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B. C. Cole to NTIS

BDX-613-1205 (Rev.)

EVALUATION OF SYNTACTIC
POLYSULFIDE STRESS
RELIEF COATINGS

PDO 6984874, Topical Report

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Published August 1976

MASTER

Prepared for the United States Energy
Research and Development Administration
Under Contract Number E(29-1)-613 USERDA

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Printed in the United States of America

Available From the National Technical Information Service, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, Virginia 22161.

Price: Microfiche \$2.25
Paper Copy \$4.00

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EVALUATION OF SYNTACTIC POLYSULFIDE STRESS RELIEF COATINGS

BDX-613-1205 (Rev.), UNCLASSIFIED Topical Report, Published
August 1976

Prepared by T. E. Neet, D/814, under PDO 6984874

The effect of phenolic microballoons on the pot life of syntactic polysulfide was evaluated. Material formulations and processing parameters were studied to develop procedures for applying a stress relief coating on electrical components to adequately protect these assemblies from stresses applied by the glass microballoon-filled encapsulating material. The static stress-strain as well as the bulk modulus characteristics of the syntactic polysulfide were evaluated. Cure schedules for the polysulfide were investigated.

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A prime contractor for the United
States Energy Research and
Development Administration
Contract Number E(29-1)-613 USERDA

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SUMMARY

Investigations into the properties and characteristics of phenolic microballoon-filled polysulfide were continued.¹ The study centered around pot life, coating applications, physical properties, and the effect of cure. The pot life evaluations were conducted on materials which did not contain a toluene diluent. The other three studies were made of polysulfide containing 10 to 15 percent toluene.

The pot life experiments revealed that using undried phenolic microballoons (PMB) in the polysulfide caused an immediate and significant reduction in the blend's pot life. The dried microballoons, on the other hand, had a more long-term effect on reducing the material's pot life. It was impossible to quantify the effect the microballoons had on pot life, because the pot life of the virgin polysulfide was also reduced as the material aged.

Another portion of the project defined the material formulations and processing conditions necessary to provide syntactic polysulfide stress relief coatings which would adequately protect electronic components from stress concentrations created by glass microballoon-filled epoxy encapsulant. This work showed that two coats of syntactic polysulfide should be applied to protect these assemblies. The coats performed two functions. They insured an adequate build-up of syntactic polysulfide, and they drastically reduced the possibility of a continuous hole occurring through the polysulfide from the epoxy to the component. A red pigment in the second coating enabled the operator to ensure that the second coating completely covered the unit. While the first coat could either be molded or brushed on the unit, the second coat was applied by brush. Also, 10 to 15 percent toluene added to the syntactic polysulfide improved brushability and coverage and increased the material's pot life.

A study of the physical properties of the material revealed that compression deflection testing of a free standing button of the polysulfide would not distinguish between syntactic and solid polysulfide. Both of these products had a compressive strength of 250 psi (1.72 MPa) at 15 percent strain. The present "hockey puck" bulk modulus test, used to simulate the actual application, does distinguish between solid and syntactic polysulfide. However, this test is expensive, it is also difficult to run, making it subject to some error. Another bulk modulus test, conducted by restraining the stress-strain test buttons in a hollow cylinder, provided equivalent test results to the hockey puck method; it was also easier and cheaper to run. However, this less expensive bulk modulus test does not show if the polysulfide is being dissolved by hot epoxy.

It is assumed that the "hockey puck" method will indicate hot epoxy attack, because this test simulates the actual material interface condition in production applications.

The project showed that 2 hours at 160°F (71°C) were needed to adequately cure the polysulfide. These experiments also indicated that the moisture content of the air surrounding the sample had a negligible effect on the polysulfide cure rate. It is possible that an excess of moisture may actually inhibit the cure.

DISCUSSION

SCOPE AND PURPOSE

Earlier work sponsored by this process development order prompted the selection of this material from several possible candidates. An evaluation of a phenolic microballoon-filled polysulfide material used as a stress relief coating was continued. The study was used to examine material formulations and coating processes required to adequately protect the electrical assembly from stress concentrations caused by the glass-microballoon-filled encapsulant.

PRIOR WORK

Initially, several material types and formulations were considered for this stress relief application.¹ This original development effort led to the selection of a phenolic microballoon-filled polysulfide as a stress relief coating.

ACTIVITY

Work on the syntactic polysulfide during this activity centered in four areas: pot life studies, coating applications, physical properties, and effect of cure. Two material formulations were used. The first was a blend of 100 parts by weight of polysulfide and 3 parts of phenolic microballoons. The reaction was initiated by adding 10 parts of the curing agent. This formulation was used for the pot life studies. In the second formulation, a similar material was used except 15 parts by weight of toluene were added to the phenolic microballoon/polysulfide blend. This was done as a processing aid to assist in the application of the coating. The coating applications, physical properties, and effect of cure investigations were conducted using the second formulation.

Pot Life Studies

One of the original concerns of using the syntactic polysulfide stress relief coating was the effect of the phenolic microballoons on the shelf life and pot life of the material. This study attempted to ascertain the magnitude of this potential problem.

One lot of PR 1201Q polysulfide was purchased for evaluation. The material was divided into thirds. The first portion remained as the original polysulfide, the second portion had three parts by weight of dried phenolic microballoons added, while the third portion received three parts by weight of undried microballoons.

Portions of each of these blends were stored in 1 pint (473 cm³) jars for periodic pot life studies.

The first of these pot life evaluations was performed on the same day that the microballoons were blended into the polysulfide. This was about 2 weeks after the PR 1201Q was originally produced by the manufacturer, Products Research and Chemical Corporation. The proper amount of curing agent was blended into the pint sample. A viscosity versus time profile (Figure 1) for each material was then determined using a Bookfield viscometer.

As shown by this graph, the material containing the dried microballoons, while starting at a higher viscosity, had a viscosity versus time profile (pot life) similar to the original PR 1201Q profile. The undried microballoons had an immediate effect in reducing the material's pot life.

Figures 2 and 3 are comparisons of the pot lives of the samples as they aged. Figure 2 illustrates the viscosity versus time profiles after 9 days of shelf aging. Figure 3 illustrates the same type data after almost 3 months of storage. From these graphs, it appears that the dried microballoons also influence the pot life of the material as the blend ages. Even the pot life of the original polysulfide decreases during storage.

A better comparison of the effect of aging on the pot life of the polysulfide blends can be found in Figures 4, 5, and 6. For example, in Figure 4, representing the original polysulfide, the pot life on the PR 1201Q decreased significantly in the first 9 days. Thereafter, the pot life gradually decreased until it became nearly stable after 6 to 8 weeks. To confirm this, the pot life of a 10-month-old lot of PR 1201Q was checked. This viscosity versus time curve, shown as the dashed line in Figure 4, conformed reasonably well to last pot life curve obtained using the original study material. The material manufacturer admits to this type aging characteristic:

It is a known fact that aging decreases application life and increases viscosity. (Of course, these two properties are tied together since the end of the application life is based on a viscosity limit.) However, we are a little surprised at your results which indicate approximately a 50 percent change in three months when we expect more like a 30 percent change in four months.³

The lessening of the pot life can also be seen for the PR 1201Q with 3 percent dried microballoons represented by Figure 5. Some of this change is undoubtedly caused by changes in the base polysulfide. However, there appears to have been more of a change in the viscosity versus time profile than can be explained by the base polysulfide alone. Thus, while it does seem that dried

Figure 1. Comparison of Pot Life for PR 1201Q Materials (Fresh Material, No Aging)

Figure 2. Comparison of Pot Life for PR 1201Q Materials (9 Days of Aging)

Figure 3. Comparison of Pot Life for PR 1201Q Materials (3 Months of Aging)

Figure 4. Comparison of Pot Life for PR 1201Q Polysulfide (No Microballoons)

Figure 5. Comparison of Pot Life for PR 1201Q Materials Containing 3 Percent Dried PMB

Figure 6. Comparison of Pot Life for PR 1201Q Materials Containing 3 Percent Undried PMB

phenolic microballoons have reduced the material's pot life, it is impossible to quantify the contribution of the microballoons to this reduction.

Figure 6 shows the effect of the undried phenolic microballoons on the viscosity versus time profile of the syntactic polysulfide. As in Figure 1, the immediate pot life reduction caused by these undried balloons is clearly evident.

Unfortunately, these studies were conducted before the decision was made to use toluene in the syntactic polysulfide formulation. The diluent effect of toluene would provide for a much longer, workable material pot life. Experiments are now being conducted to ascertain the viscosity versus time profile for these material formulations, except ten parts by weight of toluene will be added to each of the samples.

Coating Applications

The syntactic polysulfide was developed as a stress relief coating for electronic components. This was necessitated by a concern that the glass microballoon-filled epoxy encapsulant for the assembly would stress and crack some of these components. Work conducted previously at Bendix indicated that polysulfide filled with about 3 percent phenolic microballoons should give a material with a sufficient bulk modulus to isolate the components from this stress. However, to provide the required protection, the syntactic polysulfide had to completely surround these components; that is, no penetration of epoxy through the polysulfide to the component could be allowed.

A number of electronic components were coated with syntactic polysulfide by various techniques to determine the optimum procedure for processing components. After the polysulfide coating was cured, the components were encapsulated in glass microballoon-filled epoxy resin. When the epoxy was cured, the castings were cut and ground to reveal the axial cross section of the components. These cross sections were examined under a low power microscope for air voids (bubbles in the coating larger than the phenolic microballoons), epoxy beads (epoxy/GMB which had penetrated and hardened in voids in the coating), and minimum and maximum coating thicknesses.

Three groups of components were involved, with emphasis on different approaches to the coating method. Group 1 contained SA1997 capacitors with molded coatings. These devices were approximately cubical with sharp corners. Uniformity in coverage was sought, particularly at the corners (Figure 7). Group 2 contained resistors of five different sizes. These items were brush-coated with syntactic polysulfide diluted with 5, 10, and 15 percent toluene. The object was to determine the most suitable dilution from the standpoints of both application and

1

2

3

1

2

3

C

1

2

3

4

5

Figure 7. Group 1-Cross Sections of A, B, and C Series Components

coverage (Figure 8). Group 3 contained resistors and diodes of large to small size. Methods of adequately covering the ends of the components were tried, as well as determining the best way to suspend the components after coating (Figure 9).

All polysulfide mixes were deaerated prior to application. In Group 1, polysulfide discs, 0.015-inch-thick (0.381 mm) and 1/4 inch (6 mm) in diameter, were bonded to the faces of components A-1 and B-3 to ensure centering of the components in the mold. A layer of syntactic polysulfide was then applied by molding. After partial curing, additional polysulfide discs were attached as before and the part was remolded in a larger mold.

Samples A-2 and A-3 were treated like sample A-1 through the first molding operation. Syntactic polysulfide was then brushed on the corners of the part. After air drying for 1 hour, the entire surface of the part was brush-coated. The coating was allowed to air dry for 1 hour prior to curing for 6 hours at 160°F (71°C) and 44 percent relative humidity.

Pre-molded polysulfide boots were slipped over the ends of samples B-1 and B-2 and bonded in place with liquid polysulfide. The entire surface was then brush-coated, air dried for 1 hour, and cured at 160°F (71°C) and 44 percent relative humidity for 6 hours.

Except for the 5, 10, and 15 percent dilution of the polysulfide, all components in Group 2 were handled the same way. The first coat was brushed on and cured at 160°F (71°) for 1 hour; the second coat was then brushed on and cured at 160°F and 44 percent relative humidity for 6 hours.

For Group 3 samples 1L and 1S, cured polysulfide discs 0.015-inch-thick (0.381 mm) were slipped over the lead wires and butted against the end of the components. One brush coat of syntactic polysulfide was applied and cured with the component in the vertical position.

Samples 2L and 2S were processed like 1L and 1S, except the component was cured in the horizontal position. When samples 3L and 3S were processed, a toroidal syntactic polysulfide shape was painted on the component ends, allowed to air dry, and then the entire component was brush-coated. The component was held in a vertical position during cure. Samples 4L and 4S were processed like 3, except the components were cured in the horizontal position. Samples 5L and 5S received no special end preparation. The components received 2 brush coats; cure was in a vertical position, and the part was turned end for end for the second coat. Sample 7 was processed like 1. Sample 8 was processed like 1, except the discs were 0.032-inch-thick (0.813 mm) instead of 0.015 inch (0.381 mm). Samples 10L and 10S were processed like

Figure 8. Group 2-Cross Sections of 5, 10, and 15 Series Components

Figure 9. Group 3-Cross Sections of L and S Series Components

1 except two brush coats of polysulfide were applied. Samples 11L and 11S were processed like 2L and 2S, except two brush coats of polysulfide were applied. Sample 12 was processed like 3 except a second brush coat of polysulfide was applied. Sample 13 was processed like 1 except a second brush coat of polysulfide was applied. Sample 14 was processed like 1 except that discs were 0.032-inch-thick (0.813 mm) and two coats of polysulfide were applied. Samples 15L and 15S were processed like 5. The polysulfide used for brush coating all samples in Group 3 was diluted with 15 percent toluene.

The results of this study are listed in Table 1. Most of the voids listed for the samples in the table were air voids. That is, air had been entrained in the coating material in some manner. The epoxy encapsulant did not penetrate the coating to reach these voids except in four cases. Sample A-1 had three epoxy beads ranging in size from 5 to 30 mils (0.127 to 0.763 mm) in diameter in voids in the polysulfide coating. Sample A-3 had one epoxy bead 10 mils (0.254 mm) in diameter; Sample B-3 had two beads 20 mil, (0.503 mm) in diameter and Sample 10-1 had one bead 15 mils (0.381 mm) in diameter.

The tabulated data and observations made at the time of application lead to the following conclusions. For brush application, 10 percent toluene dilution provides the best combination of brushability, coverage, and pot life. End caps made from discs of 0.015-inch (0.381 mm) polysulfide sheet used on axially symmetrical components like resistors are effective. Two brush coats of polysulfide, diluted 10 percent with toluene, provide adequate thickness if properly applied. Molded polysulfide boots provide a uniform covering for components such as the SA 1997 capacitor. Symmetrical components such as resistors should be held in the vertical position for drying and curing. They should be turned end for end for application of the second coat.

The importance of operator technique cannot be over-emphasized. To aid in the application of the second coat of polysulfide, a new, red, syntactic polysulfide was developed by adding 2 percent of red iron oxide to the material. This provided a color contrast between coats. The second coat of syntactic polysulfide ensured that there would be no direct path by which the epoxy could penetrate the polysulfide to reach the component.

Physical Properties

Most of the effort on the physical property evaluations of the syntactic polysulfide was conducted during the early portion of the project. It was during this phase of the activity that the two material properties of static stress-strain and bulk modulus were determined. This work was done using syntactic polysulfide which was not diluted with toluene.

Table 1. Quantitative Evaluation of Syntactic Polysulfide Coatings

Sample	Voids	Void Size (Mils) (mm)	Coating Thickness (Mils) (mm)	
			Minimum	Maximum
A-1	13	10 to 30 (0.264 to 0.762)	30 (0.762)	65 (1.651)
A-2	0		20 (0.508)	55 (1.397)
A-3	13	10 (0.254)	30 (0.762)	65 (1.651)
B-1	9	5 to 50 (0.127 to 1.270)	20 (0.508)	60 (1.524)
B-2	0		20 (0.508)	55 (1.397)
B-3	2	20 (0.508)	15 (0.381)	60 (1.524)
C-1	8	10 to 30 (0.254 to 0.762)	20 (0.508)	70 (1.778)
C-2	5	10 to 25 (0.254 to 0.635)	30 (0.262)	70 (1.778)
C-3	4	5 to 15 (0.127 to 0.381)	25 (0.635)	65 (1.651)
C-4	5	15 to 30 (0.381 to 0.762)	25 (0.635)	75 (1.905)
C-5	14	25 to 30 (0.655 to 0.762)	10 (0.254)	75 (1.905)
5-1	0		15 (0.381)	40 (1.016)
5-6	0		10 (0.254)	25 (0.635)
5-3	1	15 (0.381)	10 (0.254)	20 (0.508)
5-4	0		15 (0.381)	30 (0.762)
5-5	0		15 (0.381)	20 (0.508)
10-1	1	15 (0.381)	15 (0.381)	40 (1.016)
10-2	0		10 (0.254)	40 (1.016)
10-3	0		10 (0.254)	30 (0.762)
10-4	0		5 (0.127)	30 (0.762)
10-5	0		5 (0.127)	40 (1.016)
15-1	1	15 (0.381)	5 (0.127)	30 (0.762)
15-2	0		5 (0.127)	25 (0.635)
15-3	0		5 (0.127)	20 (0.508)
15-4	0		0 (0.000)	30 (0.762)
15-5	0		5 (0.127)	30 (0.762)
1L	0		5 (0.127)	45 (1.143)
1S	0		10 (0.254)	35 (0.889)
2L	0		20 (0.508)	35 (0.889)
2S	0		20 (0.508)	35 (0.889)
3L	0		10 (0.254)	
3S	0		15 (0.381)	25 (0.635)
4L	0		10 (0.254)	80 (2.032)
4S	0		15 (0.381)	50 (1.270)
5L	0		10 (0.254)	50 (1.270)
5S	1	5 (0.127)	10 (0.254)	60 (1.524)
7L	0		10 (0.254)	90 (2.286)
8L	0		15 (0.381)	30 (0.762)
10S	0		20 (0.508)	45 (1.143)
10L	0		25 (0.635)	45 (1.143)
11L	0		30 (0.762)	70 (1.778)
11S	0		10 (0.254)	65 (1.651)
12	0		20 (0.508)	50 (1.270)
13	0		10 (0.254)	80 (2.032)
14	2	10 (0.254)	35 (0.880)	40 (1.651)
15S-1	0		10 (0.254)	60 (1.524)
15S-2	1	5 (0.127)	20 (0.508)	45 (1.143)
15S-3	0		10 (0.254)	50 (1.270)
15L-1	0		20 (0.508)	40 (1.016)
15L-2	0		20 (0.508)	50 (1.270)
15L-3	0		20 (0.508)	40 (1.016)

The test samples for the static stress-strain evaluations were made by molding the 3 percent phenolic microballoon-filled polysulfide between two plates spaced 0.150 inch (3.81 mm) apart. The material was cured for 8 hours at 160°F (71°C). Then 1.129-inch-diameter (29.68 mm) coupons were cut from the sheet of material. The height of each sample was measured. They were then compressed and the load required to deflect the samples 15 percent was recorded. The exact sample preparation and testing procedure is given in Appendix A. From this work, the specification limits for a compressive stress of 250 ± 50 psi (1.72 ± 0.34 MPa) at 15 percent strain was developed.

The "hockey puck" test for the material bulk modulus was more complicated. First, a 0.030 ± 0.010 -inch-thick (0.762 ± 0.264 mm) coating of syntactic polysulfide was molded onto a glass fiber-filled diallyl phthalate disc. Following a 6-hour, 160°F cure, the precise coating thickness was determined. Then a layer of glass microballoon-filled epoxy was cast on top of the polysulfide. After another 24 hour, 150°F (66°C) cure cycle, a 2.75 ± 0.20 inch-diameter by (69.9 ± 5 mm) 7/8-inch-high (22 mm) disc was machined from the resulting block. The ends of the disc were required to be flat and parallel to within 0.0005 inch (0.0127 mm). The epoxy layer had to be 0.700 ± 0.050 inch (17.80 ± 1.27 mm) thick. No more than 50 pounds (220 N) total force could be applied to the sample at any time during machining. A blank was made in the same manner, except that the polysulfide coating was omitted.

The test was conducted by determining the slope of the tangent to the stress-strain curve at 250 psi (1.72 MPa) stress for the polysulfide-containing sample. Subtracted from this was the slope of the tangent for the test blank at the same stress. This removed the effect of the glass microballoon-filled epoxy and the diallyl phthalate. The bulk modulus of the syntactic polysulfide was then determined from the resultant tangent and the known polysulfide thickness. The sample preparation and testing procedure is shown in Appendix B. From this, a bulk modulus specification limits of 4500 ± 1500 psi (31.03 ± 10.34 MPa) at 250 psi (1.72 MPa) stress was established.

All of the above experiments were conducted on syntactic polysulfide material which did not contain the additional toluene. When the coating applications work was done, a toluene dilution was found to be advantageous to aid in processing the assembly. Therefore, these defined physical properties had to be evaluated in light of this addition. The test samples were made in the same manner, except that 15 parts by weight of toluene were added to each 100 parts of base polysulfide. After the curing agent was adequately blended, the mixture was evacuated until foam collapse occurred to remove entrained air. This evacuation period did not exceed 1 minute.

The static stress-strain samples were tested in the same manner as before, except that the specimen height was determined from the point where a load was first indicated on the stress-strain curve. Because the thin buttons tended to warp, the height measurement could be in error toward the high side, thereby creating an erroneous 15-percent deflection reading. The compression testing instrument, on the other hand, compressed this warp out of the sample before any load could be noticed on the stress-strain chart. The previously established load of 250 $\pm$ 50 psi (1.72 $\pm$ 0.34 MPa) at 15 percent strain was found to be a correct requirement for the toluene containing material. However, to facilitate acceptance inspection testing, the limits for individual samples had to be widened to 250 $\pm$ 100 psi (1.72 $\pm$ 0.69 MPa). Even though the average compressive strength of the syntactic polysulfide fell within the narrower limits, it was not unusual for an individual sample to range between 150 to 350 psi (1.03 to 2.41 MPa).

The value of a test of this type (static stress-strain characteristics) is questionable. When a sample of solid polysulfide containing no microballoons and evacuated to remove air voids was tested by the specified procedure, it also met the requirements of the specification. It appears that the compression-deflection test cannot distinguish between syntactic and solid polysulfide.

The only changes in the manufacture of the bulk modulus test samples were in the addition of toluene and in the evacuation procedure. Using this method, the average bulk modulus was found to be in the region of 9000 psi (62 MPa). A possible explanation for this lies in the removal of air voids from the polysulfide. With these voids removed from the sample, the material would no longer have that empty space in which to be compressed, which would cause the bulk modulus to increase. The bulk modulus requirement was changed from 4500 $\pm$ 1500 psi (31.03 $\pm$ 10.34 MPa) to 9000 $\pm$ 6000 psi (62.05 $\pm$ 41.37 MPa) to be representative of the strengths obtained with the new process.

The increase in the tolerance limits from 1500 to 6000 psi (10.34 to 41.37 MPa) was necessary because of the inaccuracy of the bulk modulus test procedure. In general, to reach the required 250 psi (1.72 MPa) stress, the blank must be deflected 0.0007 inch (0.018 mm) while the sample is deflected only 0.0011 inch (0.028 mm). To make the bulk modulus calculation, the deflection of the blank must be subtracted from the sample deflection, leaving, in this case, a polysulfide deflection of 0.0004 inch (0.010 mm). This does not consider any inaccuracies or tolerances in the test equipment itself. In some cases, a negative bulk modulus was obtained. This was undoubtedly created by a stack-up of tolerances in the wrong direction, because no material can have a negative bulk modulus.

An equivalent and significantly less expensive test for bulk modulus was evaluated. This test used samples of the same type as those cut for the static compression-deflection characteristics. In this new test, the sample was restrained in a hollow metal cylinder having an inside diameter of the same dimensions as the diameter of the test coupon. The sample was also restrained on the bottom and compressed from the top with a ram. Thus, this procedure compresses a completely restrained test specimen and should give a reasonable approximation of the true bulk modulus of the syntactic polysulfide. Samples tested in this manner yielded a bulk modulus of between 5814 and 7418 psi (40.09 and 51.15 MPa). However, this test method for bulk modulus will not show if the polysulfide was being dissolved by hot epoxy encapsulating material. It was believed the original bulk modulus test would provide this type of polysulfide dissolution information; therefore, the bulk modulus test procedure was not changed.

The original bulk modulus test and the proposed, less expensive bulk modulus test both discriminate between syntactic and solid polysulfide. As stated, these two tests placed the bulk modulus of the syntactic polysulfide generally between 5000 and 13000 psi (34 and 90 MPa). Values for solid polysulfide were approaching infinity by these tests. The bulk modulus of the solid material was estimated to be in the range of 500,000 psi (3500 MPa).

Effect of Cure

Past experience had suggested that both heat and moisture were required to cure polysulfides. Therefore the cure cycle for the syntactic polysulfide material was placed at a minimum of 6 hours at 160°F (71°C) in an arbitrary minimum of 35 percent relative humidity. Cure times and temperatures significantly below these limits supposedly rendered the polysulfide susceptible to hot epoxy attack.

A problem occurred during the standard quality inspection of material. The 0.150-inch-thick (3.81 mm) test slabs were failing the 250 ±50 psi (1.72 ±0.34 MPa) stress requirement at 15 percent strain. This problem was traced to the high humidity cure and the elapsed time between the end of this cure and sample testing. For example, one batch of test specimens of the syntactic polysulfide material recorded a compressive strength of 105 psi (0.724 MPa) 9 hours after completion of cure. Samples tested 18 hours after cure yielded a 160 psi (1.10 MPa) compressive strength. When these slabs received an additional 6-hour, 160°F (71°C) cure in a low humidity oven, they tested at 250 psi (1.72 MPa). A sample which did not receive the high humidity cure but which was cured for 6 hours in the low humidity oven also had a compressive strength in the region of 250 psi (1.72 MPa). Even a test slab cured at 160°F (71°C) in a 5 percent relative humidity room gave the required compressive strength.

From these results, it appeared that the strength of syntactic polysulfide was in some way being reduced by the high moisture content in the curing oven. This is a reversible process that can be corrected by allowing the slabs to air dry for several days or by drying them at an elevated temperature for several hours. Also, the results of these experiments seem to show that the polysulfide can be cured at 160°F (71°C) in a low humidity area and still maintain the required compressive properties.

Other work has shown that polysulfides are indeed subject to dissolution by hot epoxy.³ However, the dissolution is only noticeable on undercured polysulfide samples. No dissolution by hot epoxy is evident when the material is sufficiently cured at high temperatures, such as 2 hours at 160°F (71°C). This work was conducted in an area in which the humidity was not controlled. Even assuming a high room temperature of 90°F (32°C) and a room relative humidity of 100 percent, this translates to relative humidity of less than 5 percent at the cure temperature of 160°F (71°C).

Therefore, it seems that time and temperature are the necessary ingredients for curing polysulfides. While moisture may tend to increase or decrease the rate of cure, no evidence was found that would suggest anything more than trace amounts of moisture are required for adequate cures.

ACCOMPLISHMENTS

This work has shown that, as the polysulfide material ages, the pot life of the product decreases. This is true for both the unfilled and dried phenolic microballoon-filled polysulfide. While the data to date does not permit a quantification of the effect of the dried microballoons, it suggests these microballoons do reduce the material's pot life. This would be in addition to the pot life reduction created by the aging of the polysulfide itself.

The experiments conducted also defined the processing procedures required to allow the syntactic polysulfide stress relief coating to adequately protect electrical components from stress concentrations created by glass microballoon-filled epoxy encapsulant. One of the recommendations was the addition of 10 to 15 percent toluene to the syntactic polysulfide to aid in brushability (for brush-on coats of material), improve coverage, and increase material pot life. Also, the addition of a red pigment to the second coating material provided a convenient operator aid to ensure the second coating completely covered the part. This work also showed an advantage in using molded boots or discs to provide good syntactic polysulfide for some components.

Studies of the physical properties of the material, while showing that the compressive strength of the syntactic polysulfide at 15 percent strain was in the region of 250 psi (1.72 MPa), provided evidence that the specified static compressive stress-strain test did not distinguish between solid and syntactic polysulfide. By this test, even solid polysulfide coupons yielded a compressive strength of approximately 250 psi (1.72 MPa). This work also showed that the "hockey puck" bulk modulus test, while reasonably estimating the true bulk modulus of the material, is difficult to reproduce. Another simpler less expensive test, conducted by completely restraining the compressive stress-strain sample in a metal cylinder, provided equally valid bulk modulus results. However, it did not provide any indication of the capability of the polysulfide to withstand dissolution by hot epoxy. It is believed that the "hockey puck" bulk modulus test can give an indication of this attack.

Other work conducted during this period cast doubt on the need for moisture in the cure mechanism of polysulfides. This project supports the conclusion that adequate time and temperature is required to cure the material. From these experiments, 2 hours at 160°F (71°C) appear to provide a sufficient cure. However, these experiments also suggest that no more than a trace of moisture is required to complete the cure.

FUTURE WORK

Recently, a distinct color change was noted in the base polysulfide. This indicated a change in either the raw materials or processes used to produce the polysulfide. The manufacturer, Products Research and Chemical Corporation, admitted to "a necessary change in raw materials". They also stated this change had not affected the end product, except for color. Because of this raw material change and to answer some remaining questions about the shelf and pot life relationship and the physical properties of the polysulfide, experiments will be conducted to determine the effect of aging on the pot life of the toluene-diluted polysulfide and on the physical properties of the cured syntactic polysulfide.

REFERENCES

¹J. W. Steele, *Syntactic Polysulfide Conformal Coating*, UNCLASSIFIED. Bendix Kansas City: BDX-613-1222, Proposed, September, 1974.

²C. J. Camp, *Final Product Investigation of Polysulfide Conformal Coating*. UNCLASSIFIED. Bendix Kansas City: BDX-613-960, November, 1963..

³Letter, "Aging of PR-1201-Q." T. E. Noet, from Donald Corkhill. Research & Chemical Corporation, PRC Coatings and Sealants Division, December, 27, 1973.

Appendix A

PREPARATION OF TEST SLABS AND TEST PROCEDURE

PREPARATION OF TEST SLABS

Mixing Ratio

Material	Grams
Base Compound	100
Phenolic Microballoons	3
Curing Agent	10

Mixing Procedure

Thoroughly mix base compound and curing agent separately in their storage containers. Add 3 grams of phenolic microballoons and stir into base compound with spatula until the balloons are thoroughly wet. Weigh 10.0 ± 0.1 g of curing agent into the container. Mix thoroughly until uniform (at least 3 minutes). Within 30 minutes from start of mixing, cast or mold the mixture between two flat and parallel surfaces spaced approximately 0.150 inch (3.81 mm) apart and parallel within 0.001 inch (0.025 mm). Cure the material in the mold for at least 2 hours at a temperature of $150 \pm 10^{\circ}\text{F}$. ($71 \pm 6^{\circ}\text{C}$). Remove slab from mold and continue cure for at least 6 hours at $160 \pm 10^{\circ}\text{F}$ ($71 \pm 6^{\circ}\text{C}$).

TEST PROCEDURE

Cut three 1.129-inch (29.68 mm) standard discs from the test slab. Measure the thickness of the specimens. While recording the force-deflection curve, compress the specimen to approximately 30 percent strain at a crosshead velocity of 0.05 inch (1.27 mm) per minute. Determine the stress at 15 percent strain.

Appendix B

PREPARATION OF BULK MODULUS SAMPLES

TEST SAMPLE A, SYNTACTIC POLYSULFIDE COATED DISC

1. Measure the actual thickness, at four points 90 degrees apart, of a 4-inch-diameter (100 mm) by 1/8-inch-thick (3.2 mm) disc made of long glass fiber-filled diallyl phthalate. The measuring points shall be 3/4 inch (20 mm) in from the edge. Wipe the surface clean with acetone, methyl ethyl ketone, or toluene.
2. Prepare the coating material as specified in Appendix A for the test slabs. Within 30 minutes from time of mixing, apply 0.030 ± 0.010 inch (0.726 ± 0.254 mm) of coating material to one surface. A build-up of material at the edge of the disc is permissible.
3. Cure for 6 hours minimum at $160 \pm 10^{\circ}\text{F}$ ($71 \pm 6^{\circ}\text{C}$).
4. Measure the actual thickness of the disc plus cured coating at the same four points. Determine the average coating thickness.
5. Wrap the disc with tape or other suitable material to create a reservoir approximately 1-1/2 inches (40 mm) high above the coating.
6. Mix the glass microballoon-filled epoxy encapsulating compound and fill the reservoir to a depth of at least 3/4 inch (20 mm). Evacuate for at least 5 minutes at an absolute pressure of 3 torr (400 Pa) or less.
7. Cure for 24 hours at 150°F (66°C).
8. Machine sample to a 2.75 ± 0.20 inch (69.9 ± 5 mm) diameter by 7/8 inch (22 mm) high disc. The ends of the disc shall be flat and parallel to within 0.0005 inch (0.0127 mm). The epoxy layer shall be 0.700 ± 0.050 inch (17.80 ± 1.27 mm) thick. No greater than 50 pounds (220 N) total force shall be applied at any time during machining.

TEST SAMPLE B, BLANK

Prepare a test blank using an uncoated DAP disc, following Step 5 through 8 above.

MODULUS DETERMINATION

Determine the slope of the tangent to the stress-strain curve for Test Sample A at 250 psi (1.72 MPa) stress. Subtract from this the slope of the tangent to the stress-strain curve for Test Sample B at 250 psi stress. Compute the modulus at the resultant tangent.

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