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ASTM D395 Short-Term Compression Set of Solid (Non-Porous) Siloxanes: SE 1700, Sylgard 184, and #New# M9787

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Disclaimer

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SUMMARY

Compression set of solid (non-porous) Dow Corning SE 1700, Sylgard 184, and "new" M9787 siloxane elastomers was measured according to ASTM D395 Method B. Specimens of SE 1700 were made using (1) the manufacturer's suggested cure of 150°C for 30 min and (2) an extended cure of 60°C for 6 h and 150°C for 1 h followed by a post-cure under nitrogen purge at 125°C for 12 h. Four specimens of each material were aged at 25-27% compressive strain at 70°C under nitrogen purge for 70 h. Final thickness of each specimen was measured after a 30-min cooling/relaxation period, and compression set relative to deflection was calculated. The average compression set relative to deflection was 6.0% for SE 1700 made using the extended cure and post-cure, 11.3% for SE 1700 made using the manufacturer's suggested cure, 12.1% for Sylgard 184, and 1.9% for M9787. The extended cure and post-cure reduced the amount of compression set in SE 1700.

MATERIALS AND METHODS

The Dow Corning SE 1700 polydimethylsiloxane clear adhesive consisted of base and catalyst components in a 10:1 mix ratio (Lot # 0007749846). The two components were mixed in a planetary centrifugal mixer (SpeedMixer DAC 150 FVZ-K, FlackTek Inc.) at atmospheric pressure for 30 s at 1600 rpm, and then again for 30 s at 2400 rpm. The mixture was transferred into a four-cavity button mold (ATSM D395, Desenco). The mold was clamped in a hot press (Carver) at 5000 lbs with the platens at room temperature. Two sets of specimens were made using different cure profiles: (1) manufacturer's suggested cure and (2) extended cure. For the manufacturer's suggested cure, the platens were heated to 150°C and held there for 30 min. For the extended cure specimens, the platens were heated to 60°C, held there for 6 h, then heated to150°C and held there for 1 h. The platens were then turned off and, after cooling, the mold was removed from the press and the SE 1700 button specimens were removed from the mold (manufacturer's suggested cure sample ID: 150205WS01, extended cure sample ID: 150311WS01). Each specimen was ~29 mm in diameter and ~13 mm thick. The extended cure specimens were finally post-cured under nitrogen purge at 125°C for 12 h.

The Dow Corning Sylgard 184 polydimethylsiloxane elastomer consisted of base and catalyst components in a 10:1 mix ratio (Lot # 0006927366). The two components were mixed in a planetary centrifugal mixer (SpeedMixer DAC 150 FVZ-K, FlackTek Inc.) at atmospheric pressure for 30 s at 2400 rpm. The mixture was transferred into a four-cavity button mold (ATSM D395, Desenco). The mold was clamped in a hot press (Carver) at 5000 lbs with the platens at room temperature. Per the manufacturer's curing instructions, the platens were heated to 150°C and held there for 10 min. The platens were then turned off and, after cooling, the mold was removed from the press and the Sylgard 184 button specimens were removed from the mold (sample ID: 150225WS01). Each specimen was ~29 mm in diameter and ~13 mm thick.

The "new" M9787 consisted of bin aged and devolatilized base material (produced by NuSil in Bakersfield, CA) and 0.47 phr Esperox 497M catalyst (as per KCP material specification 4155869). The

base material and catalyst were provided by KCP (Tom Robison). The two components were mixed in a two-roll mill for 10 min at the LLNL B231 Plastics Facility. Discs were cut from the milled sheet and stacked in a four-cavity button mold (ASTM D395, Desenco) preheated to 250°F (121°C). After heating under compression for 2 h at 250°F (121°C), the cured specimens (~28 mm in diameter and ~13 mm thick) were removed from the mold and post-cured under nitrogen purge at 400°F (204°C) for 24 h (as per KCP material specification 4003043-4003045). The post-cure resulted in darkening of the material (Fig. 1).

Compression set was determined in accordance with ASTM D395. The specimens were placed in a stainless steel compression fixture (WTF-RC-34, Wyoming Test Fixtures, Inc.) with 9.51 mm spacers, providing 25-27% compressive strain (Fig. 2). No lubrication was used on the polished steel plates. The assembly was immediately transferred to a pre-heated oven at 70°C with nitrogen purge in B132S R2775. After 70 h, the assembly was removed from the oven and the specimens were immediately removed from the fixture. After a 30-min cooling/relaxation period, the final thickness of each specimen was measured using digital calipers (293-811, Mitutoyo) and the compression set relative to deflection was calculated:

$$C_s = \frac{t_o - t_f}{t_o - t_s} \times 100 = \frac{t_o - t_f}{\varepsilon t_o} \times 100 \quad (1)$$

where t_o is the original thickness, t_f is the final thickness, t_s is the spacer height, and ε is the compressive strain.

RESULTS AND DISCUSSION

Results for each specimen are shown in Table 1 and Fig. 3. The average compression set relative to deflection was 6.0±0.1% for SE 1700 made using the extended cure and post-cure, 11.3±0.2% for SE 1700 made using the manufacturer's suggested cure, 12.1±0.1% for Sylgard 184, and 1.9±0.7% for the M9787. The extended cure and post-cure reduced the amount of compression set in SE 1700. This result underscores the importance of determining an appropriate cure profile for any material in which compression set should be minimized.

Interestingly, the compression set of porous structures made from the SE 1700 and M9787 does not correspond to the compression set of the solid materials measured in this study. After 1 month at 70°C under 25-30% compression, an SE 1700 face-centered tetragonal (FCT) ordered structure with ~50% porosity made by an additive direct ink write (DIW) process exhibited 12% relative compression set, while an M9787 stochastic foam of ~63% porosity (known as M9763 cellular silicone) exhibited 25% relative compression set (results from previous studies at LLNL). The DIW SE 1700 porous structure was cured at 150°C for 1 h (no post-cure) and the M9763 presumably was cured as described earlier according to the KCP material specification. The compression set of the ordered DIW SE 1700 porous structure is lower than that of the stochastic M9787 foam despite the higher compression set of the solid SE 1700 material measured in this study. This observation warrants further investigation of the effect of material architecture (including porosity and pore distribution) on compression set.

ACKNOWLEDGMENTS

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Table 1: Compression Set Relative to Deflection (Aged at 70°C for 70 h)

Material	Specimen #	Original Thickness (mm)	Compressive Strain (%)	Final Thickness[(a)] (mm)	Relative Compr. Set (%)
SE 1700 (extended cure and post-cure)	1	12.971	26.7	12.761	6.1
	2	12.983	26.8	12.777	5.9
	3	12.967	26.7	12.764	5.9
	4	12.949	26.6	12.744	6.0
	Avg±SD				6.0±0.1
SE 1700 (mfr suggested cure)	1	12.809	25.8	12.444	11.1
	2	12.815	25.8	12.434	11.5
	3	12.817	25.8	12.444	11.3
	4	12.805	25.7	12.436	11.2
	Avg±SD				11.3±0.2
Sylgard 184 (mfr suggested cure)	1	12.823	25.8	12.423	12.1
	2	12.831	25.9	12.426	12.2
	3	12.794	25.7	12.392	12.2
	4	12.796	25.7	12.400	12.1
	Avg±SD				12.1±0.1
M9787 (KCP cure and post-cure)	1	12.641	24.8	12.579	2.0
	2	12.676	25.0	12.587	2.8
	3	12.641	24.8	12.603	1.2
	4	12.678	25.0	12.632	1.5
	Avg±SD				1.9±0.7

[(a)]Measured after 30-min cooling/relaxation period

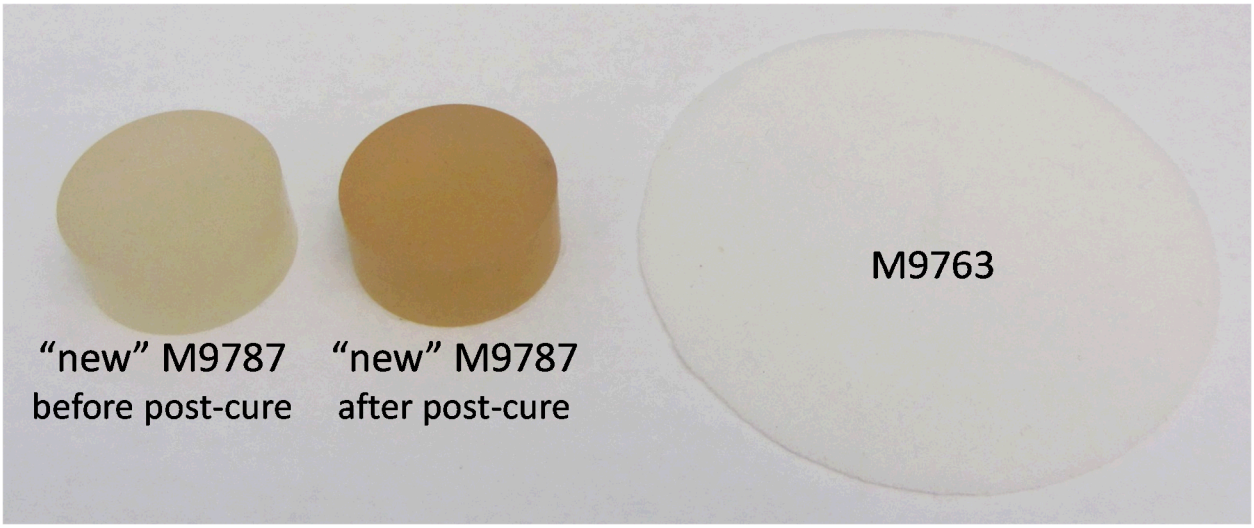

Fig. 1. "New" M9787 (non-porous) button specimens before and after post-curing at 400°F (204°C) for 24 h. A piece of legacy M9763 cellular silicone is also shown for comparison.

Fig. 2. SE 1700 specimens in the ASTM D395 compression fixture.

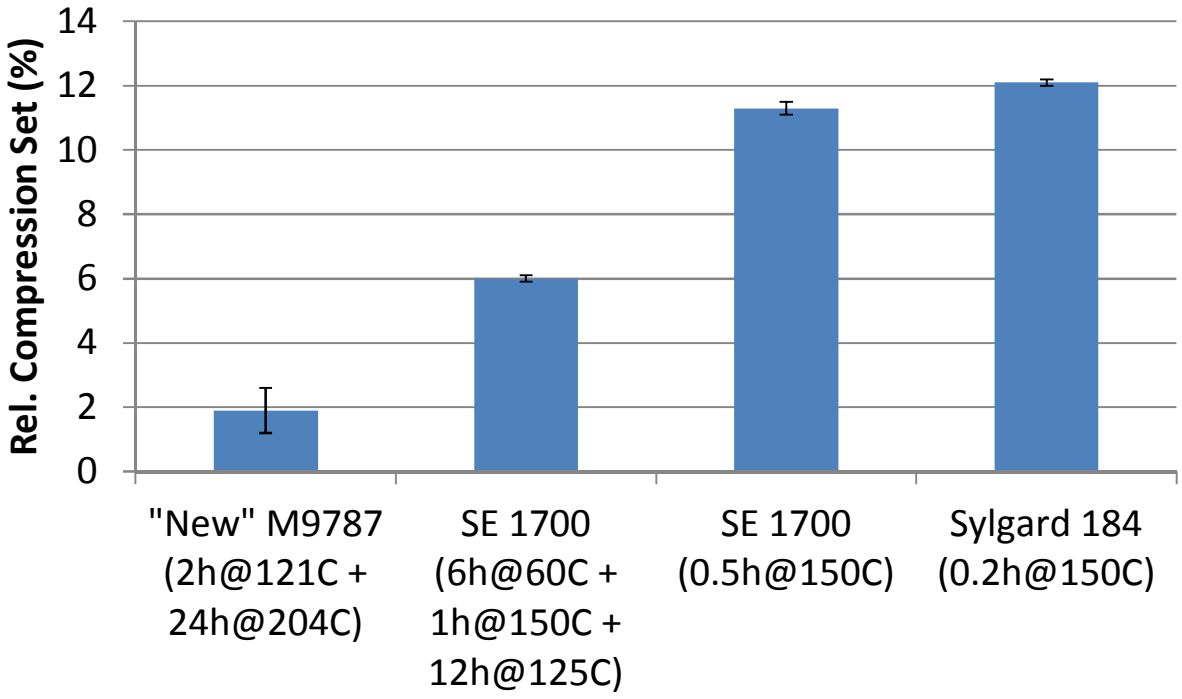

Fig. 3. Compression set relative to deflection following 25-27% compression at 70°C for 70 h. Cure profiles for each material are shown.