

# Flight Experiments on the Effects of Contamination on Electron Emission of Materials

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We report on a study of the effects of prolonged exposure to the space environment and of charge-enhanced contamination on the electron emission and resistivity of spacecraft materials. The State of Utah Space Environment & Contamination Study (SUSpECS) was deployed on the International Space Station (ISS) in March 2008 onboard the MISSE-6 payload during STS-123. The Materials International Space Station Experiment (MISSE-6) program is designed to characterize the performance of candidate new space materials over the course of its ~17 month exposure to the LEO environment, with a target return date of August 2009 on STS-127. Approximately 165 samples are mounted on three separate SUSpECS panels in the ram and wake sides on the ISS. They have been carefully chosen to provide needed information for different ongoing studies and a broad cross-section of prototypical materials used on the exteriors of spacecrafts. Design of the sample panels are detailed, including a three tiered configuration intended to provide variable atomic oxygen and ultraviolet radiation exposure. The methods used to simulate charge enhanced contamination by actively biasing samples to low positive and negative voltages are also described.

A primary emphasis of SUSpECS is the study of modifications to the electron emission resulting from exposure to the space plasma environment and to environmental contamination. There is presently little available data related to the effects of sample deterioration and contamination on emission properties for materials actually flown in space. Electron emission and transport properties of materials are key in determining the amount of charge build-up and the time for the charge to dissipate, as well as the likelihood of deleterious spacecraft charging effects. Such materials properties are essential parameters in modeling spacecraft charging with engineering tools like NASCAP-2K code. SUSpECS studies will test the validity of our predictions from ground-based studies that very thin layers of contamination can lead to severe charging effects under certain circumstance. Electron-, ion-, and photon-induced electron emission yield curves, crossover energies and emission spectra, as well as resistivity and dielectric strength, have been tested for most SUSpECS samples in their pristine conditions before flight. These measurements will be compared with post-flight measurements. Additional pre- and post-flight characterization measurements include optical and electron microscopy, reflection spectroscopy, emissivity and Auger electron spectroscopy.

## I. Introduction

A cooperative, Utah-based project named *SUSpECS* (State of Utah Space Environment and Contamination Study) has developed 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, the USU Space Dynamics Laboratory Contamination Control/Materials Chemistry Group, the ATK Space Systems Health Management Focus Group, and the USU Microgravity Research Team (MRT) have built sample trays for flight on the MISSE-6 (*Materials International Space Station Experiment*) 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.

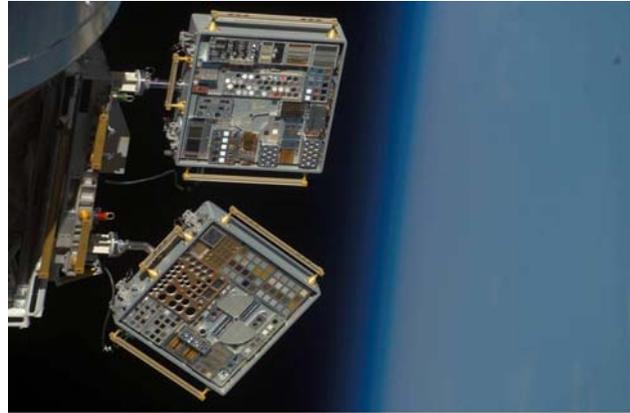
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Sample material selections, conceptual design of the *SUSpECS* sample panels, and construction of the panels were completed during 2005, led by student researchers from the USU Get-Away-Special Program. The *SUSpECS* sample panels were delivered to 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 that are 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. Figure 1 shows MISSE-6 mounted on ISS. MISSE-6 is scheduled for retrieval by STS-127 in August 2009, with return after ~17 months exposure in the LEO environment. After retrieval, sample panels will be returned to the *SUSpECS* team for post-flight analyses.



**Figure 1. MISSE 6 exposed to the space environment.** *The *SUSpECS* double stack can be seen in the bottom center of the lower case. The picture was taken on the fifth EVA, just after deployment on SYS-123. NASA Photo.*

The overall project has two key objectives: (i) basic research will extend our understanding of the materials/space environment interactions, and (ii) specific knowledge will be gained for critical materials in several on-going projects of the team members.

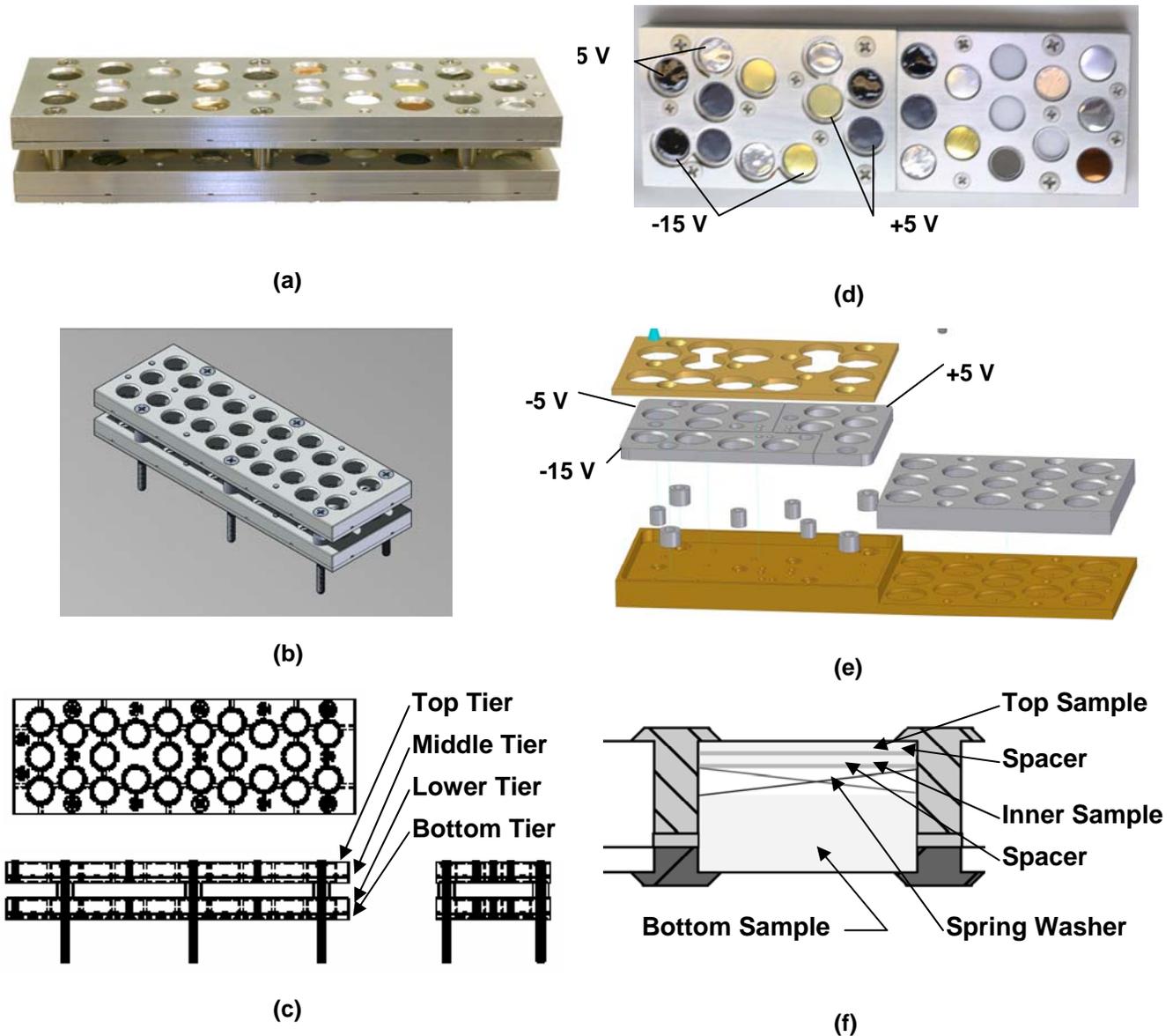
## II. Sample Panel Design

The *SUSpECS* study will expose two test panels of materials to the LEO environment for ~17 months. Environmental monitoring on board the MISSE-6 suitcases will include temperature monitoring at a number of points on each pallet. Atomic oxygen (AO) exposure will be monitored by the degradation of Kapton strips placed on the pallet frames, with an estimated sensitivity of ~25% AO variation [2], [3]. Solar ultraviolet (UV) exposure as a function of time will be monitored with UV photodiodes at several locations. Absolute absorbed radiation dosage will be monitored with several thermoluminescent detectors (TLD), as was done on MISSE-1 and -2. The Air Force MISSE-6 experiment will contain monitoring of the electron flux in the 0-200 eV regime.

### A. Ram Side Sample Panel Configuration

One *SUSpECS* sample panel will be mounted on the ram side of the ISS, with enhanced exposure to atomic oxygen. These experiments are all passive LEO exposure experiments. This panel will include 98 1.3 cm diameter (1 cm exposed diameter) conducting and insulating test samples held at ground potential, as shown in Figures 1(a-c). The specific samples are identified in Table I.

The ram-side sample holder is configured so that four stacked sample tiers will be exposed to AO+UV, AO alone (2 sets), and no AO or UV. All these materials will be tightly seated in a metal tray. The sample geometry is designed such that the sides of each tier will be 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 will experience 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, will 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 will have exposure to reduced fluxes of atomic oxygen. The lowest tier is 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* will also be monitored. AO exposure will be monitored by the relative oxidation of high purity Ag strips [2], [3], [4] and the degradation of Kapton strips [2], [3] placed on the frame of each tier. UV exposure will be monitored by the discoloration of 1.3 cm diameter, 1 cm thick borosilicate BK7 glass sample disks mounted on each tier as color centers are formed by the UV radiation.



**Figure 2. Configuration of 5 cm x 30 cm, 78 cm<sup>2</sup> SUSpECS sample panels.** (a-c) Ram side 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. (d-e) Wake side 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. (f) Cross sectional detail of typical stacked samples and sample clamping mechanism.

### B. Wake Side Sample Panel Configuration

The second *SUSpECS* sample panel will face the wake side of the ISS, with less exposure to atomic oxygen. The experiments are both passive and active LEO exposure experiments. This panel will include 13 1.3 cm diameter test samples held at ground, as shown in the right hand side of Figures 1(c-d). The specific samples are identified in Table I. Additional grounded samples are mounted underneath the exposed samples (see Table I).

There are also three separate test sub-panels of ~13 cm<sup>2</sup>, each with four conducting samples (Au, Al, Dupont Black Kapton, and Sheldahl Thick Film Black) mounted, as shown at left in Figs. 1(c-d). These three sub-panels will be held at + 5 VDC, -5 VDC and -15 VDC, respectively. Voltages for the sub-panels are provided by the ISS through the MISSE-6 bus. Current is drawn from interaction of the biased plates with the space plasma

environment. Based on a plasma current density of  $\sim 10 \text{ nA}\cdot\text{cm}^{-2}$ , the three biased plates will collectively draw  $<1 \text{ }\mu\text{A}$ . Resistors and fuses (MIL-PRF-23419) to limit arcing currents are mounted in series with each sub-panel.

A grounded sample guard is positioned above the three sub-panels to minimize possible contact biased sub-panels by astronauts during EVAs. The beveled edges of the sample clamp and guard shield have been designed to minimize fringing fields and provide nearly parallel voltage contours typical of larger biased samples. A field profile simulation is shown in Figure 3.

The biased sample configuration is designed to approximate typical conditions of materials subject to spacecraft charging. The positive test bias was chosen as +5 V. Positively charged components will typically charge to only a few volts positive [5], since low energy emitted electrons will be re-attracted to a positively charged surface and the majority of emitted electrons have energies below  $\sim 5$  to  $10 \text{ eV}$  [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  $-5 \text{ V}$  and  $-15 \text{ V}$  were chosen as representative of modest and more extreme negative charging.

### C. Sample Selection Criteria

The samples for flight have been 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.

The majority of the test samples have already undergone pre-flight analysis during an ongoing seven year study of the electron emission [7-11] and resistivity properties [7], [12-15] 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 [16]. A preliminary study of the effects of contamination of 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 CRESS satellite [15] as part of a study of spacecraft charging induced arcing [17]. The samples have recently been subjected to detailed resistivity tests using the charge storage method [18] and very successful modeling of their pulsing history during the CRESS flight [12,19]. The MISSE-6 tests will be valuable in trying to model the effects of prolonged space exposure during the CRESS flight. Relevant samples include Kapton (PI), Teflon (PTFE), Mylar (PET), FR4 PC board (PI composite) material, Alumina ( $\text{Al}_2\text{O}_3$ ), and Silicon Dioxide ( $\text{SiO}_2$ ).

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

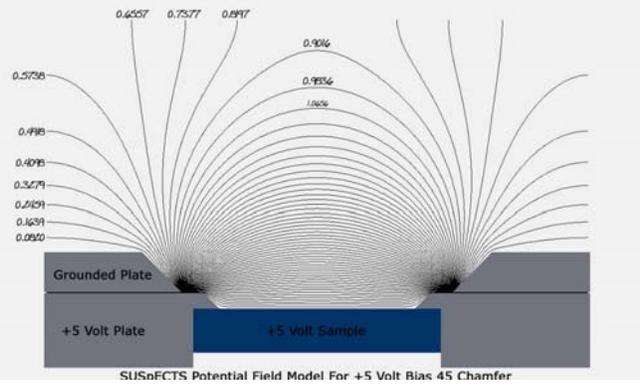


Figure 3. SIMION simulation of the electrostatic potential contour lines for a +5 VDC sample bias.

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
SiO <sub>2</sub> (Fused Quartz)	UQG Optics	3
Al <sub>2</sub> O <sub>3</sub> (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
Inconel 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

(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 of the electron emission and resistivity properties of a set of materials that were used to construct the Floating Potential Measurement Unit (FPMU) is currently underway. The FPMU is an instrument designed and built at SDL for use on the ISS [20], [21] intended to monitor spacecraft charging on the ISS [22-24] 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], [26]. The electron emission properties and resistivity of the materials, and how these properties change with exposure to the space environment and the 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.

Stringent requirements for uniform work function of the FPMU probes led to coating key elements with Au [20]. Another critical concern is non-uniform modification of the Au work function due to external contamination or to diffusion of alloying materials from underlying layers into the Au, particularly at elevated temperatures [25]. A set of samples [Au, Au(2 $\mu$ m)/Ni(2 $\mu$ m) on 316 SS, Rh(2 $\mu$ m)/Ni(2 $\mu$ m) on 316 SS, Au(2 $\mu$ m)/Rh(2 $\mu$ m) on 316 SS, Au(2 $\mu$ m)/Rh(2 $\mu$ m)/Ni(2 $\mu$ m) on 316 SS] will be included in the unexposed samples on *SUSpECS*. Although these samples will not be exposed to the external space environment, they will experience prolonged exposure to the thermal and vacuum environment. Separate ground based experiments will look at the effects of proposed heating to clean the FPMU.

Tests will be performed to study aging and degradation phenomenology of critical thermal control and optical materials currently in use or under development for SDL payloads. These include the inorganic white and black polymeric coatings used for GIFTS composites.

Composite and ceramic materials of the ATK Thermal Protection Systems (TPS) and the ATK Lightweight Structure Systems (LSS) are used on LEO bound and beyond ATK assets. These include materials for an ATK Thiokol project on snap-together structural components and solid rocket motor materials. A set of these composite materials will be evaluated for deleterious effects of AO, UV and other space environment exposure. Some ATK composite rocket motor cases include epoxy resin. Two of these epoxy resin types will be included in this LEO exposure experiment. Composite materials to be tested include a graphite fiber-filled composite, a carbon-carbon resin-impregnated graphite composite, a carbon-phenolic composite, and an anodized aluminum metal-coated composite. The proprietary ATK COI ceramic matrix composite (CMC) materials will be tested in an oxide form and a polymer derived non-oxide form.

The Health Management Focus Group (HMFG) of ATK Space Systems will perform material characterization as a function of time and/or environment using the proven ATK HMFG microspecimen technology tools for chemical and mechanical property analysis. Studies of the service life of these materials will evaluate chemical and mechanical properties of the ATK MISSE samples. Triplicate samples will be evaluated for each material: one for zero-time properties, one for ground-based aged properties, and one for space exposure. Thiokol samples will be subjected to mechanical tests, including destructive mechanical tests, microwave evaluations of changes in the relative dielectric function, and ultrasonic measurements. In addition, a series of microtome slices of the pertinent composite samples will be made to evaluate chemical and mechanical properties as a function of depth from the AO and UV exposure surface. Chemical analysis, including ATR and/or diffusive reflection spectroscopy, Auger, SIMS, XPS spectroscopies, and standard chemical suite of tests as applicable will also be performed on all microtome samples.

ATK Space Systems has worked on a series of programs during the past few years that dramatically improved our ability to assess the effects of aging based on fundamental science rather than the empirical approaches that have dominated the past. Significant progress has been made in the union of high fidelity data sources and high fidelity structural, damage, and failure models. The results of *SUSpECS* will provide much needed input data for these models as the ATK HMFG assesses the effects of exposure to the environment of LEO on these materials.

### **III. Testing**

#### **A. Materials Testing**

Comparison of post-flight analysis of these MISSE-6 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 including surface morphology tests [optical microscopy, scanning electron microscopy (SEM), scanning tunneling microscopy (STM)], chemical compositions tests, [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 tests (IR-VIS-UV attenuated total (ATR), specular and/or diffuse reflection spectroscopy), thermal tests (thermal expansion, thermal emissivity and absorptivity), and outgassing. Outgassing tests and some optical and morphology tests will be performed at SDL; other characterization will be done at Thiokol.

The electron emission properties and resistivity of most USU/SDL 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 [7]. Much of the pre-flight testing has already been done in conjunction with previous studies.

#### **D. Electrical Properties of Spacecraft Materials**

A primary focus of *SUSpECS* is the study 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. This project also investigates on 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 [27]. Thin contaminant layers readily change the optical [28] and electronic properties [16,29] 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 [25]. 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 [30]. The accelerated contamination rates can affect the long-term performance of optical, thermal control, or solar panel surfaces. Also, at all altitudes, the performance of new high efficiency multijunction solar cells is more susceptible to current loss caused by contamination than conventional single junction cells [31].

The electron emission and transport properties of materials are key parameters in determining the likelihood of deleterious spacecraft charging effects [5], [7], [32], [33] and are essential in modeling these effects with engineering tools such as the NASA NASCAP-2K code [34], [35]. The *SUSpECS* studies of electron emission and resistivity will extend more than a decade of research in the field by the USU Materials Physics Group [7-14], [16], [18], [25], [27]. Preliminary ground-based studies have shown that contamination can lead to catastrophic charging effects under certain circumstances [16]. 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.

Recent work [13], [36] has 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 [37]. Under many typical conditions 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. An apparatus to measure the decay rate of charge deposited on the surface of thin film insulators has been designed and built at USU in conjunction with an on-going NASA research project with JPL [14]. 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

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characterization measurements and sample preparation. Jim Dyer at USU Space Dynamics Laboratory was instrumental in characterization measurements and sample preparation and assembly.

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