Evaluating the effects of atomic oxygen on the outgassing and erosion yield of silicone materials

INTRODUCTION

For decades, NuSil®, a brand of Avantor®, has established a wide product offering of proven silicone-based space application solutions. These custom silicone solutions leverage unique material properties such as broad operating temperature, low modulus, high elasticity and ease of application to offer advanced protection. Such properties make these silicones ideal materials for the demanding environments of space, protecting sensitive components against vibration, shock, moisture, dust and more.

However, not all silicones are created equal; some may outgas volatile materials that can condensate and contaminate sensitive surfaces such as windows, lenses, sensors, arrays and other mission-critical equipment. These outgassed materials may degrade upon exposure to ultraviolet (UV) radiation and atomic oxygen (AO), causing adverse effects that shorten the lifetime of hardware or reduce equipment efficiency.[1] To overcome such challenging conditions, NuSil offers highly purified, low-outgassing silicone solutions for use by satellite and space vehicle manufacturers to advance mission success.

The value in having silicones designed for space-based applications is evident in the construction of space vehicles, which require advanced protection both inside and out. Satellites and vehicles in low Earth orbit (LEO) face particular challenges with exposure to AO and UV radiation. While most polymers experience significant mass loss after AO exposure,[2] the advanced silicones offered by NuSil do not and thus remain optimal material solutions for our customers.

NuSil prides itself on its long-standing customer commitment and relationships, which have led to impactful research collaborations and significant success. Recently, NuSil broadened their material testing and expanded their product offering by investigating how AO exposure affects silicone outgassing.

NASA'S TESTING AND EVALUATION STANDARDS FOR LOW AND ULTRA-LOW OUTGASSING

NASA uses ASTM E595 to evaluate and screen materials for space applications.[3] The acceptance limits for these tests are ≤1.0% total mass loss (TML) and ≤0.1% collected volatile condensable materials (CVCM). In addition, ASTM E2089 is used to characterize a material's response to AO exposure, allowing AO resistance to be considered when selecting spacecraft materials indicated for LEO environments. In this work, materials were exposed to AO for 24 hours, simulating the conditions thy would experience when spending approximately 4–6 months at an altitude of 400 km with a 28.5° incline in the ram direction, assuming an average fluence of 1 E21 atoms/cm^2/year.

Using both ASTM E595 and E2089 allows a material's AO resistance in a grounded laboratory to be compared with its AO resistance in space. Understanding this correlation ultimately allows materials intended for LEO environments to be screened for suitability. Here, AO resistance is based on erosion yield, the volume of material eroded by AO per incident oxygen atom[4] and the impact of AO exposure on outgassing.

MATERIALS TESTED

NuSil previously analyzed their suite of products via ASTM E595 and E1559, detailing the chemistry and processes used to make their controlled volatility (CV) and ultra-low-outgassing (super CV [SCV]) product lines and the effects of these lines on outgassing kinetics. Since these original analyses, three additional NuSil silicone materials have become available, CV-2500, CV2-2289-1 and SCV2-2590, and their AO resistance is evaluated here. These addition-cured (platinum-catalyzed) materials expand upon NuSil's market-proven condensation-cured (tin-catalyzed) AO-resistant coatings.

The primary goal of this research is to develop a holistic understanding of the performance of space-grade materials so that engineers can select the best materials for specific applications. Erosion yield data will provide a pragmatic view of product performance and of the contribution of these materials to mission success.

TESTING METHODOLOGY

For complete details on how ASTM E595 and E2089 were conducted, see the "Appendix — testing methodology" section.

TESTING RESULTS

As spacecraft become more advanced and increasingly sensitive to contamination, it is important for mission success that they are constructed using materials with physical properties and outgassing kinetics that are optimal for the intended application. Table 1 summarizes the AO erosion yield data for the NuSil silicones evaluated here.

Sample	Product family	Materials chemistry	AO erosion yield (g/O atom)	Avg. fluence (atoms/cm^2/year)
CV-2500	Adhesive/potting	2-Part platinum cure, dimethyl	1.08 E-26	1.26 E21
CV2-2289-1	Adhesive/coating	2-Part platinum cure, diphenyl-dimethyl	2.61 E-26	1.17 E21
SCV2-2590	Adhesive/potting	2-Part platinum cure, diphenyl-dimethyl	1.64 E-26	1.25 E21
Kapton® HN	Witness sample	Polyimide film	3.99 E-24	

TABLE 1: Mean initial AO erosion yield results for three NuSil materials.[4]

As seen in Table 1, the erosion yields of the NuSil CV- and SCV-grade materials were two orders of magnitude lower than that of Kapton® HN, indicating the suitability of these NuSil materials for space applications in LEO.

	TML, control (%)	TML, AO exposure (%)	CVCM, control (%)	CVCM, AO exposure (%)
CV-2500	0.0290	0.0198	0.0146	0.0108
CV2-2289-1	0.0528	0.0342	0.0185	0.0188
SCV2-2590	0.0213	0.0148	0.0088	0.0061

TABLE 2: Average values of TML and CVCM under ASTM E595 and E2089 for silicone materials exposed to AO.[4]

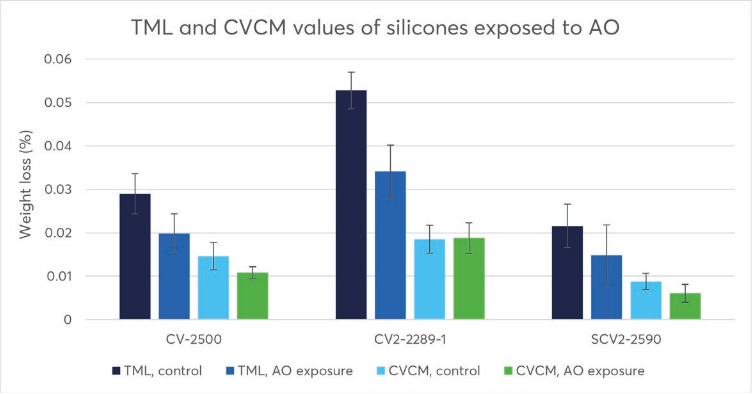

FIGURE 1: Outgassing kinetics of silicones exposed to AO.[4]

Additional testing under ASTM E2089 and E595 was performed to determine how AO exposure affects the outgassing of volatile materials. As shown in Table 2 and Figure 1, the low volatility levels of these materials decrease further after AO exposure.

This reduction in volatile condensable materials produced by cured silicone materials after exposure to AO is explained by the conversion of surface-level siloxane polymer into silicon dioxides. As confirmed through Fourier transform infrared spectroscopy (FTIR), AO removes hydrocarbons, including the dimethyl groups on a nominal siloxane chain, increasing the proportion of Si-O-Si signal relative to $Si-CH_3$. The resulting change in surface composition decreases the diffusion of residual volatile siloxanes in the bulk material.[4]

These observations support the suitability of these silicones for preparation on the spacecraft, but cured silicone rubber components could be pre-treated with AO during ground-level construction to further reduce the potential of outgassing during the mission.[4]

CONCLUSIONS

The increasing complexity and specialization of spacecraft make it necessary to have a selection of low-outgassing materials suitable for different applications. In this study, the AO resistance of three NuSil silicones was measured under ASTM E2089 and E595, and the results allow engineers to select the materials most suitable for the intended applications. While some situations may prefer or require addition-cured or condensation-cured silicones, both materials should have low-outgassing kinetics to reduce the potential contamination of sensitive equipment. These results contribute to NASA's library of test data; the comparison in Table 3 of data on CV-2500 from this study with historical data demonstrates the superior AO resistance of the new NuSil silicone. This AO-resistant material has been tested on a variety of substrates, including Delrin®, silver-coated Teflon®, Kapton® film, aluminum and Beta cloth X389-7, among others.[7] Selecting the material with the most suitable chemistry for the application does not have to come at the expense of performance because both addition-cured and condensation-cured silicones are low outgassing and AO resistant. NuSil silicones come in convenient product types and packaging to allow for simplified application in complex designs and assemblies.

Material	Substrate	Reaction efficiency (g/atom)
CV-2500	Silver-coated Teflon™	1.132 E-25
CV-1144-0	X389-7 Beta Cloth	3.158 E-25
CV-1144-0	Silver-coated Teflon™	9.831 E-25
CV-1142	Aluminum	1.299 E-24
CV-2500	Kapton® H Film	1.805 E-24
CV-1500 Black	Aluminum	2.234 E-24
CV-2566 Red	Aluminum	2.240 E-24
CV-2500	Delrin® II 900 NC	2.276 E-24
CV-1144-0	Kapton® H Film	2.657 E-24
CV-1144	Delrin® II 900 NC	4.355 E-24

TABLE 3: Reaction efficiency of NuSil silicones on a variety of substrates.[7]

NuSil's ultra-low-outgassing materials are designed for use in the most stringent applications. Compared with their low-outgassing counterparts, these materials were found here to have lower (by one order of magnitude) ASTM E595-measured TML (< 0.1%) and CVCM (< 0.01%) values and were recently[8] reported to have much lower outgassing rates. These results indicate that NuSil's ultra-low-gassing silicones are ideal for use in sensitive space applications requiring minimal contamination in order to extend the lifetime of the hardware.

Both addition-cured and condensation-cured silicones are suitable for numerous space applications. Additionally, other product types, such as tapes and film adhesives, are available for ease of use in certain applications. In all product types, these silicones demonstrate superior durability even in thin coatings, while maintaining flexibility and AO resistance, and provide engineers more options for optimizing their advanced spacecraft designs.

APPENDIX — TESTING METHODOLOGY

ASTM E2089[5] — ATOMIC OXYGEN APPARATUSES

To quantify the effects of AO exposure on volatile emissions, ground-level ASTM E2089 testing was performed. Due to differences in thermal conditions, radiation exposure and fluence, the correlation between on-ground and in-space AO resistance for a spacecraft material needs to be determined. There are multiple ASTM E2089-compliant equipment options available, including plasma ashers, pulsed CO_2 lasers, microwave electron cyclotrons and end-hall ion sources.[4] Because the strength of emitted radiation depends on the source, ASTM E2089 uses the effective fluence of Kapton®, calculated using known erosion yield values from LEO exposure. This effective fluence can be used to determine the amount of AO exposure in LEO needed to cause the equivalent sample erosion observed in the on-ground AO apparatus.

The on-ground apparatus used here was the Minimum Atmospheric eXperimentation (MAX) chamber. MAX produces an omnidirectional AO fluence with an energy range of 0.04–0.10 eV. This energy is low relative to that of AO in LEO, but the high fluence that MAX produces (approximately 1 E21 atoms/cm^2/year) leads to similar erosion levels. Within MAX, an RF generator powers an aluminum electrode that forms a capacitively coupled plasma to the ground electrode. The resulting plasma strength and shape are controlled through variable capacitors and shielding for precise exposure for the samples between the electrodes.[4] Exposure for 24 hours in MAX correlates to approximately 4–6 months of exposure in LEO at an altitude of 400 km with a 28.5° incline in the ram direction, assuming an average fluence of 1 E21 atoms/cm^2/year; these data were collected by the Materials International Space Station Experiment during the 2004–2016 solar cycle.[2]

ASTM E595[6] – TML/CVCM

ASTM E595 measures the volatile content of materials when exposed to a vacuum environment. The parameters measured are TML and CVCM. One additional parameter that may be measured is water vapor regained (WVR). All tests in this study used an environmental mass loss investigation chamber to measure outgassing at elevated temperatures under vacuum.

Before testing, the sample is preconditioned at 23 °C and 50% relative humidity for 24 hours. The sample is then weighed and placed in the test stand, and the chamber is evacuated to a pressure of ≤5 E-5 torr. The samples are heated to 125 °C and then held at these conditions for 24 hours. Dispersing volatile materials escape through an exit port and condense on a collector plate held at 25 °C. The sample and collector plate are weighed upon test completion using a Mettler-Toledo XS3DU microbalance scale capable of reading ±3 µg, with a 3.1-g max capacity.

TML % and CVCM % are measured using the following equations:

1. $TML\ \% = ((MS_i - MS_f)/MS_i) \times 100$
2. $CVCM\ \% = ((C_i - C_f)/MS_i) \times 100$

Where:

MS_i = initial sample mass

MS_f = final sample mass after testing

C_i = initial collector plate mass

C_f = final collector plate mass after testing

TESTING PROTOCOL[4]

All samples were conditioned at room temperature and 50% humidity for 24 hours before ASTM E595 testing. Next, the samples were split into 2 groups: one exposed to AO per ASTM 2089 and the other not exposed to AO (both groups were subjected to the same temperature and vacuum conditions). Finally, all materials then underwent another round of ASTM E595 testing.

ACKNOWLEDGEMENTS:

NuSil would like to acknowledge Professor Kira Abercromby and the work of Samuel Westrick in his thesis "Effects of atomic oxygen on outgassing of silicone materials" and the collaborative efforts of California Polytechnic State University of San Luis Obispo for material testing and analysis.

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