Heat Cured ONE PART™ Liquid Silicone Rubber

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Abstract

Heat cured liquid silicone rubbers are commercially available as two part systems. These parts need to be precisely mixed to produce consistent product. This requires controlled pumping and mixing equipment. Once mixed, these materials have short pot lives that are inversely related to temperature. Quality assurance of these silicones is dependent upon mixing ratios and additives.

This paper outlines the results of testing a newly developed one component heat cured liquid silicone rubber system. This silicone system is able to alleviate several of the problems associated with two part silicone rubbers. The ONE PART™ system is a material that can be fully compounded in one container, eliminating the need for a controlled mixing system. This ONE PART™ system is thermally stable during storage or transport, even at elevated temperatures. Since this material is custom mixed prior to shipping, the material is uniform throughout. The quality assurance testing performed on one portion of this material is representative of the entire lot of material.

Introduction

Heat cured liquid silicone rubbers are well known as two component systems. These systems contain a catalyst and a crosslinking agent separated into "A" and "B" parts. The catalyst, which is typically platinum, is mixed into part A and a crosslinking agent is mixed into part B. This system provides a fast curing, strong silicone that is used in a variety of industries, including the food and medical industries.

The "A" and "B" subparts are pumped in a 1:1 ratio via a controlled pumping system to the injection molding machine. Colorants are injected just prior to mixing at less than 5% of the total mass. The ratios must be kept constant to keep the properties and color of the finished product consistent. Variations in ratio and in additives contribute to quality assurance problems. Quality assurance testing of two part systems may not produce repeatable results due to variations in the mixing ratios. The injection molding machine for the silicone requires cooling of the barrel so that the mixed silicone does not cure prematurely.

The machine also needs to be cleaned, purged or dismantled at shutdown, especially if not in use for extended periods. This is done to prevent the silicone from curing inside the machine components. The cleaning process is time and resource intensive. Purging, typically done with one component of the silicone, leads to an off ratio of inventory and waste due to the extra use of one side of the two part system. Dismantling the components of the machine containing the mixed silicone to chill or freeze takes time and requires a cooling facility. Unless chilled, the typical pot life of mixed two part silicone rubber systems is three days or less.

A ONE PART™ liquid silicone rubber system has been developed which provides similar physical properties, similar viscosities, excellent thermal stability and a much simplified pumping requirement. This system does not require the molding machine to be purged or broken down for shutdown purposes. As a ONE PART™ system, the waste and off inventory material associated with purging the mixed two part streams are eliminated. The injection molding machine barrels may no longer need to be cooled to prevent onset of cure; in fact, it may provide the ability to heat the injection barrels to speed the cure time.

These ONE PART™ liquid silicone materials can also be custom formulated to fit a variety of customer requirements. It can be manufactured ready to mold or ready to color. They can be tested prior to shipment or use, providing accurate physical properties for the entire lot of material.

Physical Properties

The physical properties of these ONE PART™ liquid silicone systems are comparable to two part liquid silicone systems. Table 1 shows test values at press cure, 10 minutes at 171°C, and after a 4 hour at 200°C post cure. The materials tested were three commercially available 2 part 40 durometer general purpose liquid silicone rubbers (**A+B mixed 1:1**) and for a 40 durometer ONE PART™ silicone rubber (**One Comp**) using this newly developed system. The physical properties of these two very different systems are similar, particularly after post cure. This table also shows that there are differences between commercially available silicones.

Table 1: Physical Properties Comparison of 40 Durometer 2-part systems and a 1-part system

Materials	Physical Properties			
	Durometer	Tensile MPa (psi)	Elongation (%)	Tear kN/m (ppi)
Press Cured (10 minutes at 171°C)				
One Comp	**38.3**	**6.9 (1009)**	**536**	**55.5 (317)**
LSR #1 A+B	39.9	9.0 (1311)	573	19.3 (110)
LSR #2 A+B	38.1	9.2 (1329)	631	60.1 (343)
LSR #3 A+B	44.0	9.0 (1302)	585	40.1 (229)
Post Cured (4 hours at 200°C)				
One Comp	**40.1**	**8.1 (1181)**	**572**	**32.6 (186)**
LSR #1 A+B	40.2	9.2 (1341)	533	23.1 (132)
LSR #2 A+B	41.4	8.0 (1159)	516	36.1 (206)
LSR #3 A+B	46.2	9.3 (1353)	528	40.5 (231)

Data acquired after heat aging these materials evidences their similarities in terms of physical properties. These properties are listed in Table 2. The one component system has significantly less change in heat age tensile and elongation as compared to the two part systems.

Table 2: Physical Properties after Heat Aging for 70 hrs @ 225°C

Materials	Physical Properties			
	Durometer	Tensile MPa (psi)	Elongation (%)	Tear kN/m (ppi)
Press Cured				
One Comp	**46.9**	**8.2 (1196)**	**412**	**33.8 (193)**
LSR #1 A+B	40.6	6.9 (1006)	374	22.8 (130)
LSR #2 A+B	45.1	5.3 (768)	236	34.3 (196)
LSR #3 A+B	46.7	6.7 (976)	318	28.5 (163)
Post Cured				
One Comp	**46.2**	**8.3 (1210)**	**410**	**33.4 (191)**
LSR #1 A+B	40.9	6.9 (1001)	362	20.3 (116)
LSR #2 A+B	45.7	4.8 (694)	231	34.0 (194)
LSR #3 A+B	47.1	7.1 (1034)	342	28.0 (160)

The compression sets of these same materials are listed in Table 3. The one component silicone has good compression set at the press cure and at the post cure. Only one of the three commercially available liquid silicones has better set at press cure, while two had better post cure compression sets.

Table 3: Press Cure and Post Cure Compression Set

Materials	Compression Set (%) (22 hours at 177°C)	
	Press Cured	Post Cured
One Comp 40	**24**	**18**
LSR #1 A+B	56	38
LSR #2 A+B	46	11
LSR #3 A+B	16	11

Rheological Properties

Cure data for these materials, obtained using an MDR2000 rheometer, show that two part systems cure faster than this ONE PART™ liquid silicone rubber system, as anticipated. The ONE PART™ system is a more robust system providing a longer scorch time yet relatively fast cure times. The comparable cure times are shown in Figure 1 and Table 4.

Table 4: Scorch and Cure Data for the ONE PART™ and Two Part Silicones

Material	Rheometer Data at 171°C		
	Scorch Time TS1	Cure Time TC10 (sec)	Cure Time TC90
One Comp	**34**	**32**	**77**
LSR #1 A/B	9	8	13
LSR #2 A/B	10	9	28
LSR #3 A/B	9	8	16

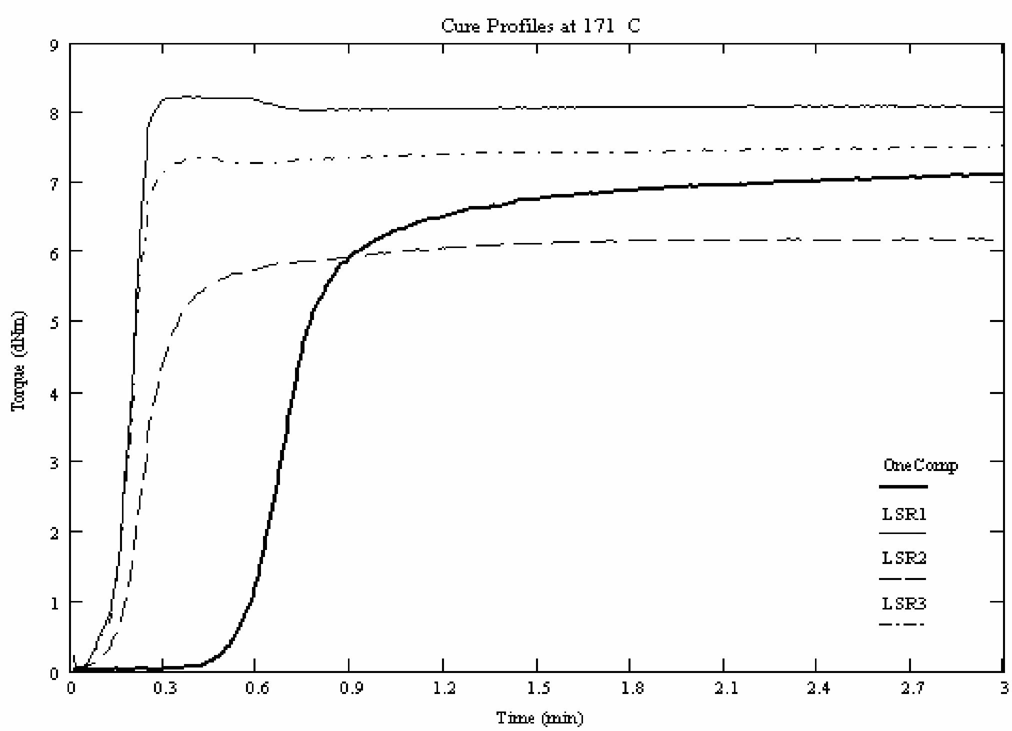

Figure 1: The Rheometer Curing of Liquid Silicones at 171°C (340°F)

Storage and Thermal Stability

Any liquid system must also be thermally stable for storage and for transportation. This ONE PART™ system is thermally stable over extended periods in storage. A test was conducted to demonstrate stability. Data was gathered over six months while storing the material at warehouse conditions. The physical properties of the material prior to storage were nearly indistinguishable from the properties after six months. This data is shown in Table 5 and graphically represented in Figure 2. Repeat analyses are being conducted.

Table 5: Storage Test Physical Properties

Materials	Physical Properties			
	Durometer	Tensile MPa (psi)	Elongation (%)	Tear kN/m (ppi)
Before Test	63.7	9.2 (1338)	382	49.0 (280)
After Six Months	66.3	9.0 (1298)	344	48.5 (277)

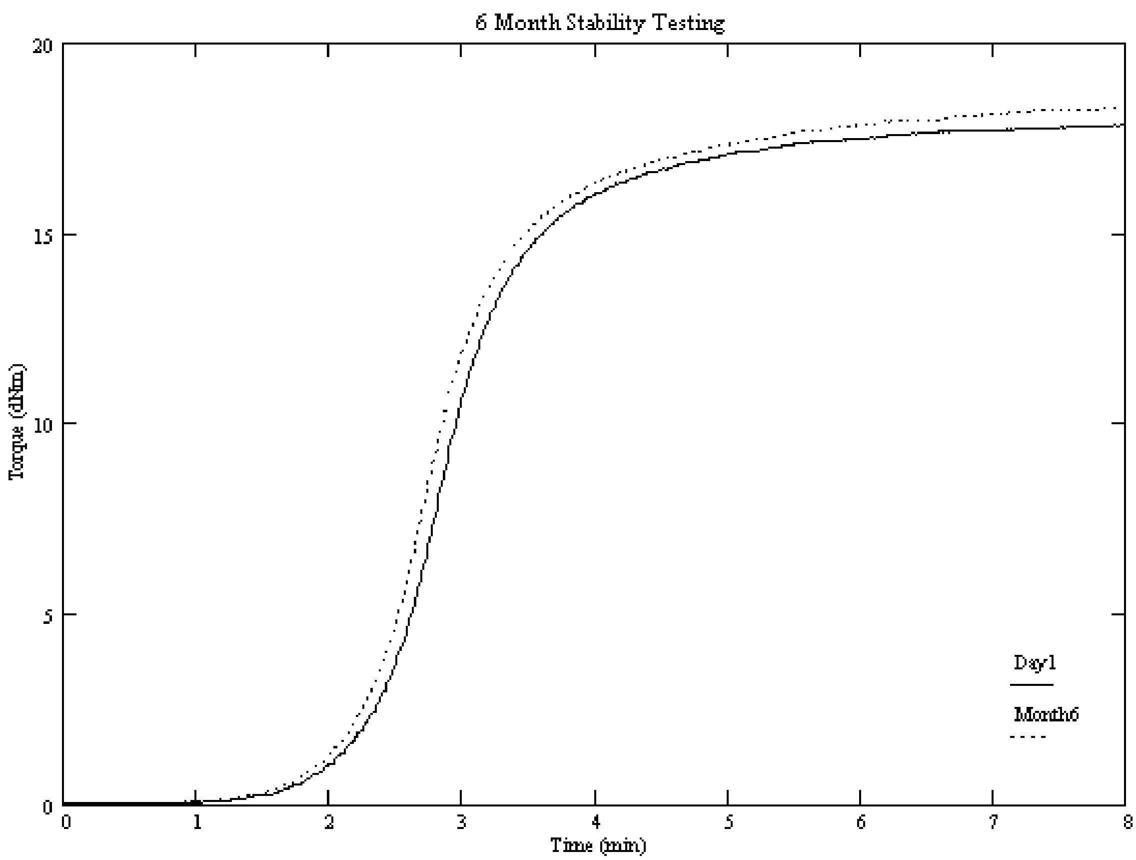

Figure 2: The results of a 6 month storage test.

A test was conducted to show thermal stability at elevated temperatures for extended periods. Samples were placed in an oven at 71°C (160°F) and rheometer samples were run daily. Figure 3 and Table 6 show the results and list the physical properties before the test and after one week in the oven. The standard ONE PART™ system is stable at temperatures of 71°C (160°F) for at least seven days. This suggests that the material will be stable for transport.

Table 6: Physical properties of Thermal Stability Test

Materials	Physical Properties			
	Durometer	Tensile MPa (psi)	Elongation (%)	Tear kN/m (ppi)
Before Test	56.2	9.2 (1338)	399	44.5 (254)
After one week	58.6	9.6 (1390)	408	50.1 (286)

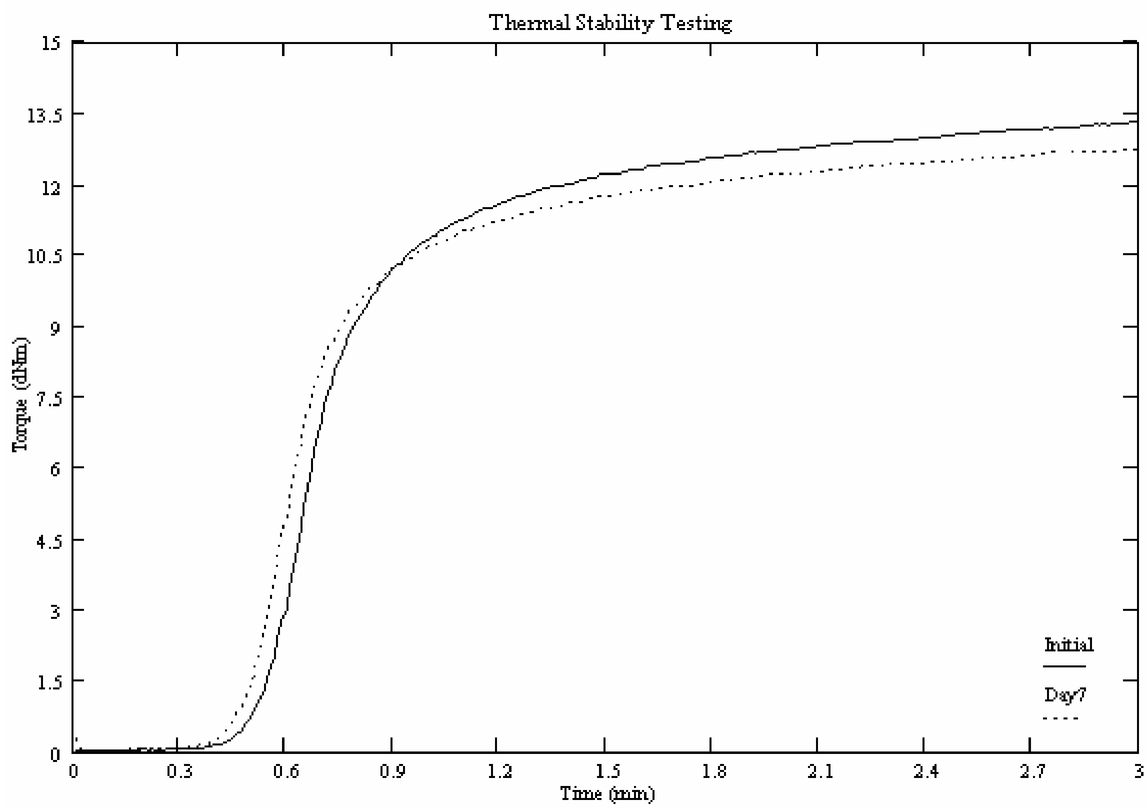

Figure 3: Rheometer Curves of material before and after 1 week at 71°C (160°F).

A test was performed by placing material samples of the ONE PART™ system into an oven to preheat for twenty minutes. The preheated samples were then transferred to the rheometer for cure. The preheat temperatures used were 100°C, 115°C and 130°C. Table 7 shows scorch and cure data for the preheated samples. As predicted, the cure begins sooner on the preheated samples. Figure 4 graphically shows the results. These results suggest that the barrels of the injection molding machines could be preheated to reduce the mold and material heat-up and cure time.

Table 7: Cure Data on Preheated ONE PART™ Silicone

Preheat Temperature	Rheometer Data		
	Scorch Time TS1 (sec)	Cure Time TC10 (sec)	Cure Time TC90 (sec)
23°C (73°F)	33	31	93
100°C (212°F)	30	28	83
115°C (240°F)	27	25	75
130°C (266°F)	22	20	61

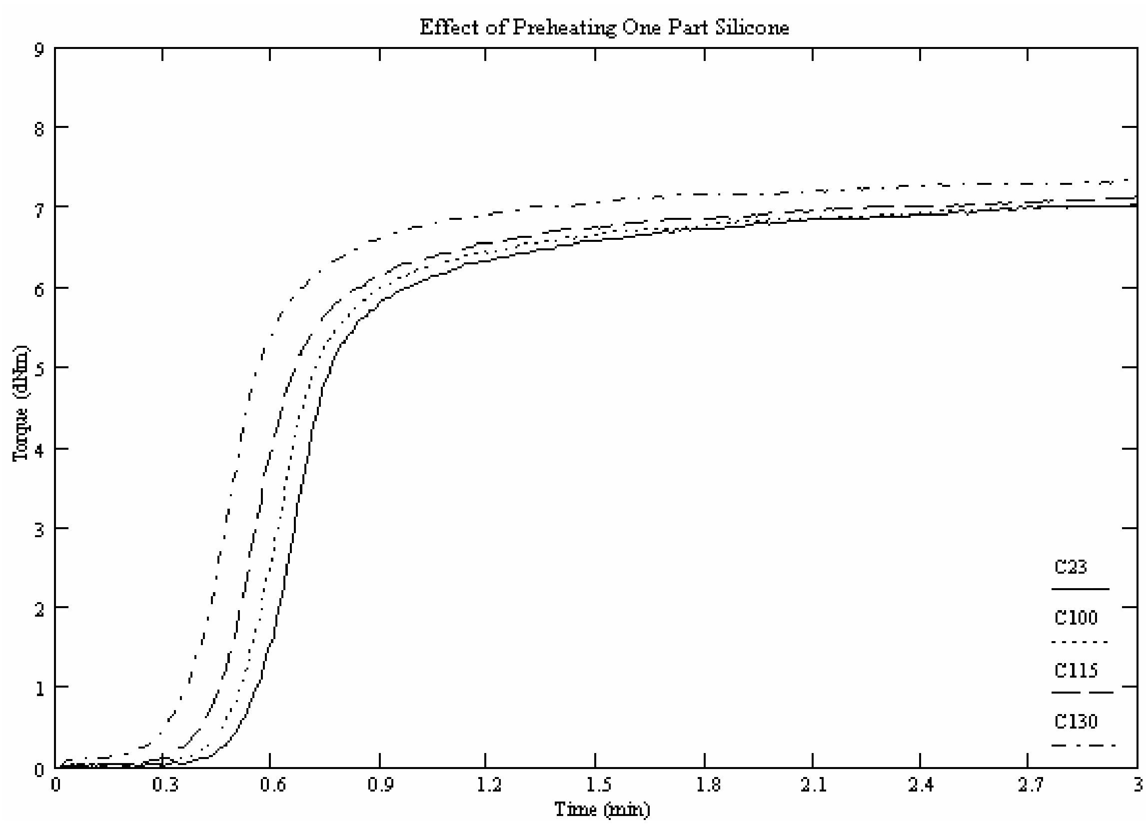

Figure 4: Rheometer Curves of ONE PART™ liquid silicone at various initial temperatures

Formulations of this ONE PART™ heat cured liquid silicone are possible that meet 21 CFR, 177.2600 for repeated food contact. These formulations were tested and will pass the specifications of the tests.

Conclusion

Based on the data collected and the analysis performed, a ONE PART™ liquid silicone rubber system is a viable alternative to the two part liquid silicone systems utilized in several markets. This ONE PART™ system provides similar physical properties and can heat age as well, or better than current two part systems.

This ONE PART™ liquid silicone system is chemically stable over six months without any degradation. It has been shown that the materials are thermally stable for extended periods at elevated temperatures. Although the temperature of the test performed did not cycle diurnally as it would during transportation, it does show the temperature stability of this ONE PART™ system.

Due to the heat stability of this uncured system, the barrel of the injection machine may not have to be cooled. The barrel may be able to be heated to assist in the preheating of the ONE PART™ silicone prior to injection into the mold. This preheating will help offset cure time lags that the ONE PART™ system shows when compared to the two part systems. Further testing in injection molding machines needs to be scheduled to verify these assertions.

The silicone in this ONE PART™ system can be formulated and mixed to color and specifications. The fluid handling equipment necessary to run this silicone system is greatly reduced. The controls necessary to monitor mixing and color injecting can be removed or simplified. The material can be tested prior to shipment to assure quality.

Patent pending