Charge Enhanced Contamination and Environmental Degradation of MISSE-6 *SUSpECS* Materials

JR Dennison, Amberly Evans, Danielle Fullmer and Joshua L. Hodges

***Abstract*—The effects of prolonged exposure to the LEO space environment and charge-enhanced contamination on optical, thermal, and electron emission and transport properties of common spacecraft materials have been investigated by comparing pre- and post-flight characterization measurements. The State of Utah Space Environment and Contamination Study (*SUSpECS*) deployed in March 2008 on board the Materials International Space Station Experiment (MISSE-6) payload, was exposed for ~18 months on the exterior of the International Space Station (ISS), and was retrieved in September 2009. A total of 165 samples were mounted on three separate *SUSpECS* panels on the ram and wake sides on the ISS. Some samples, particularly those exposed to atomic oxygen in the ram direction, showed pronounced effects due to exposure. Biased samples for the charge-enhanced contamination study showed subtle variations in visible and infrared reflectivity.**

***Index Terms*—Charging, contamination, space environment effects, materials.**

I. Introduction

A cooperative, Utah-based project named *SUSpECS* (*S*tate of *U*tah *Sp*ace *E*nvironment and *C*ontamination *S*tudy) was developed as a flight experiment to study the effects of prolonged exposure to the space environment and charge-enhanced contamination on spacecraft materials. Utah researchers from the Utah State University (USU) Materials Physics Group (MPG), the USU Space Dynamics Laboratory (SDL) Contamination Control/Materials Chemistry Group, the ATK Space Systems Health Management Focus Group, and the USU Get-Away Special (GAS) Team built sample trays for flight on the MISSE-6 (*M*aterials *I*nternational *S*pace *S*tation *E*xperiment) mission sponsored by Air Force Office of Scientific Research (AFOSR). The MISSE program objective is to "characterize the performance of new prospective spacecraft materials when subjected to the synergistic effects of the space environment" [1]. The *SUSpECS* sample panels include pertinent materials and coatings selected and characterized by each group member for a comprehensive study of the effects of the low Earth orbit (LEO) space environment and contamination on electrical, mechanical, and optical properties of materials related to several on-going projects of high relevance to manned space exploration and other long duration space missions [2].

Sample material selections, conceptual design of the *SUSpECS* sample panels, and construction of the panels were completed during 2005, led by student researchers from the USU GAS Team. Design of the sample panels are described below, including a three tiered configuration intended to provide variable atomic oxygen and ultraviolet radiation exposure. The *SUSpECS* sample panels were delivered to Boeing in spring 2006 for integration with the panels contributed by other industry, university, and government investigators. The sample panels were installed into two standard MISSE "suitcase" pallets (PECs) that were powered and instrumented to record relevant space environmental parameters during the on-orbit exposure. The integrated payload was delivered to NASA Langley Research Center in summer 2006. The Shuttle flight STS-123 transported MISSE-6 to the ISS and deployed it on the ISS "back porch" in March 2008. MISSE-6 was returned to Earth in September 2009 and the SUSpECS sample trays were de-integrated from the MISSE PECs at NASA Langley Research Center in October 2009. Photographs of various aspects of the deployment and retrieval are shown in Ref [3].

II. *SUSpECS* Sample Sets

A. *Sample Selection for Materials Studies*

The samples for flight were carefully chosen to provide needed information for several different ongoing studies and to cover a broad cross-section of prototypical materials used on the exteriors of spacecrafts. Table I lists the samples selected for inclusion on the *SUSpECS* sample panels.

Results reported here focus on the comparison of two specific sets of materials samples. The first comparison focuses on six sets of four identical samples [Au, Al, carbon-loaded polyimide (Dupont Black Kapton™ 100XC), and carbon-loaded polyester (Sheldahl Thick Film Black)]. Two sample sets were located on the top and bottom tiers of a

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three-tiered sample panel designed to provide variable atomic oxygen and UV exposure. The four other sample sets were located on the wake side sample panel, with each set held at constant bias for the duration of the flight. The biased sample configuration was designed to approximate typical conditions of materials subject to charge-enhanced contamination due to spacecraft charging by actively biasing samples to low positive and negative voltages.

The second comparison reported here focused on four materials [carbon-loaded polyimide, aluminized polyester (Dupont Mylar™), Al_2O_3 (sapphire), and SiO_2 (quartz)] that showed varying degrees of environmentally–induced changes in optical properties. Samples of each material on the wake and three-tiered sample panels were exposed to a complex environment during the flight. Identical witness samples were also exposed to a simulated subset of the environment in the Characterization of Combined Orbital Surface Effects (CCOSE) space environment test chamber at the USAF Arnold Engineering Development Center (AEDC) to mimic the space exposure profile [3-5]. The primary optical characterization methods employed for the comparison were UV/VIS/NIR and FTIR transmission of the sapphire and quartz and UV/VIS/NIR reflectance of the polyimide and polyester. Comparison of pre-flight, post-flight, and simulated exposure samples served two primary purposes: (i) to investigate the validity of simulated environmental testing methods and (ii) to help distinguish the effects of specific components of the complex space environment that samples were simultaneously exposed to during the flight. Initial results of the CCOSE tests were reported in Ref. [3].

Four additional *SUSpECS* test programs with direct relevance to spacecraft charging issues are briefly outlined below. These are studies of electron emission and resistivity of typical spacecraft materials, CRRES materials charging and contamination, ISS materials charging and contamination, and the effects of contamination on FPMU materials.

Electron-, ion-, and photon-induced electron emission yield curves, crossover energies and emission spectra, resistivity, dielectric strength, optical and electron microscopy, UV/VIS/NIR reflection spectroscopy, and emissivity were tested for pre-flight *SUSpECS* samples in their pristine conditions. The majority of the test samples have already undergone pre-flight analysis during an ongoing seven year study of the electron emission [6-11] and resistivity properties [1], [7], [12-14] of spacecraft materials sponsored by the NASA Space Environments and Effects Program. Preliminary ground-based studies at USU have shown that contamination can produce dramatic changes in electron emission that can lead to severe charging effects under certain circumstances [15,16]. A preliminary study of the effects of contamination on resistivity using the charge storage method is underway at USU. Comparison with post-flight analysis will provide the first extensive tests of space environment exposure and contamination on electron emission properties and resistivity.

Several types of samples were flown aboard the CRRES satellite [17] as part of a study of charging induced arcing [18]. The samples were the subject of detailed resistivity tests using the charge storage method [19] and very successful modeling of their pulsing history during the CRRES flight [12,20]. The MISSE-6 tests will support modeling of the effects of prolonged space exposure during the CRRES flight. Relevant samples include Kapton™ (PI), Teflon™ (PTFE), Mylar™ (PET), FR4 PC board (PI composite) material, alumina (Al_2O_3), and silicon dioxide (SiO_2).

Table I. *SUSpECS* samples.

Material	Source	# Samples
Kapton (PI) on Aluminum	Sheldahl	4
Teflon (PTFE) on Aluminum	Sheldahl	3
Mylar (PET) on Aluminum	Sheldahl	3
Nylon 6/6	McMaster-Carr	1
SiO2 (Fused Quartz)	UQG Optics	3
Al2O3 (Sapphire)	UQG Optics	2
Germanium on Kapton	Sheldahl	1
Anodized Aluminum (Chromic Acid Etch)	NASA / MSFC	1
Anodized Aluminum (Sulfuric Acid Etch)	NASA / MSFC	1
UV AR-coated Ce-doped Cover Glass	Thales	3
FR4 Printed Circuit Board (PI Composite)	CRRES NASA / JPL	3
CV-1147 RTV on Copper	Nusil/Boeing	1
DC93-500 RTV on Copper	Dow -Corning/Boeing	2
Zinc Oxide White Paint (Z93-C55)	Alion	3
Borosilicate BK7 Glass	UQG Optics	3
Gold (99.99% Purity)	ESPI	7
Aluminum (99.999% Purity)	ESPI	7
316 Stainless Steel	McMaster-Carr	2
OFHC Copper (99.9% Purity)	McMaster-Carr	1
Silver (99.999% Purity)	United Materials	3
g-C (Graphitic Amorphous Carbon) on Copper	Arizona Carbon Foil	1
Aquadag (microcrystalline C) on Copper	LADD Research	2
100XC Black Kapton	Sheldahl	6
Thick Film Black	Sheldahl	6
Inconnel on Silver on Teflon on ITO	Sheldahl	1
ITO on Teflon on Silver on Inconel	Sheldahl	1
Gold(2um)/Nickel(2um) on 316 Stainless Steel	Gold Plating Services	3
Rh(2um)/Ni(2um) on 316 Stainless Steel	Gold Plating Services	3
Au(2um)/Rh(2um) on 316 Stainless Steel	Gold Plating Services	3
Au(2um)/Rh(2um)/Ni(2um) on 316 Stainless Steel	Gold Plating Services	3
Reinforced Carbon Nano-fiber/RS-3 Cyanate Ester Composite	SDL GIFTS Satellite	2
AS/N720 Oxide Ceramic-Metal Composite	COIC/ATK Thiokol	5
S200 Nonoxide Ceramic-Metal Composite	COIC/ATK Thiokol	5
Thiokol Graphite Epoxy Foil - No Hole	ATK Thiokol	5
Thiokol Graphite Epoxy Foil - With Hole	ATK Thiokol	5
Thiokol Carbon-Carbon Composite #1	ATK Thiokol	5
Thiokol Carbon-Carbon Composite #2	ATK Thiokol	5
Thiokol Fiber-Filled Carbon-Carbon Composite	ATK Thiokol	5
Thiokol Carbon-Phenolic Carbon-Carbon Composite	ATK Thiokol	5
S400 Nonoxide Ceramic-Metal Composite	COIC/ATK Thiokol	2
S200H Nonoxide Ceramic-Metal Composite	COIC/ATK Thiokol	2
S300 Nonoxide Ceramic-Metal Composite	COIC/ATK Thiokol	2

A study of the electron emission and resistivity properties of a set of materials used to construct the ISS has been performed. This includes both basic materials [Au, Al, 316 SS, anodized Al (chromic acid etch), anodized Al (sulfuric acid etch), Kapton™, Dupont Black Kapton™, and UV AR-coated Ce-doped cover glass] [7] and a study of two RTV materials (DC93-500 and CV-1147) thought to be key contaminants of the ISS solar arrays [11]. Comparison of analysis of these MISSE-6 samples with pre-flight testing will provide valuable information for modeling the ISS spacecraft charging as the station ages.

A study was undertaken of the electron emission and resistivity properties of a set of materials used to construct the Floating Potential Measurement Unit (FPMU). The FPMU is an instrument designed and built at SDL for use on the ISS [21,22] used to monitor spacecraft charging on the ISS [23-26] through plasma measurements. The sample set includes both basic materials used to construct the FPMU [Au, 316 SS, Aquadag™] and two RTV materials (DC93-500 and CV-1147) thought to be potential key contaminants of the FPMU [7,27]. The electron emission properties and resistivity of the materials, and how these properties change with exposure to

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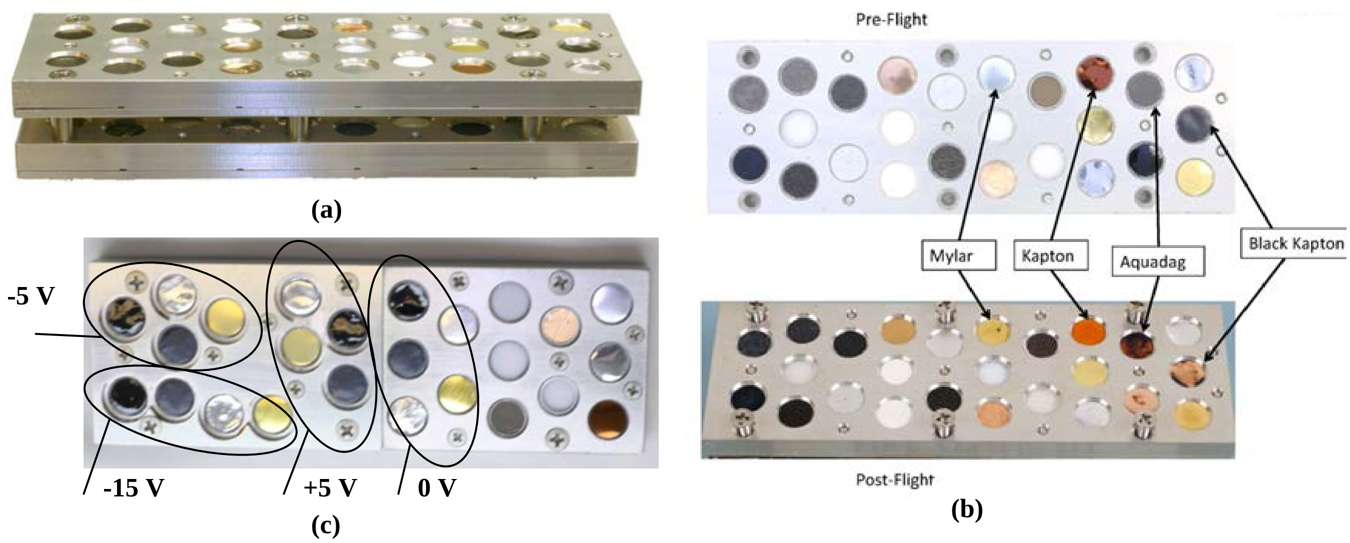

Figure 1. Configuration of 5 cm x 30 cm, 78 cm^2 *SUSpECS* sample panels. (a) Side view of ram side *SUSpECS I* sample panels. All samples are passive experiments held at ground potential. A three tiered configuration design is used with 25 samples exposed on each tier. (b) Pre- and post-flight photographs showing locations of samples shown in Figure 2. (c) Wake side *SUSpECS II* sample panel. Thirteen exposed samples at right are passive experiments held at ground potential. The three sub-panels at left each contain four identical samples held at + 5 VDC, -5 VDC and –15 VDC, respectively.

the space environment and accumulation of contamination, are critical to the precise determination of the surface potentials. Comparison of analysis of these MISSE-6 samples with pre-flight testing will provide valuable information for modeling the FPMU electron emission and the instrument effectiveness in monitoring the ISS potential as the station ages.

Additional studies of critical thermal control and optical coating materials for the USU SDL Geosynchronous Imaging Fourier Transform Spectrometer (GIFTS) composites, mechanical and thermal properties of ATK Thermal Protection Systems and Lightweight Structure Systems materials, and NASA Solar Probe Mission composite and heat shield materials have been described elsewhere [2].

B. *Space Environment Exposure of Samples*

The *SUSpECS* study exposed three test panels of materials—*SUSpECS I, II and III*—to the LEO environment for ~18 months. Environmental monitoring on board the MISSE-6 suitcases included temperature monitoring at a number of points on each pallet. Atomic oxygen (AO) exposure was monitored by the degradation of Kapton™ strips [28-30] placed on the 6A and 6B PECS; the AO fluence was $2{\cdot}10^{21}$ atoms/cm^2 with an estimated variation of ~5% [31]. Solar ultraviolet (UV) exposure as a function of time was monitored with UV photodiodes at several locations. Absolute absorbed radiation dosage was monitored with several thermoluminescent detectors (TLD). The Air Force MISSE-6 experiment also monitored the electron flux in the 0-200 eV regime. Specific details of space environment exposure for *SUSpECS* sample holders and CCOSE space simulation tests are discussed in Ref. [3].

C. *Ram Side Sample Panel Design and Configuration*

One sample panel shown in Figure 1(a), *SUSpECS I,* was mounted on PEC 6A on the ram side of the ISS, with enhanced exposure to atomic oxygen. These experiments were all passive LEO exposure experiments. Details of the sample mount are given in Ref. [3]. This panel included 98 1.3 cm diameter (1 cm exposed diameter) conducting and insulating samples held at ground potential. The specific samples are identified in Table I.

The ram-side sample holder was configured so that four stacked sample tiers were exposed to AO+UV, AO alone (2 sets), and no AO or UV. All these materials were tightly seated in a metal tray. The sample geometry was designed such that the sides of each tier were masked, allowing only front face exposure and forcing any diffusion into a one-dimensional regime. This will permit one-dimensional depth profiling of the materials to evaluate the effects of environmental exposure. The outermost tier experienced the fullest exposure to all of the variables of LEO environment, most importantly atomic oxygen and ultraviolet radiation. The lower tiers, being shielded by the outermost layer, did not have exposure to ultraviolet radiation. Due to a gap between the second and third tiers in the stacked configuration, the second and third tiers were exposed to reduced fluxes of atomic oxygen. The lowest tier was fully shielded from ultraviolet radiation and atomic oxygen by the third tier. In addition to the MISSE-6 onboard monitors of UV and AO flux, the cumulative fluence at various points on SUSpECS was also monitored. AO exposure was monitored [30] by the relative oxidation of high purity Ag strips [28], [29], [32] and the degradation of Kapton™ strips [28], [29] placed on the frame of each tier. UV exposure is monitored by the discoloration of 1.3 cm diameter, 1 cm thick borosilcate BK7 glass sample disks mounted on each tier as color centers are formed by the UV radiation.

D. *Wake Side Sample Panels Design and Configuration*

SUSpECS II and III sample panels faced the wake side of the ISS on PEC 6B, with less exposure to atomic oxygen. *SUSpECS III* was fully passive with 25 mounted in a sample holder like the bottom tier of *SUSpECS I*. *SUSpECS II* had 13 1.3 cm diameter passive exposure test samples held at ground,

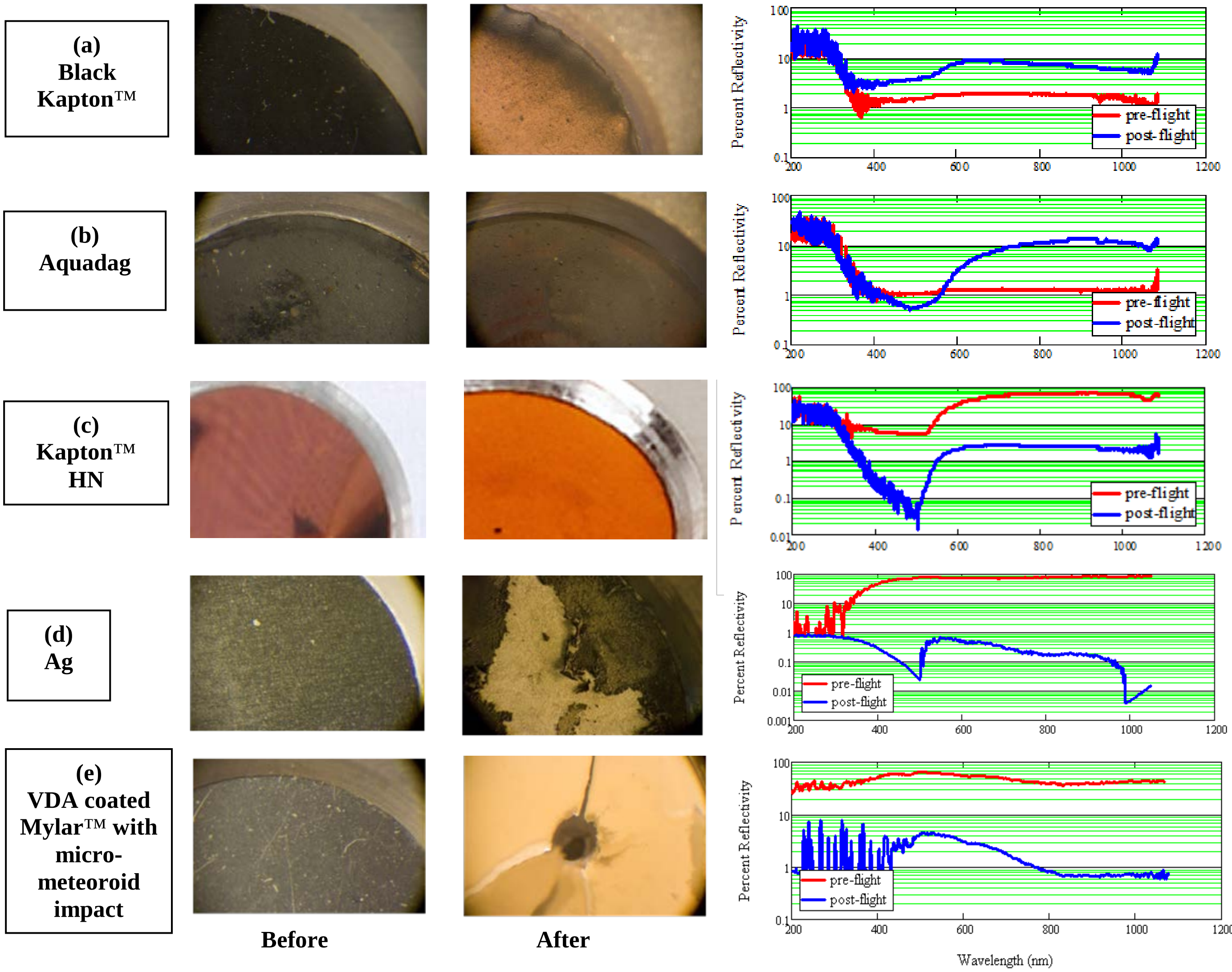

Figure 2. Comparison of pre- and post-flight photographs and UV/VIS/NIR reflectance spectra of samples from *SUSpECS II* on the ram side with high AO exposure. (a) Black Kapton™ 100XC, (b) Aquadag colloidal graphite coating on Cu substrate, (c) Kapton™ HN and (d) bulk Ag. (e) Vapor-deposited Al coated. Note the apparent micrometeoroid impact and the full AO oxidation of the Al of the VDA coated Mylar™ sample. Sample locations are shown in Figure 1(b).

as shown in the right hand side of Figures 1(c). Additional grounded samples were mounted underneath the exposed samples. The specific samples are identified in Table I.

SUSpECS II also had the sole active experiment. There were three separate test sub-panels of ~13 cm^2, each with four conducting samples mounted on *SUSpECS II*, as shown in Figures 1(c). These three sub-panels were held at + 5 VDC, -5 VDC and –15 VDC, respectively, for the full duration of the flight. Although these sample potentials could not be directly verified during flight, pre- and post-flight continuity and isolation tests confirmed intact circuitry. Voltages for the sub-panels were provided by the ISS through the MISSE-6 bus. Resistors and fuses were mounted in series with each sub-panel to limit arcing currents. A grounded sample guard was positioned above the three sub-panels to minimize possible contact with biased sub-panels by astronauts during EVAs. The beveled edges of the sample clamp and guard shield were designed to minimize fringing fields to provide nearly parallel voltage contours typical of larger biased samples, as shown by field simulations (see Figure 3 in Ref [2]).

The biased sample configuration was designed to approximate typical conditions of materials subject to spacecraft charging. Based on a space plasma environment current density of ~10 nA-cm^{-2}, the three biased plates collectively drew <1 μA. The positive test bias was chosen as + 5 V. Positively charged components will typically charge to only a few volts positive [33], since low energy emitted electrons will be re-attracted to a positively charged surface and the majority of emitted electrons have energies below ~5 eV [6]. By contrast, negatively charged materials can charge to large voltages, since emitted electrons are repelled from the charged surface and therefore do not self-limit charging, as is the case for positive biasing [6]. Biases of -5 V and -15 V were chosen as representative of modest and more extreme negative charging.

III. Testing

A. *Materials Testing*

Comparison of post-flight analysis of these MISSE-6

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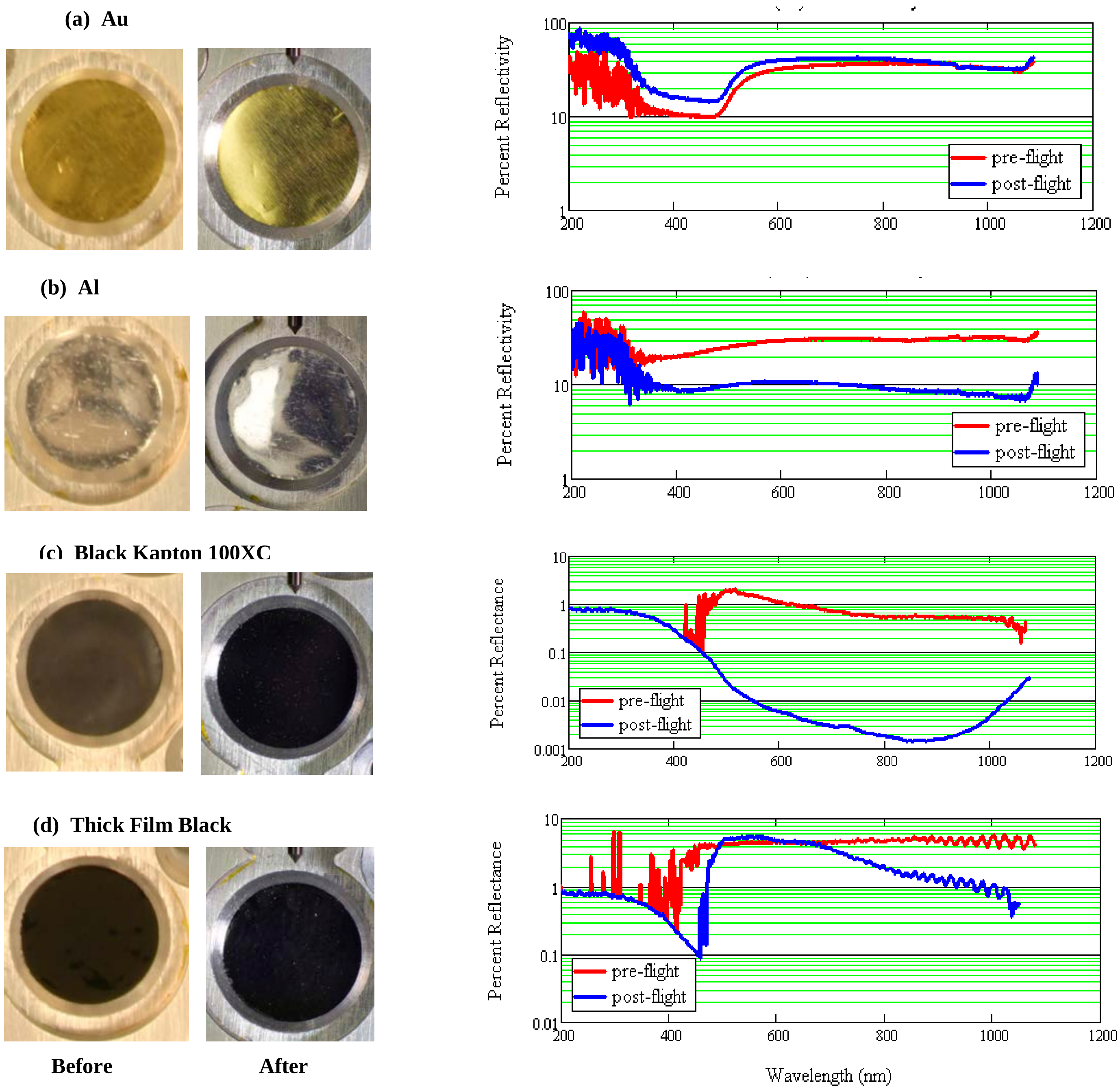

Figure 3. Comparison of pre- and post-flight photographs and UV/VIS/NIR reflectance spectra of -5 V charge samples with wake exposure on *SUSpECS I*. (a) Au, (b) Al, (c) Carbon-filled polyimide or Black Kapton™ 100 XC, (d) Carbon-filled PET or Thin Film Black.

samples with pre-flight testing will be valuable in trying to identify and model materials degradation and aging and the effects of prolonged space exposure on the samples. All samples will undergo an extensive series of pre-flight and post-flight tests to characterize the materials properties, including surface morphology [optical microscopy, scanning electron microscopy (SEM), scanning tunneling microscopy (STM)], chemical compositions, [standard suite of chemical analysis tests such as HPLC, Auger Electron Spectroscopy (AES), Secondary Ionization Mass Spectroscopy (SIMS) and X-Ray Photoelectron Spectroscopy (XPS)], optical (IR-VIS-UV attenuated total (ATR), specular and/or diffuse reflection spectroscopy [34]), thermal (thermal expansion, thermal emissivity and absorptivity), and outgassing.

B. *Electrical Properties of Spacecraft Materials*

The electron emission properties and resistivity of many *SUSpECS* materials will be tested. Specifically, the materials will be tested for resistivity and dielectric strength, and for electron-, ion- and photon-induced electron emission yield curves and emission spectra. Details of the testing procedures are described in Refs. [7,35]. Much of the pre-flight testing has already been done in conjunction with previous studies.

The electron emission and transport properties of materials are key parameters in determining the likelihood of deleterious spacecraft charging effects [7,33,36,37] and are essential in modeling these effects with engineering tools such as the NASA NASCAP-2K [38-40], SPENVIS, and MUSCAT [41] codes. The *SUSpECS* studies of electron emission and resistivity will extend more than a decade of research in the field by the USU MPG [3,6-13,19,40,42-43].

Recent work [13,44] found that dissipation of charge accumulated on thin film insulating spacecraft surfaces during on-orbit conditions is substantially slower than predicted using resistivity values acquired by standard ASTM methods [45]. This can result in charge dissipation on the order of days to months rather than minutes to hours [12]. More appropriate methods to measure charge storage decay have been developed. Apparatus to measure the decay rate of charge deposited on the surface of thin film insulators have been designed and built at USU in conjunction with an on-going NASA research project with JPL [14] and the USU electron emission test chamber [46]. Comparison of pre- and post-flight analysis of *SUSpECS* samples using these methods will provide a better understanding of modifications to these long decay times as a result of space exposure and contamination.

C. *Pre- and Post-Flight Comparisons*

Measurements of the optical microscopy and normal specular UV/VIS/NIR reflectance of selected pre- and post-flight samples that exhibited significant changes are presented in Figures 2 and 3. These preliminary results can be compared to assess on–flight degradation.

Figure 2 shows results for five samples from *SUSpECS II* on the ram side with high AO exposure. The first three materials--(a) Black Kapton™ 100XC, (b) Aquadag™ colloidal graphite coating on Cu substrate, and (c) Kapton™ HN—all exhibit significant material loss and changes in color evident in both the photographs and the reflection spectra. Presumably, these changes are due to strong AO oxidation of these carbon-based materials. The bulk Ag sample (d) also exhibits significant oxidation. Work is underway to compare the results of the Kapton™ HN and Ag AO changes, to investigate whether the Ag represents a viable alternative as an AO fluence sensor. The changes observed in the vapor-deposited Al coated Mylar™ (PET) sample (e) are perhaps the most dramatic. It appears that the AO oxidation has completely removed the ~100 nm thick VDA coating. There is also what appears to be a micrometeoroid impact site.

D. *Charge-Induced Contamination Study*

A primary focus of *SUSpECS* is the study of the effects of contamination on the accumulation, re-emission, and dissipation of charge from spacecraft surfaces and on the resulting changes in electron emission and resistivity of spacecraft materials [40]. This project also investigates the effects of charging on contamination rates. Synergistic phenomena in the space environment (*e.g.*, charging, contamination, UV exposure, atomic oxygen) can cause dramatic changes in material surface properties and performance [3,47]. Thin contaminant layers readily change the optical [34,42,48] and electronic properties [15,16] of surfaces, and often result in long-term degradation of the optical, thermal control, or electronic performance of space-based sensors and components. For example, plasma diagnostic instrumentation (such as Langmuir and plasma impedance probes) requires stable surface conductivity and charging properties, which is altered by contamination [42]. Further, at geosynchronous orbits, high spacecraft charging potentials (typically tens of kilovolts) and long Debye lengths can actually accelerate surface contamination rates by electrostatic re-attraction of ionized outgassed or vented molecules to the negatively charged satellite [49]. Accelerated contamination rates can affect the long-term performance of optical, thermal control, or solar panel surfaces. Also the performance of new high efficiency multijunction solar cells is more susceptible to current loss caused by contamination than conventional single junction cells [34].

Studies at USU have shown that very thin layers of contamination—even a few monolayers—can potentially cause significant changes in electron emission properties that can dramatically affect the charging of satellites and can lead to catastrophic charging effects under certain circumstances [15], [16]. Figure 5 in Ref. [16] shows the threshold differential charging of clean Au and carbon-contaminated Au surfaces on a hypothetical satellite in GEO orbit. However, little direct information is available on the effects of sample deterioration and contamination on the electron emission and resistivity of materials flown in space.

The comparisons presented in Fig. 3 focus on six sets of four identical samples [Au, Al, carbon-loaded polyimide, and carbon-loaded polyester]. Two sample sets were located on the top and bottom tiers of a three-tiered sample panel designed to provide variable atomic oxygen and UV exposure. The four other sample sets were located on the wake side sample panel, with sets biased for the duration of the flight at 0 VDC, +5 VDC, -5 VDC, and -15 VDC, respectively.

Comparison of pre- and post-flight photographs of the four biased wake sample sets (Figure 5), show no significant changes are apparent in the visible, in marked contrast to extensive sample modifications observed for some ram samples (Figure 3). Comparison of pre- and post-flight NIR/VIS reflectivity spectra provide a more sensitive test and are consistent with minimal changes observed in the visible region in the photographs. All four samples show little change for wavelengths less than 300 nm to 450 nm. Au shows minimal change over the full spectral range; minimal changes due to contamination would be expected for the inert Au surfaces [34]. The other three samples show reduction of already low reflectivity for most wavelengths >400 nm. Variations of the magnitude of the reduced reflectivity with wavelength—especially in the NIR/VIS above 400 nm—are more consistent with wavelength dependant absorption from contamination layers than from the generally uniform reductions in reflectivity that result from surface roughening [34]. Thin film interference fringes observed at wavelengths above ~850 nm for the carbon-loaded polyester samples suggest there is a fairly uniform ~20 μm thick polyester film above the highly absorbing carbon-loaded bulk [34]. The fact that similar fringes are still present in the post-flight spectrum suggests that this layer was not significantly modified during space exposure. The reduction in reflectivity for the post-flight film is consistent with formation of a thin film contaminate layer. While all the observed spectral changes are consistent with formation of a thin film absorbing contamination layer with preferential absorption in the NIR, further measurements and analysis are required to more fully determine the changes in materials properties that result from charge-enhance contamination.

IV. Future Work

Work on analysis of the effects of space environment exposure on the 168 samples has only begun. Measurements of optical and electron microscopy, reflectivity, FTIR, emissivity, mass loss, electron-, ion- and photon-induced electron emission, photoyield, AES, photoemission, and variable angle UV/VIS/NIR reflectivity will continue. Work will also progress in collaboration with the AEDC space simulation facility to understand the origins of these effects and quantify their impacts.

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