A CURE RATE STUDY OF ISOTHERMALLY
AGED SYLGARD 182
(AN ADDENDUM)

G. L. Clink

DEVELOPMENT DIVISION

OCTOBER - DECEMBER 1975

MASTER

Normal Process Development
Endcavor No. 102

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ABSTRACT

Isothermal aging of freshly prepared Sylgard 182 at 21 C has been completed. Chemical analysis through NMR procedures, and hardness determinations through Shore durometer measurements are reported herein.

EXPERIMENTAL

The Sylgard 182 cure study had been previously completed for four isothermal systems; 0, 21, 50, and 71 C(1). But cure behavioral patterns and environmental data obtained later (relative to the 21 C system) strongly indicated that appreciable temperature cycling occurred during the aging process. The 21 C system cure has been repeated and reported herein.

Weighed amounts of Sylgard 182 resin 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. These samples were then placed, for aging, in a controlled isothermal environment (21 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 environment.

Chemical analysis of the isothermally aged material 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 benzene/deuterated acetone, 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. A sufficient amount of the sample solution was extracted with a B-D disposable glass syringe and placed in a standard 5 mm NMR sample tube. The sample was run on a Varian HA-100, using routine integration of the proton resonance peaks of PETN and dimethylpolysiloxane, both of which are single peak species. The internal lock material was benzene, integrating in field sweep mode. Durometer measurements were obtained using a Shore "A-2" unit. Chemical analyses and durometer measurements were obtained in triplicate, using three samples for each elapsed time sampling interval. The experimental results of NMR analyses and durometer measurements are presented in Fig. 1 and Table I, and in Fig. 2 and Table II, respectively.

Graphic comparison of the chemical and hardness data is shown in Fig. 3, and shows no behavior pattern anomalies from previous studies, except the 21 C system previously studied(1), which experienced thermal cycling.

(1) G. L. Clink, *A Cure Rate Study of Isothermally Aged Sylgard 182*, January - March and April - June 1975.

A semi-log plot of elapsed time versus reciprocal temperature (Fig. 4) at a data-inferred 50% DMPS retention point for all four systems (0, 21, 50, 71 C), shows a near linear adherence for the data obtained previously in the 0, 50, and 71 C systems(1), and that obtained in this present run of the 21 C system. The 50% retention point obtained from the original (thermally cycled) 21 C system is presented for comparison of its non-linear adherence. Based on 50% DMPS retention at approximately 40 hours, the original 21 C system isothermally would have effectively experienced an aging temperature of approximately 32 C. Near adherence of the rerun 21 C system data to the experimental curve would indicate its potentially valid use in subsequent kinetics calculations.

COMMENTS

This addendum should complete the data gathering portion of the Sylgard 182 cure study. Determination of system kinetics parameters will be completed by J. Holovka, Sandia Laboratories, Albuquerque, New Mexico.

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Fig. 1. Percent Sylgard 182 Retained (21 C)

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

Elapsed Time (hrs)	Percent DMPS Extracted			Average	σ	Percent Retained
	1	2	3			
0.0	100.0	99.9	99.9	99.9	-	0.0
0.5	99.7	99.4	99.4	99.5	0.2	0.4
6.5	97.8	97.1	97.4	97.4	0.4	2.5
24.5	94.6	94.8	94.8	94.7	0.1	5.2
49.0	90.1	90.0	90.3	90.1	0.1	9.8
73.0	84.2	84.1	84.1	84.1	0.1	15.8
145.0	61.5	61.8	61.6	61.7	0.2	38.2
169.0	37.3	37.2	37.4	37.3	0.1	62.6
193.0	26.8	27.1	27.1	27.0	0.2	72.9
313.0	8.8	9.0	8.9	8.9	0.1	91.0
361.0	7.7	7.9	7.9	7.8	0.1	92.1
409.0	6.9	6.8	6.8	6.8	0.1	93.1
481.0	5.9	5.8	5.8	5.8	0.1	94.1

Fig. 2. Sylgard 182 Durometer Readings (21 C)

Table II. Sylgard 182 Durometer Readings (21 C)

Elapsed Time (hrs)	Readings			Average	σ
	1	2	3		
0.5	0	0	0	0	-
6.5	0	0	0	0	-
24.5	0	0	0	0	-
49.0	0	0	0	0	-
73.0	0	0	0	0	-
145.0	0	0	0	0	-
169.0	0	0	0	0	-
193.0	0	0	0	0	-
199.0	0	0	0	0	-
313.0	17	18	18	17.7	0.6
337.0	21	20	19	20.0	1.0
361.0	23	23	24	23.3	0.6
385.0	25	25	25	25.0	0.0
409.0	26	26	26	26.0	0.0
481.0	29	30	29	29.3	0.6
577.0	33	33	32	32.7	0.6
659.0	35	34	34	34.3	0.6
673.0	35	35	34	34.7	0.6

Fig. 3. Comparison of Percent DMPS Retained with Durometer Readings (21 C)

Fig. 4. Elapsed Time with Temperature at 50% DMP5 Retained