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E-1386

REPORT ON CLEAR RESINS

by

Samuel C. Smith

August 1963

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MIT INSTRUMENTATION LABORATORY

CAMBRIDGE 39, MASSACHUSETTS

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E-1386
REPORT ON CLEAR RESINS

ABSTRACT

After the recommendation of Epocast 1786 and Catalyst 9115 for potting of G. M. E. Modules, certain irregularities were encountered.

Two major problems in Epocast 1786 however, were solved. The problems were first, a pulling away from the corners of its potted case and second, a softening at temperatures above 120^oF. The former was corrected by pre-heating the case to 100^oF; the latter, by using Catalyst 955.

One major problem of Chomerics 202 has been that of the resin not bonding to the case. This has been solved by using a solvent-based primer known as "Resin 219". However, the susceptibility of the resin to cracking at extreme temperature changes bears watching.

In a third resin, Stycast 1264, the same softening at elevated temperatures which occurred with Epocast 1786 was corrected by the addition of Catalyst 9, making the resin a three-part component.

Hysol R8-2038 with Hardener H2-3404, a fourth resin, stood up well under preliminary tests; however, further tests indicated that it does not merit consideration. Three clear resins are recommended for use in potting the G. M. E. Modules.

by Samuel C. Smith
July 1963

REPORT ON CLEAR RESINS

INTRODUCTION

Efforts have been made to correct the irregularities in the use of Epocast 1786 and Catalyst 9115 for the potting of Gimbal Module Electronics (G.M.E.) Modules by bringing them to the attention of the manufacturer and making successive tests incorporating the manufacturer's recommendations.

Specifically, the resin exhibited certain undesirable features such as pulling away from the corners of the case in which it is cured (see Fig. 1), shrinking abnormally, and softening upon exposure to temperatures above 120°F.

The former two faults were annoyingly unpredictable; at times they would occur, and at other times there would be no occurrence even when using the same batch.

Finally, upon advice of Furane Plastics, Inc., the faults were corrected to the extent that repeated tests showed no further pull-away from the corners of the case, and the heat distortion temperature is now higher. There seems to be some improvement on the shrinkage also.

No attempt was made to check the shrinkage per se; however, some idea is obtained by using the same type case for all the samples and comparing the amount of pull on the side of the cases. Since the amount of deflection on the case is 0.002" before potting, any deflection over 0.002" after potting can be attributed to stress from the resin.

At the same time that we were ironing out the problems of Epocast 1786, we were evaluating two other resins. These are Stycast 1264, Parts A and B by Emerson and Cuming, and Chomerics 202/56.

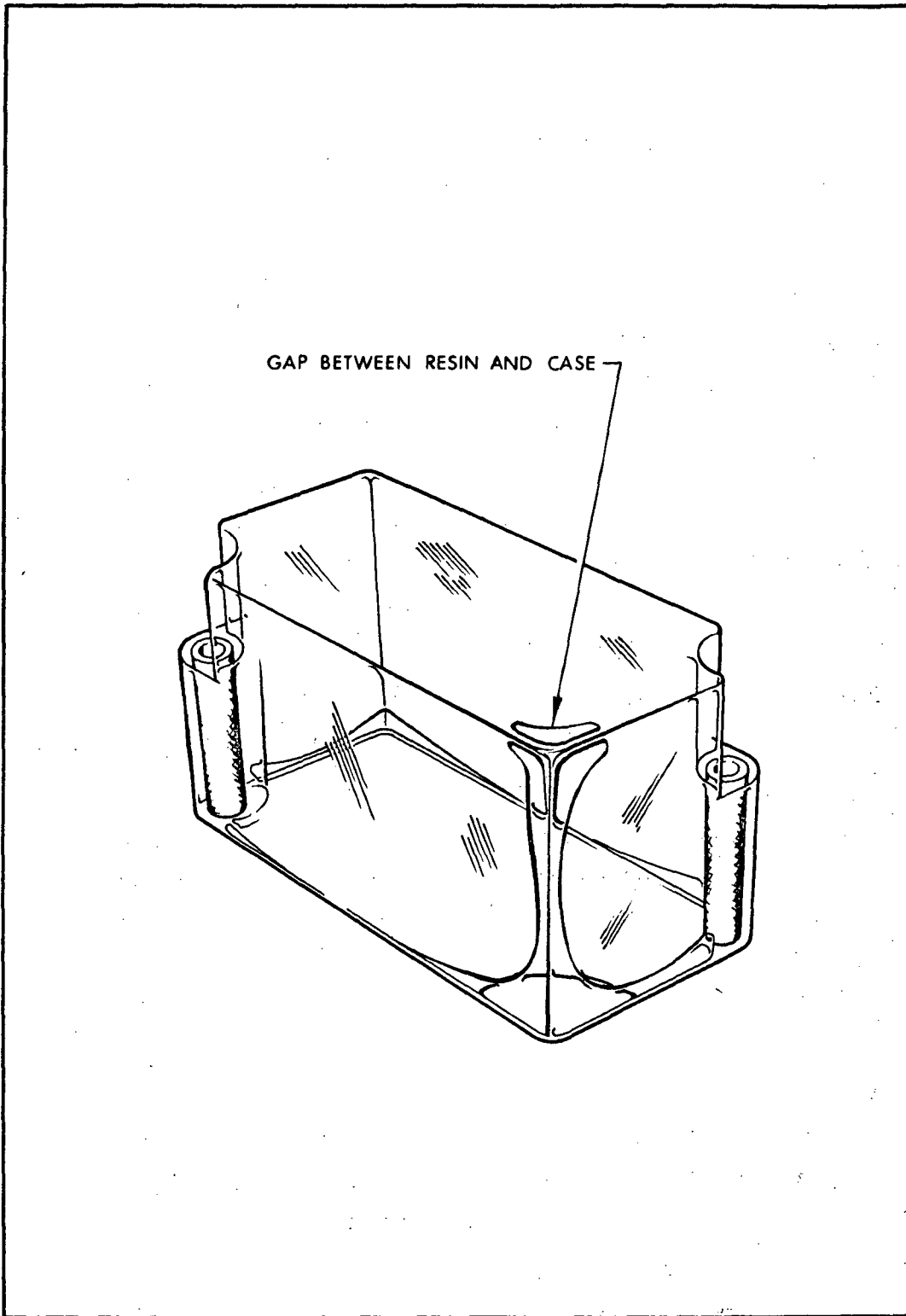


Fig. 1

STYCAST 1264 (A & B)

Tests conducted indicated that Stycast 1264 (A & B) is suitable in all respects except that, like Epocast 1786/9115, the resin would soften at temperatures above 120^oF.

The problem was finally corrected when Emerson & Cuming came up with the idea of adding its Catalyst #9 to the two-part resin, now making it a three-part resin. The resin is now light yellow in color rather than water clear as before, but this is not objectionable.

CHOMERICS 202/56

In addition to its good electrical and mechanical properties, Chomerics 202/56 remains hard when exposed to temperatures as high as 160^oF, and its clear color does not change under any circumstances.

Unfortunately, there was one bad feature. Repeated tests indicated that the resin is not compatible with the Lexan case and will not bond to it.

After some unsuccessful attempts by Chomerics, Inc. to remedy this situation, the Company finally solved the problem with a primer.

PROCEDURE

For the sake of brevity and expediency, no attempt will be made to deal with details here. In all cases, except one, the experiment consisted of filling 1 3/4" long x 7/8" wide x 15/16" high Lexan cases with Epocast 1786, Stycast 1264 or Chomerics 202.

Note that each case had a deflection of 0.002 inch in its empty state. This is reflected in the deflection column in Table 1. (See Fig. 2 for measuring deflection.)

1. EPOCAST 1786

A. EPOCAST 1786 AND CATALYST 9115

Two Lexan cases (Ser. No. F-1, F-2) were filled

TABLE I

| Ser. No. | Resin | Catalyst | Deflection | Deflection After Aging Test | Remarks |
|----------|------------------|--------------------|------------|-----------------------------|--|
| F-1 | Eurane 1786 | 9115 | 0.005 | 0.005 | Yellow after potting; almost water clear before potting. |
| F-2 | 1786 | 9115 | 0.004 | 0.004 | |
| F-20 | 1786 | 955 | 0.008 | 0.008 | High shrinkage. Pulled away at corners. |
| F-21 | 1786 | 955 | 0.007 | 0.007 | Same |
| F-22 | 1786 | 955 | 0.008 | 0.007 | Same |
| F-23 | 1786 | 955 | 0.008 | 0.008 | Same |
| F-24 | 1786 | 955 | 0.008 | 0.008 | Same |
| FH-30 | 1786 | 955 | 0.005 | 0.005 | Lower shrinkage. No pull away at corners. |
| FH-31 | 1786 | 955 | 0.005 | 0.005 | Same |
| FH-32 | 1786 | 955 | 0.003 | 0.003 | Same |
| FH-33 | 1786 | 955 | 0.004 | 0.004 | Same |
| FH-34 | 1786 | 9115 | 0.005 | 0.005 | |
| FH-35 | 1786 | 9115 | 0.006 | 0.006 | |
| E-40 | 1264 A | B + 9 | 0.002 | 0.002 | |
| E-41 | 1264 A | B + 9 | 0.003 | 0.003 | |
| E-42 | 1264 A | B + 9 | 0.001 | 0.001 | |
| E-50 | 1264 A | B + 9 | 0.002 | 0.002 | |
| E-51 | 1264 A | B | 0.005 | 0.007 | Without Cat. #9 |
| E-52 | 1264 A | B | 0.006 | 0.006 | Without Cat. #9 |
| E-53 | 1264 A | B + 9 | 0.004 | 0.005 | |
| E-54 | 1264 A | B + 9 | 0.004 | 0.004 | Deep yellow after potting. |
| C-20 S | Chomerics 202 | 56 (Primer 219) | 0.004 | 0.004 | |
| C-60 | " | 56 (Primer 219) | 0.006 | 0.006 | |
| C-61 | " | 56 (Primer 219) | 0.005 | 0.005 | Retains color. |
| C-62 | " | 56 (Primer 219) | 0.004 | 0.004 | |
| C-63 | " | 56 (Primer 219) | 0.005 | 0.005 | |
| C-64 | " | 56 (Primer 219) | 0.004 | 0.004 | |
| C-65 | " | 56 (Primer 219) | 0.005 | 0.005 | |
| S-1 | Stycast 1090 | #11 | 0.002 | 0.002 | |
| S-2 | 1090 | #11 | 0.001 | 0.001 | |
| S-3 | 1090 | #11 | 0.002 | 0.002 | |
| H-1 | Hysol R8-2038 | H2-3404 | 0.002 | 0.003 | |
| H-2 | " | " | 0.003 | 0.004 | |
| H-3 | " | " | 0.004 | 0.005 | |

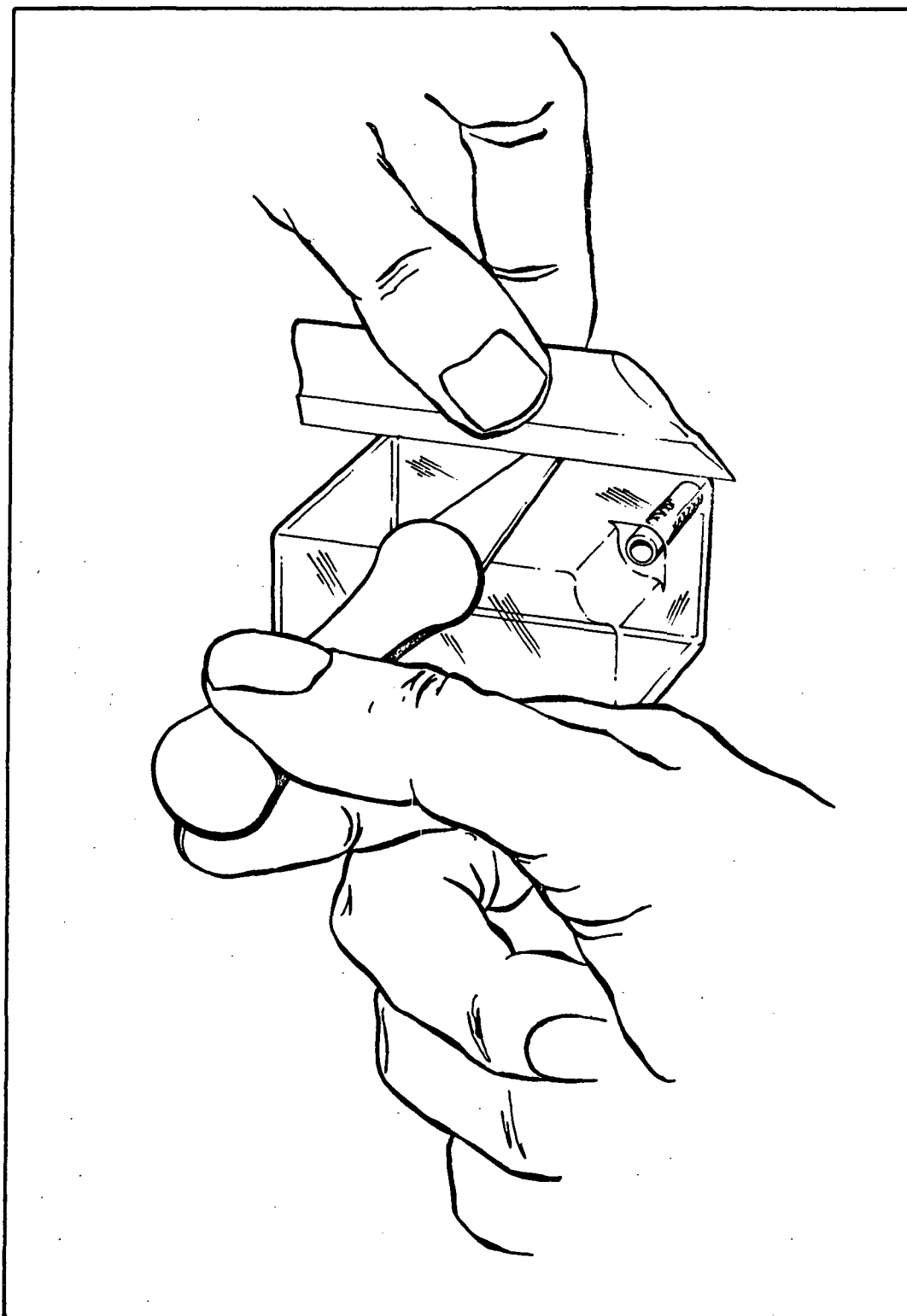


Fig. 2

with Epocast 1786/9115. After curing, it was observed that the resin had pulled away from the corners of the case.

B. EPOCAST 1786 AND CATALYST 955

Five Lexan cases (Ser. No. F-20 to F-24) were filled with Epocast 1786/955. After curing, these, too, had their corners pulled away from the case.

The experiment was repeated with six other Lexan cases (Ser. No. FH-30 to FH-35); only this time, the cases were pre-heated to 100°F. None of the cases exhibited "pull away" at the corners, shrinkage was comparatively low, and the service temperature improved.

We then slit sections 5/16" wide in one of the samples (Ser. No. F-32) and proceeded to separate the sections from the resin by pulling with a spring scale as shown in Fig. 3. An average force of 27 pounds, more than adequate, caused separation.

2. STYCAST 1264 (A&B)

Two Lexan cases (Ser. No. E-51, E-52) were filled with Stycast 1264(A&B). After curing, the samples were exposed to temperatures of 125°F. This caused them to soften.

The experiment was repeated with six other samples (Ser. No. E-40, E-41, E-42, E-50, E-53, E-54). This time E&C Catalyst 9 was added to the 1264 (A&B) in a definite ratio.

Two of the cured samples were later exposed to a temperature of 150°F, at which point they could barely be deformed with the corner of a 6-inch scale. The samples were allowed to cool and then were put back in the oven with the temperature lowered to 140°F. After one hour, they were again tested for hardness. It was found that at 140°F the resin remained hard.

As with the previous resin, 5/16" wide slits were cut and an average force of 8 pounds caused separation between the resin and the case. Although a larger separating force would have been desirable, this was acceptable.

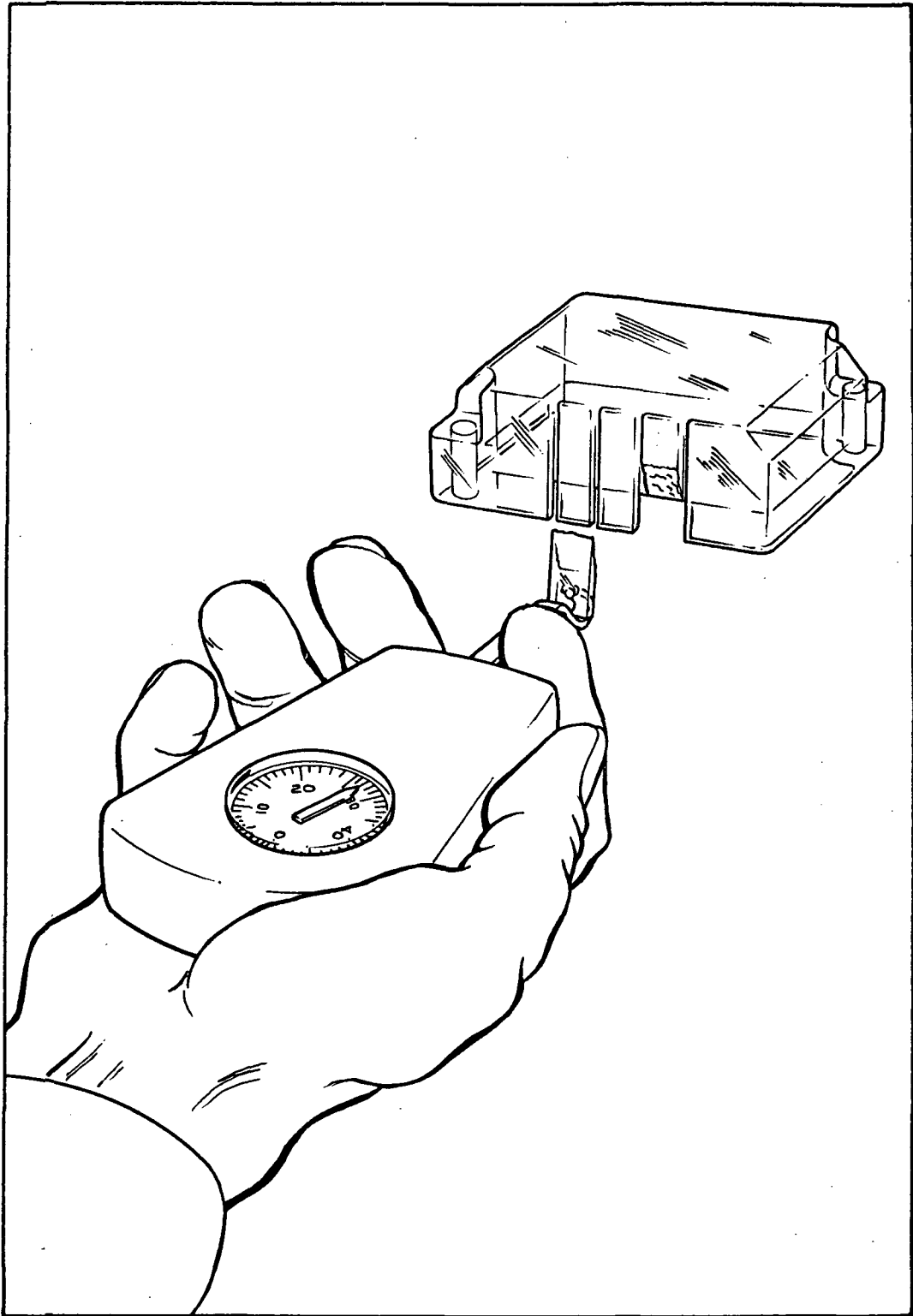


Fig. 3

3. CHOMERICS 202/16

Four samples were cured as in previous cases (Ser. No. C-10 to C-13). It was observed that the cured resin would not bond to the Lexan case. Subsequently, two other samples were first coated with the resin and then allowed to cure at 200^oF as per direction of Chomerics, Inc. These samples were then filled with the resin and allowed to cure at 160^oF. Still, there was no bond.

The experiment was repeated with six other samples, this time pre-coated with Chomerics' new primer, Resin 219. Good bonding resulted and an average force of 10 pounds was necessary to cause separation. Scratching the case before priming increased the force to 26 pounds. However, a force of 10 pounds is ample.

The resin was later exposed to a temperature of 160^oF with no material change in the hardness.

4. HYSOL R8-2038

At the time this report was being prepared, Mr. Donald Gilchrist of G. E. , who has made some inquiries into clear resins in connection with the Polaris Program, gave us a sample of a recommended clear resin, namely Hysol R8-2038. Preliminary tests indicated a favorable comparison with the other resins.

It was particularly observed that the high linear shrinkage indicated in Hysol's brochure was not apparent in the tests made for comparative shrinkage. This, perhaps, indicates the inconclusiveness of this test method.

Although it was noted at the end of an aging test that the deflection increased slightly, and although it is not uncommon with clear resins that an apparent low shrinkage may become high over a period of time, further tests indicated that R8-2038 has an initial high shrinkage and negligible aging shrinkage.

Table 1 shows the deflection of the various modules shortly after curing and 170 hours later. These modules were kept in the oven at 150°F for 170 hours to simulate possible slow shrinkage over 3 to 4 months at room temperature. All of the clear modules, except Chomerics 202, turned deep yellow in color. The color of Chomerics remains the same.

In lieu of a linear shrinkage test, which is somewhat too involved, we have proceeded to determine the comparative shrinkage by the percent volume loss of the various modules. This method is accurate to within 5 per cent.

The resin is air evacuated and poured into a 1.000 inch diameter by 1.271 inches deep (1.000 cubic inch) polished steel cylinder flush with the top.

After curing, the weight in grams of an amount of water necessary to refill the steel cylinder was noted. The water was then removed and the specimen placed in the oven at 150°F for 170 hours for aging test. At the end of this period, the cylinder was again filled with water and the weight noted. The water was emptied and the specimen placed briefly in an oven for drying.

An amount of the same type of resin necessary to fill the cylinder was added and the weight noted. This gave us the maximum volume loss over the aging test period.

The water was used to determine the volume loss, if any, between the time the resin was cured and the time the aging test ended. As shown in Table 2, slight increase in shrinkage over a period of time is expected. Contrary to our expectation, Hysol R8-2038, which exhibited a comparative high shrinkage, indicated negligible continuous shrinkage over a period of time. So does Stycast 1264 with Catalyst 9 added.

PROPERTIES

Table 2 compares the properties of Epocast 1786/955,

TABLE 2

| PROPERTY COMPARISON | | | | | |
|---|--------------------------------------|--|--|--|---|
| PROPERTIES | Chomerics 202/56 | E & C Stycast 1264 Cat. B+9 | Furane Epocast 1788/955 | Hysol R8-2038 | Stycast 1090 |
| Specific Gravity (cured) | 1.14 | 1.20 1.10 | 1.19 | 1.12 1.22 | 0.78 |
| Viscosity (cps) | 5000 at 75° F | 950 at 72° F | 4000 at 75° F | 500-1300 at 77° F | 10,000 |
| Shore D Hardness | 84 at RT 70 at 140° F* | 80 at 72° F 76 at 140° F* | 88 at 80° F 64 at 140° F* | 75 64 at 140° F* | - |
| Volume Resistivity (ohm - cm) | 7.1×10^{15} | 40×10^{14} | 1×10^{15} (75°) 6.7×10^8 (210°) | 4.1×10^{14} | 5×10^{16} 1×10^{13} |
| Dielectric Strength (v/mil) | - | - | - | - | 300 |
| Dielectric Constant | 4.3 (1 mc) | 3.5 60 to 10 ¹⁰ cps) | 3.9 (10 ⁴ cy) 3.5 (10 ⁶ cy) | 4.2 (10 ² cps) 4.0 (10 ⁵ cps) | (10 ² to 10 ¹⁰ cps) |
| Dissipation Factor | 0.035 (1 mc) | <0.02 60 to 10 ¹⁰ cps) | 0.020 (10 ⁴ cy) 0.029 (10 ⁶ cy) | 0.010 (10 ² cps) 0.027 (10 ⁵ cps) | (10 ² to 10 ¹⁰ cps) |
| Coefficient of Thermal Expansion (in/in/°C) | 58.8×10^{-6} | 45×10^{-6} | - | 61×10^{-6} | 19×10^{-6} |
| Thermal Conductivity (BTU/ft ² /hr/°F/in) | - | 1.5 | 1.2 | 1.6 | 0.8 |
| Izod Impact (ft.-lb/in notch) | 0.43 at 180° | 5.0 | - | - | 0.25 |
| H ₂ O Absorption (Wt. % gain) | - | 0.3 (24 hrs-RT) | 1.2 (2 hrs-boil) | 0.2 (24 hrs-RT) | 0.3 (24 hrs-25°C) |
| Mixing Ratios (A-B pbw) | 100-15 | 100-20 +6#9 | 100-10 | 100-11 | 100-13 |
| Pot Life | 5 hrs | 3 hrs | 20 min | 20 min | 4 hrs |
| Cure Time | 24 hrs at 140° F 16 hrs at 160° F | 48 hrs at RT 8 hrs at 110° F 3 hrs at 150° F | 16 hrs at RT +1 hr at 150° F | 2 hrs at 140° F or 24 hrs at RT | 3 hrs at 16° F then 7 hrs at 180° F |
| Discoloration Temperature (°F) | >212 | >200 | - | - | - |
| Heat of Distortion Temperature (°F) | 180 | 145* | 145* | 145* | 300 |
| Linear Shrinkage | - | - | 0.0025 in/in | 1.2% | 10.4×10^{-6} in/in/°F |
| Volume Loss (%) | 4.0* | 6.1* | 5.1* | 6.7* | 3.2* |
| Shrinkage Ratio (aging/immediate) | 1.32 * | 1.02 * | 1.22 * | 1.02 * | wetting problem |

* Per MIT Test. All other values are given by the company.

Chomerics 202/56, Stycast 1264 (A&B) and Hysol R8-2038 together with an opaque resin, Stycast 1090. It is not known at this time how the properties of Stycast 1264 are affected by the addition of Catalyst 9.

Finally, tests were made to determine the resistance to thermal shocks. The modules were placed in a chamber and temperature cycled as follows: -60°F for 30 minutes, 72°F for 15 minutes, 250°F for 30 minutes and 72°F for 15 minutes. The modules were then removed from the chamber and examined.

The cycle was repeated 5 times examining the modules at the end of each cycle.

Examination under a microscope revealed no cracks in any of the modules. Both chomerics and Stycast 1090 separated from their cases, and there was very mild separation in the case of Chomerics. Hysol R8-2038 exhibited some irregular soft spots. This irregularity was found in a number of samples (Hysol). A previous test consisted of the following: a steel washer $1\frac{1}{4}$ " diameter, $\frac{1}{4}$ " thick and having four triangular prongs at the outermost part of its faces was embedded in the resin to form a sample $2\frac{1}{4}$ " in diameter and $\frac{1}{2}$ " thick.

This sample was then placed in the chamber at 250°F for 30 minutes, then at -50°F for 30 minutes.

In all cases, including the opaque stycast 1090 sample, cracks developed.

This test was admittedly too severe in view of the geometric configuration of the washer and the difference in coefficient of expansion between it and the resin.

In addition to the previous milder thermal shock test, the above mentioned washers were embedded in the resins and examined after curing. There was a very slight crack at one point in the Chomerics 202 and in the Hysol R8-2038; no cracks appeared

in the other clear resins. This test, known as the Therman shock test, is a reliable and generally accepted one.

A point worth making is that varying degrees of hardness are obtained depending on the extent of postcuring. Tests at the end of the aging period recorded a shore D hardness of 70 for Epocast 1786/955 and 82 for the other three resins.

CONCLUSION

As a result of the tests, it is concluded that all the resins except Hysol R8-2038 are recommended for use in the G. M. E. with preference given to Stycast 1264.

In spite of its higher shrinkage factor, Stycast 1264 is chosen over Epocast 1786 because of its extremely low viscosity, which is a desirable feature in potting certain modules which present de-airing problems.

If viscosity is not a problem, Epocast 1786 is preferred to Stycast 1264 and both of these are preferred to Chomerics.

In potting G. M. E. Modules with connectors, however, viscosity is a problem and consequently Stycast 1264 should be used. It is recommended that unless Epocast 1786/955 is used, the Lexan cases should be sandblasted or otherwise roughened for better adhesion.

EQUIPMENT USED

Blue M oven

Mini Mite thermometer

Ohaus Dial Torsion Balance (0.02 gram readability)

Weights, Class S

Hunter spring scale

Starrett straight edge

Vacuum unit

Shore D hardness tester

Delta Design Chamber (-100°F to + 600°F)

American Optical Microscope Model #59M-1

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