Spacecraft Charging Test Considerations for Composite Materials

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***Abstract*—Composite materials present a growing challenge for spacecraft charging assessments. We review some recent lessons learned for charging tests of composite materials using both parallel-plate and electron beam test geometries. We also discuss examples of materials that exhibit significant variations between samples, despite them all having the same trade name.**

***Index Terms*—Aerospace testing, composite insulation, conductivity measurement, dielectric breakdown, dielectric films, electrostatic discharges, materials testing.**

I. INTRODUCTION

SPACECRAFT dielectric materials are often composite materials with combinations of constituents, each of which can have a different conductivity. Such composite materials include circuit boards, carbon/matrix composites, materials loaded or coated with nano-particles, etc.

Composites are increasing in variety and application because they can be tailored to produce desired mechanical, thermal, or electrical properties. Some are designed to be so-called leaky dielectrics, having enough conductivity to avoid the extreme buildup of charge that leads to electrostatic discharge (ESD). Composite materials present both significant challenges and opportunities for spacecraft charging assessment and mitigation. Testing in flight-like conditions is critical to ensure that the desired conduction properties will be present in flight.

Charge transport physics in highly insulating dielectric materials is challenging even for homogeneous isotropic non-crystalline materials [1-4]. Electrical properties such as bulk conductivity, electrostatic breakdown, or permittivity are quite often nonlinear. How do we define materials properties for combinations of different materials?

II. TEST METHOD COMPARISON

Spacecraft dielectrics are generally subjected to two types of charging test geometries, parallel plate electrodes and electron beam exposures, both with ground-referenced rear electrodes [5, 6]. Especially in the case of composite materials, the test geometry used may significantly impact the observed charging behavior. In both test geometries, it is necessary to test baked out samples since humidity can enhance conductivity and will not represent the outgassed in-flight case. Also, one must let the

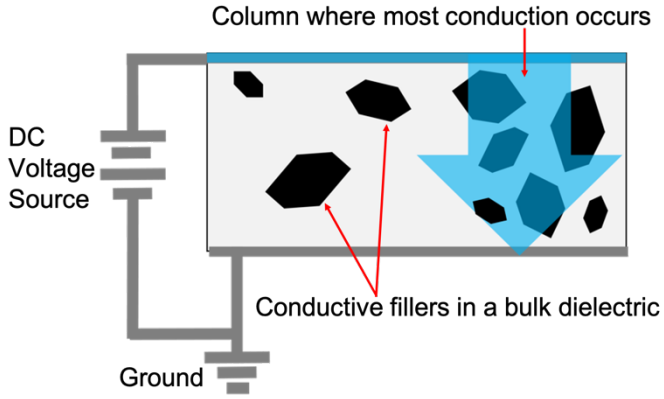

Fig. 1. Simple diagram of a composite material in a parallel-plate test geometry. Note that where some regions of the test article are more conductive, those regions will dominate the observed results.

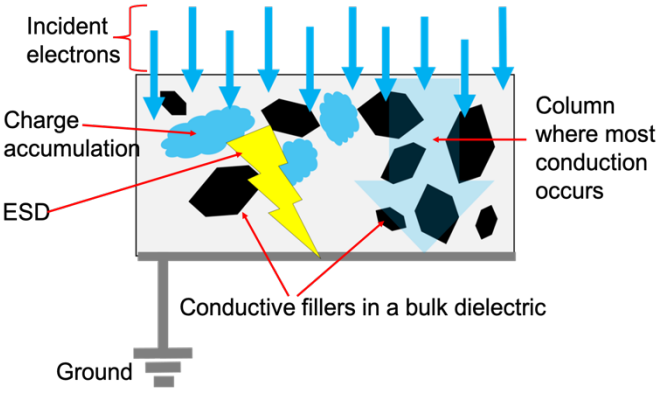

Fig. 2. Simple diagram of a composite material in an electron beam test geometry. Note that in this case, conductive regions do not prevent charge deposition in the regions of lowest conductivity.

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test reach steady-state behavior, which may take hours to weeks for highly resistive materials [5]. The ASTM D257-14 Standard Test Methods for DC Resistance or Conductance of Insulating Materials require only 60 seconds of voltage application [7], meaning that vendor or handbook data that use this standard will drastically overestimate the conductivity of most dielectrics and therefore should not be used in spacecraft charging analyses. This is also discussed in [6] and [8].

A. Parallel Plate Test Methods

Composite materials can have parallel columns of higher and lower conductivities. In parallel-plate type tests, these conductivities combine additively in series and areas of much higher conductivity will effectively short out the areas of lower conductivity (see Fig. 1). This effect can dominate the overall observed conductivity given that constituent materials' properties can differ by many orders of magnitude. For example, consider carbon fibers (10^2 to 10^{-4} Ω·cm [9]) and epoxy resin (10^{11} Ω·cm [10]). Thus, the parallel plate method tests a dielectric's ability to provide voltage isolation, driven by columnar paths of higher conductivity.

Dielectric strength tests that focus on the measurement of breakdown potential use contact methods. A discussion of breakdown tests in the context of composite materials and the ASTM standard test method is given in Appendix A. We note two recent studies presented at the same conference where the addition of fillers to dielectric materials in one case did not impact the breakdown strength [11] and another that reported both reductions and increases in breakdown strength due to fillers [12].

B. Electron Beam Test Methods

In electron-beam type tests where the entire sample is irradiated with a uniform electron beam, areas of higher conductivity will still exhibit relatively higher conduction but, unlike the parallel-plate method, areas of higher conductivity will not prevent the accumulation of charge in regions of lower conductivity since electrons will be deposited across the entire sample with the depth of deposition depending on the beam energy and material stopping power. The build-up of charge, the resulting electric fields, and with them the risk of ESD will be dominated by areas of lowest conductivity (see Fig. 2). Additionally, the interfaces between domains of dissimilar conductivity may result in local electric field enhancement. Observed surface potential decay will also be dominated by regions of lowest conductivity, especially at long time scales. In most cases, this kind of testing is more flight-like. We have previously reported examples of spacecraft thermal control paints with parallel plate conduction too high for ESD to occur in the expected environment, which nevertheless discharged under electron beam irradiation [13].

ESD tests under electron beam irradiation are useful for determining whether a test article will discharge in a simulated space environment and investigating the magnitude and rate of ESD that do occur [5, 14, 15]. These ESD tests do not measure the breakdown electric field strength. Breakdown strength tests are discussed further in Appendix A.

Fig. 3. SEM image of Cerastat sample. Note that the contrast in this figure is dominated by electron yield rather than charging.

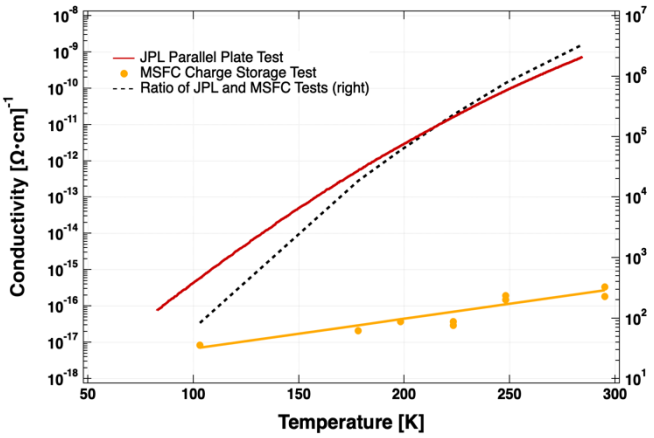

Fig. 4. Comparison of bulk conductivity of Cerastat using both parallel plate and charge storage test methods. The ratio between the results of the two results is shown by the dashed line with the scale on the right vertical axis.

III. Case Studies

In this section we present examples of testing of composite materials where the composite nature of the materials was a significant consideration in the observed results.

A. Cerastat

Cerastat is a customized proprietary ceramic material loaded with metallic particles [16]. A scanning electron microscope (SEM) image of Cerastat is shown in Fig. 3. A sample of Cerastat was tested with the parallel-plate conductivity test setup at NASA's Jet Propulsion Laboratory (JPL) as described in [13]. The same sample was also tested with the charge storage method at NASA's Marshall Space Flight Center (MSFC). In both tests, measurements were made from roughly 100 K to 300 K as shown in Fig. 4.

It is clear that Cerastat appears to be much more conductive when measured with the parallel plate method. The ratio of conductivities measured with the parallel-plate method to the

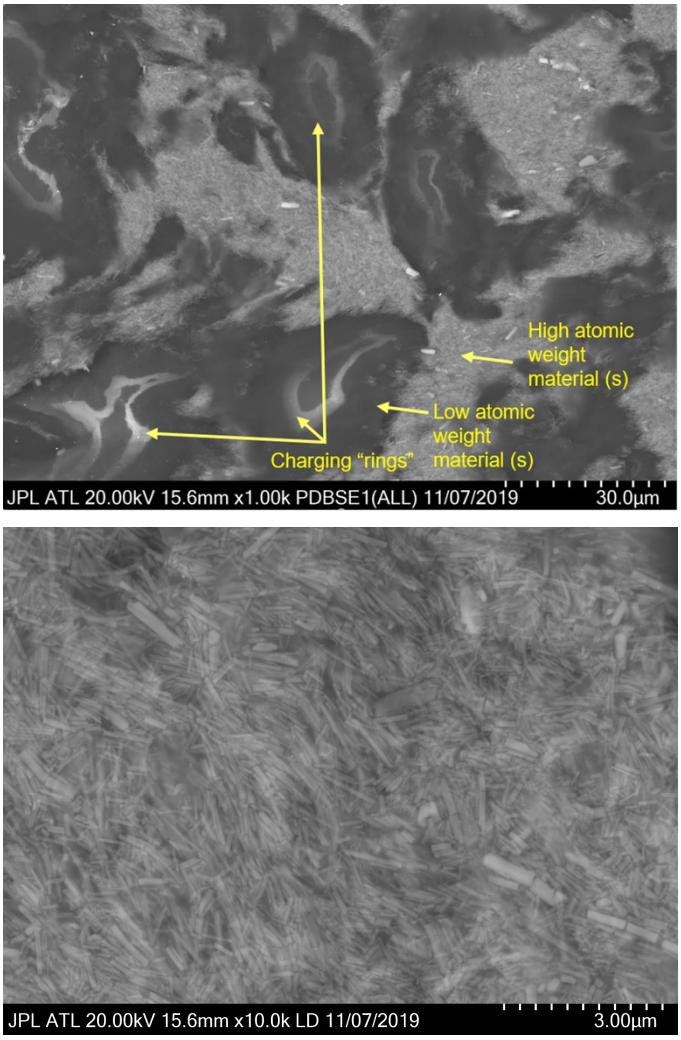

Fig. 5. Low magnification (top) and high magnification (bottom) SEM images of CNT-loaded PEEK matrix. Note that the contrast in this figure is a function of both the atomic weight of the target materials and charging. Ring-like charging regions are observed in the top figure in areas dominated by PEEK rather than CNTs.

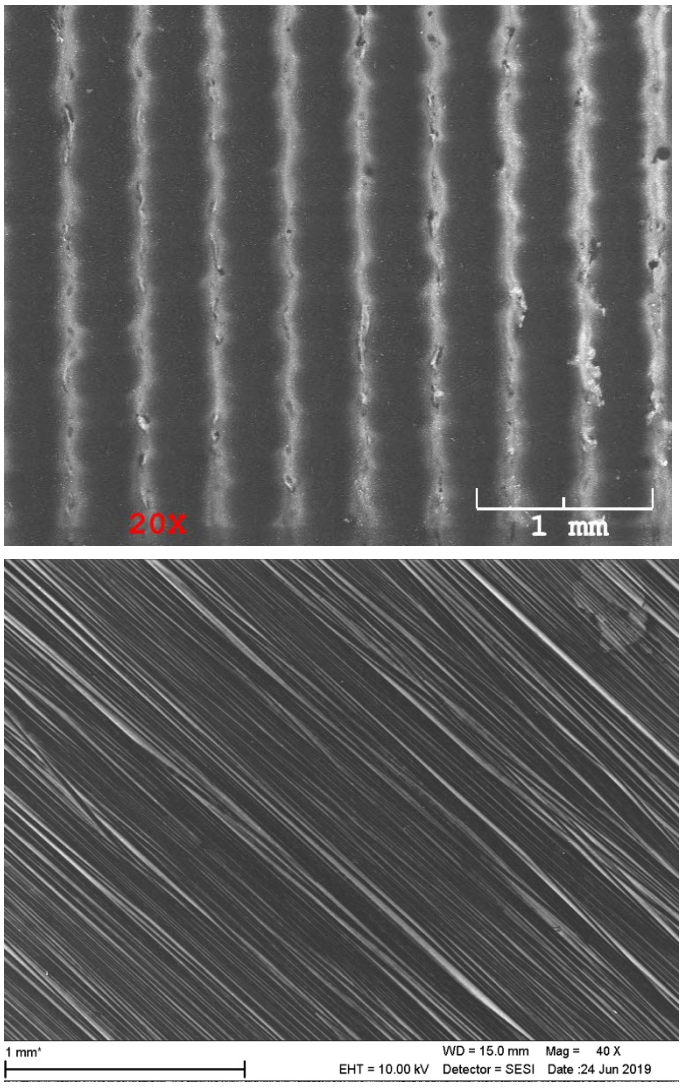

Fig. 6. SEM images of carbon composites materials. Bright regions indicate charging and therefore the presence of dielectric resin. The top image has 20x magnification and row of dielectric resin are clearly visible and consistent with those seen in previous publications. This sample produced copious discharges under electron irradiation. The bottom image, shown at 40x magnification has resin visible but much smaller than those shown in the top image. This sample did not discharge under conditions known to cause discharges in other carbon composite samples. Note that the contrast in this figure is dominated by charging rather than electron yield.

electron beam method, shown in Fig 4, increased from a factor of ~10^2 to over 10^6 as temperature increased from 100 K to room temperature.

This highlights the risk of giving composite materials a false pass based on parallel plate conductivity and the expected charging environment. These data also clearly show that with either method that room temperature data—such as what is found on materials datasheets—should not be used to estimate charge dissipation at cryogenic temperatures. Examples of how conductivity changes with temperature can be found in [17] and [18].

While such differences due to test method may not be significant in every composite material, we have clearly demonstrated that the test method can have a very significant effect on the observed conductivity. This difference may not be expected since both methods are ostensibly designed to test for the same bulk material properties. The observed difference comes from the degree of non-uniformity of the constituents. If there is sufficient uniformity, in results that will be published separately in a future publication we have seen that both test methods can yield similar results. The different temperature dependencies strongly suggest different physical conduction mechanism dominate each test method. Further analysis and explanation of the temperature dependencies is left for future work.

B. CNT-Loaded PEEK

It is known that the concentration, uniformity, and degree of anisotropy of conductive fillers impacts the conduction of filled dielectrics [19]. When filler uniformity and filler isotropy are brought into question, it can result in important considerations for a materials qualification campaign.

A recent JPL test campaign focused on material selection for dielectric components for the Europa Clipper instrument Radar

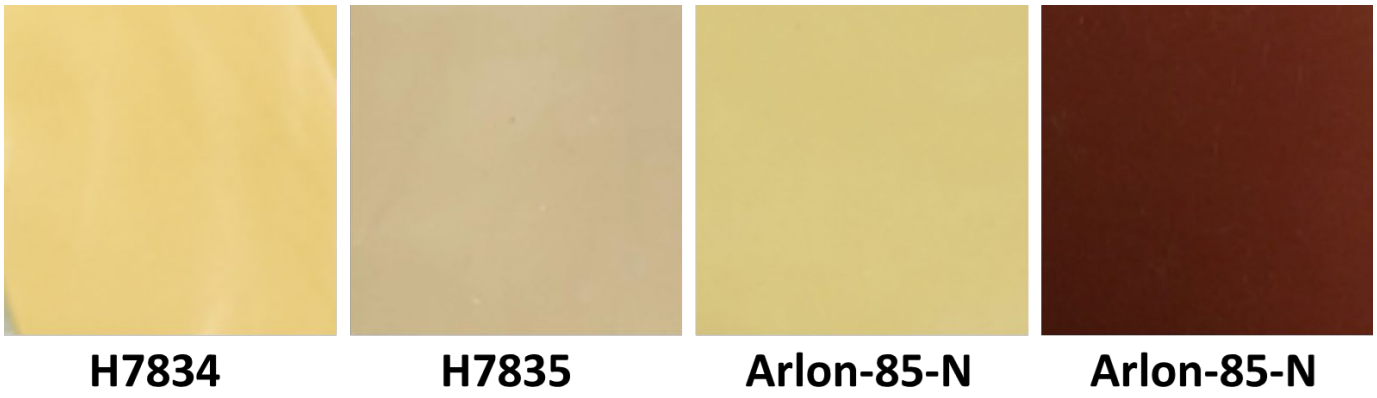

Fig. 7. Photographs of polyimide-based circuit board materials, all purchased as Arlon 85-N, from different vendors. Some materials had other, vendor-specific designations.

for Europa Assessment and Sounding: Ocean to Near-surface (REASON) provides an example of how composite materials can complicate material qualification. It is critical to instrument performance that it not be damaged by ESD or observe a rate of otherwise benign ESD high enough to degrade the science data [15, 20]. The results of this test campaign will be published separately, but here we will highlight how the effort was complicated when carbon nano-tube (CNT) loaded polyether ether ketone (PEEK) materials were considered. Static dissipative CNT-loaded PEEK was proposed as a material that would not discharge in the Europa environment.

SEM images of a CNT-loaded PEEK sample are shown in Fig. 5. We note that on the scale of hundreds of microns, there are clearly regions of high concentrations of CNTs and other regions with little or no CNTs. Additionally, within the clumps of high CNT concentration, on the scale of tens of microns, it is clear that the CNTs are not randomly oriented but come in bundles. These observations led to the following concerns:

- The effect of material orientation on dielectric performance.
- Lot to lot variations.
- Variations between samples cut from the same piece, especially at the surface. Particular attention needs to be given to the orientation of the parallel sample faces to the orientation of structural anisotropy the material (e.g., the fiber weave).
- Choosing the appropriate test method, *i.e.*, contact electrodes or electron beam (see Section II.)

These considerations are in addition to other factors such as the impact of temperature and radiation dose on conduction. These considerations can significantly increase the effort required to ensure reliable performance in the space charging environment.

C. Carbon Composites

Carbon composite materials are perhaps the most well studied composite material with regards to spacecraft charging and ESD [5, 21-27]. The dielectric resin on the surface is known to charge up and produce ESD in charging environments despite the conductive nature of the carbon fibers. Mitigation strategies include abrading the resin off of the surface, coating the surface with conductive or static dissipative materials, or using a resin that is conductive enough not to charge at all [5, 25, 27].

As part of the ESD test campaign for Europa Clipper, carbon composite materials were given special attention, particularly on the high gain antenna [26], magnetometer, and the solar array substrates. Surprisingly, one coupon of untreated carbon composite tested did not discharge, whereas all other instances of untreated carbon composites discharged in a simulated Europa charging environment, even at room temperature. SEM images comparing samples that did and did not discharge are shown in Fig. 6. In these SEM images, the bright regions are indications of charging. Although some resin is still visible in SEM images of the sample that did not discharge, the resin areas seem smaller and do not appear to protrude from the surface. In this example, we see that the variability of carbon composite materials turned out to be unexpectedly favorable in this case. While this specific carbon composite did not require additional mitigation to prevent discharges at room temperature, we recommend to implement mitigation if carbon composites are used in a charging environment. This exception depends critically on sample preparation and orientation, which are difficult to reproduce reliably. This test did not rule out discharges at cryogenic temperatures.

IV. Composite Materials Specifications

As shown in the previous section on carbon composites, large variations may be observed between different samples of composite materials procured under the same trade name. Materials specifications often give composites manufacturers a range of acceptable options for what can be called by a certain trade name.

Examples of Arlon 85-N circuit board materials were procured from different vendors. Each of these conformed to the same board construction standard [28]. Nevertheless, these boards were visually different from each other, as shown in Fig. 7. The differences in color, due to variations in material composition and construction, indicate differences in the band structure, and therefore hint that the conduction properties may differ as well.

The coupons that were thin enough to fit in the Utah State University (USU) Materials Physics Group (MPG) conductivity test fixture were measured, with the results shown in Fig. 8 and Table 1. This table includes the time intervals after

initiation of the applied voltage for which the equilibrium dark currents for each sample were calculated; these range from 6 days to >15 days. Details of this test setup have been published previously [6, 18]. There was a 3-5 day pause in testing after turning off the voltage for each sample to reestablish equilibrium before beginning the next test. This is also used to check for any drift in the zero of the baseline—zero applied voltage—current over the course of a many day test; this is typically <0.2 fA and often below the measurement capabilities. Uncertainties in conductivity of $\leq 5 \cdot 10^{-21}$ $(\Omega\text{-cm})^{-1}$ are small compared to typical conductivity measurements and result from accuracy in sample thickness and uniformity, precision in experimental resolution of current measurements, and variations of ±0.5 K in sample temperature once equilibrium was reached [6].

Variations of 10x were observed in the conductivity of the different samples. This variation is significant but also note that volume resistivity measurements in Table 1 are orders of magnitude higher than what is found in materials datasheets. As we stated before, measurement times should not be lower than the observed charge decay time constant and for 60-second tests [7] that corresponds to $\sim 10^{-15}$ $(\Omega \cdot \mathrm{cm})^{-1}$ [5]. Even longer measurements with commercially available test fixtures are limited to reliable measurements no lower than $\sim 10^{-17}$ $(\Omega \cdot \mathrm{cm})^{-1}$ due to factors such as testing in atmosphere or noise in the DC power supply [6, 29]. Test fixtures capable of measuring steady-state dark conductivity of highly resistive insulating materials have to-date been limited to custom purpose-built laboratory experiments [6, 29, 30]. Although it is worth noting again that applying datasheet values of conductivity to ESD assessments is likely erroneously characterize materials as ESD-safe in a particular environment [5], one may also incur some ESD risk by applying good test data from one example to another of the same material if the specifications controlling the manufacturing of that material allow for significant variations.

Conductivity $(\Omega \cdot cm)^{-1}$	Resistivity $\Omega \cdot cm$	Temperature (℃)	Start Time (hrs)	End Time (hrs)
H-7835 8-12-19				
$1.8 \pm 0.5x10^{-20}$	$5.5 \pm 2x10^{19}$	26.6 ± 0.7	301.14	327.42
$1.3 \pm 0.5x10^{-20}$	$7.7 \pm 3x10^{19}$	25.4 ± 0.6	350.29	372.09
H-7834 4-22-19				
$1.2 \pm 0.4x10^{-20}$	$8.8 \pm 3x10^{19}$	23.2 ± 0.3	196.84	220.27
$1.15 \pm 0.3x10^{-20}$	$8.7 \pm 2x10^{19}$	23.65 ± 0.03	281.1	294.0
Arlon 85N 7-23-19				
$5 \pm 4\,x\,10^{-21}$	$2 \pm 2x10^{20}$	25.1 ± 0.2	146.07	158.90
$4 \pm 3\,x\,10^{-21}$	$3 \pm 2x10^{20}$	24.8 ± 0.1	168.49	185.48
$3.1 \pm 0.5\,x\,10^{-20}$	$3.2 \pm 0.5x10^{19}$	40.6 ± 0.6	195.48	208.25

Table 1. Conductivity of Different Examples of Arlon 85-N. A measurement at elevated temperature is highlighted in red.

The Arlon 85-N tests presented here show that even if the same ingredients are used in two preparations of a material, differences in structure may still remain, an example being the geometry of glass fiber weave in a circuit board.

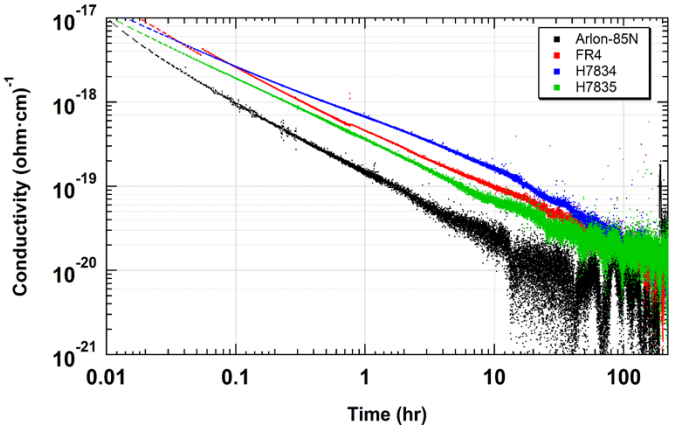

Fig. 8. Conductivity vs Time for Examples of Circuit Board Dielectrics. Three versions of polyimide circuit boards that meet Arlon 85N are shown with an example of an FR4 board. Note that hundreds of hours are needed to observe steady-state conductivity in these materials.

In addition to circuit board materials, chemical surface treatments, such as anodization and Chem Films, leave room for unexpected variations in conduction properties.

V. Conclusion

Composite spacecraft materials by design combine the properties of different constituent materials.

The measured conduction properties can vary greatly with the test method used.

- Parallel plate tests address the question "How conductive can it be?"
- Electron beam tests address the question "How resistive can it be?"

Extra caution is needed when using literature or manufacturer data on composite materials for charging calculations [18, 31]. This is true even for data from tests that specifically address spacecraft charging (e.g., baked out samples, tested in vacuum, long duration testing). Radiation-induced-conductivity (RIC) batch screen testing (published separately [31]) provides further examples of varying behavior between different fabrications of composite materials that are nominally the same when procured.

Additional material controls beyond standard specifications may be needed in sensitive charging applications.

Appendix A. Breakdown Strength Testing

DC Breakdown strength testing following the ASTM D3755-20 or some variation thereon requires the use of contact electrodes [3, 32]. In these tests, DC voltage is increased until breakdown occurs. Current-limiting resistors are used in the test circuit to protect test circuit components, typically the high-voltage DC power supply [3]. It is assumed that prior to breakdown the resistance of the sample is much larger than the current-limiting resistors R_{lm}. This ensures that the voltage drop occurs almost entirely over the sample under test. If at any time the sample resistance is on the same order as the current-limiting resistors the voltage across the sample V_{sample} (also called the device under test, *i.e.,* DUT) will be reduced

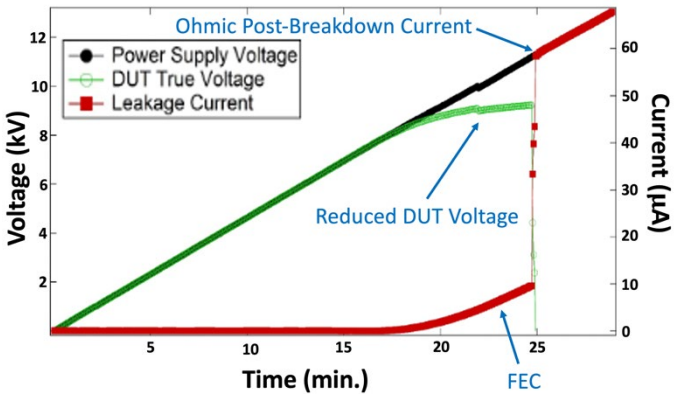

Fig. 9. Comparison of total applied voltage, sample voltage, and leakage current in Kapton E sample exhibiting field enhanced conductivity prior to breakdown. Leakage current below the observed FEC is limited by the noise floor of the test setup.

compared to the applied voltage $V_{applied}$ from the DC high voltage source as follows

$$V_{sample} = V_{applied} - IR_{lm} \quad (1)$$

where I is the leakage current through the sample [3].

A test may transition from resistance being dominated by the sample to significant contributions from the current-limiting resistance by aging, partial breakdown, or field enhanced conductivity (FEC) [3]. An example of this behavior is shown in Fig. 9, taken from [3]. In this case the breakdown voltage is still identifiable despite being reduced compared to the applied voltage, however, $V_{applied}$ at breakdown may easily be misinterpreted as the breakdown voltage (V_{sample}) and may result in an overestimation of breakdown strength. Though not shown in Fig. 9., this type of behavior has been observed to prevent breakdown altogether in this test fixture.

A similar reduction in voltage across the sample under test will occur in composite materials with columns of relatively higher conductivity on the order of the current-limiting resistors in the test circuit used. This reduction in sample voltage will not necessarily be observed in all composite materials but if the material is meant to be static dissipative it will be much more likely. Even if one corrects for V_{sample} using equation (1), the test on a composite material may underestimate the breakdown field in the non-contact case if enhanced current leads to thermal breakdown in the more conductive regions of the material. If pre-breakdown current does not result in an increasing reduction of $V_{sample}/V_{applied}$ then the test, even on a composite material will not be impacted by this preferred thermal breakdown scenario. In other words, as long as $R_{sample} \gg R_{lm}$ prior to breakdown then the parallel-plate breakdown test is a measure of the breakdown electric field, even in composite materials. Experimenters may need to reduce current-limiting resistance to measure static dissipative composite dielectrics. If the conductive regions are conductive enough to clearly result in thermal breakdown despite lowering R_{lm} or to prevent breakdown altogether, then an electron beam ESD test or voltage probe sweep test may be more appropriate.

Electron beam methods of measuring breakdown voltage often use thin electrodes that allow for penetration of the electron beam into the sample [33, 34]. Even though an electron beam is used to charge the samples, such methods are still contact methods. Non-contact methods of measuring breakdown field using a surface voltage probe sweeping the sample do not directly measure the breakdown electric field during irradiation. If the probe can be left in the beam, it will obscure the portion of the sample it is measuring. Successive sweeps between short electron exposures could be used to bound the breakdown field strength [35-37]. Voltage probe sweeps are better suited for measuring quantities such as conductivity which are determined from relative changes in potential [5, 8, 31, 38] rather than the absolute potential at which an event occurs. Further work comparing this method to the contact method of breakdown is warranted, although, to the best knowledge of the authors, the non-contact method has not been used to the same effectiveness for measuring the distribution of breakdown field strengths in a material [3, 39, 40] as is done routinely with the contact method.

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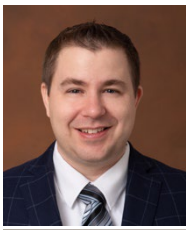

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Eric Suh, photograph and biography not available at the time of publication.

Joel Schwartz, photograph and biography not available at the time of publication.

Abdul-Majeed Azad, photograph and biography not available at the time of publication.