BOEING

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TITLE INVESTIGATION INTO REVERSION OF POLYURETHANE ENCAPSULANTS

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ABSTRACT

The effect of high humidity (95% RH) at 60°C, 70°C, 85°C and 100°C on the solid-to-liquid reversion of polyurethane elastomers (used for potting electrical connectors and conformal coating printed circuit boards) was investigated. Hardness measurements in accordance with Method 1082 of FED-STD-406 were used on eleven elastomers to track reversion for a 101-day period.

The primary purpose of the tests was to provide data to predict service life for the polyurethane elastomers. This was not accomplished as the hardness did not deteriorate rapidly enough at the lower test temperatures. The tests did determine that the potting and coating materials most widely used on the S-IC Program (PR1535 and PR1538) are susceptible to reversion but appear adequate for service in the S-IC environment. The PR1535 showed no loss in hardness at 60°C, while the PR1538 stabilized 8% down at 60°C.

KEY WORDS

Elastomer

Reversion

Potting

Conformal Coating

Humidity

This document includes the results of tests conducted per EWA 362 and EWA 362A (Reference a).

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LIST OF TABLES

* 100°C, 85°C, 70°C, and 60°C

1.0 OBJECTIVES

a. Evaluate reversion characteristics of elastomers used for potting of electrical connectors and conformal coating of printed circuit boards.

b. Provide data to predict service life of elastomers used on the S-IC Program.

c. Determine adequacy of potting and conformal coating elastomers used in S-IC applications.

2.0 BACKGROUND

Polyurethane encapsulants can revert to a liquid state under environmental stress, usually involving high humidity. The degree of this reversion, the rapidity, and the temperature intensification of humidity effect were not known until 1966 when the U. S. Navy began subjecting materials to an environment of 100°C and 95% RH and noting changes in hardness. Results indicated the reversion problem was much more severe than anticipated.

Published data (Reference b) and possible reversion of encapsulants used on the S-IC Program led to the present study at MAF.

The Shore "A" hardness test selected to track reversion has shown to be precise, convenient and inexpensive to perform.

3.0 CONCLUSIONS

a. For the encapsulants tested, the change from the original hardness values after 101 days at 60°C were very slight, indicating no significant reversion. This 101-day time period was not long enough at the 60°C temperature to establish a service life rating.

b. With the exception of Uralane 5712 and Hysol C-21, all encapsulants reverted (in 20 to 40 days at the 100°C temperature) to a very low hardness, indicating high susceptibility to reversion.

c. Based on the results of these tests, the polyurethanes used on the S-IC Program are adequate for current S-IC applications.

4.0 RECOMMENDATIONS

Humidity degradation has been shown elsewhere (reference b) to be permanent and cumulative. Hydrolytic stability tests, as employed here, are recommended for screening purposes on polyurethane elastomers to determine sensitivity to reversion. Of the polyurethane elastomers tested, the Urelane 5712 showed the greatest resistance to reversion; however, since several specimens cracked during tests, it is recommended that further testing be conducted prior to consideration of this material for S-IC or other program applications.

5.0 PROCEDURES AND RESULTS

5.1 TEST CONDITIONS

5.1.1 Temperatures

a. 60°C ± 3°C
b. 70°C ± 3°C
c. 85°C ± 3°C
d. 100°C ± 3°C

5.1.2 Relative Humidity

95% Minimum (all tests).

5.1.3 Hardness Measurements

Hardness measurements were in accordance with Method 1082 of FED-STD-406, with a type A-2 durometer and an application time of three seconds. Specimens were cooled to 23°C ± 2°C prior to the hardness check. The hardness was checked at 7-day intervals unless the rate of change indicated a different interval to be advantageous. A set of five specimens were subjected to each test condition for 101 days, or until the specimen liquified. Measurements were discontinued on any specimen when the hardness value became less than 10.

5.2 TEST SPECIMEN MATERIAL AND PROCESSING

The elastomers included in these tests were selected from the Qualified Products List (QPL) of potting, molding and conformal coating specifications and processes used on the S-IC Program. Twenty specimens of each type of elastomer listed in Table 1 were cast in discs one-half inch in thickness and one and one-half inches in diameter. Processing and curing of the test specimens were as shown in Table 2. The Hysol C-21 is the only epoxy-type potting material used in Boeing fabricated S-IC stage hardware and was included in the test for comparative data.

5.3 TEST RESULTS

Final hardness values are shown in Tables 3, 4, 5 and 6. The hardness decrease in all materials, except PR1538 (down 8%), was minimal at 60°C. All values are an average of five specimens, except as noted. Uralane specimens exhibited cracks at each of the four test temperatures, and four specimens of the Hysol C-21 cracked during testing at the 85°C temperature. Tables 7 through 17 show hardness change during the test periods for the eleven types of elastomers.

6.0 <u>REFERENCES</u>

a. Boeing Engineering Work Authorization (EWA) 362 and 362A, "Evaluation of Reversion Resistance of Elastomers", March 1969.

b. Report, "Navy Investigates Reversion Phenomena of Two Elastomers", Fred H. Gahimer and Fred W. Nieske, <u>Insulation/Circuits Magazine</u>, August 1968.

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TABLE 1

TEST MATERIALS DATA

MATERIAL DESIGNATION	MATERIAL IDENT. NO.	MANUFACTURER'S NAME	APPLICATION DATA	USING SPECIFICATION	MATERIAL TYPE
3C-900A	1	Churchill Chemical Co.	Patching and Repair	MSFC-PROC-186	Polyurethane
3C-907	2	Churchill Chemical Co.	Potting and Molding	MSFC-SPEC-202	Polyurethane
Uralane 5712	3	Furane Plastics, Inc.	Potting and Molding	MSFC-SPEC-202	Polyurethane
Uralane 5733	4	Furane Plastics, Inc.	Potting and Molding	--	Polyurethane
PR1535	5	Products Research Co.	Potting and Molding	MSFC-SPEC-202	Polyurethane
PR1538	6	Products Research Co.	Conformal Coating	MSFC-SPEC-393	Polyurethane
Hysol C-21	7	Hysol Chemical Co.	Field Potting	60B32561 (Boeing Spec.)	Epoxy
Hysol PC-22	8	Hysol Chemical Co.	Conformal Coating	MSFC-SPEC-393	Polyurethane
Pro-Seal 794	9	Coast Pro-Seal Mfg. Co.	Potting and Molding	MSFC-SPEC-202	Polyurethane
Pro-Seal 796-80	10	Coast Pro-Seal Mfg. Co.	Potting and Molding	MSFC-SPEC-202	Polyurethane
Chem Seal 3547	11	Chem Seal Corp.	Potting and Molding	MSFC-SPEC-202	Polyurethane

TABLE 2

TEST SPECIMEN PREPARATION DATA

ELASTOMER TYPE	PROCESSED PER	CURE TIME/TEMPERATURE
3C-900A	Mfg. Instructions	15 min. @ 180°F
3C-907	Mfg. Instructions	1 hr. @ 180°F
UR5712	60B32057*	16 hrs. @ 140°F
UR5733	Mfg. Instructions	4 hrs. @ 180°F
PR1535	Mfg. Instructions	6 hrs. @ 180°F
PR1538	60B32057*	16 hrs. @ 140°F
C-21	Mfg. Instructions	2 hrs. @ 140°F 4 hrs. @ 140°F (Post Cure)
PC-22	60B32057*	16 hrs. @ 140°F
PS-794	Mfg. Instructions	16 hrs. @ 180°F
PS-796-80	Mfg. Instructions	16 hrs. @ 180°F
CS-3547	Mfg. Instructions	6 hrs. @ 180°F

* Boeing Specification

TABLE 3
HARDNESS AT 60°C AND 95% RH

MATERIAL IDENTIFICATION NUMBER	INITIAL HARDNESS	HARDNESS AT ____ DAYS		HARDNESS CHANGE	REMARKS
1	77	72	101	5	
2	77	77		0	
3	67	67		0	
4	80	77		3	Two specimens cracked at 19 days.
5	79	78		1	
6	73	65		8	
7	96	91		5	
8	80	78		2	
9	84	81		3	
10	80	78		2	
11	80	76	101	4	

TABLE 4
HARDNESS AT 70°C AND 95% RH

MATERIAL IDENTIFICATION NUMBER	INITIAL HARDNESS	HARDNESS AT ___ DAYS		HARDNESS CHANGE	REMARKS
1	79	68	101	11	
2	78	71		7	
3	67	67		0	One specimen cracked in 19 days.
4	79	72		7	One specimen cracked in 19 days.
5	79	72		7	
6	75	55		20	
7	96	81		15	
8	80	68		12	
9	80	72		8	
10	80	67		13	
11	83	65	101	18	

TABLE 5
HARDNESS AT 85°C AND 95% RH

MATERIAL IDENTIFICATION NUMBER	INITIAL HARDNESS	HARDNESS AT ___ DAYS		HARDNESS CHANGE	REMARKS
1	77	27	101	50	
2	77	45		32	
3	66	54		12	Two specimens cracked at 13 days.
4	79	59		20	
5	74	47		27	
6	70	16		54	
7	96	74		22	Four specimens cracked at 68 days.
8	80	26		54	
9	80	49		31	
10	78	35		43	
11	81	25	101	56	

TABLE 6
HARDNESS AT 100°C AND 95% RH

MATERIAL IDENTIFICATION NUMBER	INITIAL HARDNESS	HARDNESS AT ___ DAYS		HARDNESS CHANGE	REMARKS
1	79	<10	28	>69	
2	81	<10	36	>71	
3	83	52	42	31	One specimen cracked at 21 days.
4	76	26	42	50	One specimen cracked at 14 days.
5	72	<10	28	>62	
6	74	<10	14	>64	
7	92	74	42	18	
8	82	<10	21	>72	
9	79	<10	42	>69	
10	79	<10	36	>69	
11	78	<10	36	>68	

TABLE 7

HARDNESS VS. TIME AT NOTED TEMPERATURES AND 95% RH
ELASTOMER TYPE 3C-900A

TIME (days)	100°C HARDNESS No.	100°C HARDNESS %	TIME (days)	85°C HARDNESS No.	85°C HARDNESS %	70°C HARDNESS No.	70°C HARDNESS %	60°C HARDNESS No.	60°C HARDNESS %
0	79	100	0	77	100	79	100	77	100
7	56	71	6	69	90	74	94	73	95
14	53	42	13	68	88	72	91	73	95
21	13	16	19	67	87	73	92	74	96
28	<10	<13	26	65	86	71	90	72	94
			33	64	83	74	94	73	95
			40	61	79	71	90	74	96
			47	61	79	71	90	76	99
			54	59	77	72	91	75	97
			61	55	71	70	89	73	95
			68	50	65	67	85	74	96
			75	40	52	67	85	73	95
			82	39	51	67	85	74	96
			89	35	45	69	87	73	95
			101	27	35	68	86	72	94

TABLE 8

HARDNESS VS. TIME AT NOTED TEMPERATURES AND 95% RH
ELASTOMER TYPE 3C-907

TIME (days)	100°C HARDNESS No.	100°C HARDNESS %	TIME (days)	85°C HARDNESS No.	85°C HARDNESS %	70°C HARDNESS No.	70°C HARDNESS %	60°C HARDNESS No.	60°C HARDNESS %
0	81	100	8	77	100	79	100	77	100
7	52	64	6	69	90	78	99	83	104
14	36	44	13	65	84	77	97	83	104
21	23	28	19	65	84	78	99	83	104
28	13	16	26	65	84	74	94	81	103
36	<10	<12	33	61	79	72	91	80	102
			40	60	78	71	90	76	99
			47	60	78	71	90	76	99
			54	58	75	70	89	74	96
			61	57	74	71	90	79	101
			68	54	70	70	89	75	97
			75	55	71	73	92	79	101
			82	50	65	71	90	74	96
			89	49	64	70	89	80	102
			101	45	58	71	92	79	101

TABLE 9

HARDNESS VS. TIME AT NOTED TEMPERATURES AND 95% RH
ELASTOMER TYPE Uralane 5712

TIME (days)	100°C		TIME (days)	85°C		70°C		60°C	
	HARDNESS			HARDNESS		HARDNESS		HARDNESS	
	No.	%		No.	%	No.	%	No.	%
0	83	100	0	61	100	67	100	67	100
7	69	83	6	56	84	61	91	65	97
14	66	80	13	58	87	61	91	65	97
21	66	80	19	60	90	63	94	65	97
28	58	70	26	60	90	62	93	66	99
36	53	64	33	59	88	64	96	65	97
42	52	63	40	60	90	65	97	66	99
			47	61	91	66	99	67	100
			54	59	88	66	99	66	99
			61	59	88	66	99	68	102
			68	57	85	66	99	68	102
			75	59	88	67	100	68	102
			82	54	80	66	99	67	100
			89	55	82	68	102	69	103
			101	54	80	67	100	68	102

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TABLE 10

HARDNESS VS. TIME AT NOTED TEMPERATURES AND 95% RH
ELASTOMER TYPE Uralane 5733

TIME (days)	100°C HARDNESS		TIME (days)	85°C HARDNESS		70°C HARDNESS		60°C HARDNESS	
	No.	%		No.	%	No.	%	No.	%
0	76	100	0	79	100	79	100	80	100
7	57	74	6	69	91	72	91	77	96
14	53	70	13	69	71	72	91	77	96
21	54	58	19	70	95	75	95	76	95
28	39	51	26	70	91	72	91	76	95
36	31	41	33	67	92	73	92	77	96
42	26	34	40	67	94	74	94	77	96
			47	68	91	72	91	79	99
			54	66	92	73	92	78	97
			61	66	94	74	94	77	96
			68	63	94	74	94	76	95
			75	63	94	74	94	78	97
			82	60	92	73	92	77	96
			89	60	94	74	94	78	97
			101	59	91	72	91	77	96

TABLE 11

HARDNESS VS. TIME AT NOTED TEMPERATURES AND 95% RH
ELASTOMER TYPE PR1535

TIME (days)	100°C HARDNESS No.	100°C HARDNESS %	TIME (days)	85°C HARDNESS No.	85°C HARDNESS %	70°C HARDNESS No.	70°C HARDNESS %	60°C HARDNESS No.	60°C HARDNESS %
0	72	100	0	78	100	79	100	79	100
7	43	60	6	71	91	78	99	79	100
14	28	40	13	70	90	76	96	79	100
21	15	21	19	70	90	78	99	79	100
28	<10	<14	26	66	85	74	94	78	99
			33	64	82	74	94	78	99
			40	62	80	75	95	78	99
			47	63	81	75	95	79	100
			54	59	76	75	95	78	99
			61	59	76	75	95	79	100
			68	54	70	74	94	78	99
			75	55	71	74	94	79	100
			82	51	65	73	92	77	98
			89	50	64	73	92	79	100
			101	47	60	72	91	78	99

TABLE 12

HARDNESS VS. TIME AT NOTED TEMPERATURES AND 95% RH
ELASTOMER TYPE PR1538

TIME (days)	100°C HARDNESS No.	100°C HARDNESS %	TIME (days)	85°C HARDNESS No.	85°C HARDNESS %	70°C HARDNESS No.	70°C HARDNESS %	60°C HARDNESS No.	60°C HARDNESS %
0	74	100	0	70	100	75	100	73	100
7	20	27	6	52	79	65	87	67	92
14	<10	<13	13	50	71	65	87	68	93
			19	47	67	62	83	67	92
			26	41	59	62	83	64	88
			33	32	46	62	83	66	91
			40	30	43	60	80	68	93
			47	30	43	59	80	67	92
			54	22	31	57	76	65	90
			61	19	27	57	76	66	91
			68	18	26	56	75	66	91
			75	18	26	57	76	66	91
			82	15	21	54	73	64	88
			89	17	24	56	75	67	92
			101	16	23	55	74	64	88

TABLE 13

HARDNESS VS. TIME AT NOTED TEMPERATURES AND 95% RH
ELASTOMER TYPE HY-SOL C21

TIME (days)	100°C HARDNESS No.	100°C HARDNESS %	TIME (days)	85°C HARDNESS No.	85°C HARDNESS %	70°C HARDNESS No.	70°C HARDNESS %	60°C HARDNESS No.	60°C HARDNESS %
0	92	100	0	96	100	96	100	96	100
7	75	82	6	84	88	85	89	91	95
14	77	84	13	84	88	85	89	88	92
21	81	88	19	85	89	85	89	89	93
28	79	86	26	84	88	84	88	89	93
36	81	88	33	81	84	86	90	89	93
42	74	80	40	82	85	85	89	90	94
			47	84	88	88	92	92	96
			54	84	88	88	92	92	96
			61	83	86	87	91	91	95
			68	76	79	83	86	89	93
			75	83	86	87	91	92	96
			82	78	81	84	88	91	95
			89	79	82	85	89	91	95
			101	74	77	81	84	90	94

TABLE 14

HARDNESS VS. TIME AT NOTED TEMPERATURES AND 95% RH ELASTOMER TYPE PC-22

TIME (days)	100°C HARDNESS		TIME (days)	85°C HARDNESS		70°C HARDNESS		60°C HARDNESS	
	No.	%		No.	%	No.	%	No.	%
7	82	100	0	80	100	80	100	80	100
7	31	38	6	67	84	74	92	80	100
14	15	18	13	66	82	73	91	81	101
21	<10	<12	19	64	80	73	91	81	101
			26	59	74	71	89	80	100
			33	56	70	72	90	79	99
			40	53	65	71	89	78	98
			47	51	64	72	90	79	99
			54	48	60	71	89	79	99
			61	43	54	71	89	79	99
			68	40	50	70	87	77	97
			75	39	49	70	87	78	98
			82	31	39	68	85	77	97
			89	32	40	69	86	78	98
			101	26	32	68	85	78	98

TABLE 15

HARDNESS VS. TIME AT NOTED TEMPERATURES AND 95% RH
ELASTOMER TYPE PRO-SEAL 794

TIME (days)	100°C HARDNESS		TIME (days)	85°C HARDNESS		70°C HARDNESS		60°C HARDNESS	
	No.	%		No.	%	No.	%	No.	%
0	79	100	0	80	100	80	100	84	100
7	57	71	6	75	94	77	96	83	99
14	40	50	13	71	89	78	97	84	100
21	27	34	19	70	88	77	96	84	100
28	16	20	26	67	84	76	95	84	100
36	11	14	33	65	81	76	95	83	99
42	<10	<13	40	65	81	74	92	79	94
			47	64	80	75	94	80	95
			54	62	77	75	94	80	95
			61	60	75	75	94	81	97
			68	58	72	73	91	78	93
			75	57	71	74	92	81	97
			82	53	66	73	91	74	88
			89	54	67	73	91	81	97
			101	49	61	72	90	81	97

TABLE 16

HARDNESS VS. TIME AT NOTED TEMPERATURES AND 95% RH
ELASTOMER TYPE PRO-SEAL 796-80

TIME (days)	100°C HARDNESS No.	100°C HARDNESS %	TIME (days)	85°C HARDNESS No.	85°C HARDNESS %	70°C HARDNESS No.	70°C HARDNESS %	60°C HARDNESS No.	60°C HARDNESS %
0	79	100	0	78	100	80	100	80	100
7	53	67	6	68	87	75	94	79	99
14	36	45	13	66	85	76	95	80	100
21	23	28	19	63	81	75	94	80	100
28	13	16	26	59	76	73	91	80	100
36	<10	<13	33	58	74	69	86	79	99
			40	56	72	66	82	76	95
			47	55	70	68	85	76	95
			54	53	68	67	84	77	96
			61	51	65	69	86	79	99
			68	46	59	66	82	75	94
			75	48	62	68	85	78	97
			82	42	54	65	81	76	95
			89	42	54	65	81	77	96
			101	35	45	67	84	78	97

TABLE 17

HARDNESS VS. TIME AT NOTED TEMPERATURES AND 95% RH
ELASTOMER TYPE CHEM-SEAL 3547

TIME (days)	100°C HARDNESS No.	100°C HARDNESS %	TIME (days)	85°C HARDNESS No.	85°C HARDNESS %	70°C HARDNESS No.	70°C HARDNESS %	60°C HARDNESS No.	60°C HARDNESS %
0	81	100	0	84	100	80	100	Not tested at this temp.	
6	71	88	6	76	90	79	99		
13	67	83	13	74	88	77	96		
19	67	83	19	74	88	76	95		
26	58	72	26	72	86	76	95		
33	56	69	33	72	86	76	95		
40	50	62	40	72	86	75	94		
47	47	58	47	72	86	78	97		
54	37	46	54	72	86	77	96		
61	40	49	61	70	83	77	96		
68	36	44	68	68	81	77	96		
75	36	44	75	68	81	77	96		
82	14	17	82	67	80	75	94		
89	28	35	89	67	80	77	96		
101	26	32	101	65	77	77	96		