NASA/TP—2018–220123

Space Environmental Effects on Additively Manufactured Materials

M.M. Finckenor
Marshall Space Flight Center, Huntsville, Alabama

October 2018

The NASA STI Program…in Profile

Since its founding, NASA has been dedicated to the advancement of aeronautics and space science. The NASA Scientific and Technical Information (STI) Program Office plays a key part in helping NASA maintain this important role.

The NASA STI Program Office is operated by Langley Research Center, the lead center for NASA's scientific and technical information. The NASA STI Program Office provides access to the NASA STI Database, the largest collection of aeronautical and space science STI in the world. The Program Office is also NASA's institutional mechanism for disseminating the results of its research and development activities. These results are published by NASA in the NASA STI Report Series, which includes the following report types:

- TECHNICAL PUBLICATION. Reports of completed research or a major significant phase of research that present the results of NASA programs and include extensive data or theoretical analysis. Includes compilations of significant scientific and technical data and information deemed to be of continuing reference value. NASA's counterpart of peer-reviewed formal professional papers but has less stringent limitations on manuscript length and extent of graphic presentations.

- TECHNICAL MEMORANDUM. Scientific and technical findings that are preliminary or of specialized interest, e.g., quick release reports, working papers, and bibliographies that contain minimal annotation. Does not contain extensive analysis.

- CONTRACTOR REPORT. Scientific and technical findings by NASA-sponsored contractors and grantees.

- CONFERENCE PUBLICATION. Collected papers from scientific and technical conferences, symposia, seminars, or other meetings sponsored or cosponsored by NASA.

- SPECIAL PUBLICATION. Scientific, technical, or historical information from NASA programs, projects, and mission, often concerned with subjects having substantial public interest.

- TECHNICAL TRANSLATION. English-language translations of foreign scientific and technical material pertinent to NASA's mission.

Specialized services that complement the STI Program Office's diverse offerings include creating custom thesauri, building customized databases, organizing and publishing research results…even providing videos.

For more information about the NASA STI Program Office, see the following:

- Access the NASA STI program home page at <http://www.sti.nasa.gov>

- E-mail your question via the Internet to <help@sti.nasa.gov>

- Phone the NASA STI Help Desk at 757–864–9658

- Write to:
  NASA STI Information Desk
  Mail Stop 148
  NASA Langley Research Center
  Hampton, VA 23681–2199, USA

NASA/TP—2018–220123

Space Environmental Effects on Additively Manufactured Materials

M.M. Finckenor
Marshall Space Flight Center, Huntsville, Alabama

National Aeronautics and
Space Administration

Marshall Space Flight Center • Huntsville, Alabama 35812

October 2018

Acknowledgments

The author gratefully acknowledges the support from the Center Investment Program in this effort. The author thanks Ken Cooper and Phil Steele of MSFC EM42, Patrick Price of Stratasys, and Matthew Napoli of Made in Space, Inc. for providing samples for study.

Available from:

NASA STI Information Desk
Mail Stop 148
NASA Langley Research Center
Hampton, VA 23681–2199, USA
757–864–9658

This report is also available in electronic form at
<http://www.sti.nasa.gov>

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF FIGURES (Continued)

LIST OF TABLES

LIST OF ACRONYMS

AO	atomic oxygen
COSMIC 2	Constellation Observing System for Meteorology, Ionosphere, and Climate
ESD-PEKK	electrostatic-dissipative polyetherketoneketone
ESH	equivalent sun-hours
GRCop-84	Glenn Research Copper 84 alloy
LPSR	Laboratory Portable Spectroreflectometer
MiS	Made in Space
MISSE	Materials on International Space Station Experiment
MISSE-FF	Materials on International Space Station Experiment Flight Facility
MSFC	Marshall Space Flight Center
NUV	near ultraviolet
PC-ISO	polycarbonate biocompatible per ISO 10993 USP Class VI
PPSF	polyphenylsulfone
VUV	vacuum ultraviolet
UV	ultraviolet

NOMENCLATURE

α_s	solar absorptance
ε_{IR}	infrared emittance

TECHNICAL PUBLICATION

SPACE ENVIRONMENTAL EFFECTS ON ADDITIVELY MANUFACTURED MATERIALS

1. INTRODUCTION

This project was a 2-yr effort to provide data on the durability of additively manufactured materials in the space environment. Materials studied were polyetherimide (Ultem 1010 and 9085), electrostatic-dissipative polyetherketoneketone (ESD-PEKK), polycarbonate biocompatible per ISO 10993 USP Class VI (PC-ISO), polyphenylsulfone (PPSF), Inconel 718, and Glenn Research Copper 84 alloy (GRCop-84). Some samples were manufactured at Marshall Space Flight Center (MSFC), while 3D printer manufacturers Stratasys and Made in Space (MiS), Inc., participated in this effort so as to compare different vendors and printing setups.

At the same time, laboratory simulators were improved for better fidelity to the space environment. Increased fidelity reduces risk. Space simulations focused on thermal vacuum, atomic oxygen (AO), and ultraviolet (UV) radiation, advancing from no data available for these additively manufactured materials to TRL5. Based on the test results, samples were prepared and characterized for flight on the Materials on International Space Station Experiment (MISSE)-9 and MISSE-10.

2. BACKGROUND

The space environment is composed of AO, UV, protons, electrons, meteoroid/space debris impacts, thermal cycling, and hard vacuum. Atomic oxygen reacts with most polymers, breaking molecular bonds. The measure of this reaction rate is referred to as erosion yield, with units of cm^3/atom. Mass loss was used to determine the AO erosion yield, but thickness loss may also be used. This study provided the AO erosion yield rates for Ultem 9085, ESD-PEKK, PC-ISO, and PPSF so that if it were necessary to deploy a 3D printed part to be exposed to space, we will know its durability and plan accordingly. Additively manufactured Ultem is being proposed for the antenna array supports for the FORMOSAT-7 Constellation Observing System for Meteorology, Ionosphere, and Climate (COSMIC-2) satellite mission.

Metals form an oxide layer when exposed to AO, and both nickel and copper are known to react with AO. Inconel and GRCop-84 may be used in high-performance metal gaskets and nozzle liners, and these may be exposed to space in an exploration vehicle.

UV radiation can cause either cross-linking or chain scission in polymeric materials. Cross-linking embrittles the material while chain scission weakens it. The optical properties of the material may be affected by exposure to UV, with generally an increase in solar absorptance. The polymer samples were exposed to 500 and 1,000 equivalent sun-hours (ESH) of UV in vacuum of 1×10^{-6} torr or better. Mass was measured before and after UV exposure. Mass was also measured before and after thermal vacuum bakeout of the MISSE-9 and MISSE-10 samples.

While providing AO erosion rate and UV degradation of optical properties of additively manufactured materials, this project also allowed for enhancement of a UV monitor for flight experiments. The original passive monitoring design was used on two experiments flown on the Mir space station for 18 mo and the MISSE 1–4 experiments. It was modified for active monitoring on MISSE-7B, but this design change was not successful. Two candidate designs were tested during the first year's effort, and a third design was tested during the second year. The best design was selected for flight and is currently flying on the zenith, ram, and wake faces of MISSE-9.

3. TEST SETUP

Ultraviolet radiation exposure was performed in the EM41 NUV/VUV Solar Simulator Facility (fig. 1). Fluence was monitored by a spectroradiometer. Atomic oxygen exposure was performed in the MSFC Atomic Oxygen Beam Facility (fig. 2). Fluence was monitored by current measurements at the neutralizer plate. A witness sample of Kapton HN was included with the samples, and AO fluence was confirmed by mass loss of the Kapton.

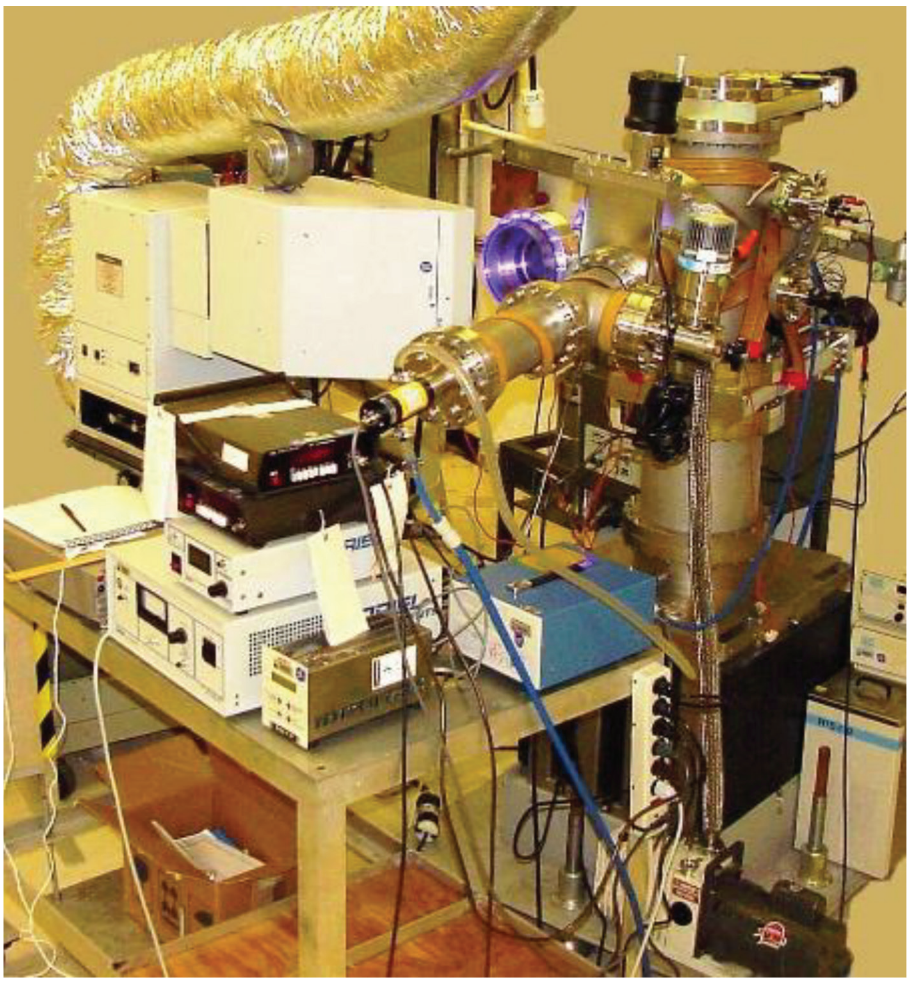

Figure 1. NUV/VUV radiation test chamber.

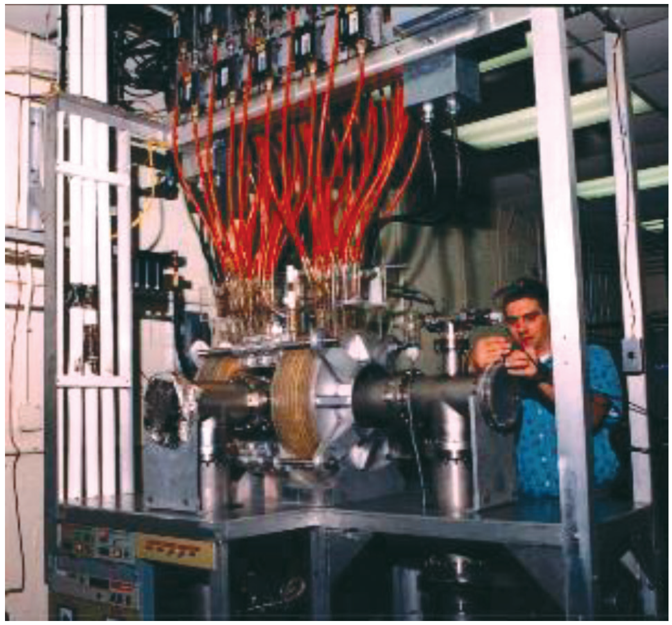

Figure 2. MSFC EM41 Atomic Oxygen Beam Facility.

Mass measurements were made using the method for hygroscopic samples to eliminate humidity effects on weight. One sample at a time was placed in a small vacuum chamber with roughing pump and pumped down to 50 millitorr. At that moment, the chamber was vented and a timer started, and the sample was moved quickly to the nearby Sartorius CPA225D balance. Mass measurements were made every 30 s from the 1-min mark to the 4-min mark, and regression analysis was used to determine mass at time zero.

Solar absorptance (α_s) for air mass zero (space) was calculated from spectral reflectance measurements made from 250 to 2,800 nm with an AZ Technology model 300. Laboratory Portable Spectroreflectometer (LPSR) ASTM E-903 was the test method used under normal laboratory conditions, and ASTM E-490 was the solar spectral irradiance data used to calculate α_s. The LPSR has repeatability of approximately ±1%.

Infrared emittance (ε_{IR}) measurements were made with an AZ Technology TEMP 2000A infrared reflectometer. This instrument measures the total hemispheric reflectance averaged over 3–35 µm wavelengths. ASTM E-408 was the test method used under normal laboratory conditions. The TEMP 2000A has repeatability of approximately ±0.5%.

4. TEST RESULTS

4.1 UV Sensor

Over the 2-yr effort, three UV sensors were tested in the MSFC Solar Simulator: Opto Diode UVG5, Opto Diode UVG100, and SpaceQuest nanoSSOC-D60 digital sun sensors. The SpaceQuest UV sensor was difficult to install and was limited to 380 nm and up. This sensor is better used for directionality of the sun than determining the UV exposure.

The Opto Diode sensors had identical coverage of the UV wavelengths (fig. 3), the same operating temperature range, and similar performance in the solar simulator (fig. 4). The Opto Diode sensors were tested in the same UV exposures as the additively manufactured samples (fig. 5). Selection of the sensor was based on size, with the 10 × 10 mm UVG100 chosen for MISSE-9 over the 5-mm diameter UVG5.

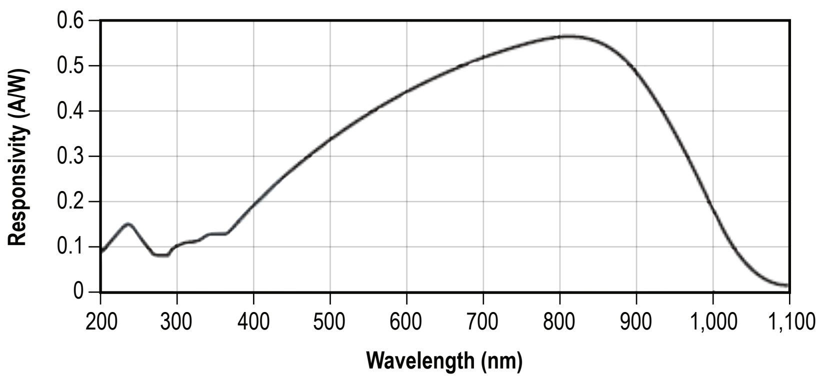

Figure 3. Response versus wavelength for Opto Diode sensors.

Figure 4. Left to right: Opto Diode UVG100 and UVG5S mounted on fiberglass, post-UV exposure.

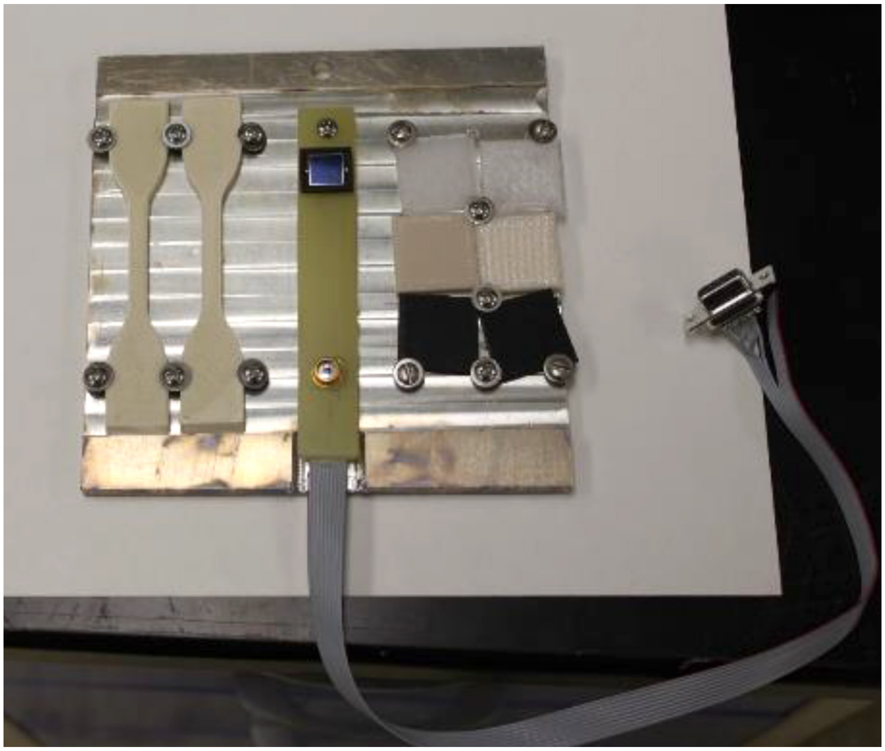

Figure 5. UV sensors mounted on sample plate with additively manufactured materials.

The UVG100 sensor was then paired with a UV-transmitting, visible-absorbing filter to improve the photodiode response to UV-only. The filter chosen was a Hoya U340, limiting the response to UV in the 255 to 380 nm wavelengths (fig. 6). Given the timing of preparing for MISSE- 9, it was decided to reduce risk and fly the UV sensors with magnesium fluoride windows to allow UV through while blocking AO. This also allows for the use of magnesium fluoride windows as contamination monitors on MISSE-9, as they are transmissive in the vacuum UV wavelengths (100 to 200 nm) and fairly durable in AO. Figure 7 is the vacuum UV transmission for the control and three flight windows. The Hoya U340 filter was chosen and tested in the same UV and AO simulators as the additively manufactured samples (fig. 8). The Hoya U340 filter did not show any degradation due to either UV or AO exposures.

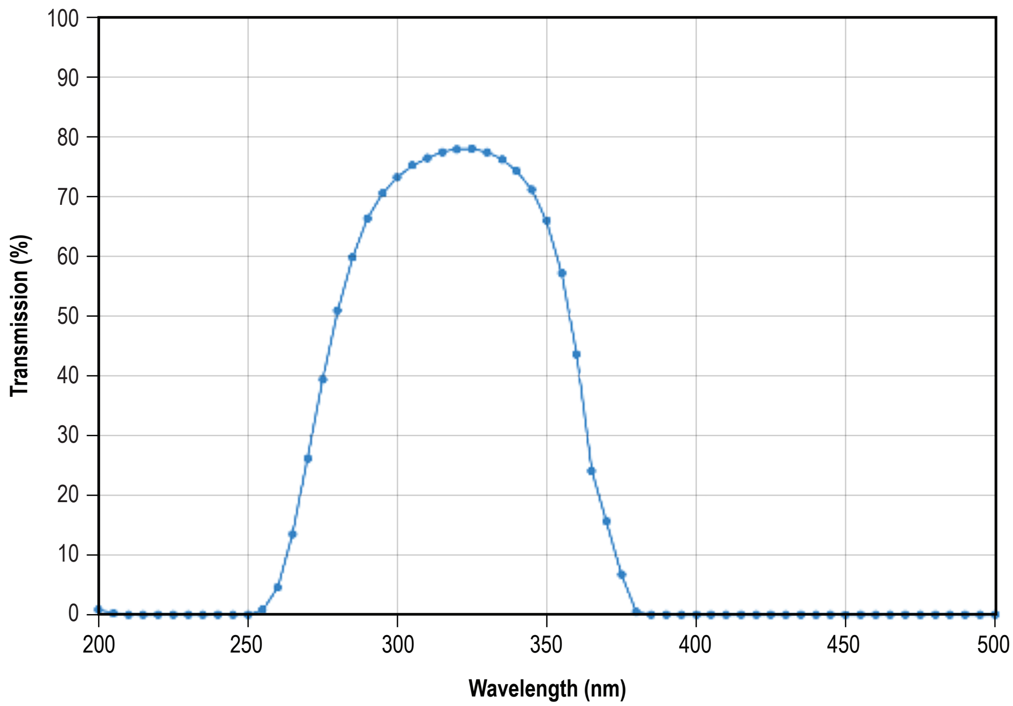

Figure 6. MISSE-9 UV filter transmission.

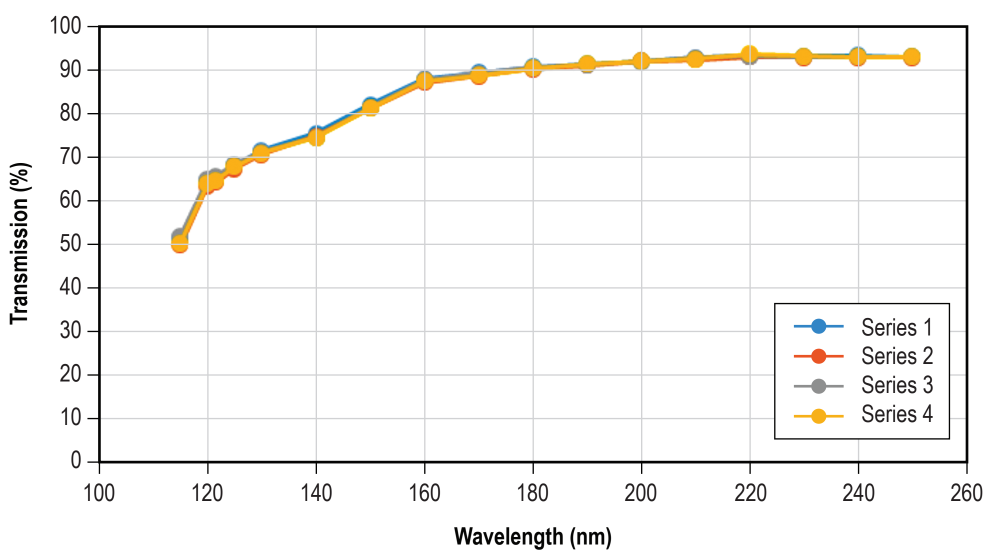

Figure 7. Control and flight magnesium fluoride sample transmission.

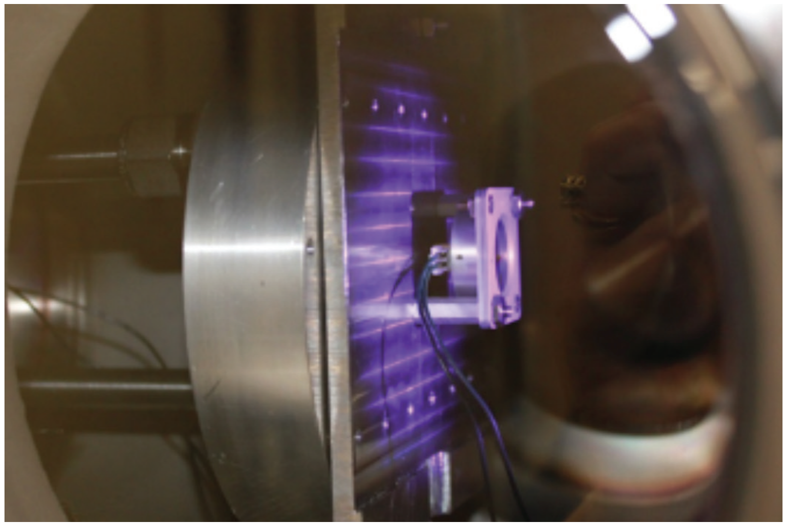

Figure 8. MISSE-9 UV sensor design undergoing acceptance testing.

4.2 Polymeric Materials

AO and UV exposures were completed for Ultem 9085, ESD-PEKK, PC-ISO, and PPSF samples. Two sets of Ultem 9085 samples were tested, one set printed by MSFC and a second set from MiS, Inc. Figure 9 shows a control sample, AO-exposed sample, and a UV-exposed sample. AO erosion rate was calculated to be 2.7 to 3.0×10^{-24} cm^3/atom after exposures totaling 1.24×10^{21} atoms/cm^2 or approximately 3 month equivalent worst-case ISS environment. De Groh et al. report an erosion rate for Ultem 1000 on the MISSE-6 flight experiment as 3.37×10^{-24} cm^3/atom,[1] so our AO simulation seems reasonable. Optical properties did not change significantly due to AO erosion.

Figure 9. Left to right: MiS Ultem 9085 control sample, AO-exposed sample, UV-exposed sample.

MSFC Ultem exposed to 500 ESH of UV experienced an increase in solar absorptance from 0.6 to 0.63/0.64, which agreed with observed darkening. MiS Ultem samples were initially lower in solar absorptance (0.49 to 0.51) but increased about the same due to the same UV exposure (0.53 to 0.54). Another 500 equivalent sun-hours of UV exposure did not further darken the samples. None of the Ultem samples indicated any measurable change in infrared emittance. The MSFC Ultem samples showed slight mass loss, slightly more for the 0°/90° than the +45°/–45° samples. The MiS Ultem samples were only +45°/–45° orientation. Samples exposed to 500 and 1,000 ESH are shown in figure 10.

Figure 10. Two EM40 Ultem samples exposed to 500 ESH, two EM40 Ultem samples exposed to 1,000 ESH, and control sample.

AO erosion rate for ESD-PEKK was calculated to be 4×10^{-24} cm^3/atom after two exposures of 5.7×10^{20} and 6.7×10^{20} oxygen atoms/cm^2 fluences, approximately 6 weeks and 7 weeks equivalent ISS environment, respectively. A Magne-Tron Instruments M-700 four-point probe was used to measure the conductivity of the ESD PEKK samples. The control sample measured $2.1\pm0.8\times10^7$ Ω/square, and the AO-exposed samples measured $4.5\pm0.8\times10^7$ Ω/square. This indicates that while AO had an effect on the conductivity, the ESD PEKK is still electrostatic dissipative. Optical properties did not change significantly due to either AO erosion or UV exposure (fig. 11).

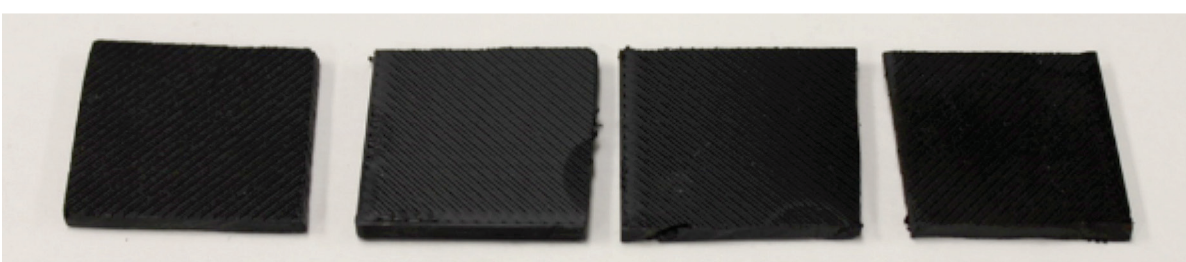

Figure 11. Left to right: control, AO1, AO2, and UV1 ESD-PEKK samples.

AO erosion for PC-ISO was calculated to be 2.7×10^{-24} cm^3/atom. Reference 1 reports an erosion rate for PEEREX-61 on the MISSE-2 flight experiment as 4.29×10^{-24} cm^3/atom, indicating the AO simulation may be low. The samples were measured with a black background, and transmission was not calculated. The UV-exposed sample was yellowed (fig. 12), with an increase in solar absorptance from 0.755 to 0.792. One of the AO-exposed samples bleached, with a decrease in solar absorptance from 0.763 to 0.681, but the other AO-exposed sample remained the same, with solar absorptance of 0.753 pre-test and 0.747 post-test. AO erosion for PPSF was calculated to be 2.9×10^{-24} cm^3/atom. The UV-exposed sample was darkened (fig. 13), with an increase in solar absorptance from 0.535 to 0.667. Optical properties of the AO-exposed samples remained unchanged.

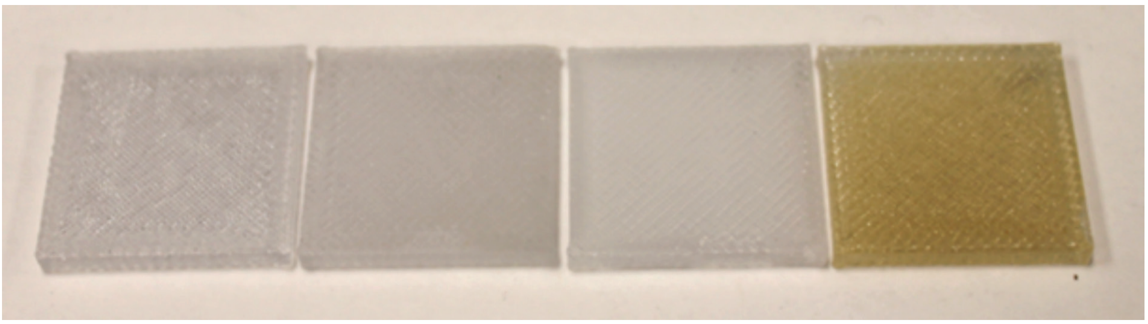

Figure 12. Left to right: control, AO1, AO2, and UV1 PC-ISO samples.

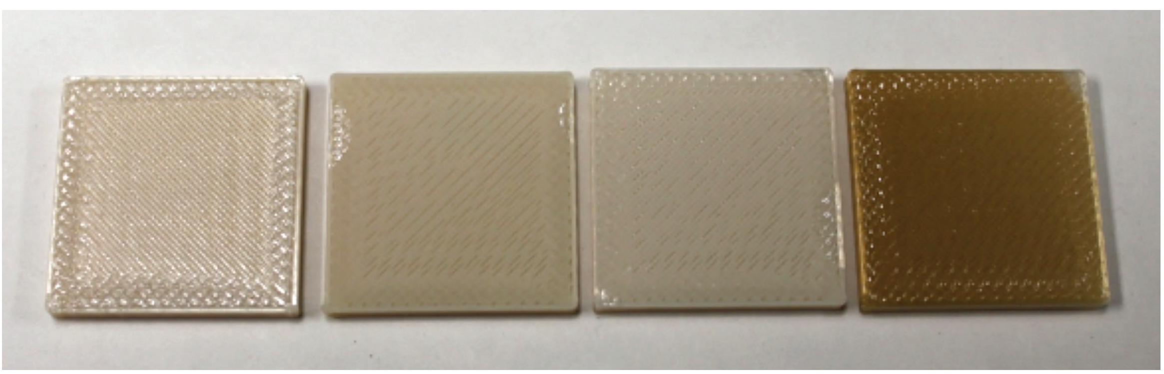

Figure 13. Left to right: control, AO1, AO2, and UV1 PPSF samples.

4.3 Metallic Materials

AO exposures totaling 1.24×10^{21} atoms/cm^2 were completed for both the Inconel and GRCop-84 samples. No significant change in mass was noted for any of the samples. Optical properties of the Inconel were unchanged (fig 14). The GRCop-84 (shown in fig. 15) increased in solar absorptance from 0.70 to 0.74 due to oxidization of copper and decrease in infrared emittance from 0.52 to 0.48.

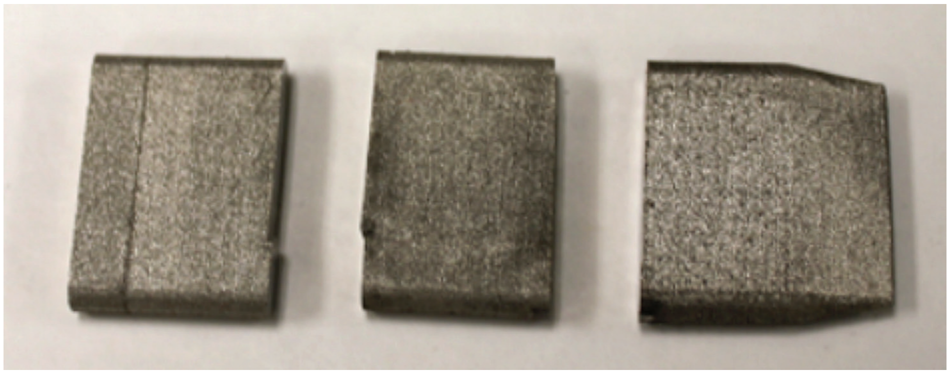

Figure 14. Left to right: AO-exposed Inconel no. 1, no. 2, and control sample.

Figure 15. AO-exposed GRCop-84.

5. SUMMARY

Table 1 summarizes the AO erosion yields for all polymeric materials tested in this effort. Tables 2 and 3 are the optical properties for all AO exposures and all UV exposures, respectively. Changes of ±0.01 in infrared emittance are not considered statistically significant.

Table 1. Calculated AO erosion yield based on mass loss.

Additively Manufactured Polymer	AO Erosion Yield (cm^3/Atom)
Ultem 9085 (MSFC)	2.7×10^{-24}
Ultem 9085 (MiS)	3×10^{-24}
ESD-PEKK	4×10^{-24}
PC-ISO	2.7×10^{-24}
PPSF	2.9×10^{-24}

Table 2. Optical property measurements for AO exposures.

Material AO1 = 5.7×10^{20} AO2 = 1.24×10^{21}	Solar Absorptance		Infrared Emittance	
	Pre-Exposure	Post-Exposure	Pre-Exposure	Post-Exposure
MSFC Ultem AO1	0.589	0.607	0.90	0.91
MSFC Ultem AO2	0.592	0.606	0.90	0.91
MiS Ultem AO1	0.493	0.515	0.90	0.92
MiS Ultem AO2	0.506	0.511	0.90	0.92
ESD PEKK AO1	0.951	0.967	0.90	0.93
ESD PEKK AO2	0.949	0.962	0.90	0.91
PC-ISO AO1	0.753	0.747	0.90	0.91
PC-ISO AO2	0.763	0.681	0.90	0.93
PPSF AO1	0.528	0.539	0.90	0.92
PPSF AO2	0.536	0.541	0.90	0.91

Table 3. Optical property measurements for UV exposures.

Material UV1 = 500 ESH UV2 = 1000 ESH	Solar Absorptance		Infrared Emittance	
	Pre-Exposure	Post-Exposure	Pre-Exposure	Post-Exposure
MSFC Ultem UV1	0.599	0.626	0.90	0.90
MSFC Ultem UV2	0.596	0.637	0.90	0.90
MiS Ultem UV1	0.506	0.541	0.91	0.90
MiS Ultem UV2	0.494	0.547	0.91	0.90
ESD PEKK UV1	0.955	0.959	0.91	0.91
ESD PEKK UV2	0.955	0.943	0.91	0.90
PC-ISO UV1	0.755	0.792	0.90	0.91
PC-ISO UV2	0.753	0.792	0.91	0.90
PPSF UV1	0.535	0.667	0.90	0.91
PPSF UV2	0.545	0.670	0.91	0.90

6. STATUS OF INVESTIGATION

Three Inconel samples per ASTM E8, twelve polymeric samples per ASTM D638, type IV, and one UV sensor have been integrated into a wake-facing tray currently flying on MISSE-9 (fig. 16). Ultem 9085 from printers at MSFC and MiS were chosen to determine the effect of printer on performance. The MSFC Ultem printer is from Stratasys, so rather than duplicate with more Ultem 9085 from Stratasys, Ultem 1010 from Stratasys was chosen. ESD-PEKK from Stratasys rounded out the MISSE-9 selection, due to its durability in the ground testing and the electrostatic dissipative properties. Teflon washers were used for three reasons: to prevent damage to the samples from the fasteners, to provide a protected area for thickness loss measurements, and to give an estimate of the AO fluence and UV exposure in addition to the MISSE-FF sensor suite. Teflon's erosion yield increases with UV exposure (ref. 2) and given the UV data from the sensor, the AO fluence can be calculated.

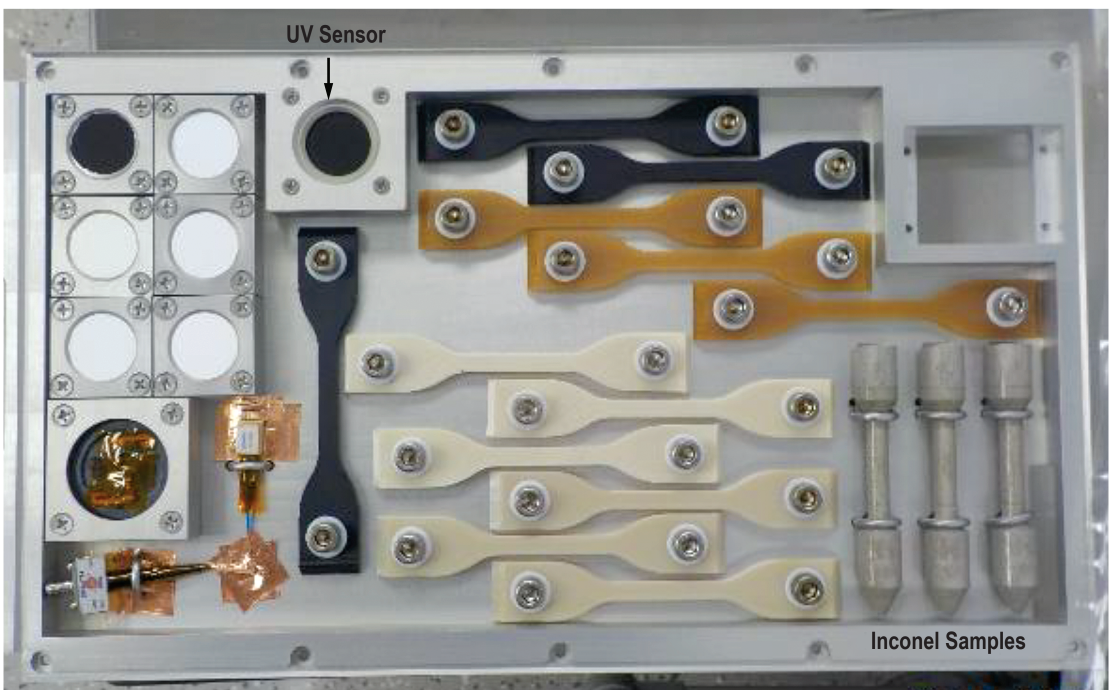

Figure 16. Additively manufactured samples and UV sensor integrated on MISSE-9 wake tray. (Photo credit: Alpha Space and NASA)

UV sensors are also included on zenith (fig. 17) and ram (fig. 18) facing trays. All were launched on the SpaceX commercial resupply mission SpX-14 April 2, 2018. The MISSE-9 experiments and the MISSE Flight Facility (MISSE-FF) were installed on the ExPRESS Logistics Carrier ELC-2 and activated on April 8, 2018. Downlinked sensor data and MISSE-FF camera photos at time of publication indicate nominal performance.

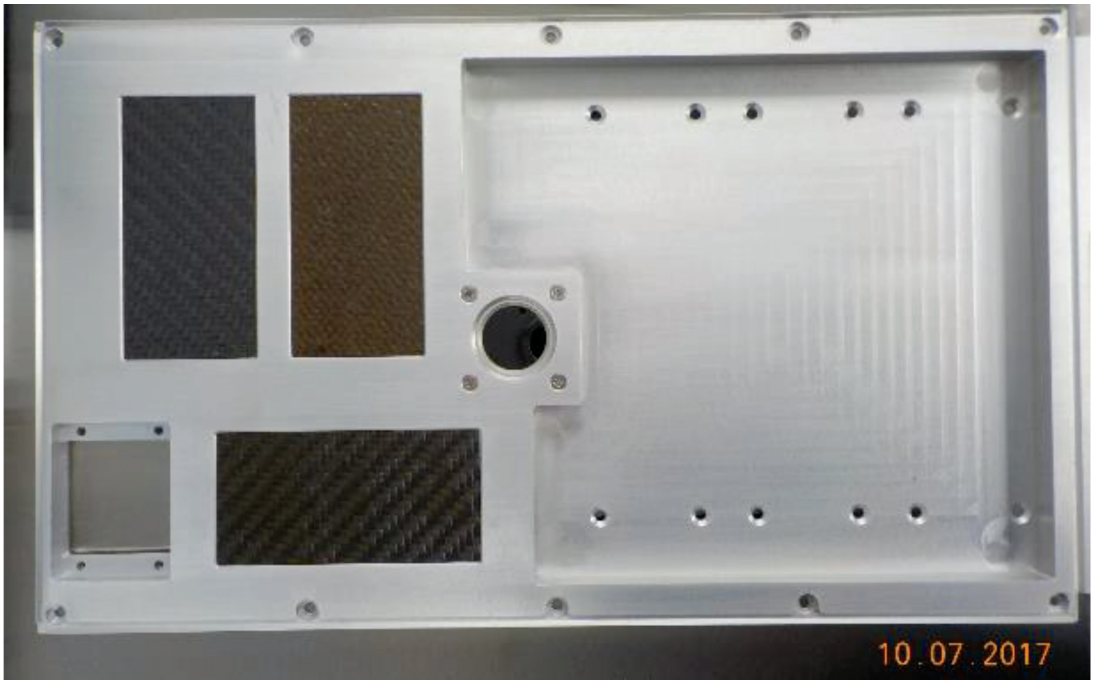

Figure 17. UV sensor in center of MISSE-9 zenith tray.

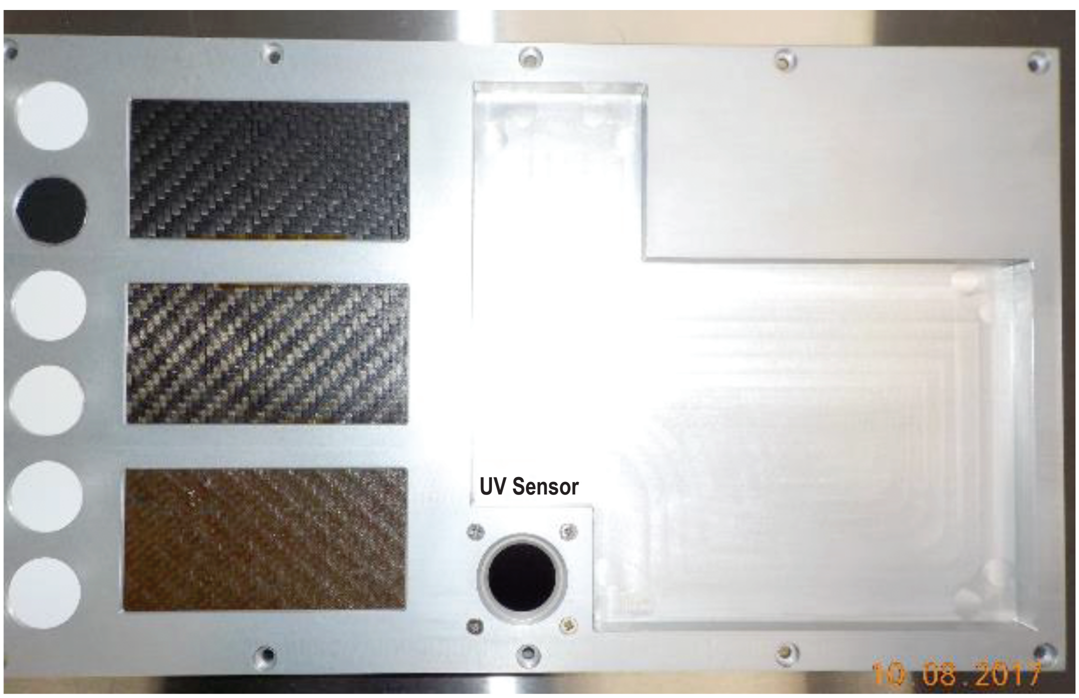

Figure 18. UV sensor integrated on MISSE-9 ram tray.

7. PLANNED FUTURE WORK

MSFC has been awarded space on the MISSE-10 nadir-facing tray. Although these samples will not be exposed to much AO and only a little albedo UV, they will be exposed to thousands of thermal cycles while in hard vacuum. A duplicate set of Inconel, Ultem 9085, Ultem 1010 and ESD-PEKK samples have already been prepared for the MISSE-10 flight. There is space for four more samples (fig. 19), so PC-ISO samples have been prepared and characterized, with the idea that by darkening, they will indicate how much albedo UV the nadir tray receives. Launch is currently scheduled for November 2018.

This phase of the project has focused on AO erosion and optical property changes. Once the MISSE-9 samples are returned, the full set of control samples and ground test samples will be mechanically tested. This will provide data on embrittlement due to UV radiation and any effects of thermal cycling.

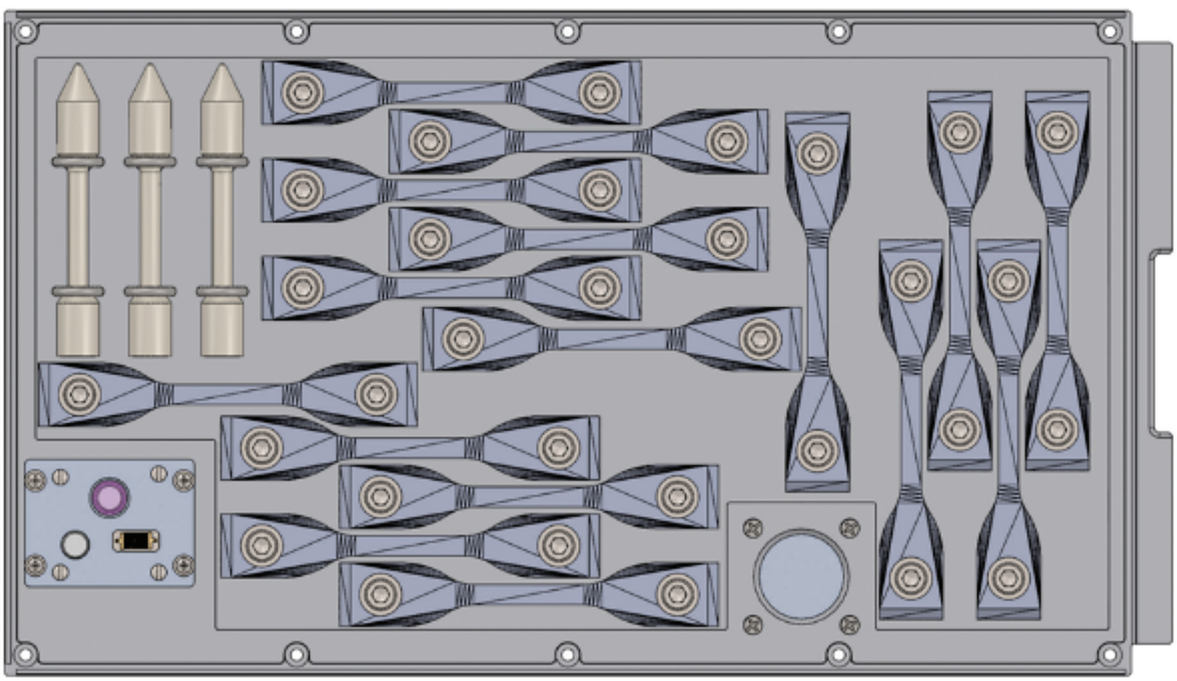

Figure 19. Proposed layout for MISSE-10 additively manufactured materials experiment.

REFERENCES

1. De Groh, K.K.; Banks, B.A.; Mccarthy, C.E.; et al.: “MISSE 2 PEACE Polymers Atomic Oxygen Erosion Experiment on the International Space Station,” *High Perform. Polym.*, Vol. 20, No. 4–5, pp. 388–409, August 2008.

2. Pippin, H. G., E. Normand, S. L. B. Woll, and R. Kamenetzky. “Analysis of Metallized Teflon™ Thin-Film Materials Performance on Satellites,” *J. Spacecraft Rockets,* Vol. 41, No. 3, pp. 322–325, May 2004.

REPORT DOCUMENTATION PAGE					*Form Approved* *OMB No. 0704-0188*

1. REPORT DATE *(DD-MM-YYYY)*
01–10–2018

2. REPORT TYPE
Technical Publication

3. DATES COVERED *(From - To)*

4. TITLE AND SUBTITLE
Space Environmental Effects on Additively Manufactured Materials

5a. CONTRACT NUMBER

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)
M.M. Finckenor

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
George C. Marshall Space Flight Center
Huntsville, AL 35812

8. PERFORMING ORGANIZATION REPORT NUMBER
M–1473

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
National Aeronautics and Space Administration
Washington, DC 20546–0001

10. SPONSORING/MONITOR'S ACRONYM(S)
NASA

11. SPONSORING/MONITORING REPORT NUMBER
NASA/TP—2018–220123

12. DISTRIBUTION/AVAILABILITY STATEMENT
Unclassified-Unlimited
Subject Category 29
Availability: NASA STI Information Desk (757–864–9658)

13. SUPPLEMENTARY NOTES
Prepared by the Nonmetallic Materials Branch, Nonmetallic Materials and Manufacturing Division

14. ABSTRACT
Space simulations including thermal vacuum, atomic oxygen, and ultraviolet radiation were performed to study the durability of various additively manufactured materials. In addition to ground simulations, additively manufactured materials were selected for a one-year flight on the Materials on International Space Station Experiment (MISSE) Flight Facility. The space environment is composed of atomic oxygen, ultraviolet radiation, protons, electrons, meteoroid/space debris impacts, thermal cycling, and hard vacuum. An improved UV sensor is also discussed.

15. SUBJECT TERMS
additive manufacturing, 3D printing, space environment, International Space Station, atomic oxygen

16. SECURITY CLASSIFICATION OF: a. REPORT	b. ABSTRACT	c. THIS PAGE	17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
U	U	U	UU	28	STI Help Desk at email: help@sti.nasa.gov
					19b. TELEPHONE NUMBER *(Include area code)* STI Help Desk at: 757–864–9658

National Aeronautics and
Space Administration
IS02
George C. Marshall Space Flight Center
Huntsville, Alabama 35812