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NASA CR-149980

ELECTRICAL PROPERTIES OF EPOXIES AND FILM RESISTORS

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March 1976

Interim Report, Phase I

NASA Technical Monitor: S. V. Caruso, Electronics and Control Laboratory

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16. ABSTRACT

The reliability of hybrid microcircuits has been enhanced in recent years by the use of organic adhesives as a replacement for solder and eutectics. The epoxies have been the most effective and widely used material for this application. Since many chemical variations of epoxies are available, selection, test, and evaluation to determine the best materials has not been completely exploited. This report develops methods for measuring the electrical and mechanical properties of epoxies. Data are given for selected conductive adhesives at high and low frequencies. Also, the temperature coefficients of resistance (TCR) of thick film resistors are presented.

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INTERIM REPORT
PHASE I

NASA CONTRACT NAS8-31172

"ELECTRICAL PROPERTIES OF EPOXIES AND FILM RESISTORS"

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PART I: ELECTRICAL PROPERTIES OF EPOXIES

I. Introduction

In recent years, the use of epoxies has all but replaced eutectic bonding in all but the most critical heat transfer and V_{CE}(SAT) applications. With this widespread use, there exists a need for information about the electrical and mechanical properties of epoxies and for methods of measuring these properties to allow epoxies to be used to their fullest extend.

This report covers the work performed under NASA Contract NAS8-31172. The objects of this study were to develop methods for measuring the electrical properties of epoxies, to measure the electrical properties of a number of conductive and non-conductive epoxies, and to determine the extent and effectiveness of their use in companies which manufacture hybrid microcircuits.

II. Determination of Resistivity of Conductive Adhesives at Low Frequencies

The first task was to develop a method for the measurement of the resistivity at low frequencies. The most common method used at the time was to fabricate two thick film contacts on an alumina substrate a known distance apart. A mold was then formed by placing two double thicknesses of scotch tape parallel to each other between the thick film contacts. The mold was then filled with epoxy and leveled with the aid of a razor blade. The tape was then removed and the epoxy cured per manufacturer's recommendations. The dimensions of the sample were then measured with a light-section microscope, the resistance was measured with a milliohmmeter, and the resistivity was calculated by the standard formula,

$$\rho = \frac{RA}{\ell} \qquad (1)$$

This method was tried and rejected for several reasons:

1. The samples prepared in this manner were not reproducible, making it difficult to compare epoxies from lot to lot.
2. Although the contact resistance of the milliohmmeter can be accounted for to a large extent, the contact resistance between the thick film contact and the epoxy is included in the measurement. Not only is this value significant in the calculation, but any problems with the metallization may be erroneously attributed to the epoxy.
3. The test samples require a considerable amount of time to prepare, especially if a statistically meaningful sample is required.
4. The equipment required (light-section microscope, millohmmeter, etc..) is expensive and difficult to operate, which eliminates the use of unskilled or semi-skilled personnel for the evaluation of epoxies prior to use.

Other methods tried were screen printing, which was rejected because the uneven surface resulted in a sample of nonuniform dimensions, and using a metal mold in contact with the substrate, which was deemed unacceptable because the epoxy tended to spread out between the mold, and the substrate where substrate camber was excessive.

The method ultimately chosen which overcame all the objections listed above was to machine a mold in Rexolite, a very high quality dielectric material, in the shape and dimension shown in Fig. 1. The mold is then filled with epoxy and cured per manufacturer's specifications. All subsequent testing is then done with the epoxy remaining in the mold.

Rexolite may be easily machined to a high degree of precision and tolerance in any standard machine shop. Once the initial setup is accomplished, a mold can be made in less than a minute. Rexolite also maintains its dimensional integrity over a wide temperature range; consequently, the curing cycle and temperature storage tests do not affect the dimensions of the sample. The molds may be machined to any desired depth so that a realistic situation may be simulated. A photograph of the mold is shown in Fig. 2.

The resistivity of the epoxy is determined by a four-point system of measurement as depicted in the schematic in Fig. 3. The mold is placed in a test fixture designed for the purpose as shown in the photograph in Fig. 4. Current is injected into the end terminals and the potential is measured across the two inner terminals. The resistivity is given by Eq. (1), where

$$R = \frac{V}{I} \tag{2}$$

$T = 0.157$ cm
$w = 8.0 \times 10^{-2}$ cm
$\ell = 1.0$ cm

$A = WT = 1.26 \times 10^{-2}$ cm^2

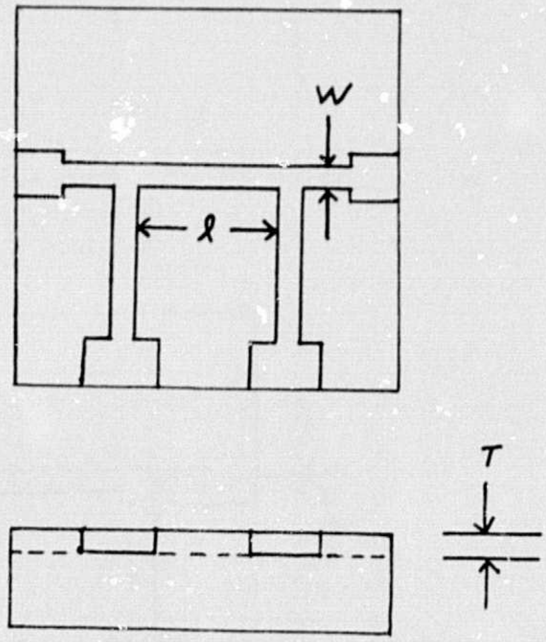

Figure 1

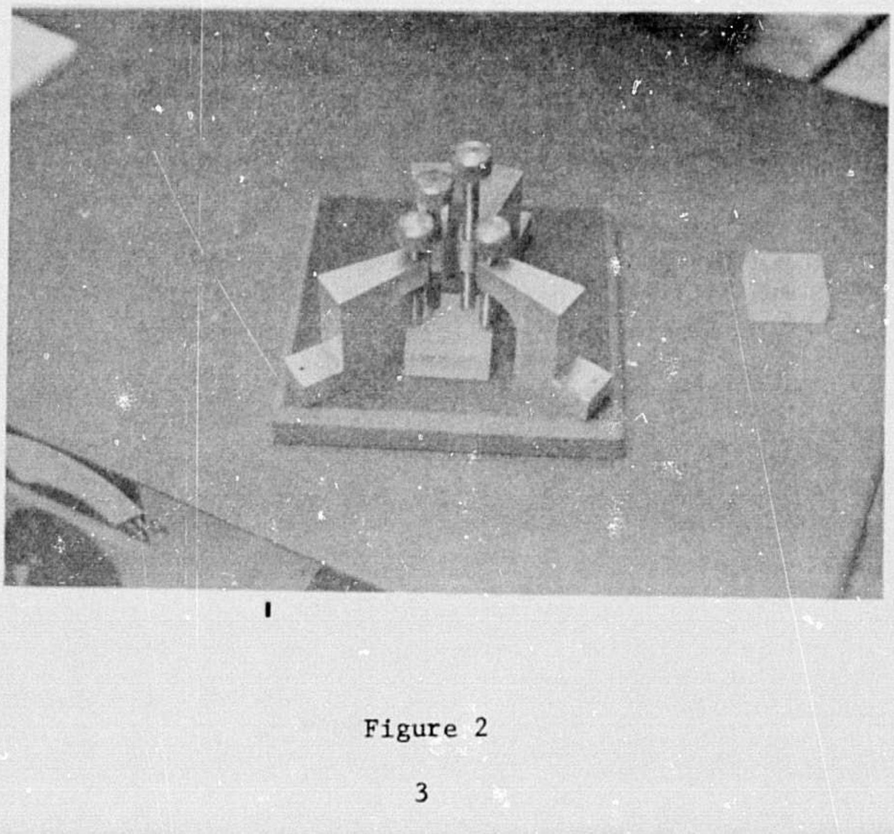

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Figure 2

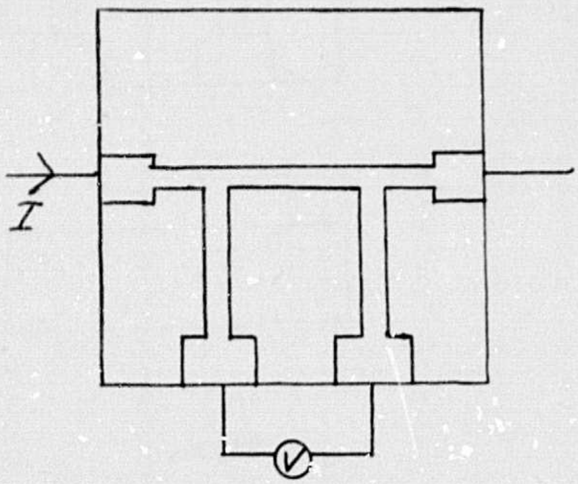

Figure 3

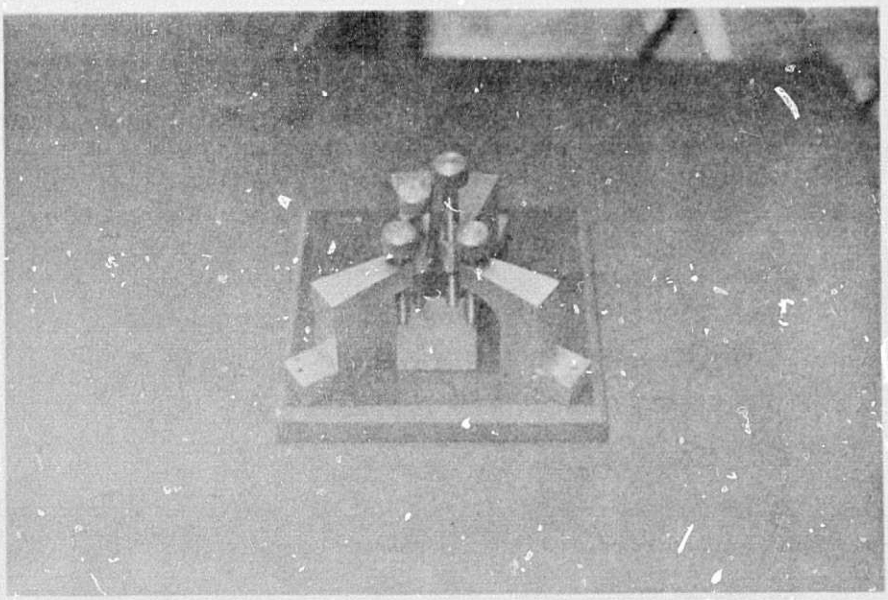

Figure 4

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and V and I are the voltage and current measurements obtained on the sample. This method is now being considered as an ASTM standard.

Resistivity measurements were obtained on a number of adhesives from different manufacturers. The measurements were taken as a function of current density and temperature and the two sets of measurements were correlated to obtain the maximum current density at which the epoxy may be used. The samples were then subjected to a temperature of 150°C for 1000 hours and the resistivity measurements were repeated to determine the effects of aging. The results of these tests were summarized in the following tables and graphs.

Epoxy	CODE	Resistivity Ohm-cm X10^{-4}	Resistivity after 1000 hrs at 150°C	Per Cent change	TCR PPM/°C	
					Hot	Cold
Epotek H20-E	B	1.55	1.50	-3.23	2050	2180
Epotek H11	B	3.09	3.95	27.33	3183	2759
Epotek H21	B	7.28	6.97	-4.26	---	---
Epotek H31	A	3.82	3.97	3.93	1800	4402
Epotek H31D	A	3.74	3.74	0	2772	2411
Epotek H40	C	6.63	6.87	3.62	5827	3030
Epotek H41	C	4.84	4.82	-0.41	---	---
Epotek H43	C	5.75	5.37	-6.61	3877	2534
Epotek H44	C	8.96	8.67	-3.24	2763	1952
Epotek H81	D	12.56	11.69	-6.93	3740	1896
Epotek 410	B	4.47	5.19	16.11	3846	3750
Epotek 410E	B	3.44	4.02	16.86	---	---
Epotek 417	B	1.84	2.21	20.11	---	---
Ablebond 58-1	C	6.26	6.04	-3.51	3323	2026
Ablebond 36-2	A	1.59	1.54	-3.14	2303	1724
ESL 1900 461-39	A	3.41	5.38	57.77	---	---

CODE:

A - 1 Component Silver
B - 2 Component Silver
C - 1 Component Gold
L - 2 Component Gold

Table 1. DC Resistivity of Conductive Epoxies - Summary

Normalized Resistivity
vs
Temperature (25°C = 1.00)

Temp (°C)	Epotek H11	Epotek H20E	Epotek H21D	Epotek H31	Epotek H31D	Epotek H40	Epotek H41	Epotek H43
-55°	0.80	0.81	0.81	0.77	0.81	0.77	0.77	0.80
-20°	0.87	0.90	0.88	0.86	0.87	0.85	0.85	0.87
0°	0.93	0.94	0.93	0.94	0.93	0.91	0.91	0.94
25°	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
60°	1.11	1.09	1.08	1.06	1.10	1.19	1.21	1.11
100°	1.23	1.21	1.20	1.21	1.21	1.41	1.59	1.25
150°	1.44	1.23	1.32	1.27	1.33	1.87	2.18	1.50

Temp (°C)	Epotek H44	Epotek H81	Epotek 410	Epotek 410E	Epotek 417	ESL LT461-39 1900	Ablebond 58-1	Ablebond 36-2	EMCA 2040-1
-55°	0.83	0.80	0.76	0.75	0.77	0.78	0.85	0.81	0.83
-20°	0.87	0.90	0.86	0.86	0.86	0.89	0.85	0.88	0.88
0°	0.93	0.94	0.92	0.92	0.92	0.99	0.91	0.93	0.95
25°	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
60°	1.08	1.10	1.10	1.12	1.10	1.10	1.07	1.08	1.09
100°	1.20	1.22	1.24	1.24	1.22	1.22	1.20	1.20	1.20
150°	1.30	1.39	1.47	1.42	1.40	1.39	1.43	1.25	1.32

Table II

Normalized Resistivity

vs

Current Density (7.96 A/cm^2 = 1)

Current Density (A/cm^2)	Epotek H11	Epotek H20E	Epotek H21D	Epotek H31D	Epotek H40	Epotek H41	Epotek H43	Epotek H44
7.96	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
15.92	1.003	0.9811	0.9853	0.9791	0.9631	0.9898	1.00	0.9901
23.89	0.9968	0.9788	0.9796	0.9921	0.9660	0.9816	0.9846	0.9967
31.85	0.9968	0.9741	0.9786	0.9686	0.9645	0.9816	0.9812	0.9856
39.81	0.9968	0.9741	0.9746	0.9791	0.9793	0.9878	0.9846	0.9901

Current Density A/cm^2	Epotek H81	Epotek 410E	Epotek 417	ESL LT 461-39 1900	Ablebond 58-1	Ablebond 36-2	EMCA 2040-1
7.96	1.00	1.00	1.00	1.00	1.00	1.00	1.00
15.92	0.9835	1.00	0.9946	0.9858	0.9923	0.9819	0.9939
23.89	0.9827	0.9857	0.9946	0.9887	0.9880	0.9639	1.00
31.85	0.9882	0.9829	0.9946	0.9717	0.9753	0.9639	0.9939
39.81	0.9851	0.9829	0.9892	0.9603	0.9646	0.9578	0.9939

Table III

III. V_{CE}(SAT) of Devices Mounted with Conductive Epoxy

A potentially serious problem with gold epoxies has been the tendency of the V_{CE} (SAT) of small-signal switching transistors which have been mounted with gold epoxies to increase beyond acceptable limits when aged at 150° C for periods as short as 100 hours. Aging tests on the epoxies alone have identified a possible failure mechanism. It is suspected that the gold filler in the epoxy tends to settle toward the bottom of the fillet, migrating away from the interface between the device and the epoxy. Tests on gold epoxies indicate that the overall bulk resistivity tends to decrease after aging, but that the surface resistivity, as measured by a conventional four-point probe, tends to increase. This is strongly indicative that the gold is migrating downward.

Since gold is much heavier than silver, the volume ratio of gold is somewhat smaller than that of silver if the filler is added to the epoxy by weight. It is planned to have special gold epoxies made with a higher gold content to see if this will alleviate the problem. However, the added gold may decrease the shear strength of the epoxy and it is also planned to conduct shear tests of these special epoxies.

IV. Determination of Relative Dielectric Constant and Loss Tangent of Nonconductive Epoxies

The technique used to measure the relative dielectric constant and loss tangent was to construct a variable capacitor with the bottom electrode fixed and the top electrode capable of vertical movement. The top electrode is attached to a precision micrometer (0.002 mm resolution) so that its position is precisely known. A guard ring to eliminate edge effects is constructed around the bottom electrode. A picture of the apparatus is shown in Fig. 5.

A disc of the epoxy to be measured is fabricated by filling the teflon mold with uncured epoxy and curing it at the time and temperature recommended by the manufacturer. The dimensions of the disc are not critical as long as the disc overlaps the guard ring and the faces are flat and parallel. The thickness of the disc is measured at several places to insure uniformity. The thickness is recorded at t_s.

The disc is then placed between the two electrodes with the top electrode close but not touching and the capacitance and Q of the ensemble are measured with a Q-meter and recorded as C_s and Q_s. The disc is then removed and the top electrode is moved downward until resonance is again observed on the Q-meter with the controls of the Q-meter remaining untouched. The Q of the apparatus is recorded as Q_a and the distance through which the top electrode must be moved to again resonate the system is recorded as t_x.

The relative dielectric constant is then calculated as:

$$\varepsilon_r = \frac{t_s}{t_s - t_x} \tag{3}$$

The loss tangent is then calculated by the following sequence of steps.

1. Calculate R_a, the equivalent parallel resistance of the apparatus, by:

$$R_a = \frac{Qa}{\omega Cs} \tag{4}$$

2. Calculate R_{as}, the equivalent parallel resistance of the sample in combination with the apparatus, by:

$$R_{as} = \frac{Qs}{\omega Cs} \tag{5}$$

3. Calculate R_s, the equivalent parallel resistance of the sample, by:

$$R_s = \frac{R_a R_{as}}{R_a - R_{as}} \tag{6}$$

4. Calculate the loss tangent by

$$\text{Loss tangent} = \frac{1}{R_s \omega Cs} \tag{7}$$

The major advantages of this method over other methods are:

1. The disc does not need to be metallized, an expensive and time-consuming process.

2. Since the dielectric constant is measured by a differential measurement, the effect of stray capacitance is negated.

3. The method is very accurate and fast.

The results of these measurements are summarized in the following tables IV and V.

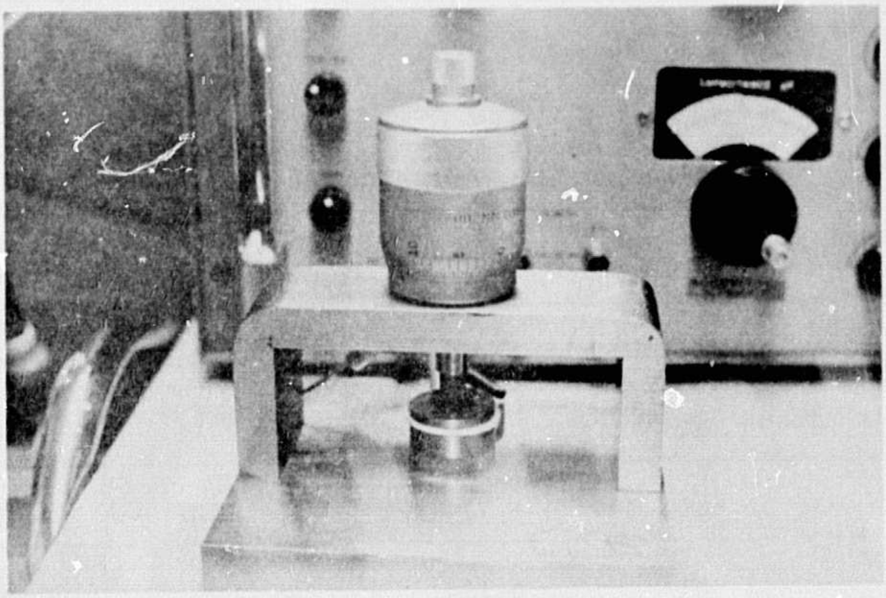

Figure 5

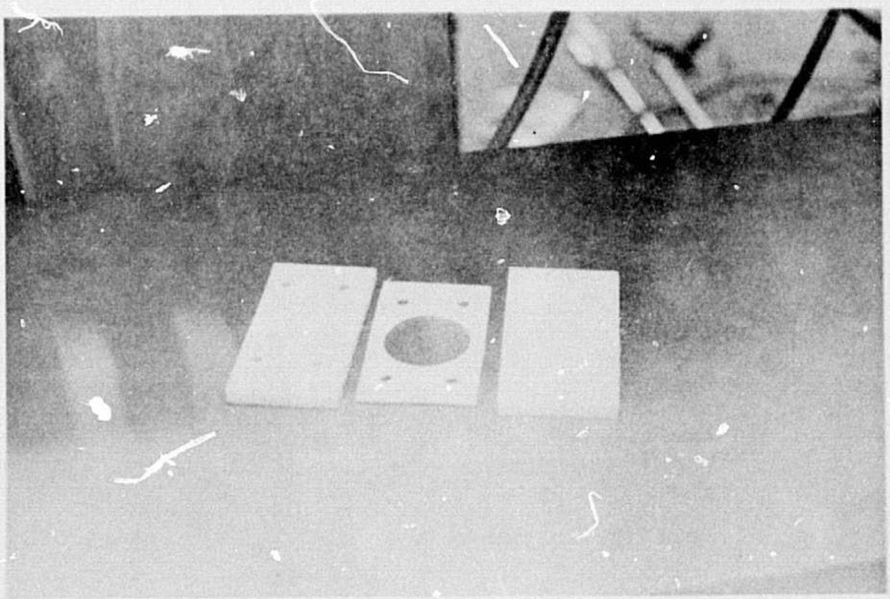

Figure 6

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ABLESTIK A806-6

	ε_r		Loss Tan	
Freq.	Initial	Aged	Initial	Aged
1 M	5.161	-	.0074	.0069
5 M	5.461	5.714	.0072	.0077
10 M	5.369	5.195	.0067	.0070
20 M	5.316	5.263	.0073	.0387
30 M	5.333	5.128	.0079	.0159
40 M	5.246	5.063	.0071	.0107

Table IV

EPOTEK H55

	ε_r		Loss Tan	
Freq	Initial	Aged	Initial	Aged
1M	9.357	---	.0064	.0067
5M	10.19	10.00	.0056	.0081
10M	10.19	10.12	.0077	.0074
20M	10.26	10.12	.0087	.0188
30M	10.06	10.12	.0072	.0112

EPOTEK 921 f3

ε_r		Loss Tan	
Initial	Aged	Initial	Aged
8.338	--	.0071	.0075
9.381	9.769	.0062	.0067
9.223	9.655	.0079	.0083
9.327	9.434	.0072	.0182
8.876	9.327	.0074	.0111

EPOTEK H74

	ε_r		Loss Tangent	
Freq	Initial	Aged	Initial	Aged
1 MHz	6.657	---	.0048	.0064
5 MHz	7.862	7.751	.0048	.0081
10 MHz	7.788	7.751	.0051	.0086
20 MHz	7.825	7.751	.0052	.0108
30 MHz	7.788	7.751	.0054	.0111
40 MHz	7.788	7.68	.0056	.0113

EPOTEK H61

ε_r		Loss Tangent	
Initial A	Aged	Initial	Aged
8.684	--	.0047	.0068
8.684	7.83	.0053	.0068
8.595	7.83	.0064	.0085
8.505	7.83	.0065	.0186
8.422	7.83	.0079	.0109
8.338	7.55	.0087	.0131

Table V

V. Measurement of Conductive Epoxies at High Frequency

Various methods have been attempted to measure the high frequency conductivity of epoxy. A resonant coaxial cavity was examined to determine if it could be used as a testing device. The cavity which was to be either one-quarter or one-half wavelength long, would have been fabricated with a center conductor of epoxy. The Q (quality factor) of the cavity would have been observed and compared to a cavity with a center conductor of known conductivity. Using this data and the calculated parameters of the coaxial line we would have been able to determine the conductivity. This method was found to be unusable due to problems in fabricating the center conductor with epoxy. A large quantity of epoxy would have been required to construct the rod. Also, air bubbles in the epoxy caused the surface of the rod to be irregular. This method was abandoned in favor of measuring the epoxy using thick film techniques.

Several different stripline filters were made on alumina substrates. Of these the simple half-wave section of transmission line seems to give the best results. A transmission line was designed with two small gaps spaced one-half wave length apart. The stripline was made of 99+ Au except for the half-wave section which was conductive epoxy. Since the epoxy had a conductivity of approximately 10^3 less than gold, any change in the Q should be attributable to the epoxy. This change in Q can then be related to the conductivity of the epoxy. The problems one encounters with this method are partly related to the thick film process. A very fine mesh screen must be used to obtain good line definition, and the screen leaves the upper surface of the epoxy line very rough. In the GHz frequencies, these parameters become critical. Another problem is the size of the gaps in the transmission line. The presence of the gap capacitiance in the line will have some effect on the data taken. This method of determining the conductivity of epoxy is still under investigation.

Another means of conductivity measurement is being examined. If the power lost across a section of stripline transmission canb e measured, then the attenuation constant of the line can be determined. This constant consists of three parts. The attenuation due to the conductive line, the dielectric substrate and the conductive ground plane. If the substrate and ground plane are made with materials of known characteristics, then their contribution to the attenuation constant can be calculated. The remainder of the constant is then attributable to the line. The attenuation constant can then be related to the sheet resistivity of the line using known formulas.

VI. General Comments

During the past year, two trips were taken to companies which use and manufacture epoxies. Some observations from these trips and other discussions are given below.

1. The curing time as specified by manufacturers is generally insufficient. Most companies double it as a standard procedure.

2. A thin, even coat of epoxy slightly larger than the device provides the best overall mechanical, thermal, and electrical joint. A coat of epoxy of this type is most easily accomplished by screen printing the epoxy. A fillet of epoxy as formed by dispensing through a hypodermic or by hand tends to form microcracks, which weaken the mechanical integrity of the joint.

3. The effect of the mixing ratio of two-component epoxies on the mechanical and electrical properties needs to be studied. Although two-component epoxies have some superior qualities over one-component epoxies, many companies shy away from their use on assembly lines because they fear problems due to mixing by untrained personnel.

Part II: ELECTRICAL PROPERTIES OF FILM RESISTORS

I. Temperature Coefficient of Resistance (TCR).

The TCR's of thick film resistors fabricated at MSFC were measured by standard methods. The results of these tests are summarized in the following tables.

DuPont 1411 - 10 ohms/square

The TCR of the DuPont 1411 ink was very unstable. After heating to 150°C, the room temperature value permanently changed from 11.939 ohms/square to 12.250 ohms/square.

The results after the change are:

Temp.	TCR (Avg. of 10 resistors)
-55°C	382.84 ppm/°C
-25°	634.55
0°	1,512.2
50°C	338.5
75°C	233.01
100°	252.12
125°	232.41
150°	223.96

DuPont 1421 - 100 ohms/square

Temp.	TCR
-55°C	-72.77 PPM/°C
-25°	-37.624
0°	-21.515
50°	-15.979
75°	33.553
100°	44.385
125°	60.172
150°	84.973

DuPont 1431 - 1000 ohms/square

Temp.	TCR	
-55°C	-30.945	PPM/°C
-25°	-15.060	
0°	10.776	
50°	38.981	
75°	52.012	
100°	66.504	
125°	79.604	
150°	91.220	

DuPont 9677 - 100K ohms/square

Temp.	TCR	
-55°C	102.38	PPM/°C
-25°	143.89	
0°	145.78	
50°	151.03	
75°	162.27	
100°	149.79	
125°	175.67	
150°	135.14	

II. Noise Figure

The noise figures of resistors fabricated at MSFC were measured on the Quantech noise measurement system. The results are summarized in the following tables.

DuPont 1411 - 10 ohms/square

Area	Noise Index μV/V/decade (db)
23 mils x 23 mils ($529\ mils^2$)	-2.94 db
39 mils x 39 mils ($1521\ mils^2$)	-10.99 db

Area	Noise Index μV/V/decade (db)
78 mils x 78 mils (6084 mils^2)	-18.93 db

DuPont 1421 - 100 ohms/square

Area	Noise Index μV/V/decade (db)
23 mils x 23 mils (529 mils^2)	-18.12 db
39 mils x 39 mils (1521 mils^2)	-24.18 db
56 mils x 56 mils (3136 mils^2)	-24.45 db
78 mils x 78 mils (6084 mils^2)	-26.15 db

DuPont 1431 - 1000 ohms/square

Area	Noise Index μV/V/decade (db)
23 mils x 23 mils (529 mils^2)	-9.625 db
39 mils x 39 mils (1521 mils^2)	-14.66 db
56 mils x 56 mils (3136 mils^2)	-17.59 db
78 mils x 78 mils (6084 mils^2)	-20.14 db

DuPont 9677 - 100K ohms/square

Area	Noise Index μV/V/decade(db)
23 mils x 23 mils ($529\ \text{mils}^2$)	9.95 db
39 mils x 39 mils ($1521\ \text{mils}^2$)	4.23 db
56 mils x 56 mils ($3136\ \text{mils}^2$)	0.13 db
78 mils x 78 mils $(6084\ \text{mils})^2$	-1.66 db

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