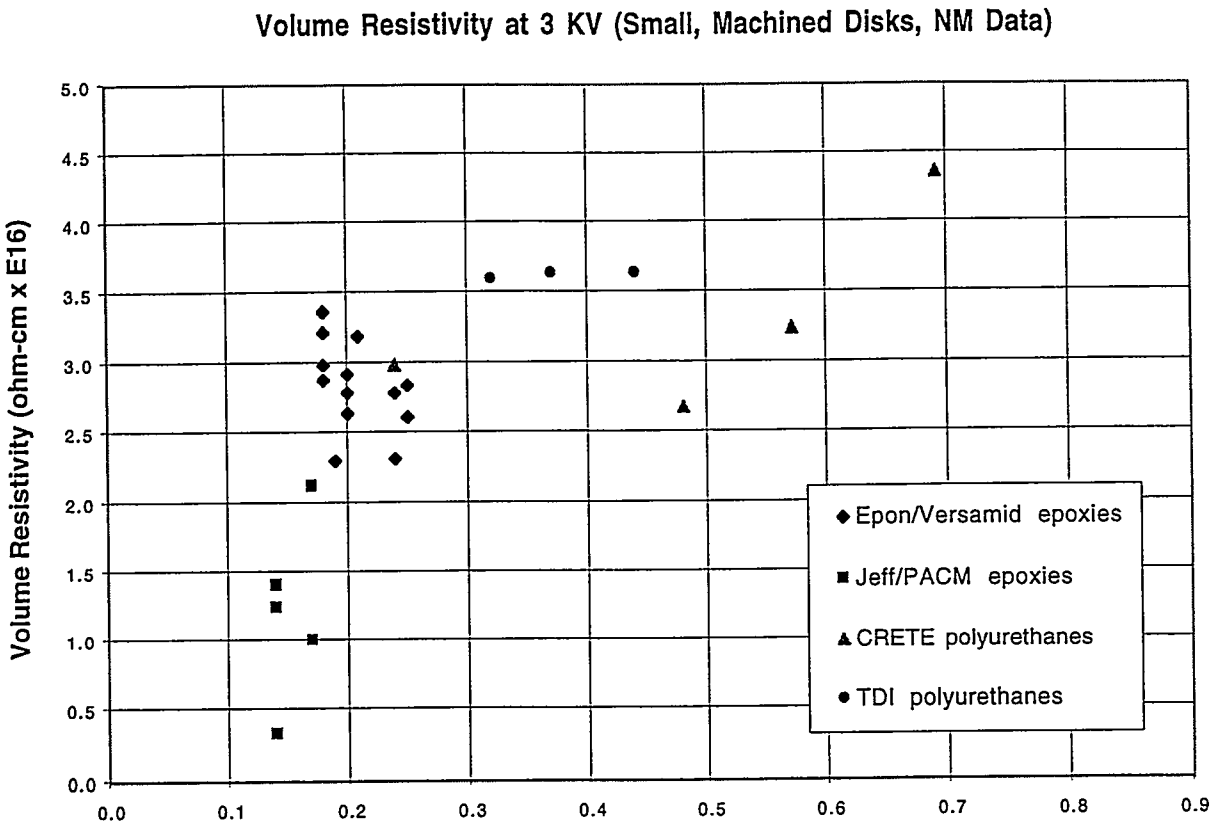
One series of tests in New Mexico measured the dependence of volume resistivity on the applied voltage. As shown in Fig. 10, neither the epoxy or urethane samples showed a strong dependence on the applied voltage. The resistivity values shown in the resistivity vs. voltage comparisons in Fig. 10 are clearly higher than seen in Figure 9, particularly at lower densities. This is attributed primarily to improvements in the quality of the samples. All the volume resistivity tests at California were conducted at 1 kilovolt.

Numerous Round Robin comparisons were made between the two Sandia Labs until the test equipment and procedures gave good agreement. One procedure considered was the insertion of a carbon cloth insert between the foam and electrode surfaces to provide better electrical contact. As shown in Figs. 11 and 12, such inserts resulted in little difference in the volume resistivity measurements and an average increase in the

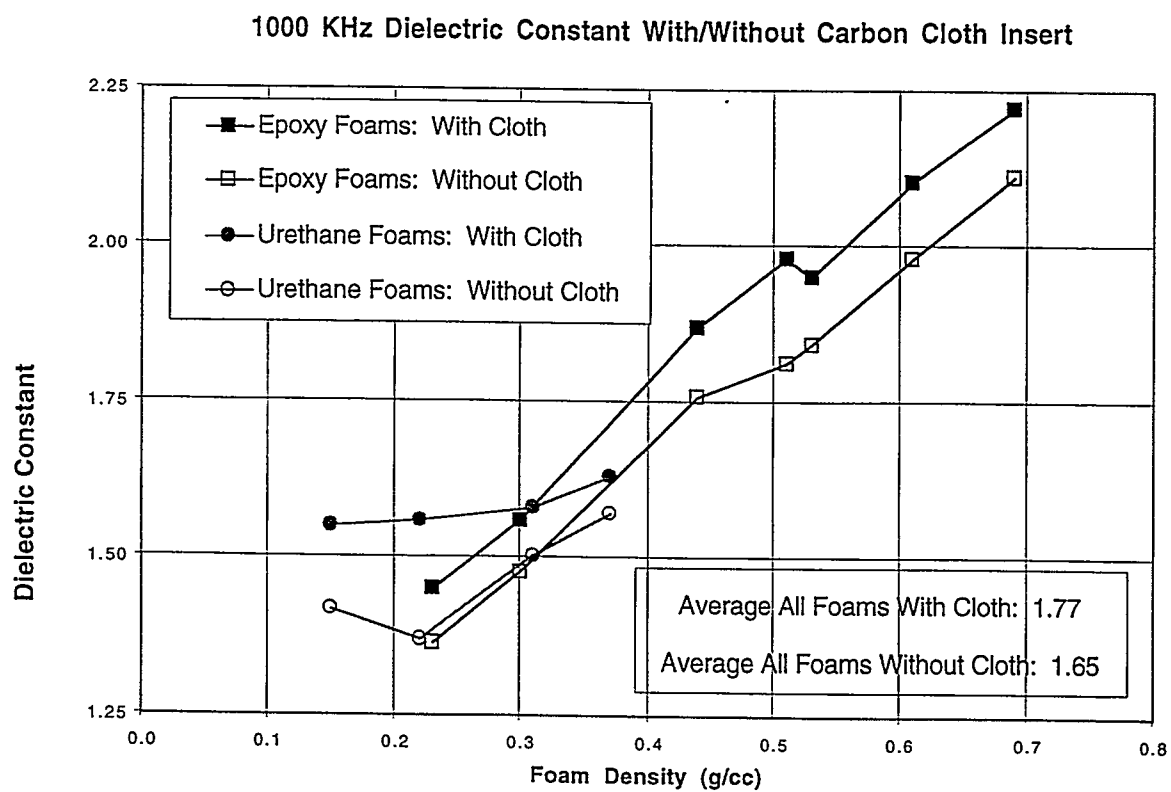
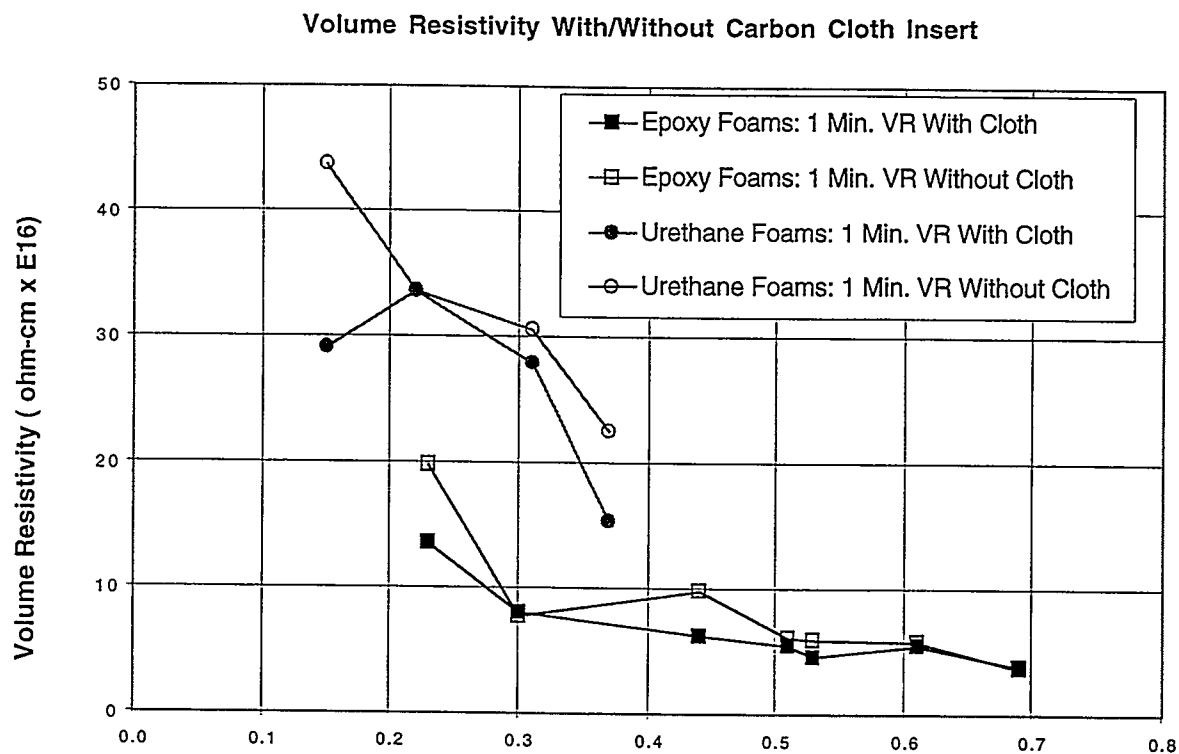
Figures 7 and 8. Dielectric Constants and Dissipation Factors of Preliminary Samples
(Sandia, NM Data)



Figures 9 and 10. Volume Resistivity of Preliminary Samples (Sandia,NM Data)



Figures 11 and 12. Volume Resistivity and Dielectric Constant vs. Carbon Cloth Insert
(Sandia, NM Data)



dielectric constant of only 0.12. Further measurements were all made without the carbon cloth insert.

Measurements on 7 mm thick machined foam disks showed a strong correlation of dielectric constant with foam density for both the epoxy and urethane materials. This is shown in Figs. 13 and 14. A slight dependence of dielectric constant on frequency is apparent. As noted above, the agreement in test values between NM and CA was generally good. Dissipation factors for these same samples, shown in Figs. 15 and 16, were less well behaved although a general correlation with density was again observed.

Volume resistivity measurements at both one minute after the voltage was applied (ASTM procedure) and three minutes are shown in Figs. 17 and 18 and indicated little dependence on foam density. All the samples had very high resistivity. The values measured in CA tended to be slightly more conservative than those made in NM, but the agreement was generally good. All these resistivities required sensitivity in the picoamp range and all showed the expected increase in resistivity between one and three minutes. The resistivity was approaching equilibrium at three minutes with much less variability.

When used as an encapsulant, the foams would not be machined and would possess a skin of higher density and varying thickness near the mold or substrate surface. A series of molded foam samples, again 7 mm thick, were prepared and tested in the same manner as the machined foams to determine the effects of such skins on the dielectric performance.

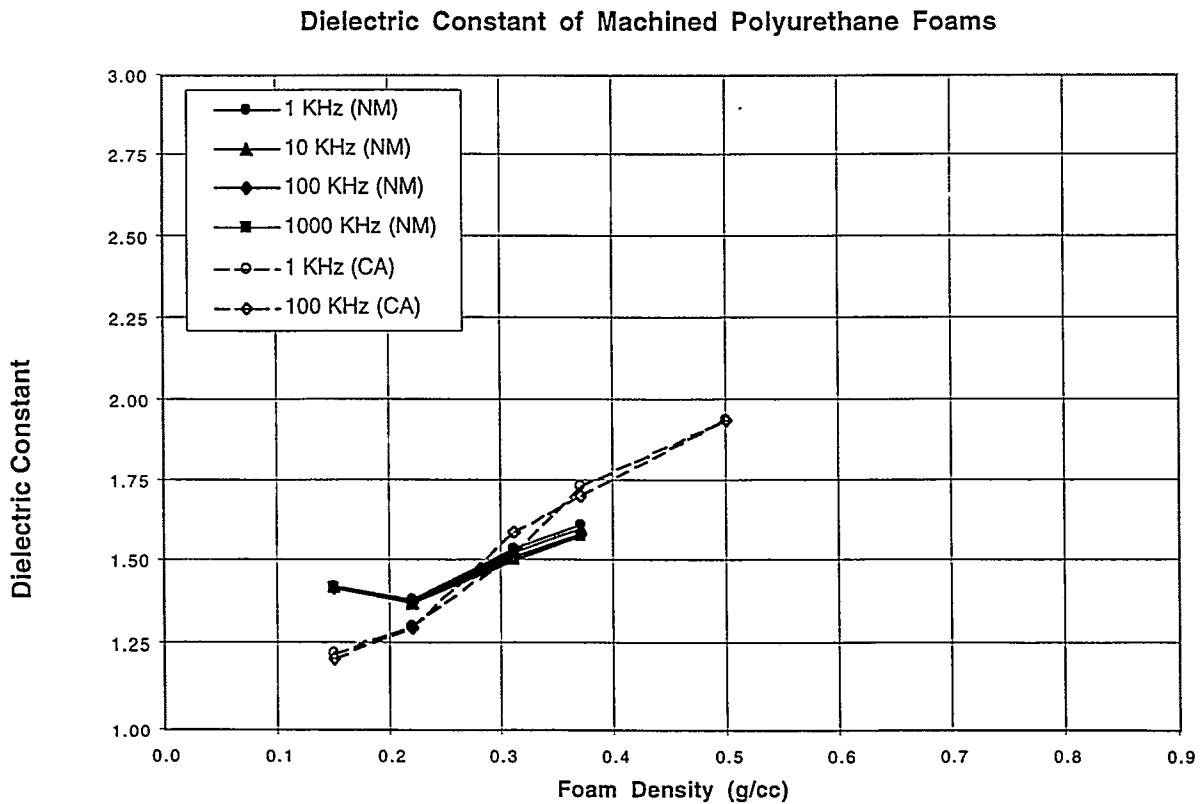
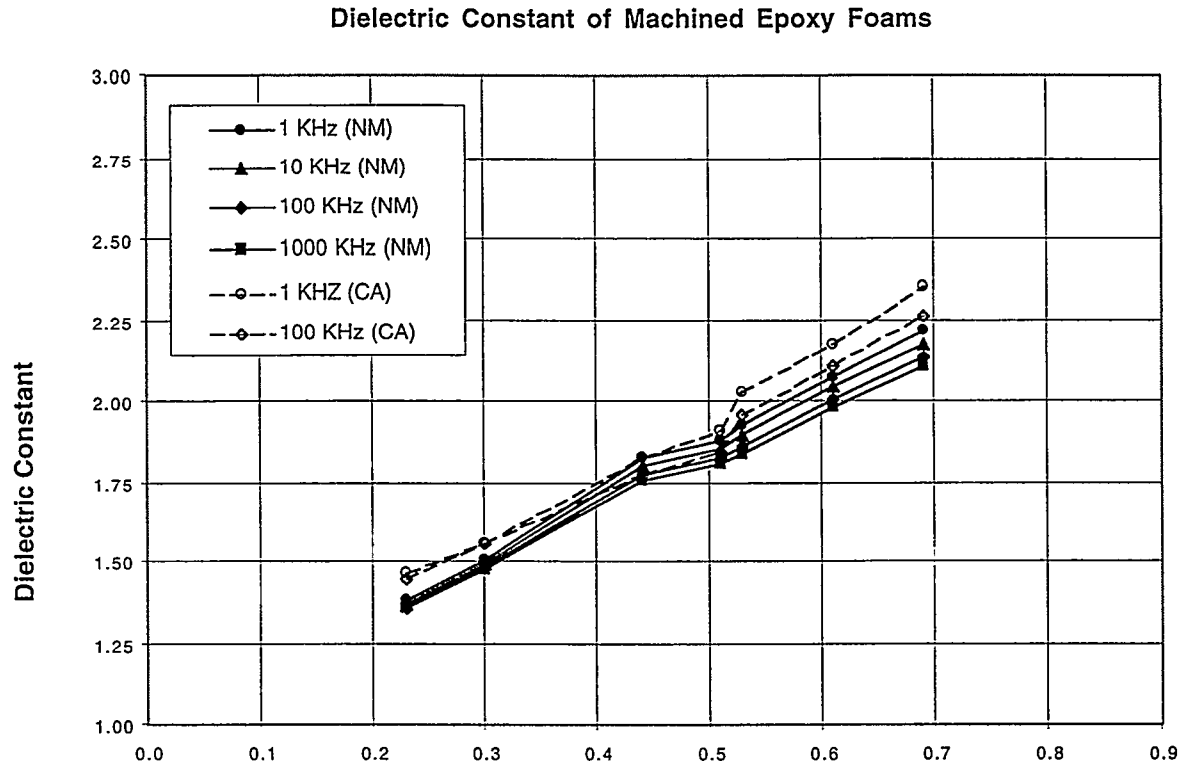
As shown in Figs. 19 and 20, the dielectric constant of the as-molded foams again showed a strong correlation with density. Two series of epoxy samples were prepared, one with Epon 828 and a second with Epon 830 and slightly higher levels of siloxane blowing agent to insure more uniform mold filling. Both series gave similar results which are combined in Fig. 19.

The dissipation factors of these same as-molded foams were better behaved than in the machined samples and are shown in Figs. 21 and 22. There was a slight correlation with foam density and a strong correlation with test frequency, particularly between 100 and 1000 KHz.

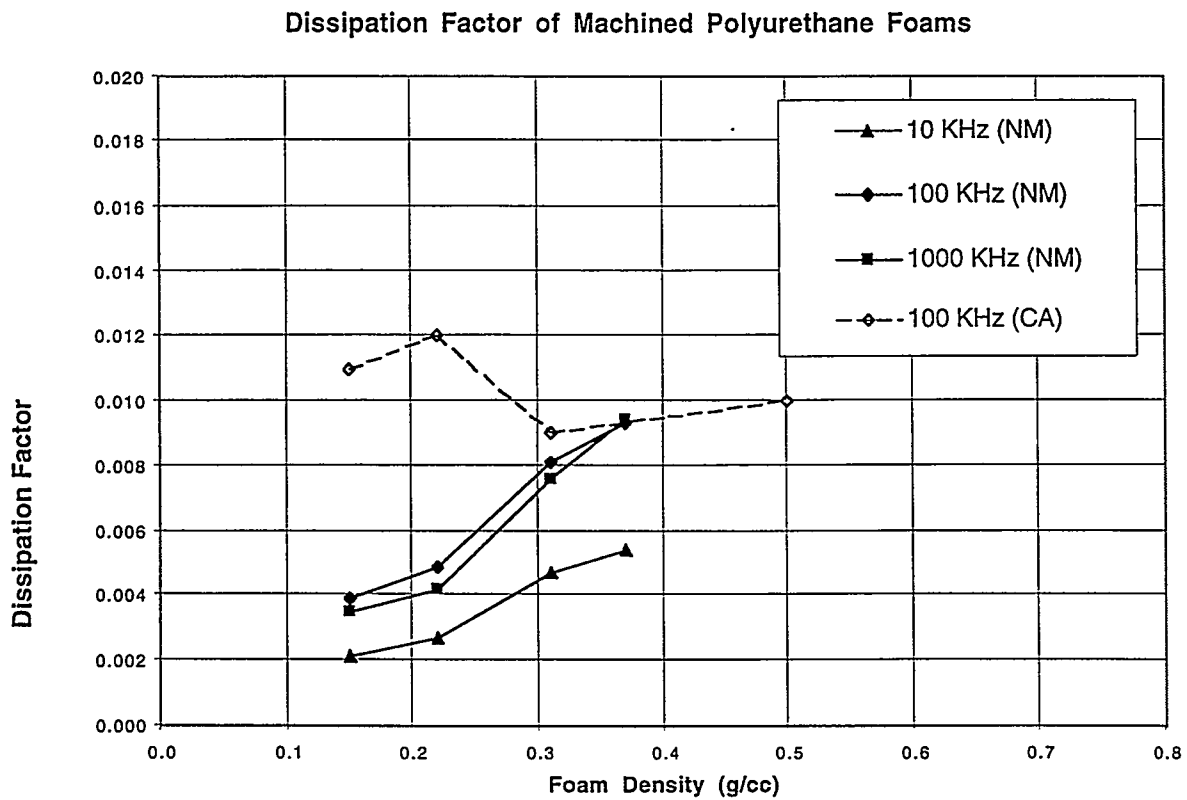
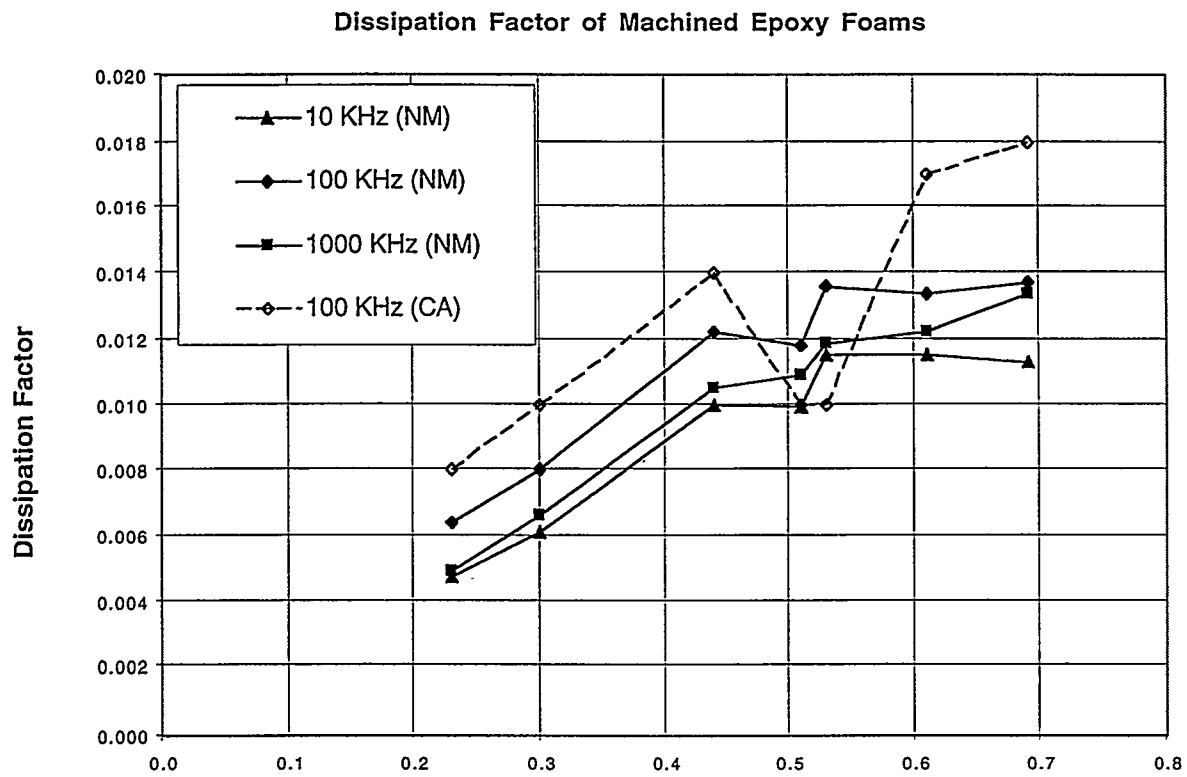
The volume resistivity results on these as-molded samples, as in the machined samples, showed little dependence on foam density except at the very lowest urethane density of about 0.2 g/cc. These results are shown in Figs. 23 and 24.

Pulling all the above results together, Figs. 25 and 26 compare, at 100 KHz, the dielectric constant and dissipation factor of both the epoxy and urethane foams with both machined and as-molded samples. The dielectric constant of all these materials was, again, clearly dependent on foam density only with no significant differences due to the material or the presence or absence of a surface skin. The dissipation factors

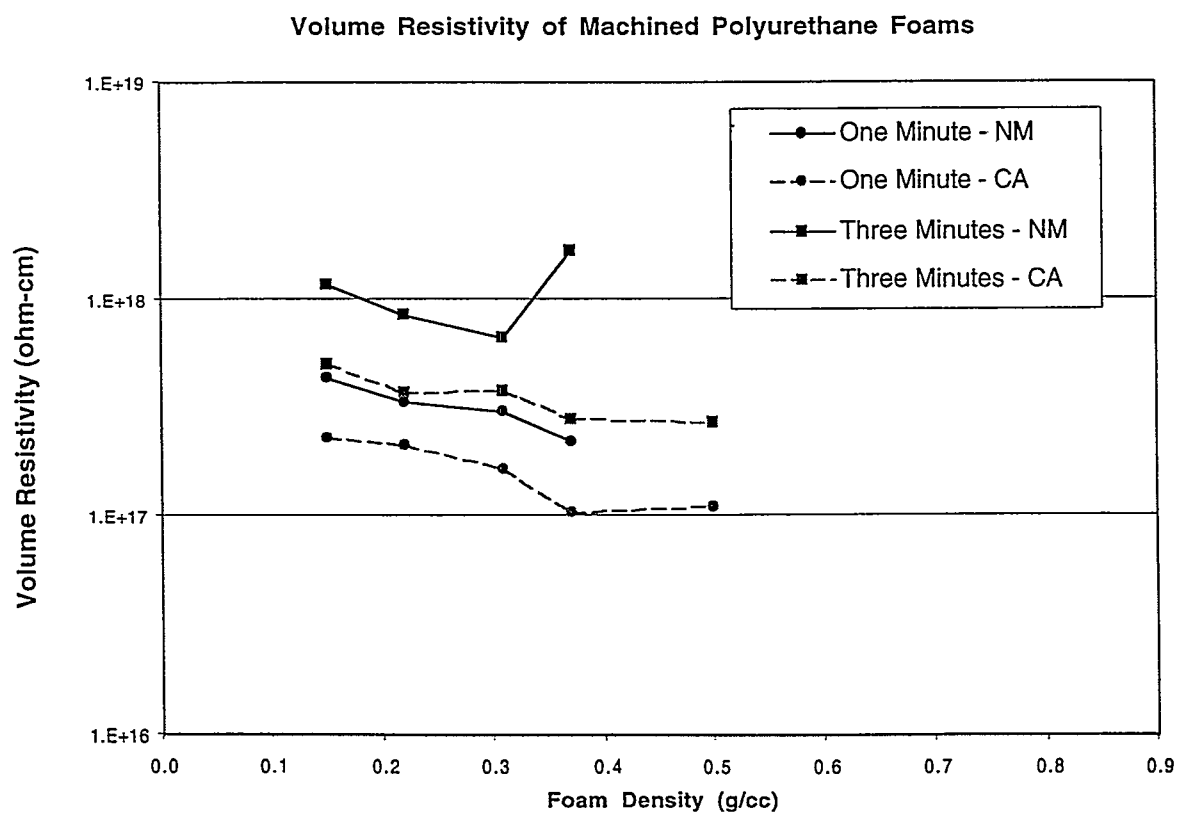
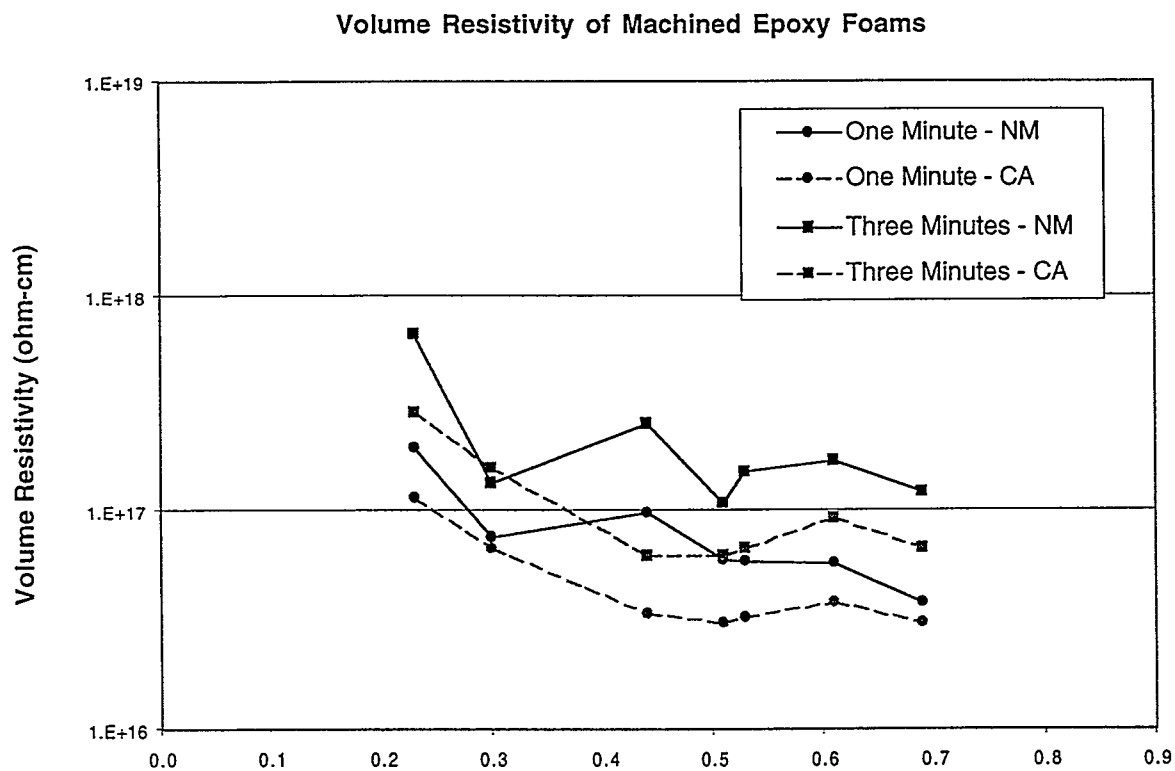
Figures 13 and 14. Dielectric Constants of Machined Epoxy and Urethane Foams
(Sandia, NM and CA Data)



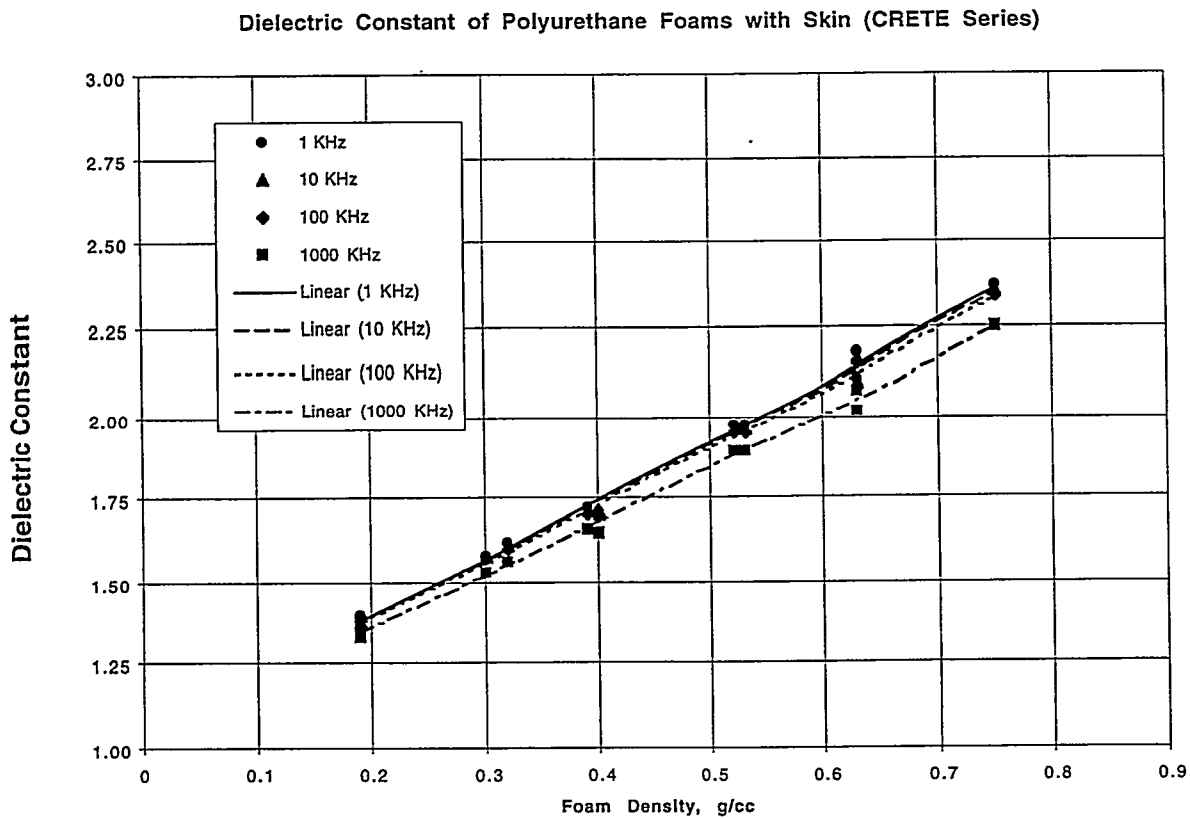
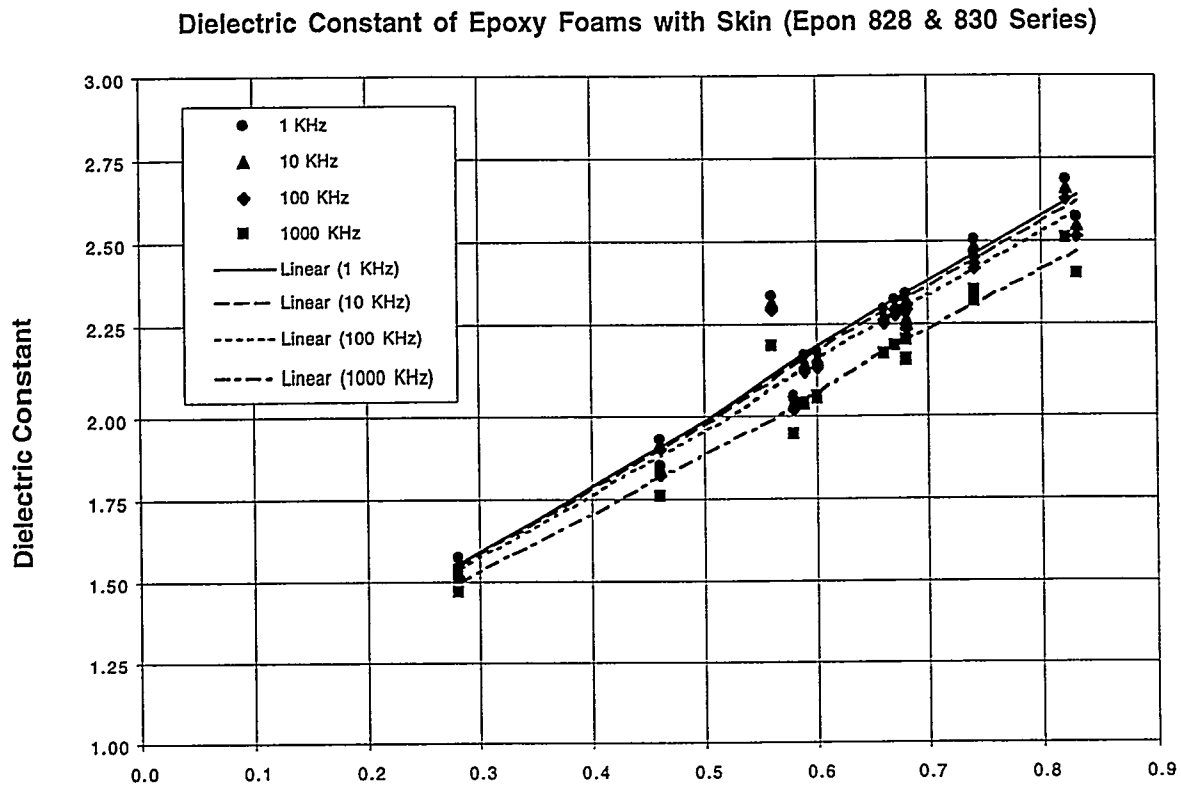
Figures 15 and 16. Dissipation Factors of Machined Epoxy and Urethane Foams
(Sandia, NM and CA Data)



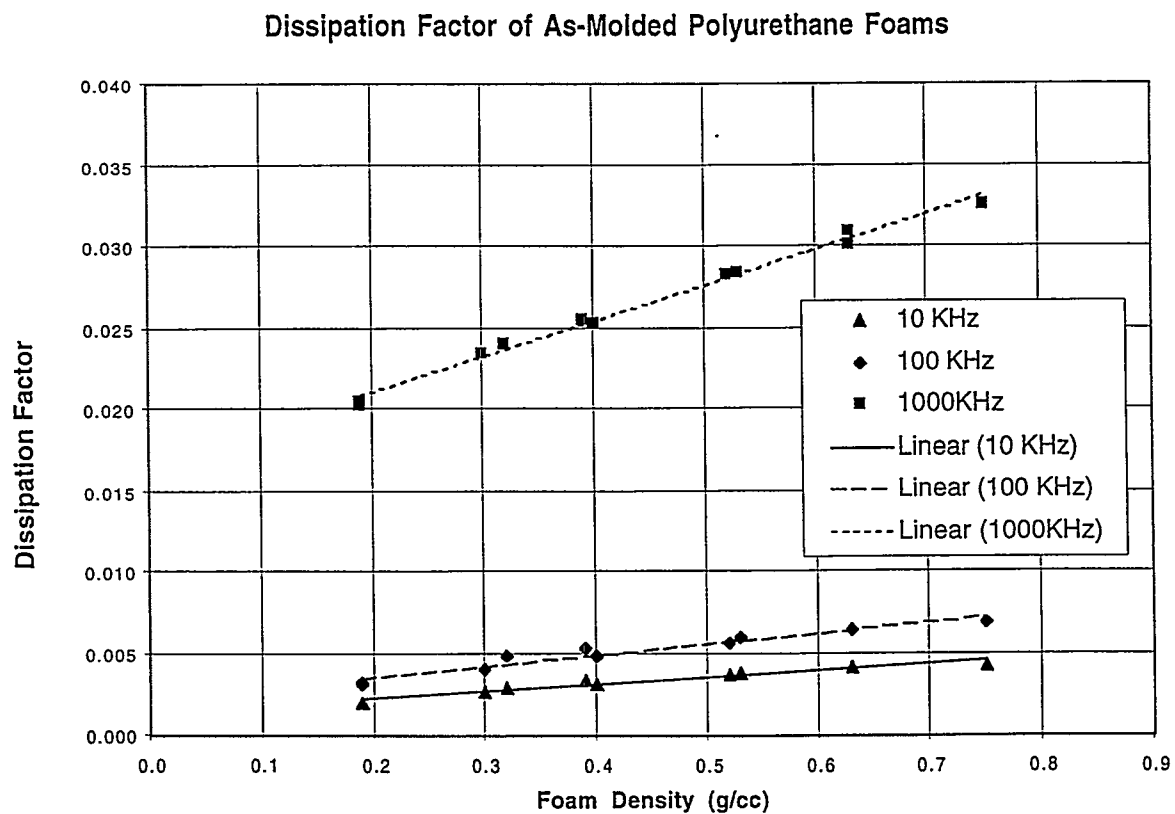
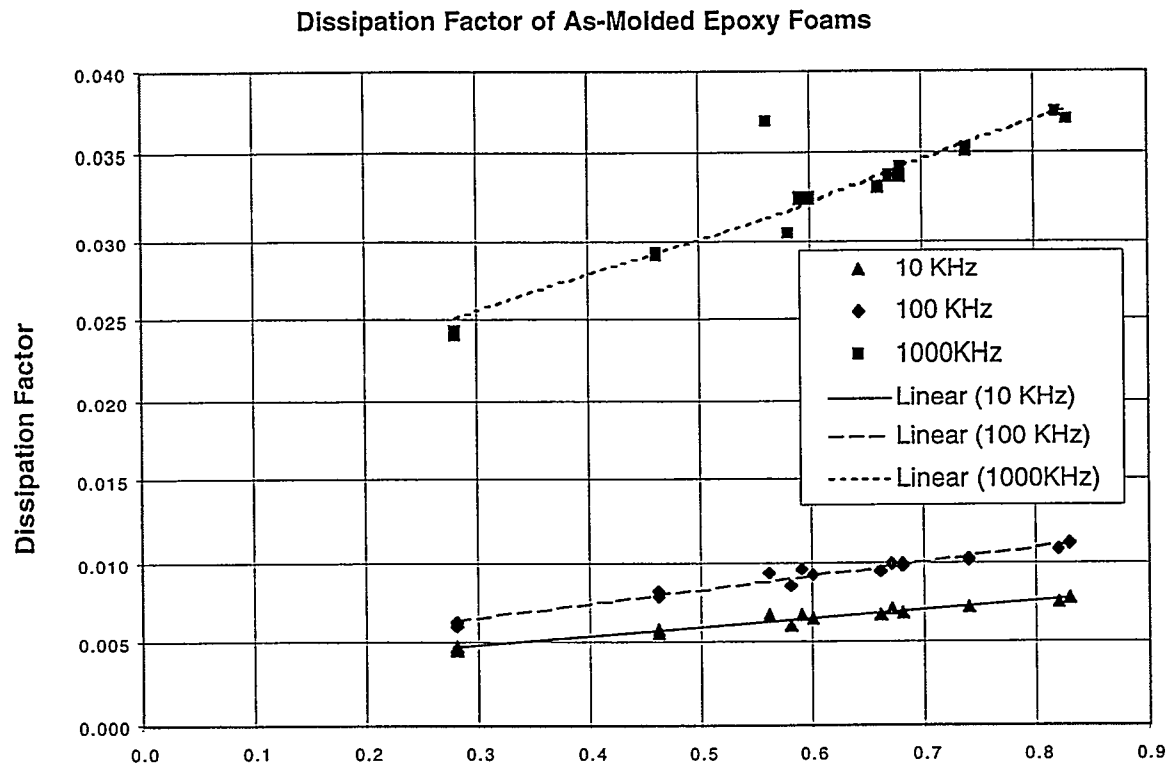
Figures 17 and 18. Volume Resistivity of Machined Epoxy and Urethane Foams
(Sandia, NM and CA Data)



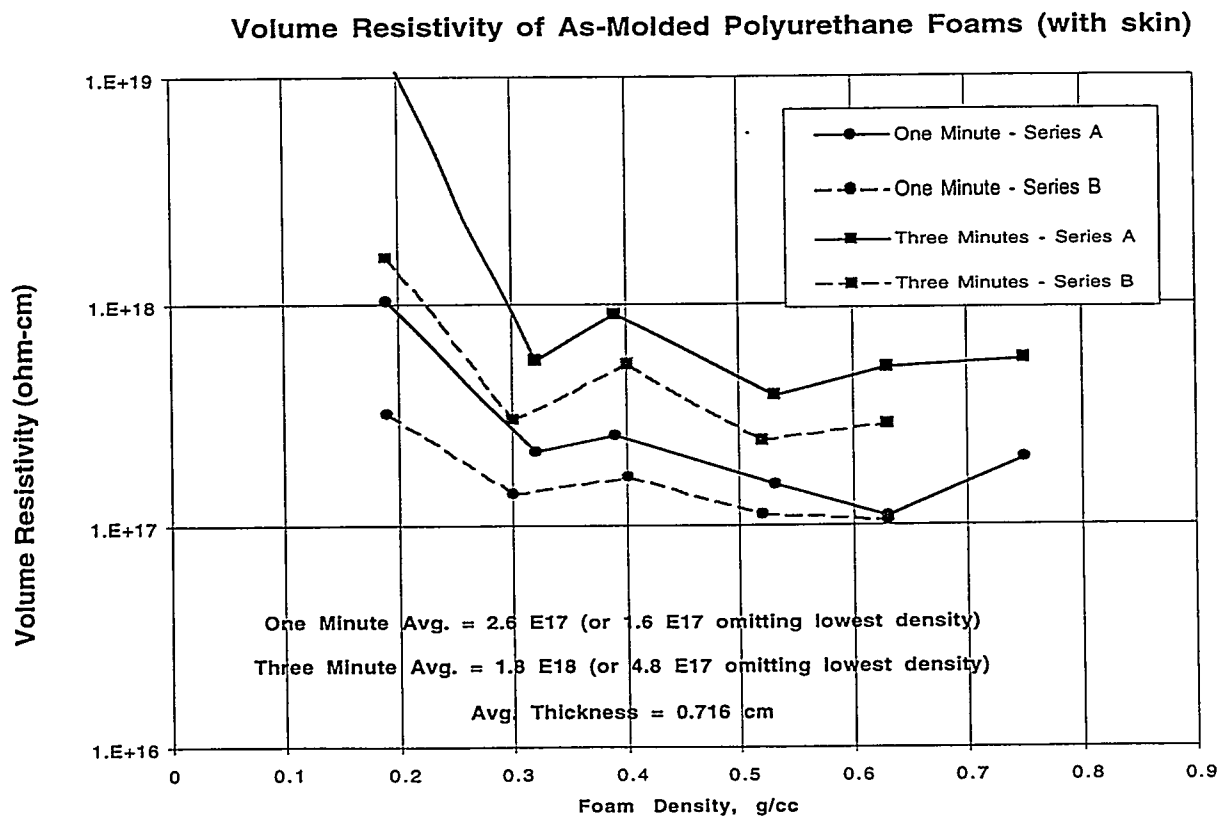
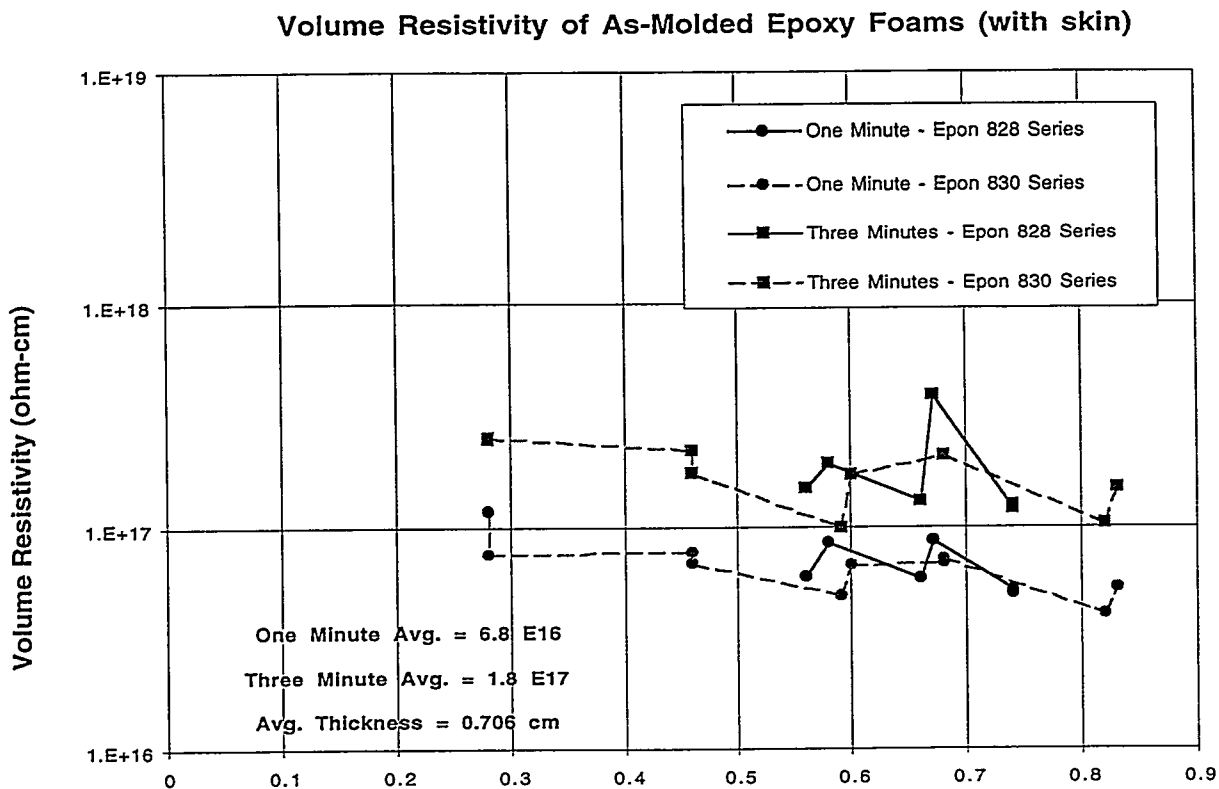
Figures 19 and 20. Dielectric Constants of As-Molded Epoxy and Urethane Foams (with skins)
(Sandia, CA Data)



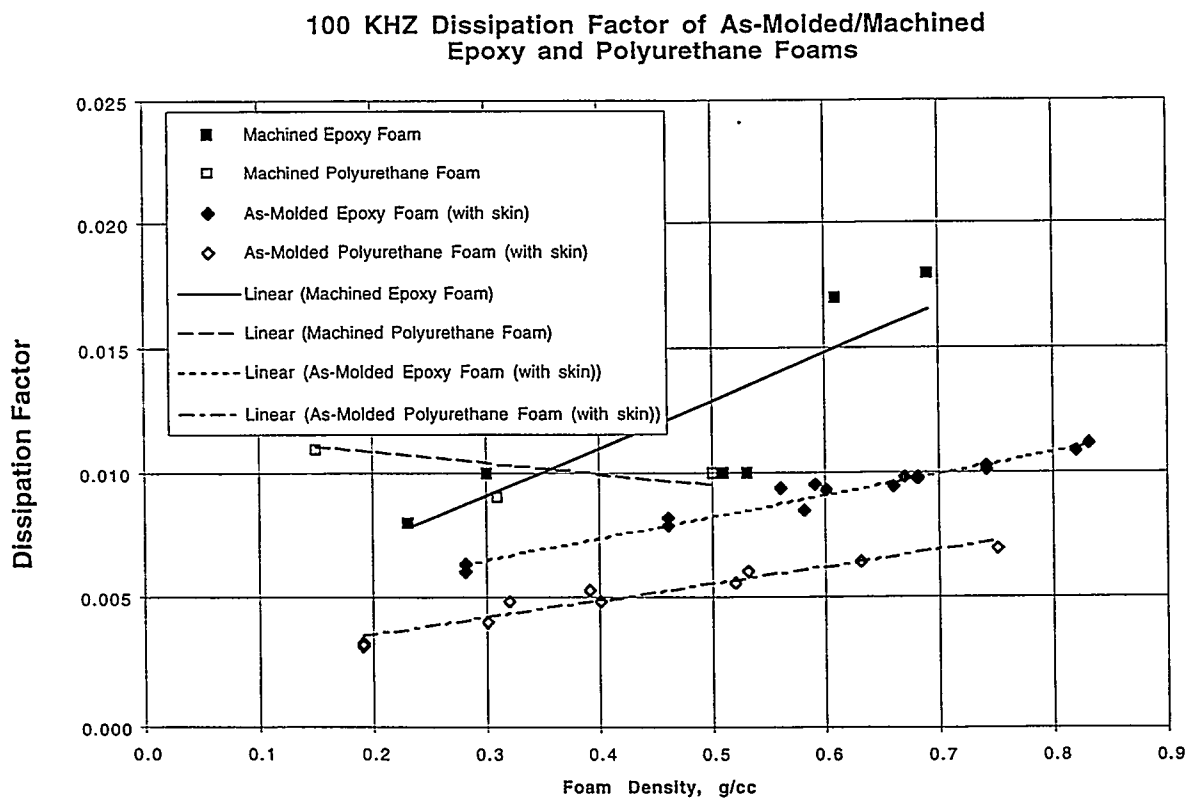
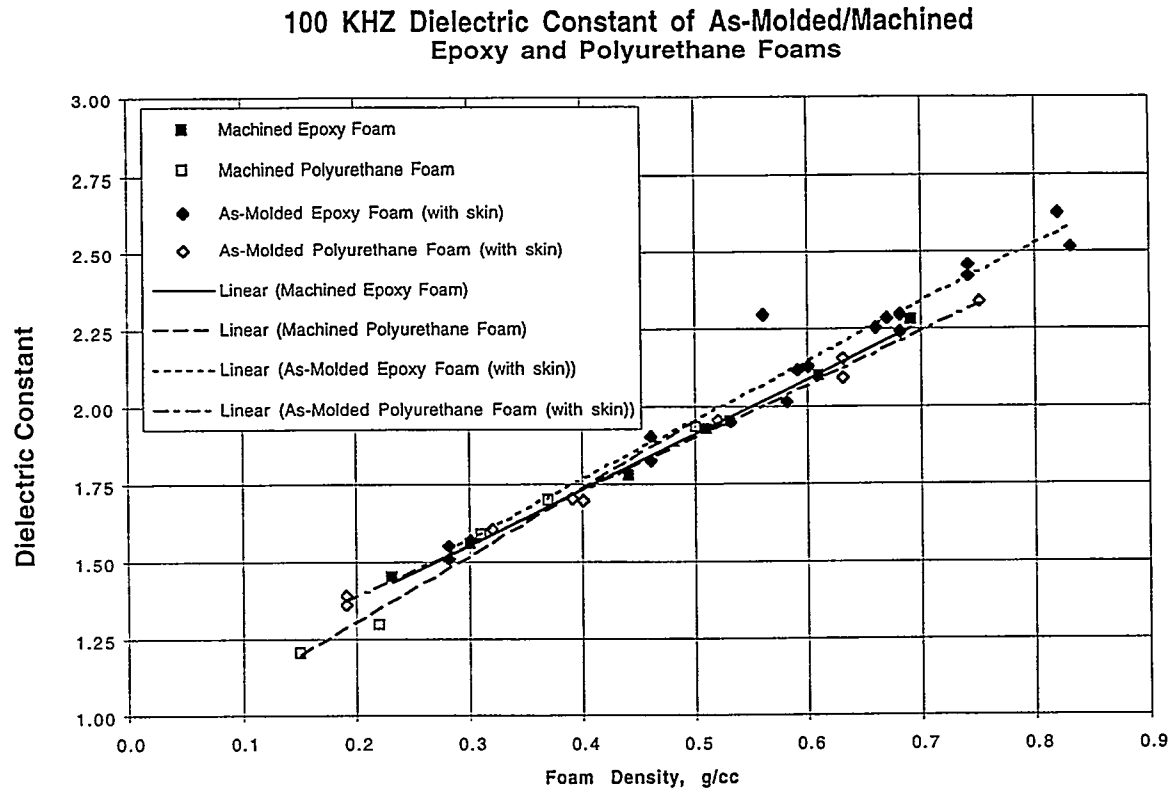
Figures 21 and 22. Dissipation Factors of As-Molded Epoxy and Urethane Foams (with skins)
(Sandia, CA Data)



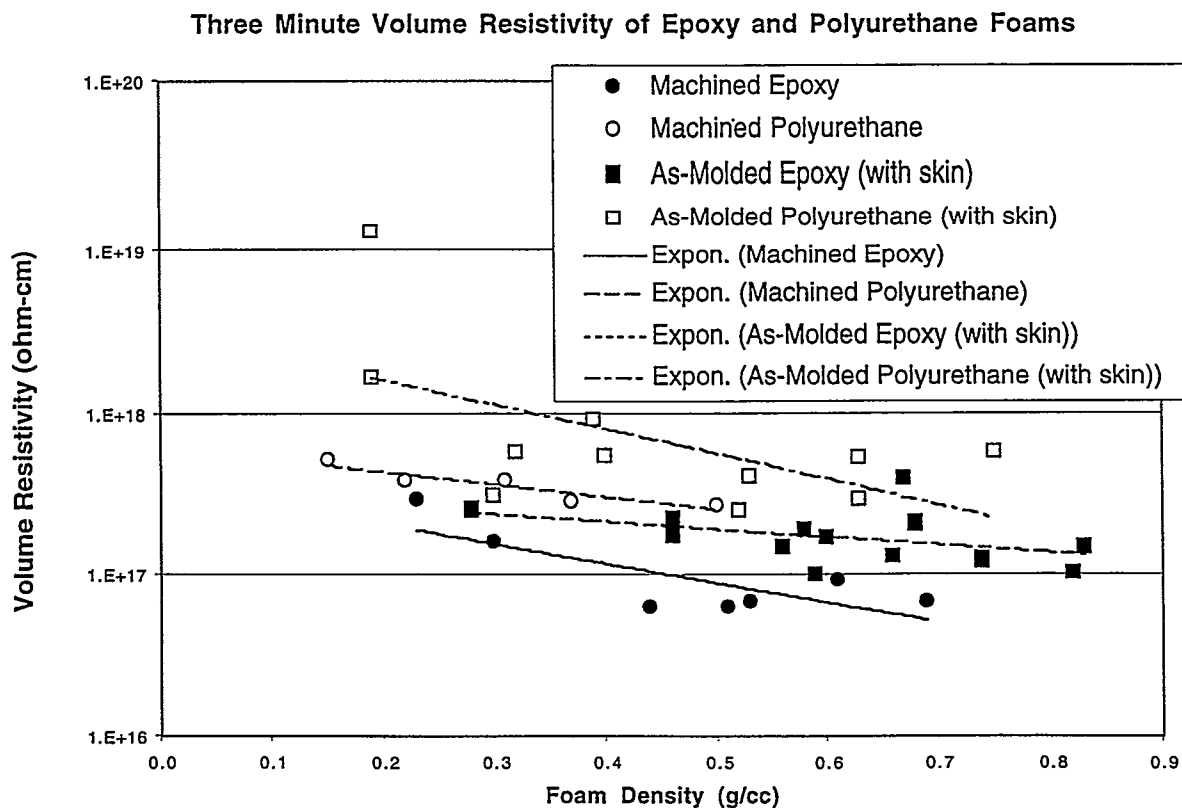
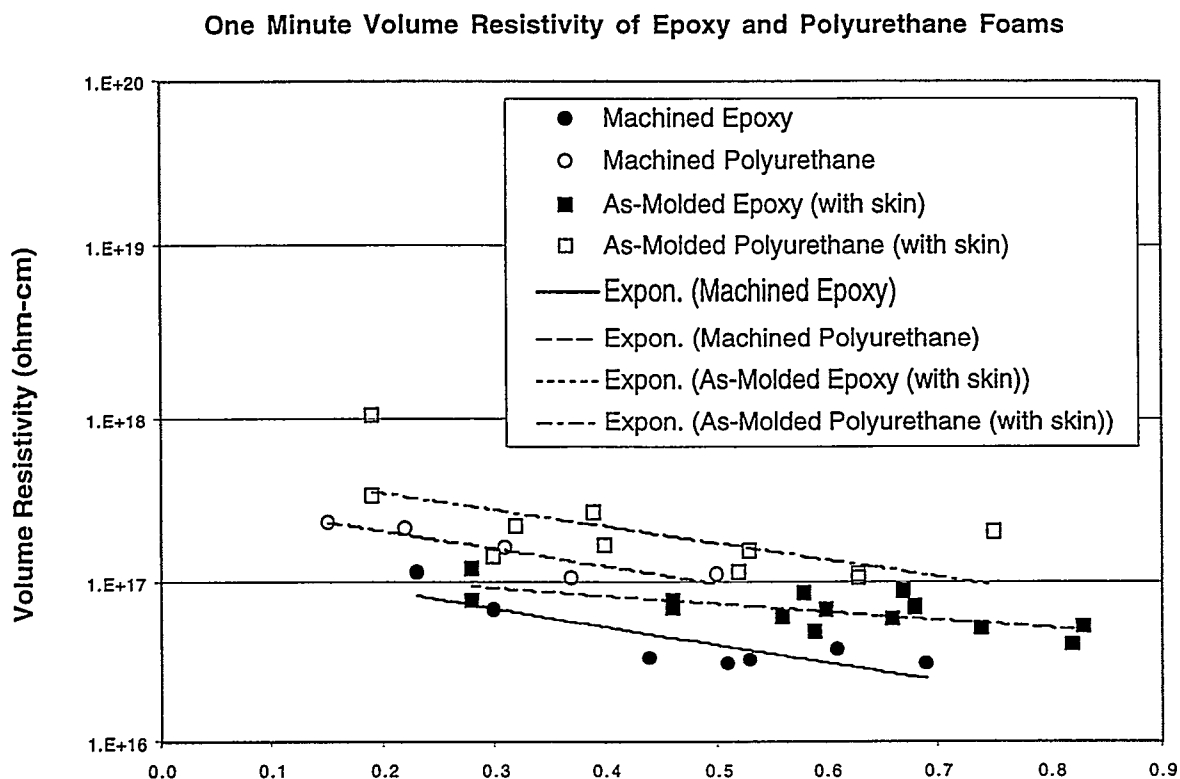
Figures 23 and 24. Volume Resistivity of As-Molded Epoxy and Urethane Foams (with skins)
(Sandia, CA Data)



Figures 25 and 26. 100 KHz Comparisons of Epoxy and Urethane Foams (Sandia,CA Data)



Figures 27 and 28. Volume Resistivity of Machined and As-Molded Epoxy and Urethane Foams (Sandia,CA Data)



were more erratic throughout these tests, but did indicate a slightly higher dissipation factor for the epoxy foams and also higher dissipation factors in the machined samples than in the as-molded samples. There was again a clear dependence on foam density.

Again pulling all the above results together, Figs. 27 and 28 show that both the one minute and three minute volume resistivities showed only a slight dependence on foam density and slightly higher values for the urethane foams. All the foams had very high volume resistivity values with the as-molded foams showing slightly higher resistance than the machined foams.

Conclusions

No dielectric advantages were found for the epoxy foams over polyurethane foams. These comparisons are evident in Figures 25-28.

Both as-molded foams with skinned surfaces and machined foams showed little dependence of volume resistivity on foam density except at very low densities. Urethane foams generally had slightly higher volume resistivities than epoxy foams although all the samples, with and without skins, had resistivities above 10^{16} ohm-cm (measured at one minute). Urethane foams were typically about an order of magnitude higher at 10^{17} ohm-cm.

Dielectric constants were clearly dependent on foam density in both the machined and as-molded samples with all samples falling along the same trendline. Neither the resin type or the presence or absence of the foam skin had additional significant effects on dielectric constant.

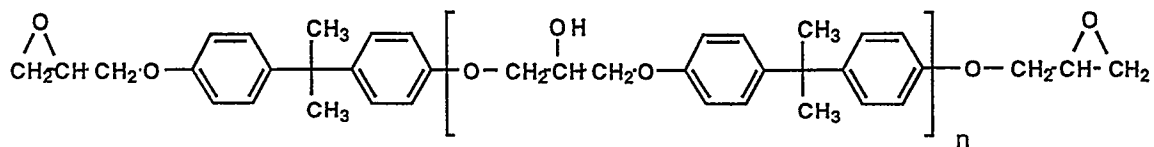
The cell sizes and foam morphologies were also examined and did not appear to have significant effects on the dielectric constant and volume resistivity.

Processing of the siloxane-blown epoxies was similar to water-blown urethanes although the cell size of the epoxies was generally larger and less uniform.

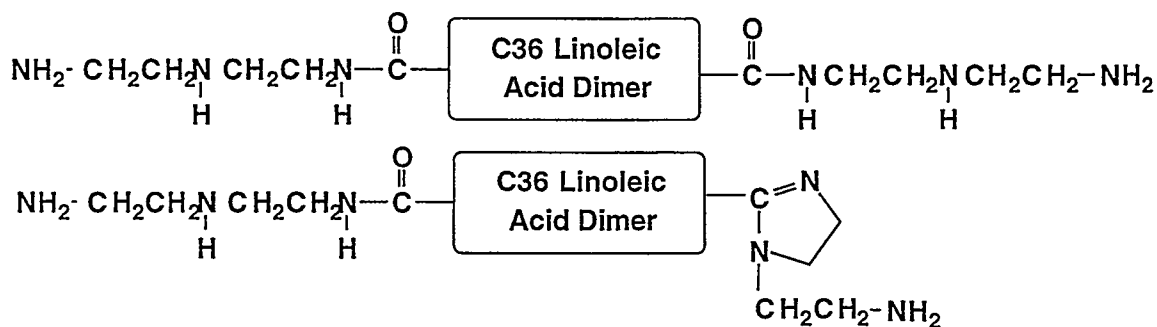
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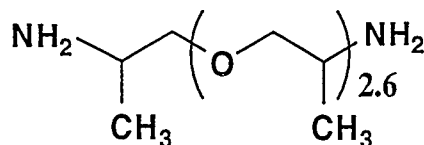
Appendix A. Structures of Epoxy Formulation Ingredients



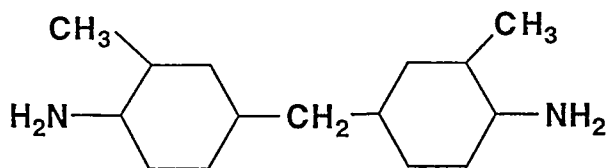
Epon 826/828/830 (increasing value of n and molecular weight in series)



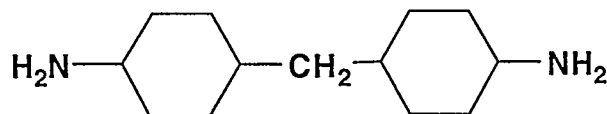
Versamid 125/140 (complex reaction product of acid dimer and diethylenetriamine)



Jeffamine D-230



Ancamine 2049



PACM

Appendix B. Surfactants Evaluated in Epoxy Foams

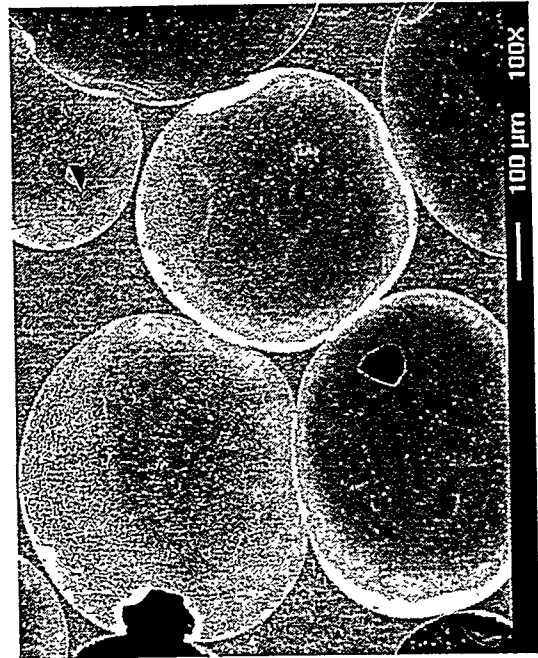
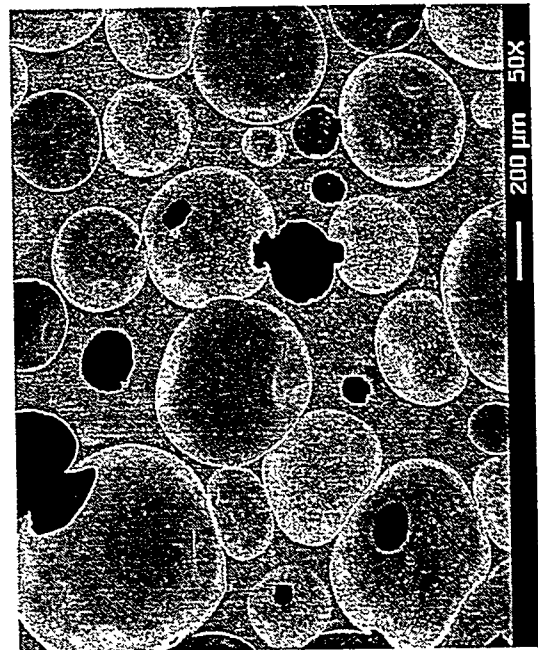
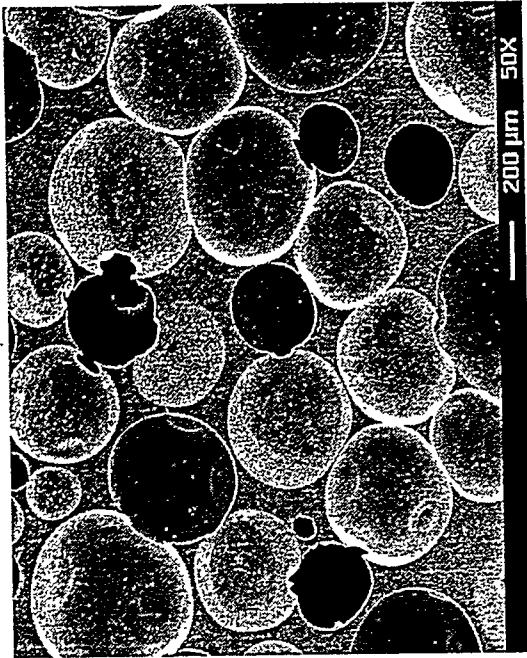
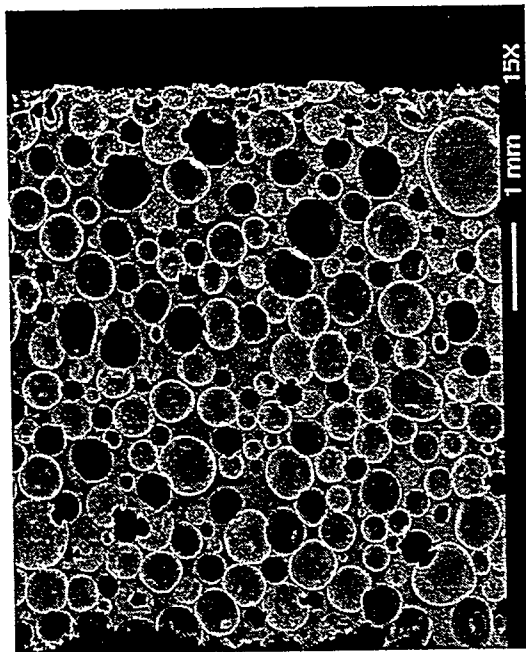
Manufacturer	Trade Name	Type	Identification
Air Products	Dabco DC-56	non-ionic	silicone polymer/copolymer?
	Dabco DC-57	non-ionic	silicone polymer/copolymer?
	Dabco DC-193	non-ionic	silicone glycol copolymer
	Dabco DC-197	non-ionic	silicone glycol copolymer
	Dabco DC-5098	non-ionic	silicone glycol copolymer
	Dabco DC-5103	non-ionic	silicone glycol copolymer
	Dabco DC-5164	non-ionic	silicone polymer
	Dabco DC-5320	non-ionic	silicone glycol copolymer?
	Dabco DC-5357	non-ionic	silicone glycol copolymer?
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	Dabco DC-5384	non-ionic	silicone glycol copolymer?
	Dabco DC-5385	non-ionic	silicone glycol copolymer?
	Dabco DC-5454	non-ionic	silicone glycol copolymer?
	LK-221	non-ionic	non-silicone polymer
	LK-443	non-ionic	non-silicone polymer
Dow-Corning	Q2-5200		silicone polymer
	Q2-8075		silicone polymer
Union Carbide	L-5420		
	Tergitol 7	anionic	0.26% monoheptadecylsulfate, sodium salt in water
	Tergitol NP-10	non-ionic	nonylphenol ethoxylate (10 EO)
	Tergitol 15-S-7	non-ionic	secondary alcohol ethoxylate (7 EO)
	Niax L-6906		polyalkylene oxide/dimethylsiloxane copolymer
ICI	Niax Y-10762		silicone
	Triton 100		
	Tween 20	non-ionic	polyoxyethylene (20) sorbitan monolaurate
	Tween 40	non-ionic	polyoxyethylene (20) sorbitan monopalmitate
	Tween 60	non-ionic	polyoxyethylene (20) sorbitan monostearate
Shin-Etsu	Tween 80	non-ionic	polyoxyethylene (20) monooleate
	KF-865	non-ionic	amine functionalized polysiloxane
Aldrich	Sorbital Monoloeate	non-ionic	sorbital monoloeate
	Glycerin	non-ionic	glycerol (1,2,3-propanetriol)
	HandiMan water		hand soap formulation

Appendix C: Foam Pictures

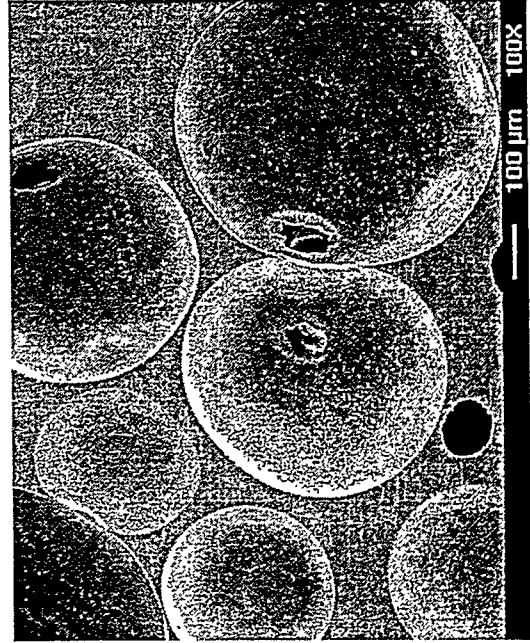
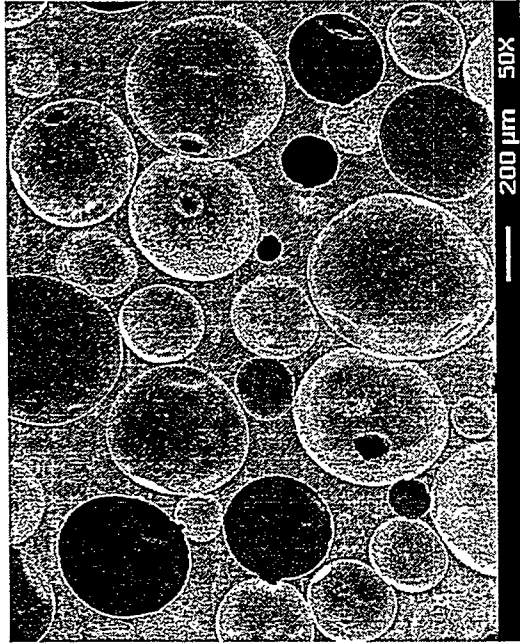
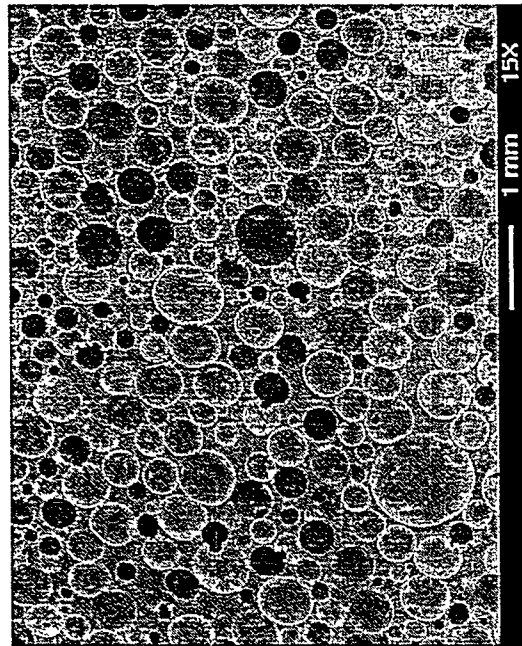
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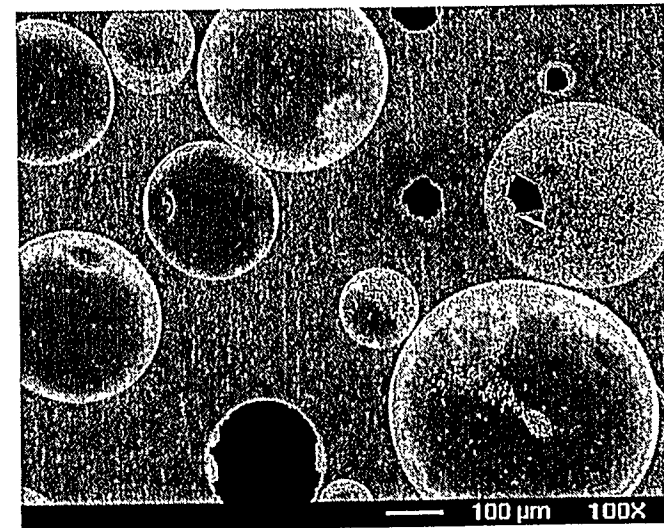
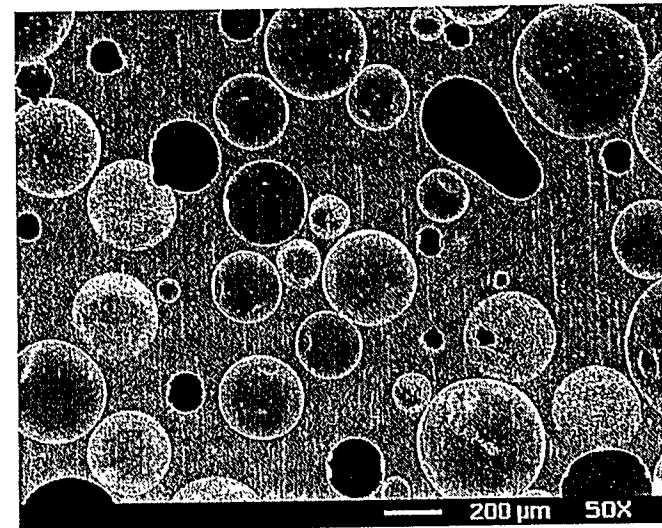
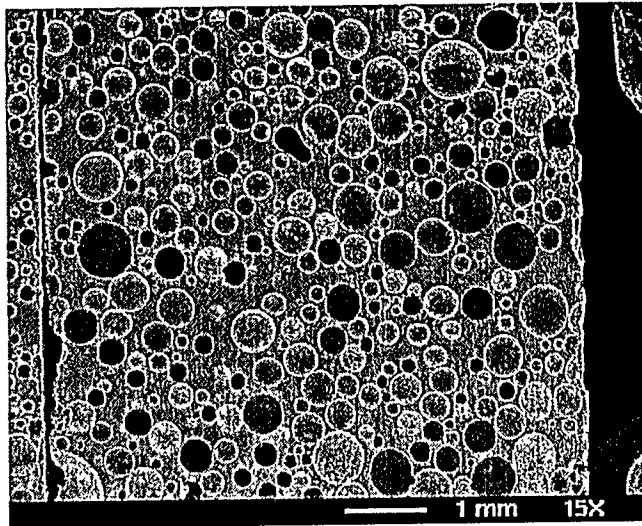
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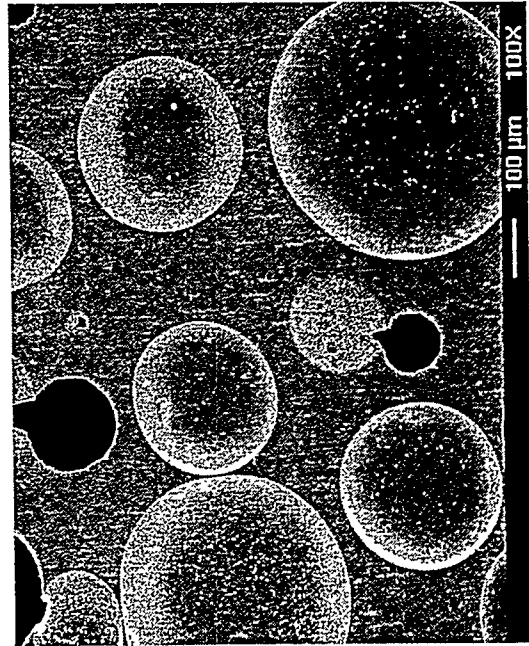
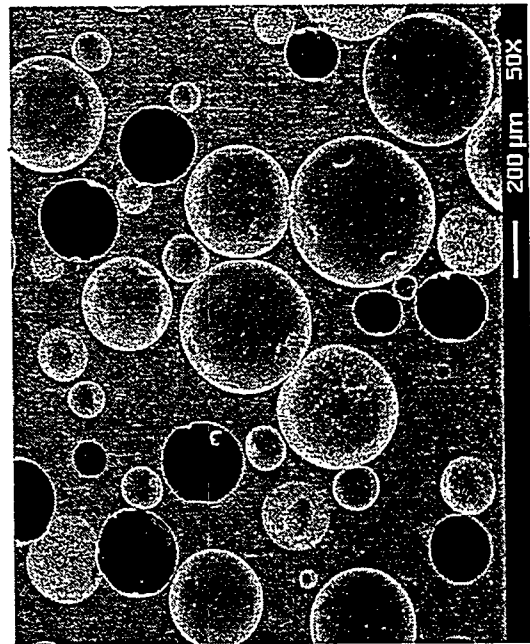
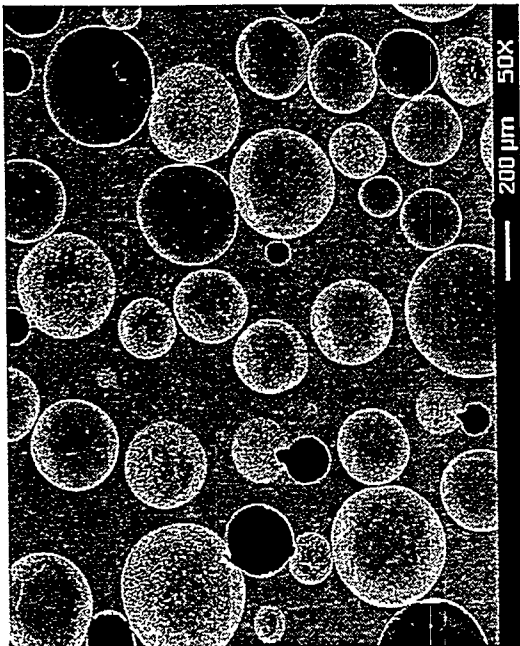
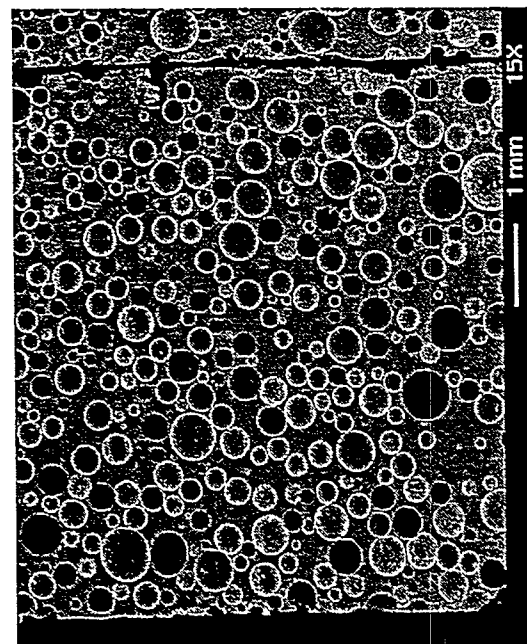
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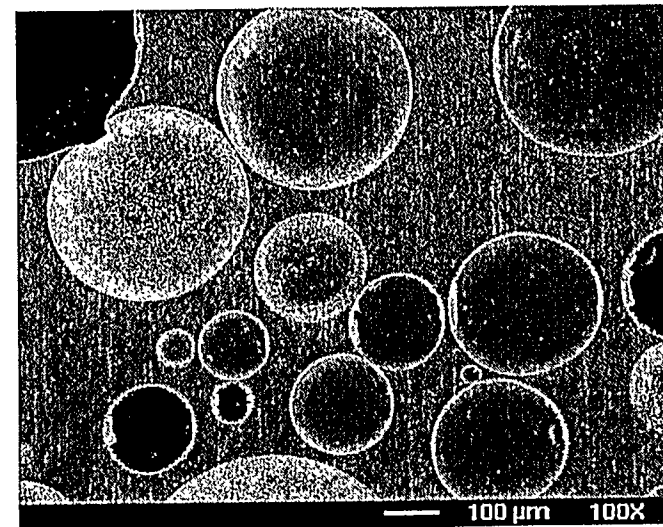
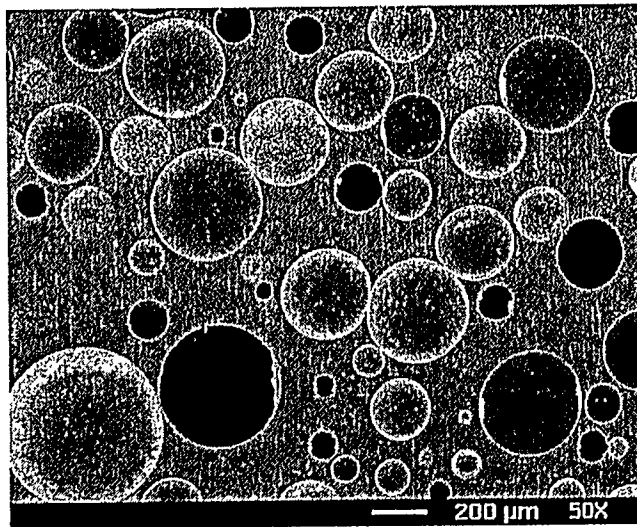
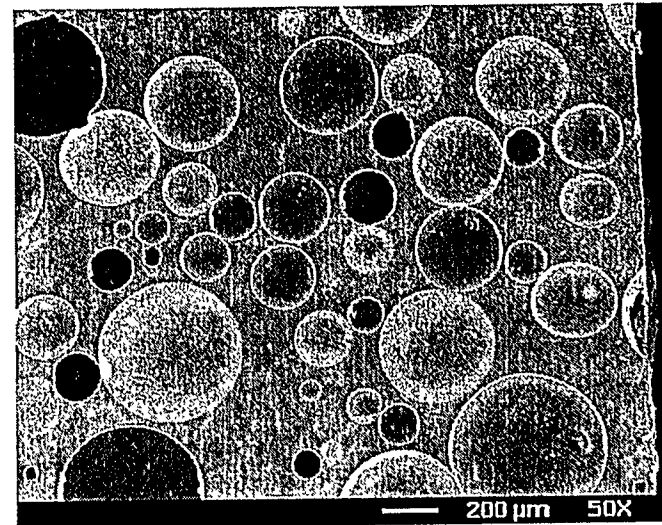
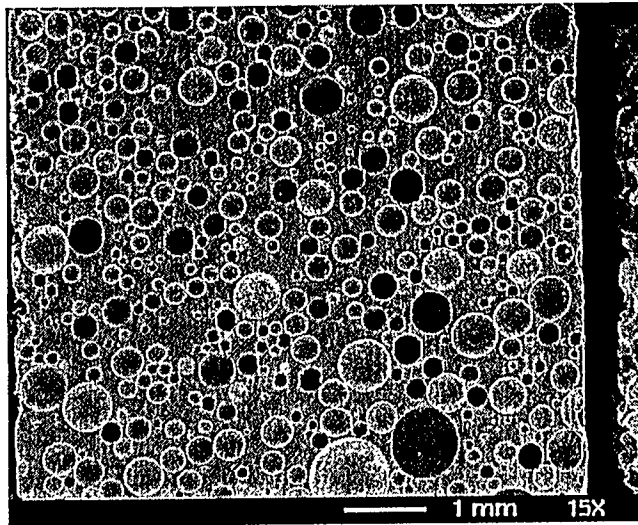
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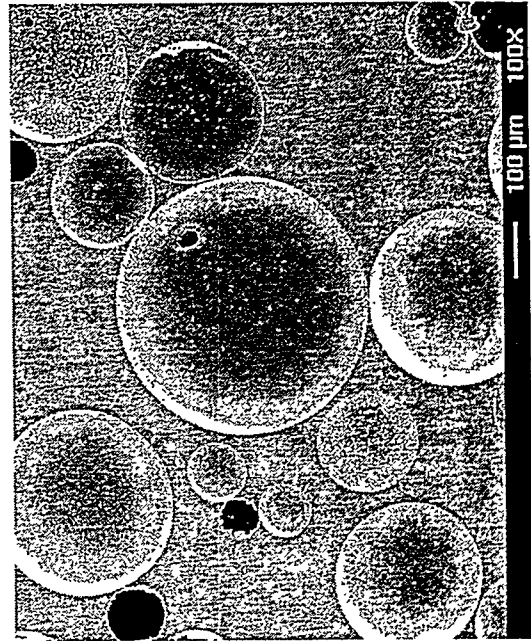
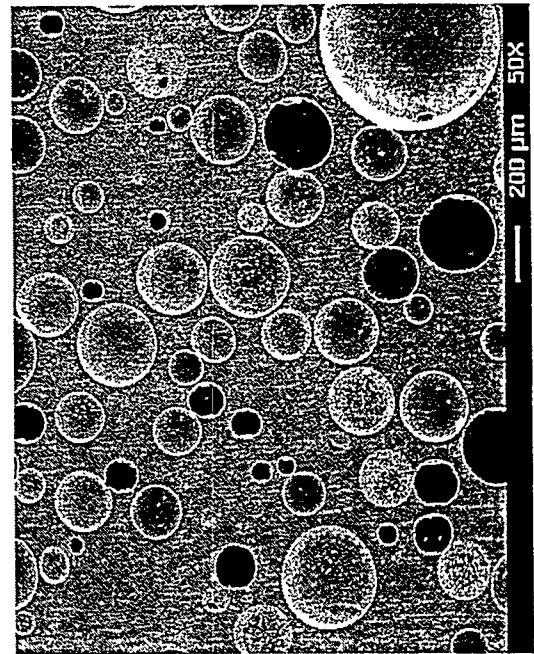
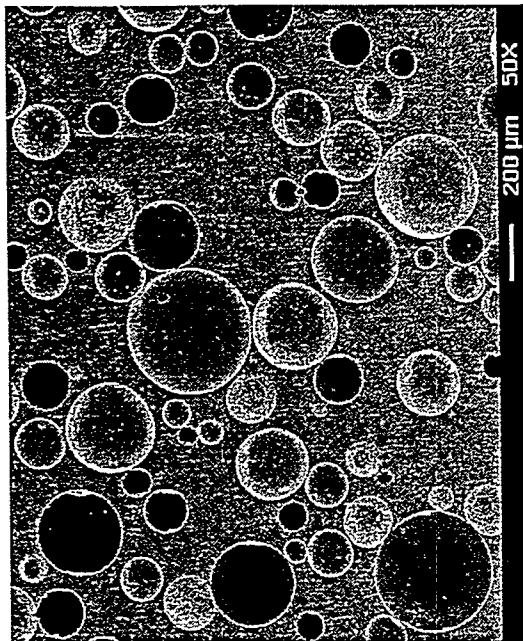
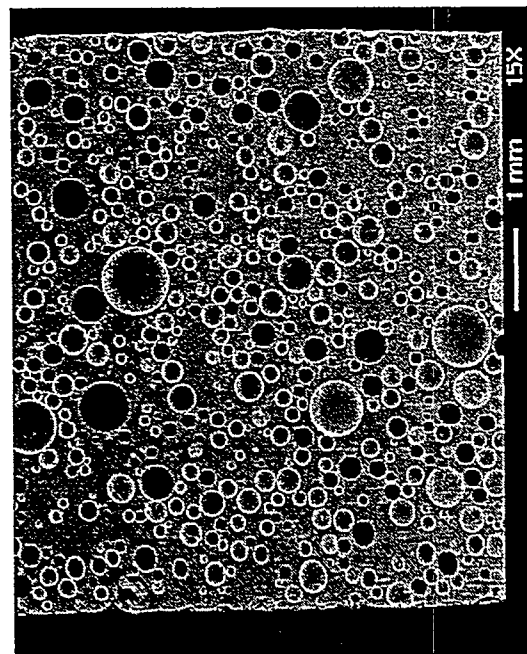
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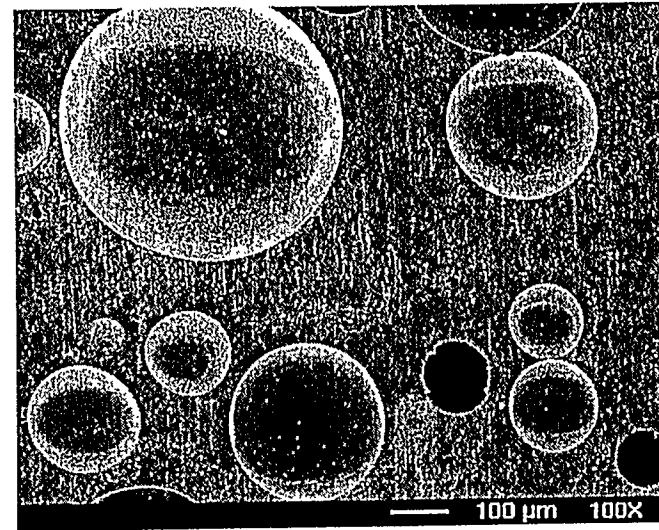
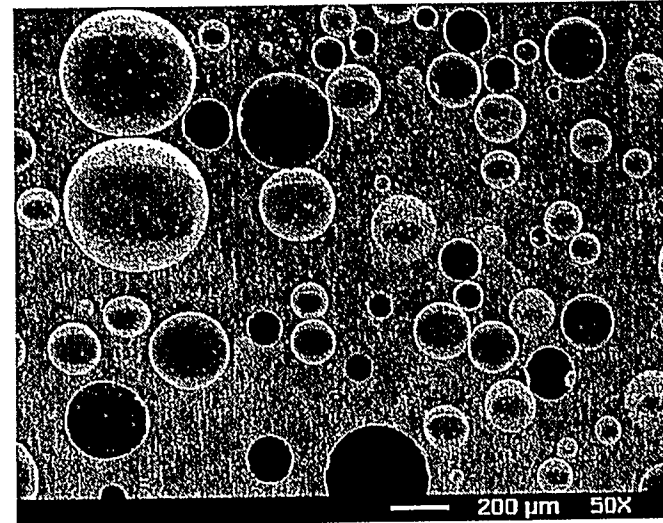
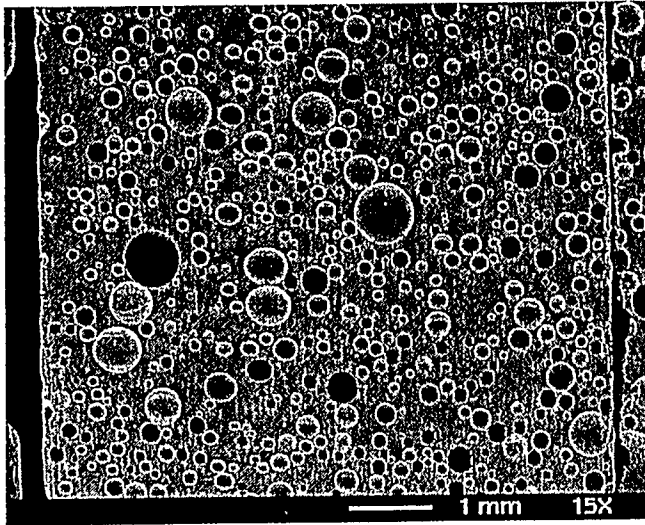
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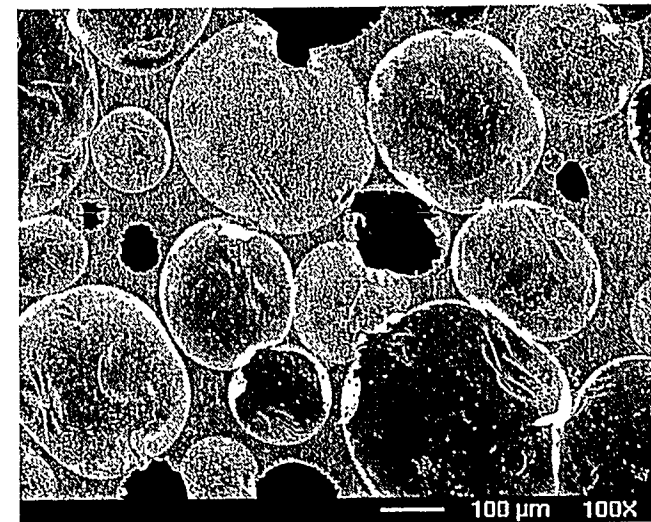
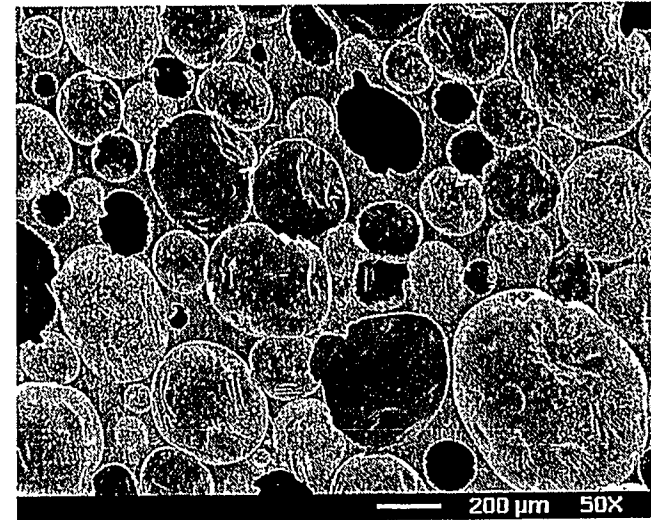
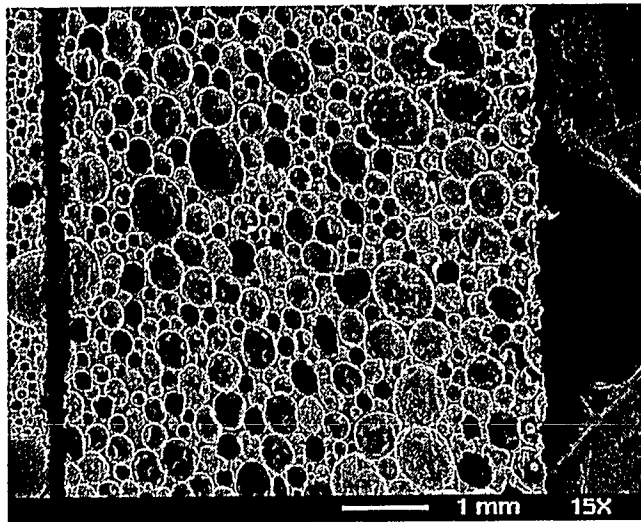
Epoxy Foam, 0.61 gr/cc (111197D2)



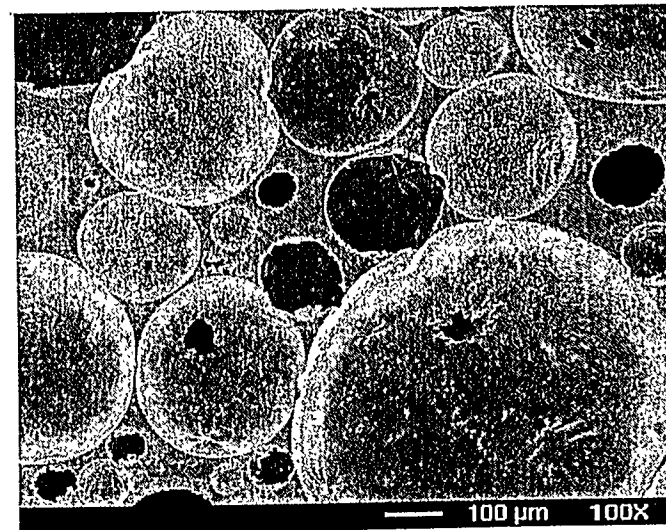
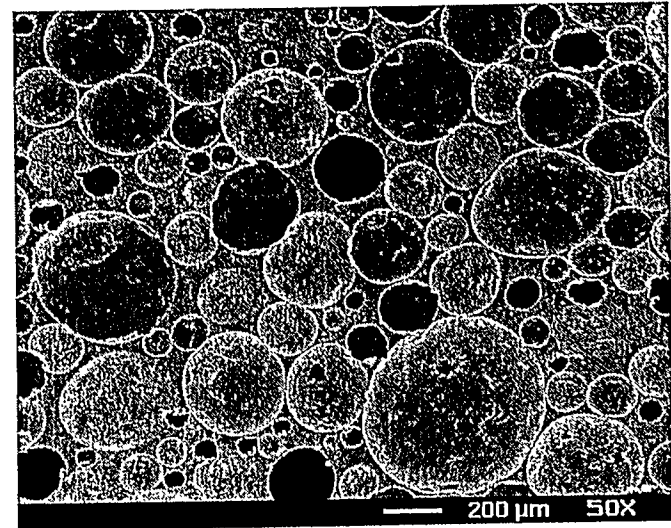
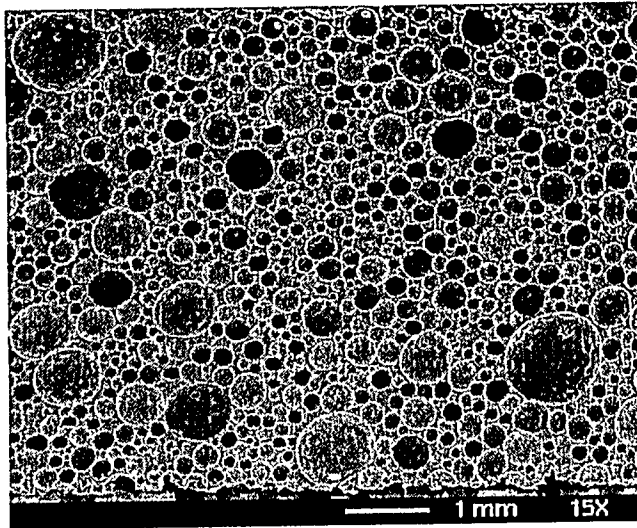
Epoxy Foam, 0.69 gr/cc (111397C3)



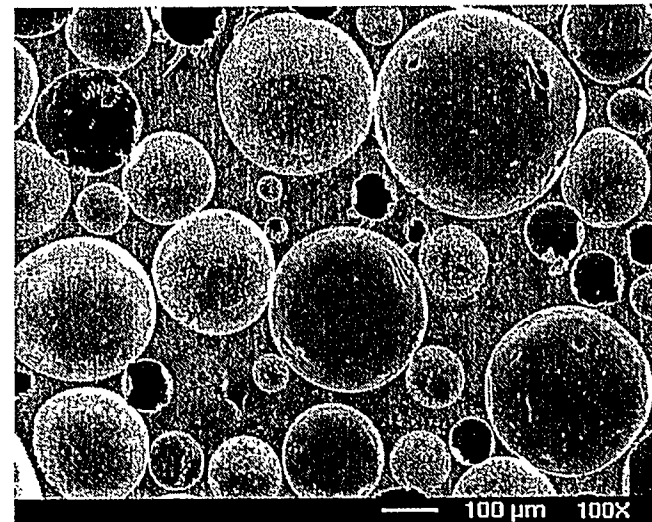
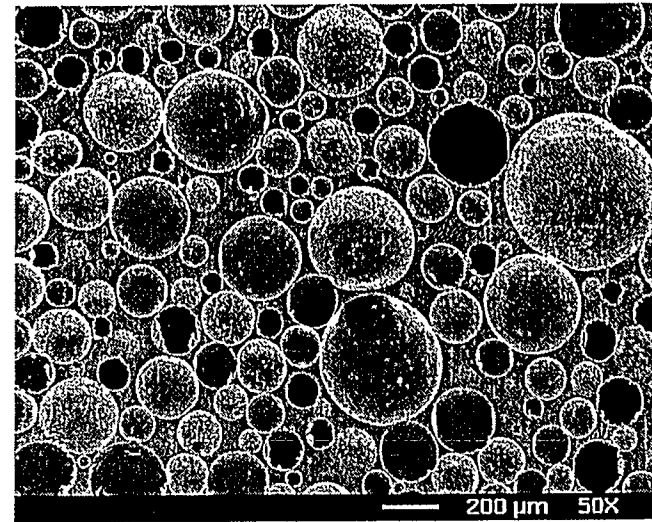
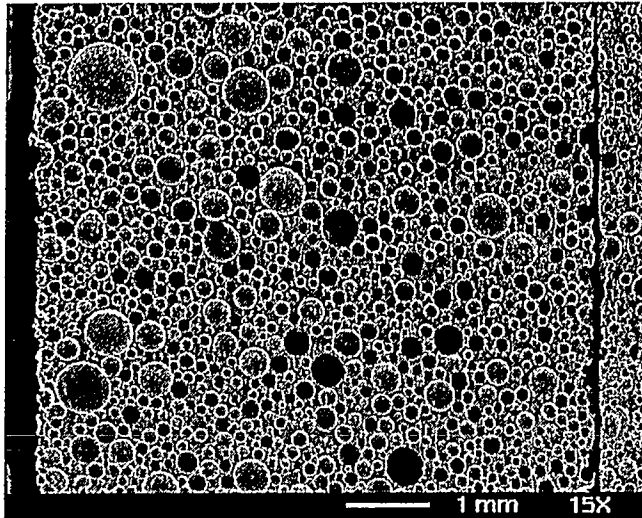
Polyurethane Foam, 0.15 gr/cc (120897A3)



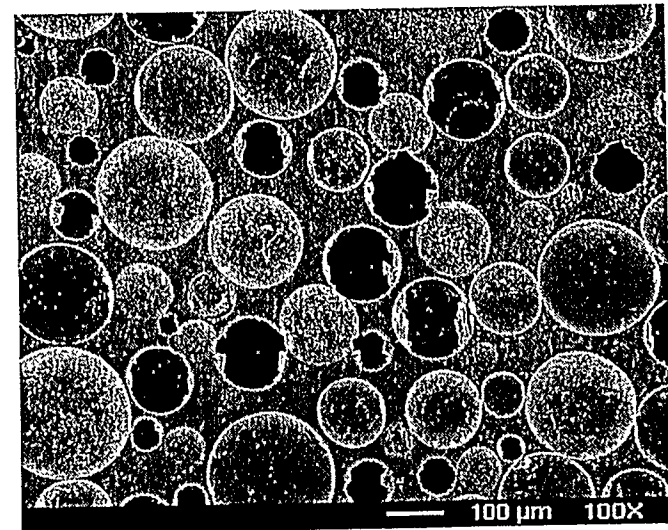
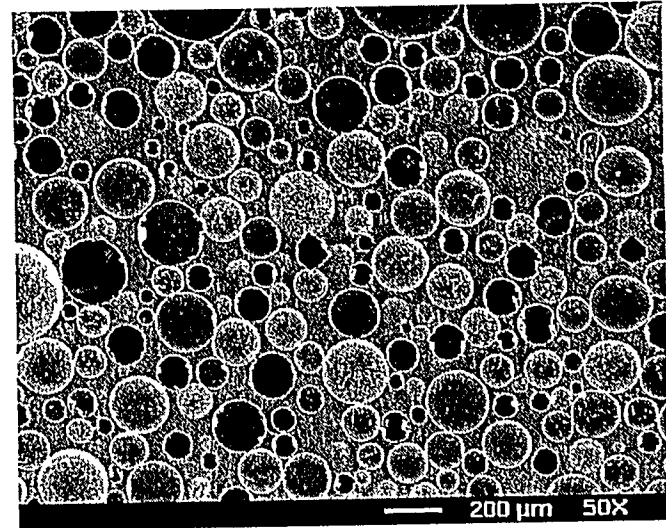
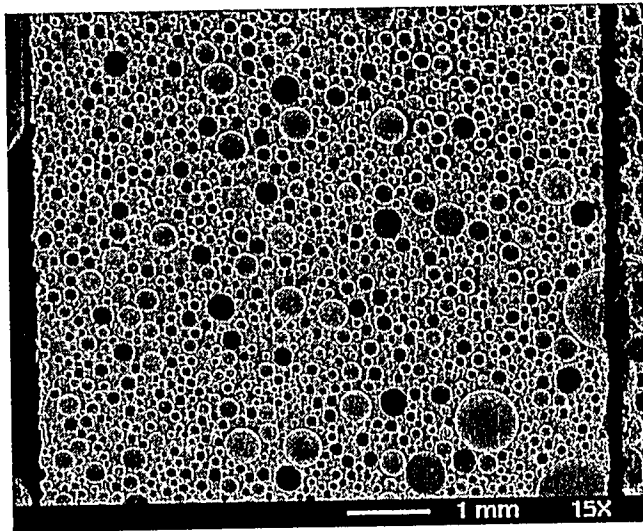
Polyurethane Foam, 0.22 gr/cc (120897B2)



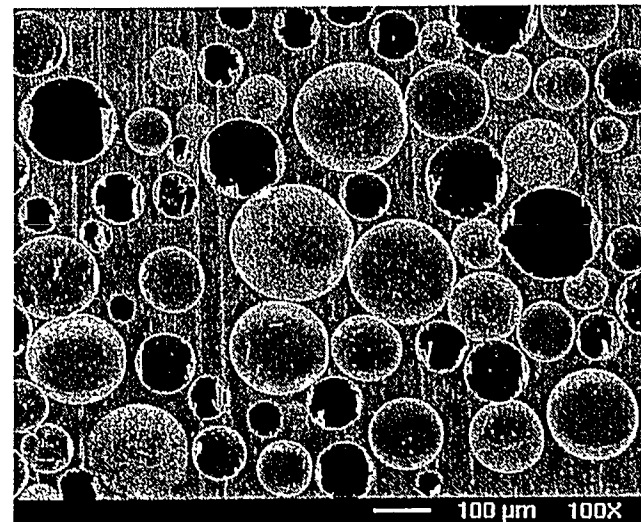
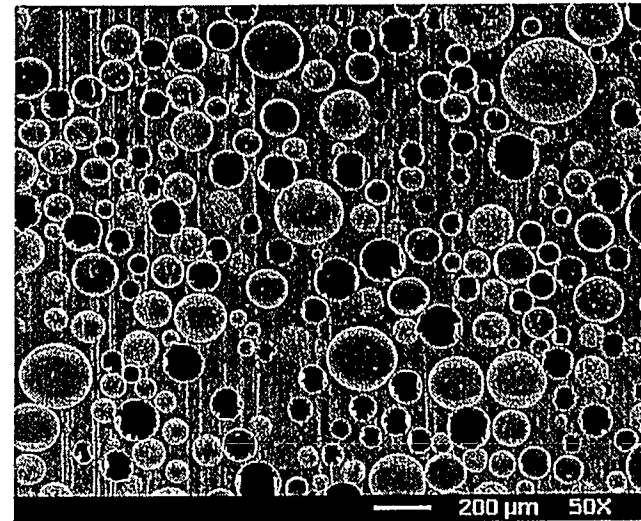
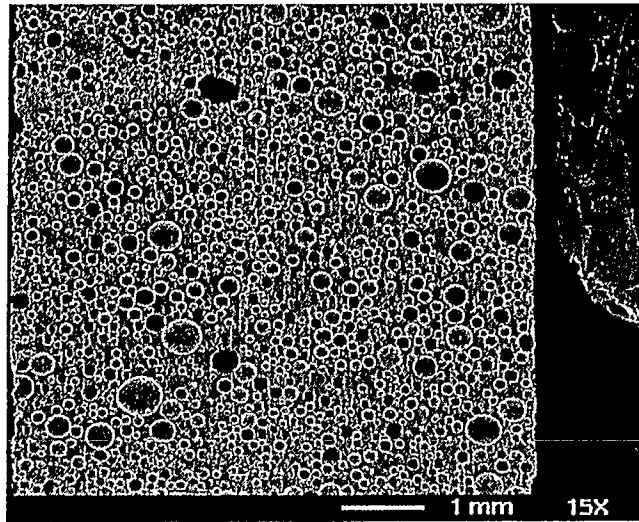
Polyurethane Foam, 0.31 gr/cc (120897C2)



Polyurethane Foam, 0.37 gr/cc (120397B2)



Polyurethane Foam, 0.50 gr/cc (120897D7)

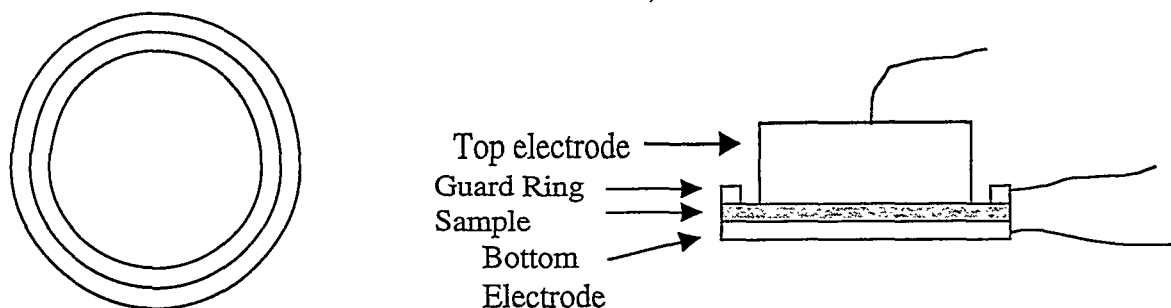


Appendix D: Dielectric Testing

Initial dielectric tests were carried out by Paul Beeson at Sandia, NM. This testing capability was later set up in Sandia, CA where most of the tests were run. Round-Robin comparisons between the two labs showed good agreement after some early trouble-shooting.

The early tests carried out by Paul Beeson in Sandia, NM used smaller brass electrodes with a 2 inch diameter top electrode, 4 inch bottom electrode and guard ring with an inner diameter of 2.3 inches. A larger set of electrodes with a 4.25 inch top electrode and 5 inch bottom electrode was designed by Paul Beeson and fabricated in the Sandia, NM machine shop for use in Sandia, CA. The guard ring had an inner diameter of 4.5 inches and the effective electrode area uses a diameter midway between the upper electrode and guard ring or 4.375 inches. The CA electrodes therefore had an effective surface area of 96.99 sq. cm. and the area of the NM electrodes was 23.55 sq. cm. The electrodes were enclosed in a oven for both faraday shielding and heating. Low noise test leads were used and all cable connections as well as the electrodes were carefully insulated.

The specific equipment used in California, all from Hewlett-Packard, consisted of a Model 4339B High Resistance Meter and a Model 4284A Precision LCR Meter along with low-noise leads (16117C).



Tests using carbon cloth inserts between the foam sample and electrodes to improve contact showed slightly higher conductivity. The differences were minor, however, and the reported tests were carried out without these inserts, unless otherwise noted, for simplicity.

All volume resistivity measurements except for some early tests and comparisons of resistivity vs. voltage in Sandia, NM were carried out at 1 kilovolt. Volume resistivities were measured both one minute, as called for in the ASTM procedures, and three minutes after the voltage was applied. The conductivity, as expected, dropped rapidly during the first minute and more slowly during the next two minutes.

Calculations:

Volume Resistivity = (Voltage x Electrode Area) / (Measured Current x Sample Thickness)

Dielectric Constant = (Measured Capacitance x Sample Thickness) / (Electrode Area x ϵ_0)

where ϵ_0 = electric field constant ($8.854 \times 10^{-12} \text{ Fm}^{-1}$)

Appendix E: Dielectric Measurements

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[illegible]

Volume Resistivity Measurements on Machined Epoxy Foam Samples (Sandia, CA and NM data with/without carbon cloth inserts)																
Sample No.	den. gr/cc	thick. cm	Volts	Electrode Area (sq. cm.)	Vol. Res. One min. (ohm-cm)	Vol. Res. Three min. (ohm-cm)	Vol. Res. One min. (ohm-cm)	Vol. Res. Three min. (ohm-cm)	Vol. Res. One min. (ohm-cm)	Vol. Res. Three min. (ohm-cm)	Vol. Res. One min. (ohm-cm)	Vol. Res. Three min. (ohm-cm)	Vol. Res. One min. (ohm-cm)	Vol. Res. Three min. (ohm-cm)	Vol. Res. One min. (ohm-cm)	Vol. Res. Three min. (ohm-cm)
Sandia, NM cal'd			NM results with no carbon cloth inserts								NM results with using carbon cloth inserts					
VR results only					1 KV	1 KV	2 KV	2 KV	3 KV	3 KV	1 KV	1 KV	2 KV	2 KV	3 KV	3 KV
111997-D2	0.23	0.700	1000	23.55	1.98E+17	6.73E+17	1.53E+17	3.96E+17	1.53E+17	4.04E+17	1.35E+17	4.81E+17	1.20E+17	2.93E+17	8.48E+17	8.34E+17
111697-D6	0.30	0.698	1000	23.55	7.67E+16	1.35E+17	9.92E+16	2.41E+17	7.61E+16	1.49E+17	8.09E+16	1.21E+17	7.58E+16	1.41E+17	5.22E+16	1.03E+17
112497-A1	0.44	0.704	1000	23.55	9.84E+16	2.57E+17	6.03E+16	1.49E+17	6.87E+16	1.65E+17	6.31E+16	1.52E+17	4.32E+16	1.08E+17	4.33E+16	9.94E+16
111697-A1	0.51	0.695	1000	23.55	6.05E+16	1.09E+17	6.33E+16	1.21E+17	5.56E+16	1.36E+17	5.47E+16	1.26E+17	4.64E+16	1.09E+17	3.77E+16	6.35E+16
110397-D1	0.53	0.702	1000	23.55	5.89E+16	1.53E+17	5.20E+16	1.43E+17	5.33E+16	1.26E+17	4.60E+16	1.20E+17	3.95E+16	9.32E+16	3.46E+16	8.32E+16
111197-D2	0.61	0.703	1000	23.55	5.74E+16	1.72E+17	5.15E+16	1.24E+17	5.52E+16	1.50E+17	5.49E+16	1.46E+17	4.89E+16	1.22E+17	3.05E+16	8.04E+16
111397-C3	0.69	0.708	1000	23.55	3.78E+16	1.23E+17	5.41E+16	1.30E+17	4.60E+16	1.28E+17	3.91E+16	1.04E+17	3.82E+16	1.01E+17	3.26E+16	8.05E+16
Sandia, CA Raw data			CA data with no carbon cloth inserts, all at 1 KV								CA data with no carbon cloth inserts, all at 1 KV					
and cal'd VR results			(4/24/98 measurements)								(4/28/98 measurements)					
111997-D2	0.23	0.700	1000	96.987	1.25E-12	4.90E-13	1.11E+17	2.83E+17	1.16E-12	4.70E-13	1.19E+17	2.95E+17				
111697-D6	0.30	0.698	1000	96.987	2.04E-12	9.30E-13	6.81E+16	1.49E+17	2.05E-12	8.10E-13	6.78E+16	1.72E+17				
112497-A1	0.44	0.704	1000	96.987	3.98E-12	2.20E-12	3.46E+16	6.26E+16	4.32E-12	2.27E-12	3.19E+16	6.07E+16				
111697-A1	0.51	0.695	1000	96.987	4.40E-12	2.23E-12	3.17E+16	6.26E+16	4.69E-12	2.24E-12	2.98E+16	6.23E+16				
110397-D1	0.53	0.702	1000	96.987	4.30E-12	2.02E-12	3.21E+16	6.84E+16	4.28E-12	2.05E-12	3.23E+16	6.74E+16				
111197-D2	0.61	0.703	1000	96.987	4.07E-12	1.86E-12	3.39E+16	7.42E+16	3.25E-12	1.23E-12	4.24E+16	1.12E+17				
111397-C3	0.69	0.708	1000	96.987	4.73E-12	2.10E-12	2.90E+16	6.52E+16	4.25E-12	1.94E-12	3.22E+16	7.06E+16				
Sandia, CA Raw data			Average of CA 4/24 and 4/28 measurements, all at 1 KV													
and cal'd VR results																
111997-D2	0.23	0.700	1000	96.987	1.21E-12	4.80E-13	1.15E+17	2.89E+17								
111697-D6	0.30	0.698	1000	96.987	2.05E-12	8.70E-13	6.79E+16	1.60E+17								
112497-A1	0.44	0.704	1000	96.987	4.15E-12	2.24E-12	3.32E+16	6.16E+16								
111697-A1	0.51	0.695	1000	96.987	4.55E-12	2.24E-12	3.07E+16	6.24E+16								
110397-D1	0.53	0.702	1000	96.987	4.29E-12	2.04E-12	3.22E+16	6.79E+16								
111197-D2	0.61	0.703	1000	96.987	3.66E-12	1.55E-12	3.77E+16	8.93E+16								
111397-C3	0.69	0.708	1000	96.987	4.49E-12	2.02E-12	3.05E+16	6.78E+16								

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