Controlled volatility silicone materials: development and analysis of ultra low outgassing™ elastomer for space

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Presented at SAMPE 2009,
May 19-21 in Baltimore, MD.

New silicone materials that aim to limit and reduce the potential for contamination while maintaining essential physical and chemical characteristics for specialized space applications are continuously being developed and evaluated. Several agencies, such as NASA, historically recommend < 1.0 % Total Mass Loss (TML) and < 0.1% Collected Volatile Condensable Material (CVCM) as a screening level for the acceptance or rejection of a material for space applications.

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ABSTRACT

New silicone materials that aim to limit and reduce the potential for contamination while maintaining essential physical and chemical characteristics for specialized space applications are continuously being developed and evaluated. Several agencies, such as NASA, historically recommend < 1.0 % Total Mass Loss (TML) and < 0.1% Collected Volatile Condensable Material (CVCM) as a screening level for the acceptance or rejection of a material for space applications. ASTM E 1559 is an additional test method used to characterize materials by monitoring the outgassing kinetics and identifying the volatile components of the material. In this paper, we compare a standard controlled volatility silica reinforced silicone adhesive that has a history of use in aerospace applications with a newly developed equivalent Ultra Low OutgassingTM version that exceeds typical ASTM E 595 requirements achieving < 0.1 % TMLs and < 0.01% CVCMs. We will compare the cured physical properties and monitor the outgassing profiles of each material based on ASTM E 1559 test method. We will also examine how different reinforcing and functional fillers can be incorporated into silicone materials while continuing to achieve low outgassing characteristics.

1. INTRODUCTION

1.1 Purpose

The purpose of this report is to demonstrate the ultra-low outgassing characteristics of SCV-2585, a newly developed silicone elastomer with specific low modulus characteristics – similar to CV-2289 – that also reduces the potential for contamination by meeting Ultra Low OutgassingTM requirements.

1.2 Situation Background and Overview of Contents

With the large amounts of adhesive/sealants currently used on a spacecraft, manufacturers must closely monitor their cumulative contaminant levels. By having materials with lower outgas levels, manufacturers would have the ability to use more material when necessary for mechanical reasons, and have the ability to choose material from a broader material range. Materials with lower TML and CVCM values could create more efficient solar cells. Lower outgassing would produce lower contamination and extend the life of a cell especially in Low Earth Orbit where equipment is exposed to the detrimental effects of molecular oxygen.7 The spacecraft would now last longer in space, therefore displacing the huge cost to build and transport spacecraft over a longer period of time.

In recent years, several low outgassing materials have been developed that Ultra Low OutgassingTM requirements (< 0.1 % TML and < 0.01 % CVCM). However, all of these systems are resin reinforced silicone materials that have limited physical properties. Engineers at NuSil recognized a need to develop a low modulus silicone elastomer that also meets Ultra Low OutgassingTM requirements.

To date, CV-2289 is widely used controlled volatility (CV) silicone material for space applications offered by NuSil Technology. This silicone elastomer is an ideal material for potting or encapsulating due to exceptional physical and mechanical properties. This includes high tear strength, low modulus for CTE mismatch, and a broad operating temperature range required for materials used in extreme environments such as space, where thermal temperatures can range from -115°C to 300 °C. Nevertheless, a low modulus silica reinforced elastomeric silicone that meets Ultra Low OutgassingTM requirements has not previously been achieved.

In a previous study the thermal outgassing characteristics of CV-2289 and SCV2-2590 were extensively compared.1 This report compares the cured physical properties of the controlled volatility silicone elastomer, CV-2289, with an equivalent Ultra Low OutgassingTM version, SCV-2585. The outgassing profiles of each material based on ASTM E 15593 test method are examined in detail. We also look at how different reinforcing fillers such as silica or silicone resin affect outgassing characteristics.

1.3 Audience

This paper targets engineers in the aerospace, photonics, and laser industries where low outgassing materials are needed to prevent contamination of sensitive equipment. Some understanding of ASTM E 5952 and ASTM E 15593,4 test standards is beneficial, but is not required.

1.4 Background of Silicone Materials for Use in Space

Silicones have been used as adhesives and coatings for over five decades in the Aerospace industry. They are ideal materials for the protection of electrical components and assemblies against shock, vibration, moisture, dust, chemicals and other environmental hazards particularly encountered in space environments. They also have the ability to maintain elasticity and low modulus over a broad temperature range. A more detailed description of silicone materials used for space applications can be found in Appendix A.

A major drawback to using many silicones compounds for space applications is that silicone materials can outgas from the polymer matrix and cause subsequent contamination of expensive equipment and devices. Early NASA flights that used silicones around the space capsule windows and other areas observed an oily residue caused by low molecular weight species that had not cross-linked into the silicone polymer matrix and subsequently outgassed and deposited on the shuttle surfaces. Based on these discoveries, NASA and other space agencies realized the importance of using low outgassing materials, known as controlled volatility (CV) materials, and recommends all adhesives used in extraterrestrial environments be tested prior to use in space for volatile species that may outgas form the material.

ASTM E 5952 is a widely accepted test standard used to screen materials for volatile content that may outgass from a material in a vacuum or space environment. NASA and the European Space Agency (ESA) recommend testing low outgassing materials per ASTM E 595 prior to use in space. A maximum Total Mass Loss (TML) of 1% and Collected Volatile Condensable Material (CVCM) of 0.1% are base requirements set out by these agencies. Although a standard for many years, some question whether these specifications are stringent enough. In response, NuSil Technology developed an Ultra Low OutgasssingTM line of silicone materials with limits set out at less than 0.1 % TML and 0.01 % CVCM. The characteristics and outgassing kinetics of many of these materials were recently discussed at length.1

2. EXPERIMENTATION

2.1 Materials

Samples

Table list the materials that are compared in this study.

Silicone Property	CV-2289	SCV-2585	SCV2-2590
Silicone Polymer	Dipheny-dimethyl	Dipheny-dimethyl	Dipheny-dimethyl
Cure Mechanism Mix Ratio (A:B)	2-part Pt Addition (1:1)	2-part Pt Addition (1:1)	2-part Pt Addition (10:1)
Reinforcing Filler	Silica	Silica	Resin

2.1.1 Processing for Controlled Volatility

The critical contaminating species typically outgassed from silicone materials are primarily caused by the low molecular weight silicone cyclics and polymers that are not covalently bound into the silicone matrix. These species are eliminated in NuSil Controlled Volatility materials to prevent subsequent outgassing and contamination.

A distillation process can remove low molecular weight linears and cyclics from the polymer formed during the polymerization process. The low molecular weight materials condense on the cold finger and are separated to a collection vessel. Depending on the size of equipment and the ultimate use of the polymer, one to multiple passes through the distillation process can be performed to remove a sufficient amount of low molecular weight species based on the ultimate requirement of polymer. To produce the Ultra Low Outgassing materials, extensive processing time is needed to achieve lower levels. See Appendix A section 6.2 for more details on this process.

2.2 Methods

2.2.1 ASTM E 5952

This test method is used to determine the volatile content of materials when exposed to a vacuum environment (i.e. space). The two parameters measured are total mass loss (TML) and the collected volatile condensable materials (CVCM). Water vapor recovery is an additional parameter that can also be obtained after the completion of the exposures and measurements required for TML and CVCM.

2.2.2 ASTM E 595 Test Parameters

Each material sample is preconditioned at 50 % relative humidity and ambient atmosphere for 24 hours. The sample is weighed and loaded into the test chamber, see Figure 11, within the ASTM E 595 test stand. The sample is then heated to 125°C at less than 5x10-5 torr for 24 hours. The volatiles that outgass under these conditions escape through an exit port, and condense on a collector plate maintained at 25°C. Once the test is complete the samples are removed from the chamber and the collector plate and samples are then weighed.

2.2.3 Data Analysis

The CVCM is the quantity outgassed from the sample that condenses on the condenser plate and presented as a percentage calculated from the difference in mass on the collector plate before and after the test. The percent TML, the percent total mass of the material outgassed from the initial sample, is calculated from the mass of the sample measured before and after the test.

After the specimen is weighed to determine the TML, the WVR can be determined. The specimen is stored at 50% humidity for 24 hours at 25 C to permit sorption of water vapor. The specimen is then weighed again which is then subtracted by the he mass determined after vacuum exposure to obtain the WVR.

The standard criteria for low outgas materials is a 1.0 % TML and 0.10 % CVCM. For the Ultra Low Outgassing™ materials, the specification requires < 0.1 % TML and < 0.01 % CVCM, as designated by NuSil Technology.

2.2.4 ASTM E 15593

ASTM E 1559 experiments were conducted at OSI laboratories and test reports were provided. The isothermal outgassing test apparatus is explained in detail by Garret et al. and will only be discussed here briefly.3,4,5 A schematic of the ASTM E 1559 test stand is shown in Figure 12, Appendix B. The material sample can range from 0.5 g to 10 g and is placed in a temperature-controlled effusion cell in a vacuum chamber. All samples are preconditioned in accordance with ASTM E 5951 unless otherwise specified.

Outgassing flux leaving the effusion cell orifice condenses on four Quartz Crystal Microbalances (QCMs) that are controlled at selected temperatures. The QCMs and

effusion cell are surrounded by liquid nitrogen shrouds to ensure that the molecular flux impinging on the QCMs is due only to the sample in the effusion cell. The TML and outgassing rate from the sample are determined as functions of time from the mass deposited on an 80 K QCM and normalized with respect to the initial mass of the sample.

The amount of outgassing species that are condensable, VCM, is measured as a function of time from the mass collected on the 298 K QCM. After the outgassing test is complete, the QCMs are then heated to 398 K at a rate of 1K/min. As the QCM heats the deposited material evaporates. The species that evaporate can be analyzed by a quadrupole mass spectrometer to quantitatively determine the species observed.

2.2.4.1 ASTM E 1559 Sample Preparation

The CV-2289 and SCV-2585 were cured at 150°C for 15 minutes into discs 1.49 inches diameter by 0.125 inches thick. SCV2-2500 was cured at 65 °C for 4 hours into to discs: one disc was 1.491 inches diameter by 0.100 inches thick, the other disc was 1.491 inches diameter by 0.090 inches thick. The surface area is calculated for both faces and the edge of the disc.

One of the supplied discs of material was placed in the effusion cell as the test sample. The samples were tested with no additional preconditioning.

2.2.4.2 Test Parameters

The following parameters were set for each sample:

- **Chamber Pressures :**10-8-10-10 torr
- **View Factor from QCM to sample:** 415 cm2
- **Test Duration:** 72 hours
- **Sample Temperature:** 125 °C
- **QCM Temperatures:** 80 K, 160 K, 220 K, and 298 K
- **QCM Sensitivity:** 4.43 x 10-9 g/cm2/Hz

2.2.5 Data Analysis

2.2.5.1 Outgassing Rate

The outgassing rates for species condensable on the warmer QCMs can be calculated from curve fits to the data. The total outgassing rate from the 80 K QCM then can be compared to the outgassing rates for species condensable on the warmer QCMs to determine the rates of very high volatility species (water and solvents) and the rates of the remaining species (high, medium, and low volatility).

3. RESULTS AND DISCUSSION

3.1 Comparison of Physical Properties

In Table 2 the results for the typical material properties for each sample were measured according to ASTM protocols, are listed and compared. These properties were measured at NuSil Technology.

ASTM	Typical Properties	CV-2289	SCV-2585	SCV2-2590
Uncured	Viscosity	50,000 cps	50,000 cps	3,500 cps
	Worktime	1 hour	1 hour	2 hours
Cured	Cure Time	15 min @ 150 °C	15 min @ 150 °C	4 hrs @ 65 °C
D412	Durometer, Type A	30	36	40
D412	Tensile Strength	650 psi	684 psi	500 psi
D412	% Elongation	3.5	3.02	0.85
D1002	Lap Shear Strength	525 psi	506 psi	200 psi
E 595	% TML	0.0052	0.0006	0.0005
E 595	% WVR	0.0004	0.0004	0.0001
E 595	% CVCM	0.0005	0.0001	0.0001

TABLE 2: Typical Physical Properties Evaluated for Silicone Elastomers

Currently, the only silicone materials to achieve Ultra Low OutgassingTM requirements are resin filled systems like SCV2-2590. The primary goal of this study was to develop an ultra low outgassing platinum cure, diphenyl-dimethyl copolymer, similar to CV-2289, for satellite and space application but to achieve TMLs < 0.1% and < 0.01 % CVCMs similar to SCV2-2590. These goals were accomplished by achieving 0.06 %TML and 0.01% CVCM for SCV-2585. These results are ten times lower than CV-2289. Comparison of the physical properties of CV-2289 and SCV2-2585 in Table 1 show that the desired physical properties are not compromised to achieve these requirements.

3.2 % Total Mass Loss

3.2.1 Comparison of % TML from ASTM E595 and %TML at the end of the ASTM E 1559 Test

The %Total Mass Loss (TML) of each sample for the results obtained from ASTM E 595 are compared to the results obtained from ASTM E 1559.

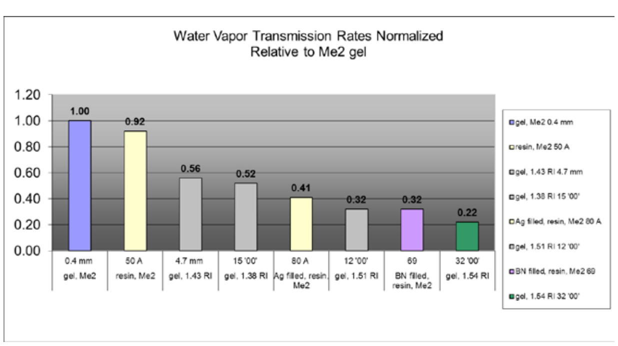

FIGURE 1: Comparison of % TML for each silicone elastomer and comparing the results from ASTM E 595 and ASTM E 1559 at the end of the test.

3.2.2 % TML ASTM E 1559

3.2.2.1 Amount of TML Due to Different Outgassed Species

Molecular weight ranges for the species in the different volatility categories can be estimated based upon engineering experience related to species condensability and mass spectrometer data. The "very high volatility" group of species in Table 3 is primarily due to water and solvents. The "high volatility" species most likely have molecular weights of 50 to 200 amu, the "medium volatility" species fall in the 200 to 400 amu range, and the "low volatility" species have estimated molecular weights above 400 amu.4

ASTM E 1559		Cumulative Amounts of Estimated Volatility of Different Outgassed Species			
Silicone Material	Total % TML at end of test	very high volatility	high volatility	medium volatility	low volatility
SCV-2585	0.08%	0.08%	0.00%	0.00%	0.00%
	100.00%	89.74%	5.50%	3.69%	1.07%
CV-2289	0.43%	0.09%	0.19%	0.14%	0.01%
	100.00%	21.84%	43.71%	31.79%	2.67%
SCV2-2590	0.07%	0.05%	0.01%	0.01%	0.00%
	100.00%	80.15%	9.45%	9.05%	1.35%

TABLE 3:

The % TML of the Cumulative amounts of estimated volatility ("very high, high, medium, and low") of different out gassed species are plotted in Figure 2. The majority of the outgasssed species for the SCV materials are of high volatility with low probability of causing contamination.

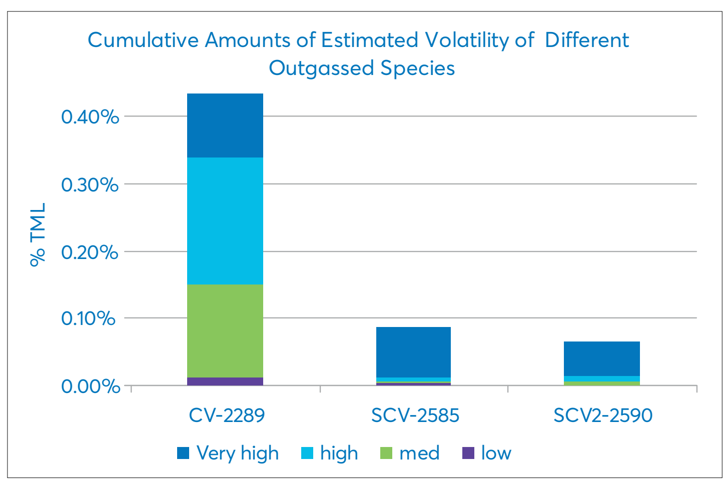

FIGURE 2: % TML of the cumulative amount of estimated volatility of different outgassed species

3.2.3 Total Mass Loss as a Function of Test Time

Figure 3 is a plot of the total mass loss of each sample over the 72 hour period. After the initial loss and compared to CV-2289, SCV2-2590 shows relatively no mass loss over the entire course of the experiment.

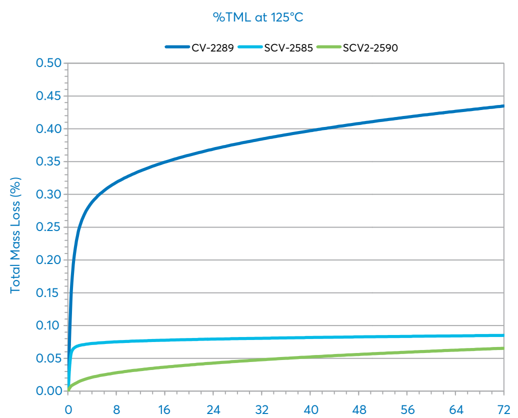

FIGURE 3: Total Mass Loss of CV-2289, SCV-2585 and SCV2-2590 as a function of time

3.3 % Volatile Condensable Material (ASTM E 1559)

Figure 4 is a plot of the volatile condensable material from the 298 QCM as a function of test time.

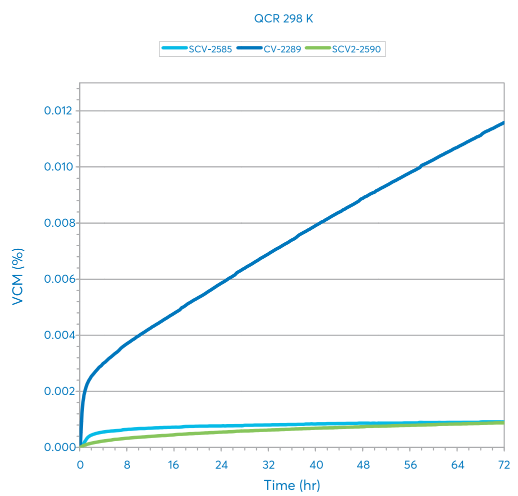

FIGURE 4: Volatile Condensable Materials from CV-2289, SCV-2585, and SCV-2590 condensed on the 298 K QCM

3.4 Outgassing Rate

Total outgassing rate data for the sample were calculated by differentiating the data obtained from the 80 K QCM. The rates of very high volatility species (water and solvents) and the rates of the remaining species (high, medium, and low volatility). These data are presented in Table 4.

	Total Outgassing Rate	Cumulative Amounts of Estimated Volatility of Different Outgassed Species	
	end of test	very high volatility	high, med and low volatility
	207.5 $pg/cm_2/s$	105.7 $pg/cm_2/s$	1,352 $pg/cm_2/s$
CV-2289	100%	51%	49%
	35.1 $pg/cm_2/s$	26.9 $pg/cm_2/s$	8.1 $pg/cm_2/s$
SCV-2585	100%	77%	23%
	108.5 $pg/cm_2/s$	80.7 $pg/cm_2/s$	27.8 $pg/cm_2/s$
SCV2-2590	100%	74%	26%

TABLE 4:

3.5 Comparison of the Total Outgassing Rate as a Function of Time

Figure 5 shows the total outgassing rate data as a function of test time for CV-2289, SCV-2585, and SCV2-2590. These outgassing rates are for species condensable at 80 K and thus would not include certain gases such as nitrogen and oxygen.

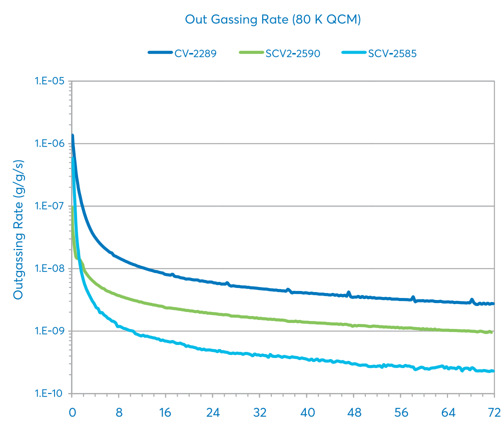

FIGURE 5: Total Outgassing Rate for CV-2289, SCV-2585, and SCV2-2590 as a Function of Test Time (Species Condensable on 80 K QCM) w

3.5.1 Predicted Outgassing Rate for SCV-2585

As previously discussed,1 outgassing kinetics show that the majority of the sample loss for both silicone samples occurs in the first few hours of the test then mass is lost at a steady rate for the remainder of the test. This indicates that there is a large initial burst of volatiles, most likely concentrated near the surface of the sample. The slow steady release thereafter can be due to either a steady state diffusion of gaseous molecules distributed throughout the polymer matrix that migrate out the material at a constant rate.

In Figure 6, the data for the outgassing rate of SCV-2585 shown in Figure 5 were fit using a general power function:

$$y = 3*10^{-9} x^{-1.1107}$$

$$R^2 = 0.9516$$

This model provides a conservative analysis of the predicted amount of volatile components that will be outgassed over a year.6

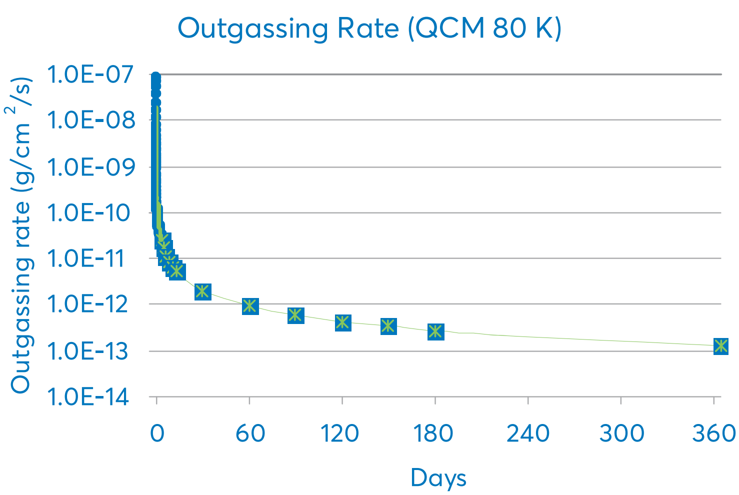

FIGURE 6: Predicted Outgassing Rate for SCV-2585 as a Function of Time Extrapolated for 1 year.

3.6 Desorption Rate at 80 K QCM

The QTGA test data can be used to determine the relative amounts of the species outgassed. Recall, at the conclusion of the isothermal outgassing test, the 80 K QCM is heated at increments of 1K/min while the outgassed species that deposit on the surface evaporate from the crystal. As the temperature of the QCM is increased during QTGA, the collected species will evaporate from the QCM in order of their relative volatilities. As the temperature of the QCM increases, the evaporation rate of the species also increases until it reaches a peak. The slope of the leading edge is characteristic of the species being volatilized. In Figure 7, the QTGA data for all three samples are plotted together as evaporation rate from the QCM as a function of QCM temperature.

cThe major outgassing species for CV-2289 are projected to be low molecular weight siloxanes as discussed previously.1

4. CONCLUSIONS

As devices and processes become more advanced and sensitive to molecular contamination, more details of characterization of the construction materials must be obtained. Ultra low outgassing specification requirements of < 0.1% TML and < 0.01% CVCM can be useful in the overall management of outgassing species. The results from kinetic outgassing data allow engineers to better predict the levels of contamination, migration, and deposition once the materials are in space. Achieving these lower levels does not show to compromise physical properties and thus a broad range of silicone elastomers with unique and specific properties can be developed. An area of future research is to develop a ULO silicone elastomer incorporated with functional fillers that impart thermally or electrically conductive properties.

5. References

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2. ASTM E-595, "Standard Test Method for Total Mass Loss and Collected Condensable Materials from Outgassing in a Vacuum Environment."
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4. J.W. Garrett, A.P.M. Glassford, and J. M. Steakley, "ASTM E1559 Method for Measuring Material Outgassing/Deposition Kinetics", Journal of the IEST, pp. 19- 28, Jan/Feb 1995

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6. Appendix a

6.1 Silicone Materials for Use in Space

Silicones typically used in space applications are called Silicone Thermosets, which are comprised of moldable elastomers and adhesives. These types of silicone materials are made up of low viscosity polymers, reinforcing fillers, and adhesion promoters. Each component can be modified for a specific process or application.

6.1.1 Polymer Chemistry

The silicone polymers used for the materials discussed in this report are diphenyldimethylpolysiloxane co-polymers. Silicone polymers are composed of repeating R2Si-O units, having no carbon atoms in the backbone, and are named polysiloxanes. The structure of silicone accounts for its high degree of flexibility. The siloxane backbone can be formulated with different types of constituent (R) groups integrated onto the polymer backbone. Typical constituent groups include dimethyl, methylphenyl, diphenyl, and trifluoropropylmethyl functionality.

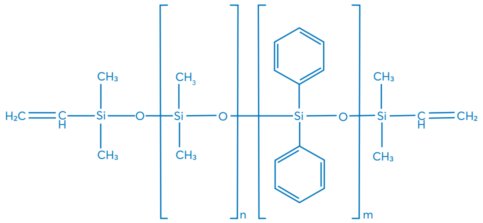

FIGURE 8: Diphenyl-dimethyl Vinyl Endblock Silicone Co-polymer

6.1.2 Reinforced Silicone Elastomers

Silicone polymers in this study are reinforced to achieve specific physical properties. Polymers are typically reinforced with silica and/or resin fillers. Reinforcement fillers such as silica are the most commonly added to a silicone elastomer systems in order to improve mechanical properties such as tensile, tear, elongation and adhesion. Filler particles reinforce an elastomer by reducing the mobility of the siloxane chains. The uniform distribution and the particle surface area available to make contact with the siloxane chains have the most influence on the physical properties of a reinforced elastomer. The bond angles of the silicon-oxygen bonds create large amounts of free volume in silicone elastomers.

Resin reinforced silicone elastomers on the other hand utilize highly branched polysiloxanes, also called Polysilsesquioxanes, to reinforce the silicone polymer. Silicone resins reinforce silicone polymers in three general ways: by covalent bonding – crosslinking of functional groups on the polymer, chain entanglement of resin and polymer, and through weak non-covalent intermolecular interactions, such as hydrogen bonding, between the silicone polymer and the surface of the resin. Resin reinforced silicones are typically low in viscosity, have high optical clarity, high durometer and modulus (typically have durometer > 40 Type A), high stress at low elongations, and tear values of < 100 ppi.

6.1.3 Addition Cure Mechanism

The cure chemistry gives a silicone adhesive its cohesive strength. The silicones used in this study are two-part platinum addition curing silicones. These two-part systems cure to a rubbery type hardness and often contain reinforcing fillers to improve physical properties.

Once the polymer is produced and reinforcing filler is added to make a base, it is separated into two parts. Catalyst is introduced to Part A while a functional crosslinker and inhibitor is added to part B. The ratio between the components must be balanced to give optimal properties. Addition cure silicones utilize a platinum complex as a catalyst to participate in a reaction between a hydride functional siloxane polymer and a vinyl functional siloxane polymer. The result is an ethyl bridge between the two polymers. The reaction mechanism is pictured below:

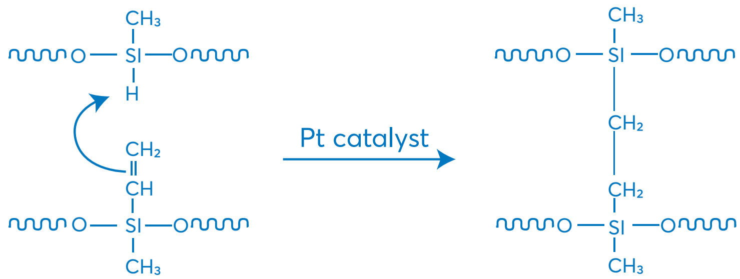

FIGURE 9:

The product of this polymerization reaction is a mixture of various molecular weights of cyclics, short chained linear molecules and higher molecular weight polymers where the concentrations of each species is based on its thermodynamic equilibrium, Figure 3. The oily substance and fogging associated with silicones are primarily caused by the low molecular weight species represented in the smaller peak to the left and the lower molecular weight portion of the larger peak. These species are eliminated in low outgassing materials to prevent contamination.

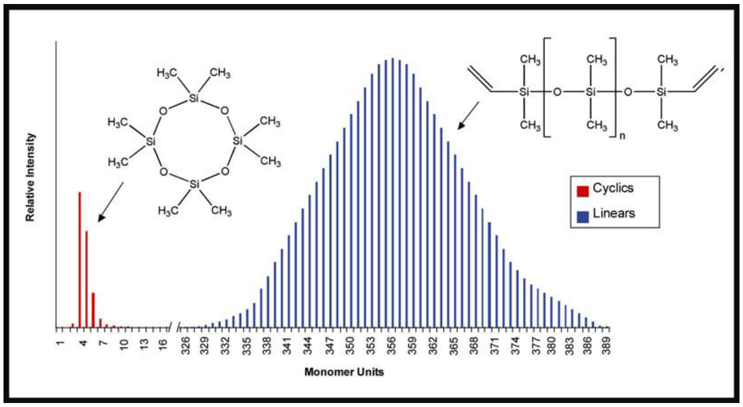

FIGURE 11: Molecular weight distribution of final ROP reaction products of PDMS.

Platinum systems are often cured quickly with heat but can be formulated to cure at low temperatures and room temperature if necessary. The advantages of these systems include fast cure and no volatile byproducts allowing these types of systems may be used in closed environments. Most systems will cure at room temperature; however, some need heat to cure. The moldable materials can be casted or injection molded into various configurations.

6.2 Material for Processing for Controlled Volatility Silicones

Ring Opening Polymerization (ROP) is commonly used for commercial production of these silicone polymers. The process begins with polyorganosiloxane cyclics reacting with a chain terminating species, or "end blockers," in the presence of an acid or base initiator as shown in Figure 2.

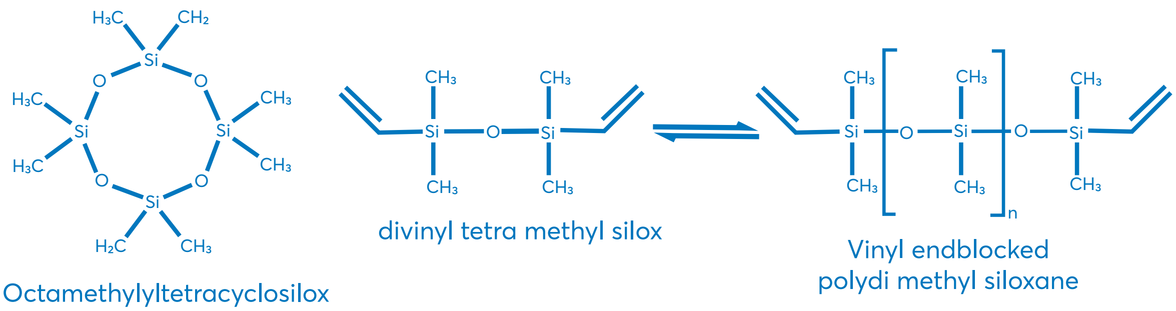

FIGURE 10: Basic Ring Opening Polymerization (ROP) reaction for a vinyl terminated polydimethylsiloxane.

Stripping the polymer via a distillation process removes low molecular weight linears and cyclics from the polymer formed during the polymerization process. The distillation apparatus is typically an evacuated chamber with heated walls and a central cooling finger designed for condensing low molecular weight molecules. Polymer is driven into the heated chamber and wiped onto the chamber walls. This exposes a thin film of the polymer to heat under vacuum conditions. The low molecular weight materials condense on the cold finger and are separated to a collection vessel. Depending on the size of equipment and the ultimate use of the polymer, one to multiple passes through the distillation process can be performed to remove a sufficient amount of low molecular weight species based on the ultimate requirement of polymer. To produce the Ultra Low Outgassing™ materials, more low molecular weight linears need to be removed via distillation. This adds more processing time to achieve these lower levels.

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