BDX-613-1496 (Rev.)
Distribution Category UC-38

**MATERIAL AND PROCESSING COMPARISONS
OF FOUR CONFORMAL COATINGS**

Published March 1977

Project Leader:
D. J. Caruthers
Department 814

Project Team:
F. E. Meisner

PDO 6984462
Final Report

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Prepared by D. J. Caruthers, D/814, under PDO 6984462

This evaluation compares the processing and properties of four materials in common use as conformal coatings. The processes were optimized to obtain the highest level of electrical properties and direct comparisons of physical properties at three temperatures are included. A design manual, BDX-613-1216, was developed as part of this effort.

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The Bendix Corporation
Kansas City Division
P.O. Box 1159
Kansas City, Missouri 64141

A prime contractor with the United States
Energy Research and Development
Administration under Contract
Number EY-76-C-04-0613 USERDA

CONTENTS

Section	Page
SUMMARY.	6
DISCUSSION	8
SCOPE AND PURPOSE.	8
ACTIVITY	8
<u>Materials</u>	8
<u>Processing of Materials</u>	10
<u>Aging Characteristics</u>	27
<u>Solvent Swell</u>	29
<u>TMA Analysis</u>	31
<u>Dynamic Shear Modulus</u>	31
<u>Adhesion</u>	34
<u>Electrical Properties</u>	45
<u>Physical Properties</u>	48
<u>Design Manual</u>	54
ACCOMPLISHMENTS.	54
FUTURE WORK.	55
REFERENCES	57
DISTRIBUTION	58

ILLUSTRATIONS

Figure		Page
1	Electrical Breakdown Versus Surface Preparation of Copper.	15
2	Breakdown Voltage Versus Thickness of Five Coating Materials.	16
3	Stainless Steel and Gold Plated Radii Test Specimens (P-91020)	20
4	Thickness of Conathane EN-4 Coating Compared to Diameter of Sphere.	21
5	Breakdown Voltage of Conathane EN-4 Showing Effect of Conductor Material	22
6	Breakdown Voltage Versus Thickness of Conathane EN-4 on Copper Showing Effects of Removing Solid Particles.	25
7	Appearance of Conathane EN-4 After Solvent Soak in the Indicated Solvents and Air Drying (P-82069)	32
8	Thermomechanical Analysis of Conathane EN-4, Change in Length Versus Cure Temperature . .	33

TABLES

Number		Page
1	Conformal Coating Materials.	9
2	Breakdown Voltage Range of Laminar X500 at Test Point A on Different Metals	18
3	Breakdown Voltage Range of Laminar X500 at Test Point B on Different Metals	19
4	Breakdown Voltage Range of EN-4 Coating Thickness Versus Dipping Position.	24
5	Shrinkage of Urethane Elastomers at Room Temperature	28
6	Thermal Coefficient of Expansion	29
7	Coefficient of Expansion From Humidity Changes.	30
8	Dynamic Physical Properties Chart.	35
9	Tensile Shear Values for EN-4 as an Adhesive	38
10	Peel Values of EN-7 Against Kapton at 68°F (20°C).	39
11	Peel Values of EN-7 and Adiprene L-100/MOCA to Insulation Material	40
12	Peel Values of EN-7 to Rigid Plastics and Metals	42
13	Adhesion of EN-7 to EN-7	43
14	Electrical Properties Data for Three Materials Tested at 77°F (25°C) Obtained From 0.08 Inch (2.03 mm) Thick Slabs.	46
15	Electrical Properties Data for Five Materials Tested at 77°F (25°C) Obtained From 0.08 Inch (2.03 mm) Thick Slabs.	47
16	Physical Properties Data for Three Materials.	49
17	Physical Properties Data for Five Materials.	52

SUMMARY

This project was initiated to aid in the selection of conformal coating materials for high voltage circuits based on electrical, mechanical, and processability characteristics. The candidates were the most likely commercial representatives of epoxy, polysulfide, silicone, and urethane materials.

The highest resistance to electrical breakdown was obtained with the epoxy coating tested at 11,000 to 15,000 volts per mil (0.025 mm) compared with the urethane coatings which ranged from 7000 to 10,000 volts per mil. The electrical breakdown of the polysulfide and silicone materials occurred in the 2000 to 3000 volts per mil range. The breakdown voltage was directly affected by the surface finish of the material to be coated. Copper surfaces prepared with either 600 grit paper or by being polished with rubbing compound provided breakdown voltages of 11,000 to 15,000 volts per mil when an epoxy coating material was used. Copper surfaces prepared with 240 grit abrasive to illustrate the effect of small scratches drastically lowered the breakdown voltages, with the same coating, to the 2000 to 3000 volts per mil range.

Four metallic surfaces (tin-lead, gold, copper, and nickel) had little effect on the electrical breakdown voltage when the surfaces were coated with the epoxy coating. As the conformal coating thickness approached 0.5 mil (12.5 μm) no effect resulting from the use of different metals could be discerned.

A determination was made of how well conformal coatings will cover small radii by measuring the coating thickness and resultant electrical breakdown on five spherical surfaces ranging from 0.015 through 0.063 inches (0.381 through 1.600 mm) in diameter.

The urethane coating retained on the 0.015 inch (0.381 mm) sphere measured from less than 0.0001 to 0.0002 inch (2.5 to 5.0 μm) while a thickness of 0.002 to 0.004 inch (50 to 100 μm) was found on an adjacent flat surface. The coating thickness on the larger spheres (0.063 inch or 1.600 mm) measured from 0.0004 to 0.001 inch thick (10 to 25 μm) while the adjacent flat remained at 0.002 to 0.004 inch (50 to 100 μm) thick. Insulation coating thickness could not be measured on nickel, copper, and stainless steel 0.015 inch (0.381 mm) diameter spheres. The gold plated 0.015 inch (0.381 mm) sphere insulation ranged from 1000 to 4000 volts per mil (0.025 mm). Breakdown voltage ranged from 1000 to 4000 volts per mil on the 0.063 inch (1.600 mm) diameter spheres for all four metal surfaces tested.

The Conathane EN-4 used contained desiccant and gel particles. Filtering this conformal coating to remove these particles increased the breakdown voltage protection from approximately 3000 volts per mil (0.025 mm) to 6000 to 8000 volts per mil.

The adhesion of the epoxy, polysulfide, and silicone coatings could not be tested because the adhesion always exceeded the tensile strength of the materials. The adhesion of urethane materials is affected by the substrate material chosen and the surface conditions. Sandblasting surfaces improved adhesion over that of unblasted substrates. Conathane EN-7 adhesion to nylon improved from 19.3 to 54 pounds per linear inch width (p/li) (3380 to 9457 N/m) where adhesion for aluminum improved from 19.6 to 35.4 p/li (3433 to 6200 N/m) after the substrate surface was sandblasted. The surface finish required for maximum adhesion is in opposition to the surface finish required for optimum breakdown voltage.

The polyether based urethane Adiprene L-100 (du Pont) required a primer for effective adhesion to metals while the polybutadiene based urethane required none. Aging, natural or accelerated, improves the adhesion of the polybutadiene based urethane Conathane EN-7 for substrates of Kapton (du Pont) and Neoprene rubber used as cable jackets.

A major portion of effort was applied to the analysis of a conformal coating used as a stress relief coating to mechanically protect electrical components from the stresses imposed by potting compounds during temperature cycling. Mathematical models have been developed which allow for calculations of stress that would result from various shapes and combinations of electrical components, potting compounds, circuit boards, and conformal coatings. The work is reported in BDX-613-1216,¹ a separate report, and is intended for use as a design manual.

This endeavor did not show a clear picture of a superior class of material. No method of application or material studied produced a conformal coating with significant thickness on component edges or small diameter test items. Where high voltage levels are critical or components must function in a vacuum, isolation by encapsulation is recommended.

Compressive bulk modulus data of the materials tested ranged from 190,000 psi (1.31 GPa) for the silicone to 900,000 psi (6.2 GPa) for the polysulfide; therefore, these conformal coatings cannot be counted on to give significant mechanical stress relief in compression as encountered in encapsulated assemblies.

DISCUSSION

SCOPE AND PURPOSE

A high voltage circuit required a conformal coating capable of withstanding 6000 volts in a vacuum. Repeated attempts to upgrade both the materials used and the techniques for application failed to obtain the required results. The purpose of this endeavor was to correct this deficiency by providing materials and processes to meet the design objective.

During the development of a family of high voltage circuits, cracks formed in the epoxy-glass microsphere potting matrix. One method for correcting these defects was by using a conformal coating over the affected component to absorb the differentials of thermal expansion between the component and the epoxy-glass matrix. This project includes investigation of materials used in conformal coatings as mechanical stress absorbers to relieve mechanical stress generated by thermal processes and subsequent product testing.

The investigation determined the physical properties of each material and related effects caused by processing. The primary methods of evaluating these effects were by electrical breakdown tests and thickness measurements. This investigation also included variables such as solvent compatibility, the effects of filtering the resin, effects of surface finish of metals, ability to cover small radii, adhesion to materials found in electronic assemblies, techniques for cleaning surfaces, and effects of production sequences.

ACTIVITY

Materials

There are many conformal coating materials on the commercial market. This investigation was limited to the materials listed in Table 1.

A urethane resin, Adiprene L-100 cured with MOCA, has been a standard for a number of years. It is made from a 2,4-toluene diisocyanate end-capped polyether (polyoxy-1,4-butylene) and an aromatic diamine curing agent (methylene bis [0-chloroaniline] or MOCA). It is classed as a polyether-TDI-diamine type. It can be used in thick or thin sections as a 100 percent solid or diluted with selective solvents to be used as a thin coating. Adiprene

Table 1. Conformal Coating Materials

Class	Type	Manufacturer
Urethane	Adiprene L-100/MOCA	E. I. du Pont de Nemours and Company
Urethane	Conathane EN-7 and EN-4	Conap Incorporated
Silicone	Dow Corning 3140	Dow Corning Corporation
Polysulfide	Chem Seal 3100	Chem Seal Corporation of America
Polysulfide	P.R.C. 1201Q	Product Research and Chemical Corporation
Polysulfide	Pro Seal 727	Coast Pro Seal Manufacturing Company
Epoxy	Laminar X500 (7-C-23)	Midland Division Dexter Corporation

L-100/MOCA is considered to be a high modulus elastomer and can be processed as a medium to low viscosity liquid of short pot life curing at temperatures from ambient through 200°F (93°C). All cures develop similar properties using normal test procedures.

Another urethane investigated was the polybutadiene-based urethane, Conathane EN series by Conap Incorporated. This material is a hydroxylated polybutadiene toluene diisocyanate prepolymer cured with a mixture of polyols containing a catalyst of phenyl mercuric oleate (EN-4) or ferric acetylacetonate (EN-7). This series of urethanes is similar in ambient temperature properties to the Adiprene L-100/MOCA but exhibits improved electrical properties over the du Pont polyether. The EN series must be processed near room temperature because high exotherm accompanied with short pot life will occur if either the resin or hardener is much above room temperature when combined. Both EN-4 and EN-7 require elevated temperatures for curing in reasonable times. This series has been reported hydrolytically stable; however, no data in this report describes this physical parameter. Processors will find that the EN series resins exhibit a room temperature viscosity one order of magnitude higher than the polyether based Adiprene L-100 resin at its processing temperature of 160°F.²

Three softer materials investigated include the polysulfides represented by Coast Pro Seal's 727, Chem Seal's C3100, and Product Research Corporation's 1201Q. These materials are based on the moderate viscosity polysulfide LP-2 using metallic oxides,

in combination with an alkaline environment as curing agents. The polysulfides have been a production item for a number of years and one of their major uses is as a sealer. These materials have a lower tensile modulus than the epoxy or urethane coatings and may be cured either at ambient or elevated temperature.

The silicone material, Dow Corning DC3140, is a room temperature vulcanizing, low durometer coating material having the capability of remaining rubbery over the intended product temperature range. Since no acetic acid is present and no corrosive by-products are generated during cure, DC3140 is suited for use as a conformal coating. Moisture is required for cure and this limits the thickness of the coating because the cured material is an excellent moisture barrier. The reported low modulus at sub-ambient temperature makes it attractive for use as a mechanical stress relief material.

The epoxy coating, Laminar X500 (7-C-23) resin, is a two-component system. The resin portion is a polyester epoxy resin dissolved in a mixture of methyl ethyl ketone and ethylene glycol/monoethyl ether acetate (MEK in Cellosolve acetate). It has a solid content of 42.6 percent. The hardener portion is a toluene diisocyanate prepolymer dissolved in a mixture of xylene and ethylene glycol/monoethyl ether acetate (Cellosolve acetate). The solid content of the hardener solution is 63.2 percent.

Because of the polyether, polyurethane, and polyester linkages the polymer chain is very linear and the molecular weight is high with a fairly narrow molecular weight distribution. The coating is obtained by the stoichiometric addition of the isocyanate prepolymer to the resin. It also is designed for use as a thin coating because of the high solvent content.

Processing of Materials

Many factors determine the protection resinous coatings give to electrical components. One of the most universal questions asked concerns the electrical breakdown voltage of the cured polymer. This parameter is affected by the resin used, the coating thickness and its cure, and filler materials either put in to enhance a specific property or as a foreign material like dirt or gel particles. The surface of the component to be coated also affects the breakdown voltage. Other factors including surface roughness, contaminants such as oil, grease, fingerprints, and solder flux must be considered along with basic compatibilities of the surface to be coated and the resin.

The parameters specifically addressed in this section are grouped into categories such as the cleaning of assembly components and solvent compatibilities to the coating materials used. The

effects of surface roughness along with the effects of dielectric strength of five thin coating materials are studied. Since breakdowns were reported occurring along edges of metallic circuits, the effects of four metallic surfaces were studied. The relationship of radii magnitude and its limitations to organic coating thickness was investigated by breakdown voltage studies. Processing parameters related to the thickness of the coating obtained by variations in a dipping process were examined along with the aftereffects of removing the foreign material found in one resin system.

Each material is handled using procedures compatible to the function of the product involved. These procedures usually optimize on cost and properties. Within limits, specific properties may be tailored to meet unique conditions depending upon the equipment used, quantity of product to be handled, skill of process designers, and economics available to integrate all of these into a production situation. Several types of equipment were available and, where possible, processing equipment identical to that available in the production area was used.

Where possible, power driven equipment was employed for repeatability of the involved process. Observations of production techniques and the resultant coating indicated that some materials were not being used under optimum conditions. These materials tended to be too viscous to flow, caused bridging between component parts, trapped bubbles in the coating, and would not wet some components resulting in poor adhesion. Polysulfide and silicone presented problems in that they required hand mixing procedures because of their high viscosity which was beyond the available equipment capabilities. Further problems were found when specimens were built to test the resins electrically; these were, uniformity of coating thickness, and the cleanliness of specimens to be coated.^{3,4}

Cleaning Components

Clean surfaces are paramount for achieving repeatable optimum results from thin coating material. Cleaning techniques, along with the solvents or compounds used, determine the effectiveness of the surface presented for coating. The most common contaminants are greases, oils, dirt, oxides, and similar materials accumulated due to prior processing, aging, and storage. Each contaminant must be cleaned using materials and processes which are effective for removing those contaminants present yet will not degrade or destroy the coating material surface. At times this may involve the use of an abrasive compound to remove the outer layer of the component. For example, vapor blasting molded thermoset plastics was used to remove mold releases concentrated or entrapped in the as-molded surface.

Solvents are used to loosen and flush away gross foreign material. Trichloroethylene (TCE), alcohol, Freon TF, methyl ethyl ketone, and acetone are all effective on selected contaminants. Vapor degreasing was effective because of the increased solvent activity due to the higher temperatures. This has an added benefit of using the nonflammable solvents TCE or Freon; however, many electronic component packages are degraded by this heating process. The effectiveness of a series cleaning consisting of degreasing in TCE followed by a dip in alcohol followed by an ultrasonic dip in Freon TF was found to be the most effective. Isopropyl alcohol was used to remove fingerprint salts not removed by the TCE. The ultrasonic Freon TF removed some metallic particles from polished copper test panels resulting in higher voltage breakdown levels than were obtained with Conathane EN-4 coatings without an ultrasonic cleaning.

The ultrasonic generator has been effective on production assemblies only if the solvents are changed frequently. The frequency that the solvent is to be changed must be determined by the amount of foreign material to be cleaned from the assembly because continued use of any quantity of solvent will soon add to the problem by uniformly coating all parts of the assembly with foreign material cleaned from prior assemblies. High pressure solvent spray is used to minimize some of these problems. Trichloroethylene and Freon TF are good because no fire or explosion hazards are prevalent, yet all solvent spray cleaning units require specialized facilities to remove and condense vapors for disposal.

Each assembly must be studied to determine the surface and material effect of the cleaning solvent, especially the effect of any absorbed solvent upon subsequent encapsulants or conformal coatings. Many devices and proprietary materials are used to manufacture individual components and are likely to absorb portions of cleaning solvents depending upon the conditions used. Effective drying can be obtained by baking at elevated temperatures as well as by exposure to vacuum environments. Heated vacuum techniques have also been used successfully to remove surface moisture when its presence has been determined to be detrimental to the processes or materials.

Solvent Compatibility

Conap Incorporated recommends methyl ethyl ketone (MEK) for diluting their Conathane resin series. Specimens prepared using the diluent contained three major visual discrepancies. A slight discoloration occurred that was thought to be a reaction with the copper test panel but which also outlined thickness variation areas on the test panels. Two categories of foreign material occurred; these were clear gel-like particles and black dirt-like

objects only the largest of which were visible to the unaided eye. Localized areas of reduced coating thickness without discoloration (fish eyes) also occurred.

Acetone, methyl cellosolve, toluene, a 50/50 mixture of MEK/methyl cellosolve, and a 50/50 mixture of toluene/methyl cellosolve were used to determine if more suitable solvents could be found for the polybutadiene based Conathane EN series. Mix ratios of two parts Conathane EN-4 to three parts solvent were used with the following indicated results:

Acetone: Mix turned cloudy when acetone was added. Resin appeared to precipitate when allowed to set at room temperature.

Methyl cellosolve: Large flakes of material precipitated when solvent was added.

Toluene: No apparent problem. Clear solution.

MEK/methyl cellosolve appeared cloudy and precipitated when allowed to stand at room temperature.

Toluene/methyl cellosolve: No apparent problem. Clear solution.

All specimens were cured for 24 hours at 160°F (71°C). Specimens using methyl cellosolve, MEK/methyl cellosolve, and toluene/methyl cellosolve contained surface tack after a 24-hour, 160°F (71°C) cure and were cured an additional 72 hours at 160°F (71°C) with no improvement. Fish eyes appeared when both MEK and acetone were used as diluents; however, these defects were not evident in coatings using toluene as the diluent.

Toluene was found to be the most compatible solvent with the urethane, polysulfides, and silicone resins tested. A proprietary blend of solvent, 66-C-28 (Midland Division of the Dexter Corporation), worked best with the epoxy urethane resin Laminar X500 (7-C-23).

Surface Finish of Component to Be Coated

Copper-clad, glass-epoxy circuit board material (MS 2141891) was used as a testing specimen. In the as-received condition, the copper surface showed the usual fingerprints and hairline scratches expected from a standard production process. It soon became apparent that large numbers of electrical failures in conformal coatings were caused by scratches or tool marks left by the manufacturing operation. The effect of conductor surface on coating protection was studied by measuring the voltage required to breakdown the coating after the conductor surface was prepared with different silicone carbide abrasives.

The conductor surface was abraded and the conformal coating was applied in the same direction. Some rounding occurred along the specimen cut edges eliminating any burrs caused by the cutting shear. Several strokes with the abrasives were used to present a uniform appearance on all boards. Those boards to be polished were first prepared with 600 grit paper then polished by rubbing in the same direction using du Pont "7" rubbing compound.

After the boards were dusted and the thickness was measured, a meticulous cleaning procedure was employed to obtain the maximum electrical protection. This procedure included ultrasonic cleaning in trichloroethylene, isopropyl alcohol, and electronic grade PCA Freon (du Pont) after masking one surface. Laminar X500 (7-C-23) was used to study the surface effect and was prepared with 66-C-28 reducer and 10-C-32 hardener, both Dexter Corporation products. After the ingredients were blended, the solution was allowed to stand for 30 minutes prior to dipping the panels. The panels were cured for 16 hours at 160°F (71°C).

The improvement in breakdown voltage as it relates to panel finish is significant even though the range of breakdown voltages in panels finished with the 500 grit abrasive was extensive (Figure 1). Microscopic ridges created by the coarse abrasive action reduced the actual coating thickness and effectiveness by allowing for higher voltage stress levels along the top of the raised microscopic metallic ridges. The first coating work with Conathane EN-4 showed small gas bubbles trapped on the surface of prepared panels when using a more viscous resin solvent solution; however, no bubbles were apparent on the panels coated with Laminar X500 (7-C-23).

Dielectric Strength of Five Thin Coatings

Four classes of coating materials are in general use for either mechanical, environmental, or electrical isolation of components. In specific applications, several functions may be served by one coating. To obtain thin coating, solvents are normally used to control viscosity and extend pot life; however, solvents must be compatible with both the resin system and the component to be coated.

A comparison of breakdown voltage versus coating thickness is presented (Figure 2). The substrate was copper clad glass epoxy circuit board material (MS 2141891). All specimens were polished with 600 grit to provide a uniform surface for reproducible electrical results. Test panels were washed in solvent prior to measurement and again prior to an oven drying at 160°F (71°C).

Figure 1. Electrical Breakdown Versus Surface Preparation of Copper

Toluene was the solvent selected as the diluent for all resin systems except the epoxy urethane complex Laminar X500 where the commercial solvent Laminar 66-C-28, a mixture of methyl ethyl ketone and ethylene glycol monoethyl ether acetate, was used because other solvents caused a precipitate to form and were considered incompatible. Dilution was used to obtain a coating thin enough to allow electrical failure without exceeding the test equipment limit of 10,000 volts dc. All specimens were coated at room temperature and cured in a vertical position.

The Laminar X500 (7-C-23) coating yielded the highest breakdown voltage followed by the polybutadiene based urethanes, Conathane EN-4 and EN-7. The room temperature vulcanized Dow Corning silicone DC 3140, although showing thicker coatings, also shows a higher voltage breakdown than the polysulfide, Chem Seal Corp. 3100.

Figure 2. Breakdown Voltage Versus Thickness of Five Coating Materials

The polysulfide mixture showed a slight degree of separation as small dark brown spots appeared over the surface after the mixture was cured for a short time. The EN-7 had the same surface character as EN-4 as well as a similar solid particle content. These solid particles were found to be in the resin component and can be strained out by filtering the solvent resin mixture. The particles are thought to be particles of reacted resin and are undesirable in coating materials. Bubbles formed on the copper surface in the dipping solution; however, they broke as the panel was lifted out of the solution.

The thickness change noted between the two test points is similar in all materials and ranges from 4.3 to 5.3×10^{-4} inches (0.010 to 0.013 mm). Thinner coatings, while showing higher average breakdown voltages per unit thickness, are affected more by foreign material or perturbations on the surface. Also, standard deviation was greater on the thinner test area for each material.

Effect of Four Metallic Surfaces on Breakdown Voltage

The breakdown voltage of a single coating of Laminar X500 (7-C-23) was measured when the coating was cured against gold, plated tin lead, nickel, dipped tin lead, and a matte copper surface similar to the plated metals. Tables 2 and 3 are a listing of the metal showing the average thickness range and the average breakdown voltage range for each checkpoint.

Plated tin lead has a lower breakdown voltage level than dipped tin lead because the plated surface is porous and contains microscopic spikes which can cause concentrated electrical stress in the coating. For this reason the copper surface was not polished as on other test sets but prepared to simulate the plated specimens. Also, many gold plated components are matte finished to improve wet-ability to soldering. However, here again, the matte surface condition does not promote the best breakdown voltage in conformal coatings. The plated nickel specimens approach the dipped tin lead values and both are equal to or above copper in breakdown voltage. As the Laminar X-500 (7-C-23) coating increases in thickness, the resin metal interface has less affect upon standoff voltages and it approaches a value controlled by the resinous material alone.

Relationship of Radii Magnitude and Its Limitation to Coating Thickness by Breakdown Voltage

One pressing production problem involved electrical failure at corners and edges of components and along bends in the wire where the resin coating thins out. At first it was thought failures occurred where tools or mishandling caused marks, sharp edges, or upsets on the metal contour to create sharp projections which reduced the coating thickness. An experiment was used to determine if the magnitude of a radius was contributing an affect and how large the minimum required radius would be.

Stainless steel balls of five radii were vacuum brazed onto stainless steel bars in a manner which retained the spherical surface of the ball. Three sets of ten were then plated, one set with gold, one with nickel, and one with copper (Figure 3).

All specimens were cleaned by vapor degreasing in TCE, measured, and re-degreased followed by baking in a 160°F oven (71°C).

Conathane EN-4 was prepared for coating by weighing out the resin (Part A), adding the thinning solvent and mixing, then adding the hardener (Part B), and mixing. The following ratios were used.

Table 2. Breakdown Voltage Range of Laminar X500 at Test Point A on Different Metals

Base Metal	Average Coating Thickness		Voltage	
	Mil	μm	V/Mil	V/ μm
Plated Tin Lead	0.24	6.1	7763	305
Dipped Tin Lead	0.23	5.8	12931	509
Plated Gold	0.27	6.9	8597	338
Matte* Copper	0.31	7.9	10764	424
Plated Nickel	0.27	6.9	11635	458

*600 Grit Finish

Specimens were measured and tested on a jig so the same point measured for thickness would be tested electrically.

Specimens were 1 by 3 inches (25.4 by 76.2 mm) by thickness of material.

Test point A is 1/2 inch (12.7 mm) from 3 sides.

EN-4 Part A 400 grams

Solvent 200 grams

EN-4 Part B 70 grams

This mixture was allowed to stand for 30 minutes before the hot 160°F (71°C) specimens were dipped into the resin/solvent mixture. One hour at room temperature was allowed for the excess resin to drip off the specimens, then a cure of 24 hours at 160°F (71°C) was started.

Dimensional measurements were made after cure using the same locations as were used prior to coating. These are compared in Figure 4, along with corresponding measurements made 0.25 inch

Table 3. Breakdown Voltage Range of Laminar X500 at Test Point B on Different Metals

Base Metal	Average Coating Thickness		Voltage	
	Mil	μm	V/Mil	V/ μm
Plated Tin Lead	0.52	13.2	9074	357
Dipped Tin Lead	0.54	13.7	10657	420
Plated Gold	0.54	13.7	11241	443
Matte* Copper	0.64	16.3	10608	418
Plated Nickel	0.58	14.7	10656	420

*600 Grit Finish

Test point B is 1/2 inch from the opposite end.

Test point B is the first part of the specimen to be coated with resin and hangs in the lower position when cured vertically.

(6.35 mm) away and on the same level as the ball showing the corresponding thickness of EN-4 resin coating on the flat surface.

A positive coating could not be ascertained consistently by measurement on diameters smaller than 0.032 inch (0.813 mm). Coating presence was verified by testing for electrical breakdown after the thickness was measured. Stainless steel balls consistently picked up a thicker coating of EN-4 while nickel and copper plated balls held a thinner coating. The flat surface of the copper plated specimens consistently picked up a thinner coating of EN-4 than the other metals. However, overall the flat surface measured 4 to 10 times thicker than the coating over the spheres.

Electrical breakdown voltage tests were made on the spheres where the EN-4 thickness was measured (Figure 5). All specimens with 0.032 inch (0.813 mm) diameter spheres and larger had standoff

Figure 3. Stainless Steel and Gold Plated Radii Test Specimens

LOWER DATA CORRESPONDS TO COATING THICKNESS OVER FIVE SPHERES INDICATING COATING THICKNESS RELATIVE TO DIAMETER AND MATERIAL: LETTER G INDICATES GOLD, N NICKEL, C COPPER, AND S STAINLESS STEEL.

UPPER DATA CORRESPONDS TO COATING THICKNESS 0.25 IN. (6.25 μm) FROM SPHERE ON SAME LEVEL AS SPHERE INDICATING COATING THICKNESS ON FLAT SPECIMEN SURFACE

LETTER POSITIONS CORRESPOND TO AVERAGE THICKNESS VALUES

Figure 4. Thickness of Conathane EN-4 Coating Compared to Diameter of Sphere

LETTER G INDICATES GOLD, N NICKEL, C COPPER, AND S STAINLESS STEEL.

0.015 INCH (0.38 mm) DIAMETER. NO ELECTRICAL VALUES FOR NICKEL, COPPER, AND STAINLESS STEEL.

0.025 INCH (0.63 mm) DIAMETER. NO ELECTRICAL VALUE FOR COPPER.

Figure 5. Breakdown Voltage of Conathane EN-4 Showing Effect of Conductor Material

voltages greater than 1000 volts/mil (39.4 volts/μm). Several instances occurred where no measurable coating existed; however, some coating was detected electrically on gold plated specimens. No measurable voltage existed for nickel, copper, and stainless steel on the 0.015 inch (0.381 mm) diameter balls and on copper for the 0.025 inch (0.635 mm) diameter balls. Stainless steel resulted in lower average breakdown voltage than the other metals tested. The breakdown voltage of the coating on the plate could not be measured because it was too thick for the hipot tester available.

The large variation noted, especially when including the panel area beside the spheres, is indicative of the results which occur on production wiring board assemblies. Sharp points have little chance of being effectively coated by dipping with any thin resinous material.

Variation in Thickness Relative to a Dipping Process

The coating thickness variation on flat test panels was noted on all dipped specimens. The lower portion, or first part of the panel to contact the coating resin (Point B), always had a coating approximately twice the thickness of the upper portion of the board (Point A), the section of board under the surface of the resin for the shortest duration of time. Several technique changes were tried to eliminate this phenomena; however, no single dip process would eliminate the thickness variance of the panels.

One set of panels was coated twice, using opposite positions for each dip, along with a companion set done in the normal manner, coating from the same end each dip. Laminar X500 (7-C-23) was the resin used and the data was measured (Table 4).

The panels that were dipped twice were not cured before the second coating but were dried 20 to 40 minutes at 160°F (71°C) to evaporate most solvents prior to the second coating. These panels were then cooled for 10 minutes at room temperature prior to the second dip.

Reversing the dipping position did not provide thickness equalization when components were mounted on circuit boards because any uniform material flow was disturbed by the components.

Effects of Removing Foreign Material From a Resin System

Conathane EN-4 hardener (Component B) contains a desiccant added to trap any moisture contacted during storage or the manufacturing process thereby extending the usable shelf life of this compound. The desiccant settled to the bottom of the package and agglomerated; on re-mixing, the desiccant could not be re-dispersed and visual evidence indicated the electrical breakdown was occurring at the agglomerated desiccant locations.

A test was designed to show differences, if any, in electrical standoff voltage by comparing as-received commercial material with special material where the agglomerates had been removed by use of a centrifuge or with filter paper.

Test panels were prepared identically for each set. The resin component (Part A) was diluted with toluene in a 1:2 ratio, and mixed with hardener (Part B) as received (Set 15), centrifuged (Set 14), and filtered (Set 17) (Figure 6).

Table 4. Breakdown Voltage Range of EN-4 Coating Thickness Versus Dipping Position

Point Held Up		Average Coating Thickness		Voltage	
Dip 1	Dip 2	10 ⁻³ in.	μm	V/Mil	V/μm
A	A	0.7	17.8	12108	477
A	B	1.04	26.4	*	*
B	B	1.36	34.5	*	*

*Thickness was too great for electrical equipment.
 Specimen size is 1 by 3 inches (25.4 by 76.2 mm).
 Point A is 1/2 inch (12.7 mm) from three sides.
 Point B is 1/2 inch from opposite end of specimen.

The mixed resins for each set were allowed to stand for 30 minutes to allow included air bubbles to rise and then the resins were agitated in an ultrasonic generator to assist in removing these bubbles. The test panels were dipped vertically, touched to a Kaydry brand paper towel to remove most of the resin bead formed along the bottom edge, and positioned horizontally in a forced air oven to cure 24 hours at 160°F (71°C). Removing the desiccant resulted in two changes: a slight increased average thickness of coating, and double the average standoff voltage of unfiltered coatings.

On thin coated test specimens, the first part of the specimen to contact the resin mix is thicker, even when specimens are cured horizontally. Test Point B is at the lower end of the panel, the first part of the panel to contact the resin when coating by dipping. The use of filter paper also appears to improve the average electrical standoff voltage over the centrifuging method of removing desiccant (Figure 6, Set 17).

To determine if changes occurring when filtering the EN-4 resin (Part A) would also affect the electrical breakdown of a film coating, sets 22 and 23 were processed with identical conditions. Set 22 was used as received and the resin of set 23 was filtered through an ED 612 filter (Eaton Dikeman Company). Filtering took place after the EN-4 resin had been diluted with two parts toluene for one part resin (Part A). Both sets (22 and 23) used Part B which had been centrifuged to remove the desiccant.

- 14 RESIN AS RECEIVED, HARDENER CENTRIFUGED
- 15 RESIN AS RECEIVED, HARDENER AS RECEIVED
- 17 RESIN AS RECEIVED, HARDENER FILTERED
- 22 RESIN AS RECEIVED, HARDENER CENTRIFUGED
- 23 RESIN FILTERED, HARDENER CENTRIFUGED

Figure 6. Breakdown Voltage Versus Thickness of Conathane EN-4 on Copper Showing Effects of Removing Solid Particles

When the copper clad panels were submerged into the resin solvent mixture, small bubbles appeared on the copper surface as the gases collected along the scratches in the surface. These bubbles would break when the specimens were removed leaving localized thinner coatings (fish eyes) where the bubbles had been.

The filtering removed a number of small, clear soft particles of gelled resin. Although the average thickness of Test Point B of both sets is almost identical, little improvement in breakdown voltage was noted by filtering the mixed resin. The gel particles found in EN-4 Part A had little effect on the breakdown voltage in comparison to the effect of filtering or centrifuging the hardener solution to remove the desiccant.

A microscopic examination of the gel particles found them to be agglomerated desiccant, added by the manufacturer, as well as clear resinous particles. The desiccant had been added to improve storage life of the packaged resin. Although the desiccant was ground to a 2 to 5 micron range, it had settled to the bottom of the container during storage and could not be redispersed into individual particles by available techniques.

Both centrifuging and filtering solvent solutions were found to improve the foreign material problem. All commercial coating materials tested contained some extraneous particles requiring removal to provide for optimum standoff voltage protection. Similar considerations, discussed for Conathane EN-4 resin coating, were employed with all materials.

Nonuniformity of Wire Coating

The coating uniformity of small diameter wire and small components was disappointing. Manufacturers of small electrical components furnish these items encased with materials having good electrical resistance. These materials surfaces are usually inert or have a quality of not reacting with conformal coating materials. This causes the coating materials to bead due to differences in surface tension, leaving the edges bare of protection. The wire industry applies coating in very thin layers programming a partial cure between each layer and wiping each layer before the partial cure to assure a complete coverage as well as a uniformly thin coating. These methods are applicable to wire but not to an assembly or individual components. More recent designs treat such cases as the electrical component manufacturers do by grouping these components and encapsulating these special assemblies to isolate those affected circuits. Numerous periodicals have dealt with these problems within the past 5 years.

Aging Characteristics

As polymer materials age, dimensional changes occur which are caused by chemical restructuring, stress relaxation, molding conditions, temperature changes, and moisture absorption or loss. Some or all of these factors affect coating materials to some extent; however, the effects of aging time, temperature cycling in ambient condition, and the influence of relative humidity changes have been measured on Conathane EN-4 and compared to those of du Pont's Adiprene L-100/MOCA. The largest dimensional change is caused by the cure schedule and the following temperature drop to ambient conditions. These changes normally occur without the benefit of a stable moisture condition which takes place over a longer period of time causing continuous change (Tables 5 and 6).

As expected, the higher temperature cures cause greater shrinkage. A further study was made covering the effect of time, temperature, and relative humidity on the dimensions of EN-4 and Adiprene L-100/MOCA where the separate effects of aging temperature and aging relative humidity are computed. This indicates it takes the polybutadiene based EN-4 45 days to reach 90 percent of its weight stabilization when cured at 160°F (71°C) for 24 hours. Much shorter times are required for the 180°F (82°C) cure of EN-4 to stabilize; however, the linear shrinkage data favors the 160°F (71°C) cure showing values one-half of the high temperature cure. These estimates were made using day-to-day changes measured at ambient conditions. The relative humidity varied from 15 to 43 percent throughout the one-hundred day measuring period.

A comparison of the changes caused by ambient humidity and temperature, experienced during storage in the laboratory, were made on Adiprene L-100/MOCA and Conathane EN-4. The change in weight of Adiprene L-100/MOCA with a change in relative humidity was two to five times greater than for Conathane EN-4, depending upon the cure cycle used. The change in dimension from a change in weight with changes in relative humidity was from 0.25 to 0.45 $\mu\text{m}/\text{m}/\text{g}$ of water absorption for EN-4. Adiprene L-100/MOCA when corrected for the difference in density and moisture pickup ratio would be at least 0.61 to 0.69 m/m for a corresponding volume of EN-4 material. The change in dimension with change in temperature gave thermal coefficients of 63 to 88.5 $\times 10^{-6}$ $\text{in.}/\text{in.}/^{\circ}\text{F}$ (113 to 159.3 $\mu\text{m}/\text{m}/^{\circ}\text{C}$) for Adiprene L-100/MOCA and 63 to 79 $\times 10^{-6}$ $\text{in.}/\text{in.}/^{\circ}\text{F}$ (113 to 142.2 $\mu\text{m}/\text{m}/^{\circ}\text{C}$) for EN-4, depending upon the cure cycle used (Table 7).

Table 5. Shrinkage of Urethane Elastomers at Room Temperature

Elastomers	Cure Schedule		Shrinkage			
	Time (Hours)	Temperature		After Cure (Percent)	After 30 Days (Percent)	After** 60 Days (Percent)
		°F	°C			
EN-4*	24	160	71	0.36	0.56	0.48
EN-4	16	180	82	0.76	0.90	0.88
EN-4	10	200	93	0.90	1.26	1.22
L-100/MOCA	96	RT		-0.08	-0.08	-0.08
L-100/MOCA	4	160	71	0.68	0.7	0.72
L-100/MOCA	40	160	71	0.92	0.94	0.92
L-100/MOCA	3	200	93	1.08	1.10	1.02
L-100/MOCA	16	200	93	1.26	1.30	1.36

*The EN-4 specimens cured at 200°F (93°C) exhibited a concave surface on the ends which caused a correction to be made at that value. These specimens also showed excessive shrink marks on the flat 1 by 5 inch (25.4 by 127 mm) surfaces.

**Changes in these polymers after 60 days aging may be caused by the absorption or desorption of small quantities of moisture dependent upon the ambient storage history.

The dimensional changes occurring in Conathane EN-4 in a vacuum at zero relative humidity as well as at controlled environments of 20, 40, and 70 percent RH environments were studied for the three temperature cures of 160°, 180°, and 200°F (71°, 82°, and 93°C). The specimens used had been aged at room temperature conditions several months prior to this study. The weight loss in vacuum was nearly alike for specimens from all three cures. Since the material was mixed in a vacuum, it was expected that all volatile materials should have been removed.

The change in dimension in vacuum ranged from 0.24 to 1 percent for the 160° to 200°F cures (71° to 93°C). The difference between cures is not explained by weight loss alone; it appears to be caused primarily by a stress relaxation after removal from the mold.

Table 6. Thermal Coefficient of Expansion

Elastomers	Cure Schedule			Coefficient of Expansion	
	Time	Temperature		μin./in./°F	μm/m/°C
		°F	°C		
EN-4	24 Hours	160	71	71.3	128
EN-4	16 Hours	180	82	65	117
EN-4	10 Hours	200	93	69	124
L-100/MOCA	7 Days	RT		86.9	156
L-100/MOCA	4 Hours	160	71	92	166
L-100/MOCA	3 Hours	200	93	78.8	142

The change from vacuum to 20 percent RH gave a small increase in weight while the dimension did not increase; it appeared to have a slight decrease. This appears to be a further relaxation of the material.

Increasing the relative humidity from 20 to 40 percent caused a similar dimensional change for all three cures; however, the weight response is reduced with increased cure temperature.

The total change from vacuum to 70 percent RH showed a small decrease in moisture pickup with increased temperature cure. The differences in weight are relatively small. The dimensional change is greater for the higher temperature cure. The higher shrink with increased cure temperature suggests a low temperature cure when using EN-4 as an encapsulant.

Solvent Swell

Solvents of one type or another are used to thin resins to lower viscosities or solid contents compatible to the application techniques utilized or to the coating thickness required. Compatibility of the resin/solvent combination is mandatory and the solvent should be capable of leaving the polymer, allowing the resin to remain in a tightly adhered coating. Unfortunately, solvents create drastic dimensional changes which allow for stresses due to shrinkage to remain in the final coatings. Manufacturers also slow the evaporation of solvents by adding those which volatilize slowly. These solvents cause problems because the coating material requires an excessive cure time or aging

Table 7. Coefficient of Expansion From Humidity Changes

Elastomer	Cure Schedule			Humidity Coefficient
	Time	Temperature		
		°F	°C	μm/m/%RH
EN-4	24 Hours	160	71	22.6
EN-4	16 Hours	180	82	17.0
EN-4	10 Hours	200	93	33.8
L-100/MOCA	7 Days	RT		37.7
L-100/MOCA	4 Hours	160	71	24.1
L-100/MOCA	3 Hours	200	93	13.05

time to become stable. Removing the undesirable solvent can also be a safety hazard requiring specific techniques for explosive or otherwise dangerous solvents. Almost all cleaning required the use of solvents, either as a vapor or a liquid. The effect upon the functionality of the polymer being cleaned is dependent upon the temperatures used as well as on the solvent remaining.

Several tests have been performed showing the effects of cleaning techniques on these materials. Measurements have been made concerning the effect upon dimensions of EN-4 due to cleaning thin films (0.003 inch or 0.762 mm), in several solvents. Changes in the ascending order of severity caused by room temperature soaking for five minutes were: water, isopropyl alcohol, Freon, and trichloroethylene.

The rate of solvent loss from 50 percent solvent mixtures of three comparable solvents used for dip coating (50/50 ratio of MEK/toluene, TCE, and 28/72 isopropyl alcohol/TCE) showed the 50/50 ratio of MEK and toluene to be completely evaporated in 24 hours at room temperature.

The response of three cures of EN-4 to soaking in six different cleaning solvents (acetone, alcohol, Freon, MEK, toluene, and TCE) was measured. No significant difference was shown to have been caused by the three cures used; however, the rate of solvent pickup was rapid. Of these six, alcohol had the lowest rate of absorption by the EN-4 by weight or volume. It also had the least total absorption after a 7-day soak. The rapid absorption

of these volatile materials indicates some EN-4 was dissolved in the solution. Cured EN-4 had the least solubility in Freon and alcohol and the greatest solubility in trichloroethylene. Upon removal from the solvents, alcohol and Freon did not evaporate completely after a 2-week room temperature air exposure. MEK, toluene, and TCE were practically evaporated in 5 days at room temperature; however, the EN-4 is definitely too absorbent for MEK, toluene, or TCE over more than a 1-day period because gross cracking of the polymer can occur when specimens are removed from these solvents (Figure 7).

TMA Analysis

TMA incorporates the use of a weighted probe to measure the penetration over a programmed temperature change. Analysis of three cures of Conathane EN-4 showed these cures to be equivalent when enough time was allowed after cure to age the material to equilibrium conditions. TMA charts also help locate the phase change temperature if it is present in the range of test temperatures used. TMA curves indicate one phase change occurs near -100°F (-75°C) and another occurs near 122°F (50°C) (Figure 8). The change at low temperature is beyond any expected environment; however, the upper temperature phase change is of direct concern because it is well within the use temperature of most electrical assemblies. The upper temperature portion of the curve may not be real because excessive penetration may allow the probe shoulder to contact the specimen material being measured.

Dynamic Shear Modulus

To be able to predict the effects of grouping polymers within one package, it was necessary to define a moduli value by measuring the elastomeric element of the material. The American Society for Testing and Materials has published a method of test for *Dynamic Mechanical Properties of Plastics by Means of a Torsional Pendulum*, D-2236-70, describing equipment and formulas which can be used. By comparing the value of shear modulus (G'), loss modulus (G''), and loss tangent (G''/G'), the changes in second order transition can be determined as well as changes caused by formulation, processing, environmental conditions, and aging. Data show the room temperature shear modulus to be very similar for Conathane EN-4 and Adiprene yet the loss modulus G'' for EN-4 is much higher than G'' for Adiprene L-100 indicating the greater damping capabilities of EN-4 over Adiprene L-100/MOCA at room temperature.

Comparison of modulus data at room temperature for a polysulfide (Coast Pro Seal 727) indicates a loss tangent, G''/G' , intermediate between EN-4 and Adiprene L-100/MOCA. Since Pro Seal 727 does not exhibit weight stability, care must be exercised in using this data to identify the cure condition and aging criteria.

TOLUENE

ACETONE

METHYL ETHYL KETONE

ISOPROPYL ALCOHOL

TRICHLOROETHYLENE

FREON TF

Figure 7. Appearance of Conathane EN-4 After Solvent Soak in the Indicated Solvents and Air Drying (X1.5)

Figure 8. Thermomechanical Analysis of Conathane EN-4, Change in Length Versus Cure Temperature

Additional dynamic data are to be found in Table 8 where data was taken at three temperatures: -65°F (-54°C), room temperature, and 165°F (74°C). Differences at both temperature extremes are quite noticeable showing the real difference between the Conathane EN and Adiprene/MOCA systems. Adiprene tends to have a similar shear modulus to the EN resin from -65°F through room temperature yet little change is noted as the Adiprene L-100/MOCA temperature is increased to 165°F (74°C). EN-7, however, drops an order of magnitude above room temperature through 165°F . This indicates the material becomes more like soft putty at elevated temperatures acting more like a hydraulic fluid.

The time that the material spent at elevated temperature was the shortest time for the test specimen to reach stabilization. Loss modulus data indicates a similar trend; however, note the reverse trend of loss tangent data especially for 160°F (71°C) cured polymers.

Adhesion

To obtain the desired level of soundness in mechanical as well as electrical properties of assemblies, adhesion of coatings and encapsulants to components and structural members is imperative. Lap shear data of EN-4 adhesive was obtained for several materials found in a typical electrical assembly. Average shear values for five specimens are shown (Table 9).

Pull out data using polyolefin and Kynar (Pennwalt Corporation) insulated wire surrounded with EN-7 showed better adhesion to Kynar especially after thermocycling test assemblies between 200°F (93°C) and -65°F (-54°C).

Further testing was done with another polybutadiene based system, Conathane EN-7. The only difference is that the mercury catalyst of EN-4 has been replaced by ferric acetylacetonate which does not volatilize at temperatures as low as 93°C .

It has been generally noted that Adiprene L-100/MOCA adheres poorly to metals. Values obtained immediately after cure may be good to excellent; however, as the bond ages and the Adiprene L-100 becomes impregnated with small quantities of moisture (found at ambient conditions), the adhesion deteriorates to a level where a thermocycle from -65 to 165°F (-54 to 74°C) will cause the Adiprene to release metal surfaces. To combat this tendency, primers (XAD 772, Dayton Laboratories) have been employed which are compatible to both the metals involved as well as the Adiprene L-100 encapsulant.

Table 8. Dynamic Physical Properties Chart

Test Material and Conditions	-65°F (-54°C)	77°F (25°C)	165°F (74°C)
G' Shear Modulus (kpsi) (MPa)			
EN-4			
24 Hours at 160°F (71°C)	12.9 (88.9)	3.4 (23.4)	0.236 (1.63)
16 Hours at 175°F (80°C)	29.7 (205)	3.3 (22.7)	0.271 (1.87)
10 Hours at 200°F (93°C)	16.3 (112)	3.2 (22.1)	0.251 (1.73)
L-100/MOCA			
7 Days at 77°F (25°C)	65 (448)	3.1 (21.4)	2.7 (18.6)
4 Hours at 160°F (71°C)	61.4 (423)	3.2 (22.1)	3.4 (23.4)
3 Hours at 200°F (93°C)	54 (372)	3.1 (21.4)	3.68 (25.4)
EN-7			
16 Hours at 175°F (80°C)	15.2 (105)	3.5 (24.1)	0.183 (1.26)
CS3100		0.154 (1.06)	
Coast Pro Seal 727	0.26 (1.79)	0.154 (1.06)	
Silicone DC3140		0.039 (0.269)	

Table 8 Continued. Dynamic Physical Properties Chart

Test Material and Conditions	-65°F (-54°C)	77°F (25°C)	165°F (74°C)
G" Loss Modulus (kpsi) (MPa)			
EN-4			
24 Hours at 160°F (71°C)	1.1 (7.58)	0.598 (4.12)	0.048 (0.331)
16 Hours at 175°F (80°C)	1.41 (9.72)	0.59 (4.07)	0.073 (0.503)
10 Hours at 200°F (93°C)	1.19 (8.20)	0.586 (4.04)	0.054 (0.372)
L-100/MOCA			
7 Days at 77°F (25°C)	2.57 (17.7)	0.17 (1.17)	0.09 (0.620)
4 Hours at 160°F (71°C)	3.4 (23.4)	0.120 (0.827)	0.05 (0.345)
3 Hours at 200°F (93°C)	2.2 (15.2)	0.11 (0.758)	0.034 (0.234)
EN-7			
16 Hours at 175°F (80°C)	1.89 (13.0)	0.74 (5.10)	0.05 (0.345)
CS3100		0.035 (0.241)	
Coast Pro Seal 727	0.034 (0.234)	0.035 (0.241)	
Silicone DC3140		0.011 (0.758)	

Table 8 Continued. Dynamic Physical Properties Chart

Test Material and Conditions	-65°F (-54°C)	77°F (25°C)	165°F (74°C)
Loss Tangent G"/G'			
EN-4			
24 Hours at 160°F (71°C)	0.085	0.175	0.2
16 Hours at 175°F (80°C)	0.047	0.185	0.27
10 Hours at 200°F (93°C)	0.037	0.185	0.21
L-100/MOCA			
7 Days at 77°F (25°C)	0.039	0.055	0.033
4 Hours at 160°F (71°C)	0.073	0.035	0.016
3 Hours at 200°F (93°C)	0.033	0.035	0.009
EN-7			
16 Hours at 175°F (80°C)	0.11	0.21	0.29
CS3100		0.226	
Coast Pro Seal 727		0.133	
Silicone DC3140		0.295	

Table 9. Tensile Shear Values for EN-4
as an Adhesive

Substrate	Shear	
	psi	MPa
Epoxy Glass Laminate	918	6.329
Tin Lead Plating	1025	7.066
Gold Plating on Aluminum	949	6.542
Nickel Plating on Aluminum	839	5.784
Nickel Foil	935	6.445
Cured EN-4	1051	7.245
Acrylic Sheet	655	4.515

The polybutadiene based urethane, Conathane EN-7 has not shown these tendencies. EN-7's hydrolytic stability is much improved and it has a natural adhesion for most metals which was not found in the polyether based Adiprene L-100. Another plus factor for EN-7 is an improvement in dielectric strength on 0.08 inch (2 mm) slabs after aging. Using the ASTM test for dielectric strength on the 0.08 (2 mm) slabs, the dielectric strength of Adiprene L-100 starts near 500 volts/mil (20 kV/mm) after curing and will deteriorate to values near 300 volts/mil (12 kV/mm) with room temperature aging. EN-7 starts around 500 volts/mil (20 kV/mm) and values increase with aging to near or above 1000 volts/mil (40 kV/mm).

Table 10 relates peel values found using Conathane EN-7 cast against Kapton film per MS 2320012. Values were obtained using 180 degree peel specimens pulled at 10 inches per minute (0.254 m per minute).

It was noted that aging by temperature shock cycles or humidity cycles improved adhesion but it is not yet known if this is due to the environment's effect upon the EN-7 or the Kapton or both. Gas plasma cleaning techniques yielded no improvement on one set of five specimens over solvent cleaned Kapton where a second set doubled the gas plasma cleaned values over solvent cleaned values. Two etch preparations did improve adhesion; however, these techniques would be limited to specific situations where no detrimental effects would occur to metals involved in assemblies.

Table 10. Peel Values of EN-7 Against Kapton at 68°F (20°C)

Surface Treatment	Kapton Thickness		Peel Strength	
	Mils	μm	p/li	N/m
Solvent Cleaned	5	127	27.7	4851
Gas Plasma Cleaned	5	127	8.9-17.5	1559-3065
Tetramethyl Ammonium Etch	5	127	40.9	7163
Isoprep 177 Etch	5	127	25	4378
Aged 16 Hours at 300°F (148°C)	5	127	62	10,858
Coated With Nylon	2	508	5.6	981
Nylon Coated, Aged 16 Hours at 300°F	2	508	11.3	1979
Solvent Cleaned	2	508	3.9	683
Aged 16 Hours at 300°F	2	508	21.3	3730
1 Percent Union Carbide A187 Silane	5	127	6.7	1173

The time between the plasma cleaning and the specimen casting was not recorded; however, this phenomenon may be dependent upon the timing between cleaning and the bonding operation.

Heat aging peel test assemblies at 300°F (149°C) for 16 hours did improve adhesion; however, like temperature shock and humidity aging, we do not yet understand the cause of improved adhesion. No improvement was noted by using one Silane primer.

Adhesion of Adiprene L-100/MOCA is better for three of the four materials compared in Table 11. Even so, it is felt that, with the proper joint design, the EN-7 values should be adequate for production uses. The uses of additives and their effects have not been studied in depth. These could be added to the resin encapsulant in lieu of being applied to the substrate as precoats.

Table 11. Peel Values of EN-7 and Adiprene L-100/MOCA to Insulation Material

Surface Feature*	Peel Strength in Pounds/Linear Inch** at Indicated Temperatures				
	-65°F (-54°C)	69°F (20°C)	165°F (74°C)	68°F*** (20°C)	68°F† (20°C)
EN-7 to Kapton	13.3	8.6	2.6	28.9	24.8
L-100 to Kapton		27.7			
EN-7 to Neoprene	7.7	15.8	5.3	21.1	22
L-100 to Neoprene		29.3			
EN-7 to Polyolefin	7.1	8.6	1.4	4.1	4.2
L-100 to Polyolefin		2.9			
EN-7 to Urethane	34.6	17.2	1.2	15.3	5.1
L-100 to Urethane		45			

*All specimens were solvent wiped with a 50/50 solution of alcohol/toluene prior to casting the EN-7 or Adiprene L-100.

**p/li = 175.13 N/m.

***After temperature shock consisting of 1 cycle starting at room temperature, place unit in a -54°C (-65°F) chamber and hold for 8 hours. Transfer to a 71.1°C (160°F) chamber within 1 minute and stabilize the unit. Return to the -54°C (-65°F) chamber (within 5 minutes) and hold unit for 8 hours. Place unit in room ambient and stabilize temperature.

†After humidity cycling consisting of 10 consecutive 48 hour cycles starting at 30°C (86°F), increasing to 65°C (149°F), at a rate of 9.4°C (15°F)/hour minimum, holding at 65°C (149°F) 11 hours. Reduce the temperature to 30°C (86°F) at a rate of 9.4°C (15°F)/hour minimum, holding at 30°C (86°F) for 21 hours. Reduce the temperature to 20°C (68°F) in 1 hour holding at 20°C (68°F) 4 hours returning to 30°C (86°F) in 1 hour holding at 30°C (86°F) 5 hours. Relative humidity at constant temperature was controlled at 93 percent ±5 percent.

Temperature shock improves the room temperature adhesion of EN-7 toward Neoprene and Kapton, has little effect upon the adhesion of EN-7 toward urethane sleeving, but drops 50 percent of the adhesion for polyolefin. After humidity cycling, both the polyolefin and urethane substrates produced lower adhesion values indicating a moisture effect upon these two substrates.

The adhesion of EN-7 to metal is good, unlike Adiprene L-100/MOCA, showing highest values for vapor blasted aluminum at all temperatures tested. This adhesion is expected to hold even during extended periods after encapsulation because of the excellent moisture stability of the EN-7. These higher peel values for metals were obtained without the use of intermediary primers or adhesives which were necessary when adhesion was required with the Adiprene L-100/MOCA system. Non-metallic materials including nylon and etched Teflon indicate excellent relative adhesion. The adhesion to epoxy glass circuit board material NEMA grade G10 was tested at 60 p/li (10,508 N/m) at ambient conditions (Table 12).

The lower shear modulus of EN-7 at elevated temperature was such that a force of 8 p/li (1401 N/m) would not be exceeded and failure occurred within the EN-7 not in the bond areas.

Studies using 90 degree T peel specimens to determine the adhesion of EN-7 to EN-7 were made to optimize the cleaning techniques which were available. The first casting was formed in 0.080-inch (0.2 mm) thick slabs in TFE Teflon coated aluminum molds cured at temperatures and times listed under the first casting cure (Table 13). Trifluoroethylene releases transfer the smallest amount of material of any release system; however, the EN-7 surface contacting the Teflon may contain a character of Teflon by assuming a non-polar surface condition as found on Teflon.

The aging times used correspond to actual production schedules of double-layered encapsulated components. Values listed are averages of five individual tests.

Abrasion offers by far the best treatment for adhesion between layers of EN-7. This could be due to an increase of the surface area or because of removal of the non-polar character on the surface. Abrasion can be obtained in several ways, even with a high rpm brush. All abraded specimens broke in a cohesive mode with the break usually occurring in the first casting which was usually slightly thinner than the second casting.

When partial cures are used to build systems, the as-cast condition shows improvement by aging at room temperature. Solvent cleaning systems showed poorer adhesion after aging one week.

Table 12. Peel Values of EN-7 to Rigid Plastics and Metals

Surface Feature	Peel Strength in Pounds/Linear Inch* at Indicated Temperatures		
	-65°F (-54°C)	68°F (20°C)	165°F (74°C)
G10 Laminate	31.3	60	**
Nylon As Received		19.3	
Nylon Vapor Blasted		54	
Teflon Etched		17.9	
Gold Plated Copper	7.6	18.3	
70-30 Brass Bright Dipped		16.6	**
Tin Lead Dip Coated on Copper		20.4	
Aluminum As Wrought	2.8	19.6	1.8
Aluminum Analyzed	21	26.3	4.3
Aluminum Vapor Blasted	26.9	35.4	7.9
Stainless Steel	11.9	28.3	**

*p/li = 175.13 N/m.

**Could not test because of excessive elongation of Conathane EN-7 in temperature control box which indicated adhesion in excess of value observed for vapor blasted aluminum.

Adhesion held constant on as-cast surfaces or improved with up to 14 days aging. These surfaces were exposed to ambient conditions found within the laboratory during aging.

Table 13. Adhesion of EN-7 to EN-7

Surface Treatment	Average Peel Strength (5 Specimens) After Indicated Aging Time Before Second Casting of 16 Hours at 175°F (80°C)					
	4 Hours (p/li)†	1 Day (p/li)	4 Days (p/li)	7 Days (p/li)	14 Days (p/li)	21 Days (p/li)
First Casting Cure at 175°F (80°C) for 4 Hours						
As Cast	50.8			51.8		
Abraded*	65.5			78.4		
TCE Wipe	54.4			49.0		
Toluene/Alcohol	53.4			37.9		
First Casting Cure at 175°F for 16 Hours						
As Cast		42.1	39.6	45.8	49.1	44.9
Abraded**		75.4	73.8	84.5	74.6	74.6
TCE Wipe***		41.3	51.6	36.1	46.1	46.1
Toluene/Alcohol		53.7††				
TCE Spray						
First Casting Cure at 175°F for 20 Hours						
As Cast		49.8		50.5		
Abraded		66.8		79.8		
TCE Wipe		47.6		60.2		
Toluene/Alcohol		46.4		54.7		

Table 13 Continued. Adhesion of EN-7 to EN-7

Surface Treatment	Average Peel Strength (5 Specimens) After Indicated Aging Time Before Second Casting of 16 Hours at 175°F (80°C)					
	4 Hours (p/li)†	1 Day (p/li)	4 Days (p/li)	7 Days (p/li)	14 Days (p/li)	21 Days (p/li)
First Casting Cure at 175°F for 24 Hours						
As Cast		40.1		47.8		
Abraded		76.3		73.3		
TCE Wipe		55.7		53.2		
Toluene/Alcohol		50.0		57.6		
<p>*All abraded specimens broke in cohesion. All other specimens failed in adhesion.</p> <p>**Second Casting, 3 days, 45.4 p/li.</p> <p>***Second Casting, 3 days, 39.4 p/li.</p> <p>†pli = 175.13 N/m.</p> <p>††Average of 10 specimens.</p>						

Solvent cleaning improved the adhesion on the over-cured specimens having 20 and 24 hour cure periods. It is known that EN-7 is not completely cured in the 16-hour 175°F (80°C) cure cycle; therefore, these data indicate those surfaces overcured do respond to solvent cleaning over no cleaning. Solvents are also convenient for removing certain classes of contaminants from a polymer surface which would only smear with abrasion.

No conclusive difference was noted between spray and wipe trichloroethylene. Spray cleaning techniques are recommended because clean solvent is always used, reducing chances of spreading any contamination.

Electrical Properties

Electrical properties are affected by many things both externally as stress environment, and aging as well as internally due to initial cures, contaminants, fillers, mix uniformity. It would be difficult to measure the effects of each factor let alone isolate the portion assigned to each cause. Some properties are felt to be general in nature and a few are felt to be pertinent to conformal coating work. Dielectric strength and constant along with dissipation factor were measured in the traditional manner; however, the Laminar X500, (7-C-23) and Conathane EN series could not be tested to dielectric strength failure as both systems show superior electrical properties. These material properties along with the polysulfide and silicone DC 3140 properties are found in Tables 14 and 15.

Breakdown voltage was a nonstandard test designed to quickly determine the effects of processing upon both the resin system as well as on a copper clad laminate. The technique has been described showing the progression of cleaning, effects of different solvents, the ratio of solvent to resin system, and cure. All these were measured and tested in a similar manner and can be, in that form of reference, compared with other compounds tested in a similar manner. To be able to electrically break down each material it was necessary to test thin coatings built with solvent solutions which were compatible yet would be removed during normal cures specified for the material being tested.

Laminar X500 (7-C-23) was found to be far superior electrically to the other systems tested. Values above 8000 volts per mil (0.025 mm) in thickness less than one mil were obtained. Conathane EN-4 and EN-7 tested above 4000 volts per mil (0.025 mm). DC 3140 silicone tested above 2500 volts per mil, while the polysulfides indicated 1200 volts per mil minimum value. Variations in all systems were large ranging up to twice the minimum in individual values.

Table 14. Electrical Properties Data for Three Materials Tested at 77°F (25°C)
Obtained From 0.08 Inch (2.03 mm) Thick Slabs

Electrical Properties	L-100 MOCA	EN-7			EN-4		
	77°F (25°C)	-65°F (-54°C)	77°F (25°C)	165°F (74°C)	-65°F (-54°C)	77°F (25°C)	165°F (74°C)
Dielectric Strength							
Volts/Mil*	388	1380**	1372	1279	1312	1312	1309
Dielectric Constant							
1 kHz	6.50	2.35	2.65	3.17	2.46	2.64	3.23
10 kHz	6.11	2.33	2.59	2.87	2.45	2.61	2.96
100 kHz	5.68	2.28	2.58	2.77	2.48	2.57	2.79
1 MHz	5.26	2.26	2.53	2.66	2.48	2.53	2.69
10 MHz	4.93	2.23	2.52	2.57	2.43	2.51	2.61
Dissipation Factor							
1 kHz	0.034	0.00543	0.00092	0.0532	0.0037	0.0105	0.0525
10 kHz	0.040	0.00525	0.00658	0.0474	0.0038	0.0089	0.0423
100 kHz	0.045	0.00572	0.00995	0.0353	0.0027	0.0111	0.0420
1 MHz	0.047	0.00322	0.00885	0.0253	0.0027	0.0086	0.0220
10 MHz	0.062	0.00312	0.01006	0.0253	0.0101	0.0128	0.0255

*Volts/Mil = V/25.4 μ m.

**Exceeded tester limit of 10 kVdc.

Table 15. Electrical Properties Data for Five Materials
Tested at 77°F (25°C) Obtained From 0.08 Inch
(2.03 mm) Thick Slabs

Electrical Properties	Polysulfide*			Silicone DC 3140	Epoxy/ Urethane Laminar X500
	CS3100	Pro Seal 727	PRC 1201Q		
Dielectric Strength					
Volts/Mil**	252	257	274	232	1163†
Dielectric Constant					
1 kHz	8.43	7.83	7.68	2.83	3.81
10 kHz	8.37	7.80	7.71	2.83	3.57
100 kHz	8.36	7.74	7.73	2.82	3.49
1 MHz	8.36	7.68	7.68	2.82	3.19
10 MHz	7.65	7.55	7.20	2.76	2.98
Dissipation Factor					
1 kHz	0.00255	0.00173	0.00066	0.00054	0.03958
10 kHz	0.00197	0.00267	0.00136	0.00052	0.04162
100 kHz	0.00484	0.00683	0.00531	0.00073	0.04189
1 MHz	0.02395	0.02238	0.02183	0.00121	0.04797
10 MHz	0.17038	0.01160	0.01160	0.00250	0.00606
*Polysulfide materials were cured for 24 hours at 160°F (71°C).					
**Volts/Mil = V/25.4 μm.					
†Exceeded tester limit of 10 kVdc.					

Toluene was found to be the most compatible solvent in the polysulfides, silicone, and urethane resins tested. A proprietary blend of solvents, 66-C-28 (Midland Division of the Dexter Corporation), worked best with the Laminar X500 (7-C-23).

These studies also showed that cleanliness and surface finish of the article to be coated play major roles in obtaining optimum results. The solvent cleaning system found most effective on copper clad laminate consisted of a vapor degrease in trichloroethylene followed by an alcohol and Freon TF wash. The use of ultrasonic energy during the two washes improved the measured dielectric strength of these coatings.

Development of these techniques was made using EN-4 and Laminar X500 (7-C-23) film coatings and greatly reduced the work required for optimizing the dielectric strength of other material and processes.

Filler materials appear to have detrimental effects on the electrical properties possibly due to the difficulty in dispersing the filler agglomerate and to the resins variable wetting of the filler. The desiccant found in the purchased EN-4 reduced the electrical standoff voltage by 50 percent and a similar phenomena was reported with the Laminar X500 (7-C-23).

Physical Properties

These are the usual parameters used to determine if materials are suitable for a given mechanical application. While a specific property may not be utilized, data is generated to determine the normal character, possibility for quality control purposes. These properties were measured on rather massive samples of material, much more massive than the conformal coating they were designed to produce. Comparison of these data with actual product performance gives confidence that more meaningful choices of material can be made for future design.

The bulk of testing was done at the ambient conditions of the testing laboratory; however, data is being generated at the use temperature as great variations in dynamic properties are noted as the materials are used at temperatures other than ambient (Tables 16 and 17).

Data is being generated using a mechanical rheometer capable of recording changes in shear properties at various temperatures in easy succession. The data presented should be considered as being about a specific state of cure and at the time of test.

The three cures of Conathane EN-4 and Adiprene L-100/MOCA were established to give similar product; however, it is noted that particular properties are favored by a selection of cure temperature. Compressive modulus and 200 percent tensile modulus are two examples. The differences of values at temperature other than ambient for compressive modulus and bulk modulus show the dependence on cure temperature these values have.

Table 16. Physical Properties Data for Three Materials

Properties	Conathane EN-4 Cure			Adiprene L-100/MOCA Cure			Conathane EN-7 Cure
	24 Hours at 160°F (71°C)	16 Hours at 175°F (80°C)	10 Hours at 200°F (93°C)	7 Days at 77°F (25°C)	4 Hours at 160°F (71°C)	3 Hours at 200°F (93°C)	16 Hours at 175°F (80°C)
Tensile Strength, Ultimate							
psi at -65°F							6390
MPa							44.1
psi at 77°F	2000	1830	1580	3250	4680	4260	2070
MPa	13.8	12.6	10.9	22.4	32.3	31.9	14.3
Tensile Modulus at 200 Percent							
psi at 77°F	1320	1200	1200	1190	1470	1520	1250
MPa	9.1	8.3	8.3	8.2	10.1	10.5	8.6
Elongation at Break							
Percent at 77°F	320	330	280	600	430	420	380

Table 16 Continued. Physical Properties Data for Three Materials

Properties	Conathane EN-4 Cure			Adiprene L-100/MOCA Cure			Conathane EN-7 Cure
	24 Hours at 160°F (71°C)	16 Hours at 175°F (80°C)	10 Hours at 200°F (93°C)	7 Days at 77°F (25°C)	4 Hours at 160°F (71°C)	3 Hours at 200°F (93°C)	16 Hours at 175°F (80°C)
Tear Strength							
p/li at -65°F MPa							663 4.57
p/li at 77°F MPa	280 1.93	214 1.48	253 1.74	370 2.55	344 2.37	3.11 2.14	250 1.72
p/li at 165°F MPa							
Hardness, Shore A							
-65°F							98
77°F	89	89	89	92	92	90	91
165°F							66
Coefficient of Thermal Expansion (microunit/unit/°F)							
-65°F to RT	97						
RT to 165°F							

Table 16 Continued. Physical Properties Data for Three Materials

Properties	Conathane EN-4 Cure			Adiprene L-100/MOCA Cure			Conathane EN-7 Cure
	24 Hours at 160°F (71°C)	16 Hours at 175°F (80°C)	10 Hours at 200°F (93°C)	7 Days at 77°F (25°C)	4 Hours at 160°F (71°C)	3 Hours at 200°F (93°C)	16 Hours at 175°F (80°C)
Specific Gravity							
77°F	1.0	1.0	1.0	1.1	1.1	1.1	1.0
Compressive Modulus							
psi at -65°F				9590		6020	28900
MPa							
psi at 77°F	5600	4950	4900	9500	9000	7530	3030
MPa	38.6	34.1	33.8	65.5	62.0	51.9	20.9
psi at 165°F				3950		9400	1300
MPa				27.2		64.8	8.96
Bulk Modulus (psi x 10 ³)							
psi at -65°F				706	706	540	586
GPa				4.87	4.87	3.72	4.04
psi at 77°F	375	375	375	408	409	403	355
GPa	2.59	2.59	2.59	2.81	2.82	2.78	2.45
psi at 165°F				3.83	3.83		2.84
GPa				2.64	2.64		1.96
Poisson's Ratio	0.44-0.48	0.48	0.47	0.45	0.47	0.47	0.49

Table 17. Physical Properties Data for Five Materials

Properties	Polysulfides			Silicone	Epoxy Urethane
	CS3100	Pro Seal 727	PRC 1201Q	DC 3140	Laminar X500 7-C-23
Tensile Strength					
Ultimate (psi)*					
-65°F (-54°C)	6200	5000	6000		
77°F (25°C)	250	420	267	146	1740
165°F (73°C)	165	260	182		
Tensile Modulus at					
200 Percent (psi)					
77°F				61-70	
Elongation at Break					
in Percent					
77°F				240	100
Tear Strength					
(p/li)**					
-65°F	175	80	135		
77°F	32	60	43	18	174
165°F	20	60	23		
Hardness (Shore A)					
-65°F	77	88	83		
77°F	50	69	53	20	
165°F	45	65	50		

Table 17 Continued. Physical Properties for Five Materials

Properties	Polysulfides			Silicone	Epoxy Urethane
	CS3100	Pro Seal 727	PRC 1201Q	DC 3140	Laminar X500 (7-C-23)
Coefficient of Thermal Exp. (in./in. x 10 ⁶ /°F)					
-65°F to RT				164	
RT to 165°F				164	
Specific Gravity					
77°F	1.73	2.04	1.86		1.22
Compressive Modulus (psi) at 300 psi					
-65°F	6300	5630	5200		
77°F	2200	1910	1900	375***	
165°F	3150	1960	2262		
Bulk Modulus (psi x 10 ³)					
-65°F	700-800	909		360	600
77°F	500	550	525	244	580
165°F	500	500		190	470
Poisson's Ratio	0.45	0.48			

*Pa = psi x 6895.

**p/li = 175.13 N/m.

***At 200 psi (1.38 MPa).

The three polysulfides reported to be manufactured to the requirement of the same specification show considerable difference in tear strength, hardness, specific gravity, and compressive modulus. It should be noted that polysulfides are manufactured as sealing materials and normally are not considered materials engineered for mechanical properties.

Design Manual

In conjunction with this material work a design concept was developed which can be used by engineers as an aid in material selection when they are faced with problems involving composite materials for multiple functions. Composite groupings of materials were subjected to strain analysis as they were subjected to temperature gradients, and structural stresses. Data gathered in these analyses along with data gathered on each of the materials involved has allowed for understanding some causes of failure and to correct marginal design concepts as well as adding a dynamic basis for the selection of material. This design manual, published separately (BDX-613-1216), is considered necessary to complete this endeavor. The design manual is intended as a ready reference for the working engineer and as a place to collect the philosophy, techniques, and material properties necessary to make good engineering judgements. Both generalized models and specific solutions to some particular problems are included. The organization is flexible so that revision and addition will be easy as new information becomes available through advances in coating and potting technology.

The first edition of the design manual is divided into three parts. The first part covers design philosophy. Concepts and models are defined and the philosophy of materials and component characterization are discussed. The second part covers techniques, models, and applications. Proven techniques are collected, models are used, and methods are applied to the solution of general and specific problems. The third section covers material properties selection. This includes comparisons of material for mechanical and thermal compatibility and a compilation of material property data. Copies of the current edition can be obtained from Bendix Kansas City technical publications department. BDX-613-1216, *Design Manual for Potting Electrical Assemblies*, November, 1975. Useful information for future editions is welcome from all sources.

ACCOMPLISHMENTS

A properties chart for selected conformal coating materials was developed. Specimens were prepared and tested by essentially identical test methods.

Several materials exhibited excellent dielectric breakdown properties such as the epoxy (11,000 to 15,000 volts/mil); however, they remain basically unsuited for critical high voltage conformal coating application because of thinning over edges and around small diameters. The conformal coatings using standard application techniques of spraying, brush coating, or dipping do not remain in significant thickness on sharp edges and small diameters to stand off high voltages in near vacuum environments.

The conformal coatings tested are not satisfactory as stress relief coatings due to high bulk modulus exhibited in compression. Where shear stress only is involved, the silicone and polysulfide coatings would be satisfactory.

The conformal coating concept to provide a high dielectric strength assembly failed to meet design requirements. Corrective action incorporated regrouping high voltage elements for encapsulation, thereby eliminating the need for a conformal coating.

The commercial materials tested are not suitable for critical electrical application. An improvement in dielectric strength was accomplished by removing dirt and large solid particles from the packaged resins.

The material having the highest electrical breakdown, epoxy, (11,000 to 15,000 volts/mil) also showed poor adhesion between second applications of material due to the nonpolar surface generated during cure.

A design manual was published covering technique, mathematical models, and a dynamic basis for the selection of materials for stress relief applications. The manual was prepared for use by material and electrical design engineers to aid in design of packaging as well as in material selection for reducing stresses due to encapsulation.

FUTURE WORK

The design manual will require revision as this technology develops; however, no specific work is planned at this time.

Liquid dip coatings are not capable of covering edges, small diameter wire, or burrs. Vapor phase coatings of Parylene could solve the defects encountered on this effort; however, problems of adhesion as well as any other drawbacks have not been established at this time. The production problems pointed out in this paper have been solved by other means such as regrouping components for localized potting of high voltage areas; therefore, no additional work is planned at this time.

Conformal coatings for mechanical stress relief has been approached successfully by the development of syntactic polysulfide coatings on PDO 6984874. This type coating has a bulk compressive modulus from 6,000 to 10,000 psi compared to the 190,000 to 900,000 for materials investigated in this project. The syntactic coating is based on a polysulfide which has a significant modulus increase in the -60°F (-51°C) area as well as poor electrical characteristics. A new project is planned which will find a new matrix material for the syntactic formulation.

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