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A CURE RATE STUDY OF ISOTHERMALLY
AGED SYLGARD 182

G. L. Clink

DEVELOPMENT DIVISION

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P.O. 64-3655

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ABSTRACT

Isothermal aging of freshly prepared Sylgard 182 at 0, 21, 50, and 71 C, has been completed. Chemical analysis through NMR procedures, and hardness determinations through Shore durometer measurements, are reported herein for the four systems. Finalized data collation for determination of system kinetic parameters will be reported later.

EXPERIMENTAL

Weighed amounts of Sylgard 182 and Sylgard 182 catalyst, in a 10/1 ratio, were mixed for five minutes in a glass beaker with an air operated Teflon stirrer. The resulting mixture was immediately poured into 30 X 10 mm culture dishes, which were placed in a vacuum desiccator and evacuated for two minutes to remove the bulk of trapped air in the material. The samples were then placed, for aging, in controlled isothermal environments. Four isothermal systems were chosen, i.e., 0, 21, 50, and 71 C. At preselected elapsed times, three samples were removed for chemical analysis and for Shore durometer hardness measurements. Analysis of these samples was initiated immediately following removal from their isothermal environments.

Chemical analysis of the isothermally aged materials was effected through NMR analytical procedures. Weighed amounts of the samples, along with weighed amounts of high purity PETN (99.7%; used as the internal quantitative standard) were mixed with a 50/50 volume mixture of deuterated acetone/benzene, all contained in a Teflon septummed vial. The vials were placed on a Kahn mechanical shaker for several hours, until extraction of the unbound Sylgard was effectively complete. The sample was extracted with a disposable glass syringe (B-D) and placed in a 5 mm NMR sample tube. The sample was run on a Varian HA-100, using routine integration of the proton resonance peaks from PETN and DMPS (dimethylpolysiloxane), both of which are single peak species. The internal lock material was benzene. Durometer measurements were obtained using a Shore "A-2" unit.

Chemical analysis and durometer measurements were obtained in triplicate, using three samples for each elapsed time sampling interval.

Figs. 1 and 2 and Tables I and II present the results of chemical analysis and Shore durometer measurements, respectively, for the 0 C system, with a comparison of these two system properties presented in Fig. 3. The corresponding data for the 21, 50 and 71 C systems are presented in a temperature sequential manner, in Tables III through VIII and Figs. 4 through 12.

Comparison of the dimethylpolysiloxane retention of the four isothermal systems studied is presented in a semi-log plot of elapsed time with percent DMPS retained (Fig. 13). The graphic behavioral patterns are

quite similar for the 0, 50, and 71 C systems. Although the cure behavior pattern of the 21 C system is, in general, broadly similar to the other systems, there is a distinct deviation in its behavior in the cure onset portion of the curve (up to 60-80% retention). A corresponding semi-log comparison of the durometer measurements of the four systems is presented in Fig. 14.

In considering the four systems, at a common percent DMPS retention point, there should exist a linear relationship between the reciprocal temperature and the log of the elapsed time. An arbitrary DMPS retention of 50% was chosen, and the corresponding elapsed times for that temperature determined. The resulting relationships are presented in Fig. 15, a semi-log plot of the elapsed time versus the reciprocal temperature (K). The results show an obvious and substantial deviation of the 21 C system in its nonadherence to the expected linear relationship to the other members of the system. This deviation indicates a positive cycling of the isothermal conditions of the system, occurring in the initial stages of the aging. This behavior coincides with the relative non-adherence seen to occur in the systems comparison plot of elapsed time versus percent DMPS retained (Fig. 13). The isothermal conditions for the 21 C system were effected by the static 21 C environment maintained in the NMR Laboratory. Records show that a air conditioner malfunction did occur during this initial period of aging, and would explain the abnormal behavior indicated by the data from the 21 C system. Accordingly, the data obtained from this particular system are not valid for subsequent calculation of kinetic parameters for the Sylgard 182 system. However, the three system data groups obtained from the 0, 50 and 71 C studies are sufficient for this purpose.

COMMENTS

This essentially completes the data gathering portion of the cure rate study. Subsequent collation and computerized processing of data, for the determination of system kinetics parameters, will be performed by J. Holovka, Sandia Laboratories, Albuquerque New Mexico.

Fig. 1. Percent DMPS Retained (0°C)

Fig. 2. Sylgard 182 Durometer Readings (0 C)

Table I. Dimethylpolysiloxane (DMPS) Extractables (0 C)

Elapsed Time (hrs)	% DMPS Extracted			Average	σ	Percent Retained
	1	2	3			
0	99.8	99.8	99.9	99.8	0.1	(0.2)
153	70.3	70.5	70.4	70.4	0.1	29.6
333	64.3	64.7	64.7	64.6	0.2	35.2
501	61.2	61.0	61.3	61.2	0.2	38.6
671	58.2	58.4	58.5	58.4	0.2	41.4
837	48.8	49.1	49.0	49.0	0.2	50.8
1005	35.8	36.1	35.8	35.9	0.2	63.9
1053	29.5	29.4	29.7	29.5	0.2	70.3
1077	28.8	28.5	28.7	28.7	0.2	71.1
1101	26.1	26.2	25.9	26.1	0.2	73.7
1197	22.3	22.0	22.1	22.1	0.2	77.7
1269	19.2	19.5	19.4	19.4	0.2	80.4
1365	16.5	16.3	16.5	16.4	0.1	83.4
1413	14.2	14.2	14.4	14.3	0.1	85.5
1557	13.0	13.1	12.9	13.0	0.1	86.8
1677	11.4	11.5	11.4	11.4	0.2	88.4
1845	10.5	10.3	10.3	10.4	0.1	89.4

Table II. Sylgard 182 Durometer Readings (0 C)

Elapsed Time (hrs)	Readings			Average	σ
	1	2	3		
837	0	0	0	0	0
1005	1	2	1	1.3	0.6
1029	2	3	2	2.3	0.6
1053	4	4	5	4.3	0.6
1077	5	5	6	5.3	0.6
1101	6	7	6	6.3	0.6
1197	9	8	8	8.3	0.6
1225	10	10	9	9.7	0.6
1246	10	11	10	10.3	0.6
1269	11	11	10	10.7	0.6
1365	13	14	14	13.7	0.6
1389	14	14	15	14.3	0.6
1413	15	15	15	15.0	0.0
1557	18	18	18	18.0	0.0
1581	18	19	18	18.3	0.6
1605	19	19	18	18.7	0.6
1677	19	19	20	19.3	0.6
1749	19	19	19	19.0	0.0
1773	19	19	19	19.0	0.0
1845	19	20	19	19.3	0.6

Fig. 3. Comparison of Percent DMPS Retained with Durometer Reading (0 C)

Table III. Dimethylpolysiloxane (DMPS) Extractables (21 C)

Elapsed Time (hrs)	% DMPS Extracted			Average	σ	% Retained
	<u>1</u>	<u>2</u>	<u>3</u>			
0	99.8	99.8	99.9	99.8	0.06	0.2
8	82.1	82.3	82.1	82.1	0.12	17.9
22	62.4	62.6	62.3	62.4	0.15	37.6
45	47.3	47.1	47.3	47.2	0.12	52.8
69	36.2	36.1	36.3	36.2	0.10	63.8
75	32.6	32.5	32.6	32.6	0.06	67.4
93	19.8	20.0	20.1	20.0	0.15	80.0
141	15.3	15.4	15.4	15.4	0.06	84.6
189	12.6	12.5	12.6	12.6	0.06	87.4
213	12.1	12.1	12.3	12.2	0.12	87.8
285	10.8	10.6	10.8	10.7	0.12	89.3
309	10.7	10.8	10.8	10.8	0.06	89.2
357	10.6	10.4	10.6	10.5	0.12	89.5
381	10.2	10.4	10.2	10.3	0.12	89.7
453	10.0	9.8	10.2	10.0	0.20	90.0
501	9.8	9.7	9.8	9.8	0.06	90.2
621	9.7	9.7	9.8	9.7	0.06	90.3
717	9.7	9.6	9.7	9.7	0.06	90.3
885	9.7	9.5	9.6	9.6	0.10	90.4
1056	9.6	9.5	9.4	9.5	0.10	90.5
1293	9.5	9.4	9.4	9.4	0.06	90.6

Table IV. Sylgard 182 Durometer Readings (21 C)

<u>Elapsed Time</u> <u>(hrs)</u>	<u>Readings</u>			<u>Average</u>	<u>σ</u>
	<u>1</u>	<u>2</u>	<u>3</u>		
0	0	0	0	0	0
21	0	0	0	0	0
45	0	0	0	0	0
69	0	0	0	0	0
75	0	0	0	0	0
93	8	9	8	8.3	0.6
117	21	21	21	21.0	0
141	30	30	31	30.3	0.6
189	36	36	36	36.0	0
213	39	39	40	39.3	0.6
285	42	42	43	42.3	0.6
309	44	43	44	43.7	0.6
357	45	46	46	45.7	0.6
453	47	46	46	46.3	0.6
477	49	46	47	47.3	1.5
501	47	48	47	47.3	0.6
525	48	47	47	47.3	0.6
621	47	47	49	47.7	1.2
717	47	48	47	47.3	0.6
813	48	48	47	47.7	0.6
957	47	48	47	47.3	0.6
1005	49	48	47	48.0	1.0
1173	49	47	47	47.7	1.2
1293	47	47	48	47.3	0.6

Table V. Dimethylpolysiloxane (DMPS) Extractables (50 C)

Elapsed Time (hrs)	% DMPS Extracted			Average	σ	% Retained
	<u>1</u>	<u>2</u>	<u>3</u>			
0	99.8	99.8	99.7	99.8	0.06	0.2
1.5	86.8	86.9	86.6	86.8	0.15	13.2
3.5	84.9	84.8	84.8	84.8	0.06	15.2
5.0	74.4	74.3	74.1	74.3	0.15	25.7
6.0	68.1	68.3	68.3	68.2	0.12	31.8
7.0	18.9	19.1	18.9	19.0	0.12	81.0
16.5	7.9	8.1	7.9	8.0	0.12	92.0
21.0	5.5	5.7	5.7	5.6	0.12	94.4
24.0	5.2	5.4	5.2	5.3	0.12	94.7
27.0	4.5	4.3	4.3	4.4	0.12	95.6
45.0	4.1	4.0	4.1	4.1	0.06	95.9
69.0	3.9	3.9	3.9	3.9	0.00	96.1
75.0	4.0	3.9	3.8	3.9	0.10	96.1
141.0	3.9	3.9	3.8	3.9	0.06	96.1

Table VI. Sylgard 182 Durometer Readings (50 C)

<u>Elapsed Time</u> <u>(hrs)</u>	<u>Readings</u>			<u>Average</u>	<u>σ</u>
	<u>1</u>	<u>2</u>	<u>3</u>		
0	0	0	0	0	0
1.5	0	0	0	0	0
3.5	0	0	0	0	0
5.0	0	0	0	0	0
6.0	0	0	0	0	0
7.0	13	13	13	13.0	0
16.5	27	25	27	26.3	1.2
21.0	32	32	30	31.3	1.2
24.0	37	36	36	36.3	0.6
27.0	39	40	38	39.0	1.0
45.0	44	45	44	44.3	0.6
69.0	45	47	45	45.7	1.2
75.0	46	46	45	45.7	0.6
141.0	48	47	46	47.0	1.0

Table VII. Dimethylpolysiloxane (DMPS) Extractables (71 C)

Elapsed Time (hrs)	% DMPS Extracted			Average	σ	% Retained
	<u>1</u>	<u>2</u>	<u>3</u>			
0	99.9	99.7	99.9	99.8	0.12	0.2
0.17	91.7	91.9	91.6	91.7	0.15	8.3
0.33	87.5	87.5	87.7	87.6	0.12	12.2
0.50	85.2	85.4	85.4	85.3	0.12	14.5
0.67	79.9	80.1	79.8	79.9	0.15	19.9
0.92	74.9	74.9	74.7	74.8	0.12	25.0
1.50	34.0	33.8	34.0	33.9	0.12	65.9
2.08	8.9	9.0	8.8	8.9	0.10	90.9
2.33	6.9	6.8	6.8	6.8	0.06	93.0
2.67	6.6	6.6	6.7	6.6	0.06	93.2
3.58	5.0	5.1	4.9	5.0	0.10	94.8
6.00	4.8	4.8	4.9	4.8	0.06	95.0
24.00	4.6	4.5	4.4	4.5	0.10	95.3

Table VIII. Sylgard 182 Durometer Readings (71 C)

<u>Elapsed Time</u> <u>(hrs)</u>	<u>Readings</u>			<u>Average</u>	<u>σ</u>
	<u>1</u>	<u>2</u>	<u>3</u>		
0	0	0	0	0	0
1.17	0	0	0	0	0
0.33	0	0	0	0	0
0.50	0	0	0	0	0
0.67	0	0	0	0	0
0.92	0	0	0	0	0
1.50	0	0	0	0	0
1.75	0	0	0	0	0
2.08	7	7	6	6.7	0.6
2.33	16	17	16	16.3	0.6
2.67	27	26	26	26.3	0.6
3.57	35	35	34	34.7	0.6
4.60	40	41	40	40.7	0.6
6.00	42	44	43	43.0	1.0
7.00	44	44	44	44.0	0
8.00	45	45	46	45.7	0.6
24.00	47	47	47	47.0	0

Fig. 4. Percent DMPS Retained (21 C)

Fig. 5. Sylgard 182 Durometer Readings (21 C)

Fig. 6. Comparison of Percent DMPS Retained with Durometer Readings (21 C)

Fig. 7. Percent DMPS Retained (50 C)

Fig. 8. Sylgard 182 Durometer Readings (50 C)

Fig. 9. Comparison of Percent DMPS Retained with Durometer Readings (50 C)

Fig. 10. Percent DMPS Retained (71 C)

Fig. 11. Svlgard 182 Durometer Readings (71 C)

Fig. 12. Comparison of Percent DMPS Retained with Durometer Reading (71 C)

Fig. 13. Isothermal Systems Comparison (DMPS Retention)

Fig. 14. Isothermal Systems Comparison (Durometer Hardness)

Fig. 15. Elapsed Time with Temperature at 50% DMPS Retention