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Effects of Exposure to Atmospheric Humidity on Breakdown Field Strength Measurements of Polymers

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Abstract
This study investigates the effects of absorbed water introduced via exposure to atmospheric humidity on electrostatic breakdown field strength measurements of polymers. Conducting breakdown tests under sample conditions appropriate for different applications is essential. If the breakdown field strength is overestimated for an application, an insulator may be used inappropriately in high electric fields where they are more likely to break down. Comparisons are made between: sets of pristine samples, samples that underwent a thorough vacuum bake out to remove absorbed water, and samples subject to subsequent incremental prolonged atmospheric exposure. These investigated the effects of absorbed water and determined how quickly samples reverted to an unbaked state. Specifically, we compared: changes in measured electrostatic breakdown field strength, pre-breakdown arcing (DC partial discharge) rate, rates of flashover signatures, and images of the arc damage sites. The polymeric dielectric materials chosen were hydrophobic low density polyethylene (LDPE), intermediate polyether-etherketone (PEEK), and more hydrophilic Nylon 66.

Introduction

Electrostatic discharge events are a concern for the success of spacecraft missions, the safety of high voltage direct current power (HVDC), and the performance of microelectronics [1]. Knowing how insulating materials will react to different physical situations is of the utmost importance in these and other applications. Electrostatic breakdown occurs when an insulator is exposed to a high voltage, breaks down, and no longer blocks significant current flow [1]. The breakdown field strength is an intrinsic property of a material which can be calculated using the thickness and electrostatic breakdown voltage found during testing. The primary focus of this study is to examine how absorbed water introduced via exposure to atmospheric humidity affects the breakdown field strength and other electrical behaviors of polymers.

One of the biggest threats to the success of spacecraft missions is the dielectric breakdown of insulators and compromise of critical electronic equipment [2]. If severe, these electrostatic breakdown events can lead to the total failure of the spacecraft. In the plasma environment of space, charge accumulates on materials and creates localized electric fields due to a lack of ground and inefficient charge transport in insulators. Knowledge of the dynamic interaction between the spacecraft and its environment and materials' responses to different environments can help when predicting their long term behavior in space [3]. In space, materials are often exposed to high vacuum, heat, and outgassing which affects the contaminants present on their surface. The development of satellites requires extensive testing of many kinds of surfaces ranging from pristine to highly contaminated [4].

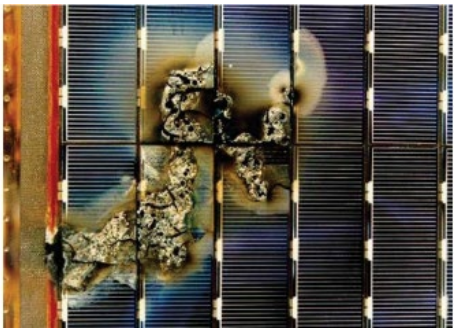

Figure 1 Damage to spacecraft solar panel due to electrostatic discharge event [6].

Since the 1950s, polymers have been used to insulate transmission cables [5]. As higher voltages are used and lines are made longer, it is increasingly important to understand the breakdown strength of insulators to maintain proper safety [6]. Of particular concern are joints in these HVDC lines in which charge may build up at the dielectric interface between the joint and the insulation [7]. Another terrestrial application is the development of increasingly small microelectronics. As the area available for insulation becomes smaller, dielectric breakdown

becomes more likely which could destroy the sensitive electrical systems [6]. The insulators that are used in terrestrial settings would generally be exposed to the atmosphere which can deposit contaminants such as water vapor on their surface even if all other precautions are made to keep them clean.

Testing of insulators should reflect the wide variety of applications that they are used in. Behavior changes of samples contaminated by the atmosphere versus samples that have been fully outgassed in space is one of the important differences to characterize during electrostatic breakdown testing. If these differences are not emphasized, material breakdown strength values used in spacecraft charge models could be based on measurements not relevant to the specific mission, which increases the associated risk [8].

Although this experiment was originally planned to focus entirely on the polymer polyether-etherketone (PEEK), two other polymers were added to provide information on how the hydrophobicity of the material affects its change in breakdown field strength with exposure to humidity. Three commonly used insulating polymers with varying hydrophobicities were used in this study: hydrophobic low density polyethylene (LDPE), intermediate polyether-etherketone (PEEK), and more hydrophilic Nylon 66. Increasing contact angle provides a measure of increasing hydrophobicity; materials with contact angles >90° are deemed hydrophobic [9]. Contact angles are reported as 90° for polyethylene [9], 78° [10] to 88° [11] for PEEK, and 68° for Nylon 66 [9]. Breakdown field strength comparisons were made between sets of pristine samples and ones that underwent a thorough vacuum bake out to eliminate absorbed water and volatile compounds. Additionally, baked samples subject to incremental prolonged atmospheric exposure and humidity were tested to determine how quickly the samples reverted to an unbaked state. The exposure time it takes for samples to revert to an unbaked state is important in designing tests for different application conditions and for how long baked test samples can be exposed to ambient conditions without adversely affecting electrostatic breakdown test results.

Theory

Highly Disordered Insulating Materials (HDIM) are used in a wide variety of applications to insulate electronic equipment. HDIM are materials which have a non-crystalline structure and are very electrically insulative. They build up charge well and so are at risk of electrostatic breakdown [12]. The polymers chosen in this study are examples of HDIM.

Charge transport in HDIM is characterized by the thermally-assisted hopping conductivity model where electrons hop from one randomly distributed localized trapping site to another [13]. When an electric field is applied or increased through a sample, the potential energy per transition decreases, leading to increased charge transport as shown in Figure 2 [14].

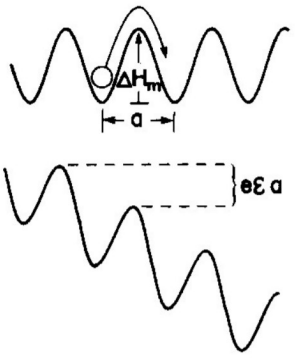

Figure 2 Diagram of hopping conductivity model [11].

If exposed to a high enough electric field, an electron will often have enough energy to displace another electron as it moves from one trap site to the next through the material. When each electron displaces more electrons, they become an electron cascade which produces high heat, breaks bonds, and can permanently damage the insulator [15]. Electrostatic breakdown occurs as a result of an electron cascade and is defined by the voltage at which an insulator no longer blocks significant current flow [1].

Another phenomenon to examine during electrostatic breakdown testing are surface flashovers. The signatures of electrostatic breakdown and related phenomena in current versus voltage IV curves are shown in Fig. 3; further discussion is provided in [16]. This is when an electron cascade occurs along the surface of an insulator, often through a small layer of gas, as an alternate path to ground. As desorbed gasses facilitate the movement of current along the surface, higher amounts of surface contamination may encourage more surface flashovers [17]. Water vapor on the surface of polymers may especially increase the likelihood of surface flashovers because of the high conductivity of water compared to the polymer. Additionally, studies have been conducted which suggest that a small layer of water along the interface of two objects will have a higher conductivity than bulk water when electric fields are applied [18].

Methods

Thin film samples of LDPE, PEEK, and Nylon were cut to 25 mm diameter discs and cleaned using methanol. Eighty-four samples of each material were divided into 7 groups of 12 samples each, an unbaked control group, a fully baked group, three groups of samples that will be left at atmosphere after bakeout for one, three, and five months, and two groups that were exposed to a high humidity environment for two days and two weeks.

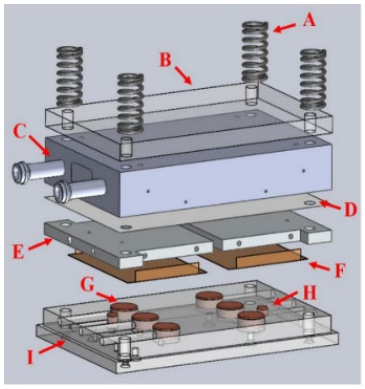

Figure 3 ESD assembly stack [18].

Samples for the unbaked control group were tested after cutting and cleaning without further treatment. The rest of the samples were baked out under vacuum for 3 days at 375 K. This bakeout was done to evaporate water vapor and other volatile contaminants on the surface of the sample that are not removed with methanol. After being cooled to room temperature, the fully baked group of samples were immediately tested.

To determine how quickly samples revert to an unbaked state, three groups of samples were kept out at atmosphere in a clean container left slightly open which allowed for some air exchange inside the box. Humidity in the lab was monitored and found to be 20±5% relative humidity [19]. At the one, three, and five month intervals, samples were tested to gather data from different exposure times allowing us to observe how quickly the samples reverted to an unbaked state. The high humidity samples were put into a closed container with a small dish filled with water. The humidity was monitored and found to be 90±10% relative humidity [19]. At two days and two weeks, samples were removed from the container and tested.

All samples were tested using the Materials Physics Group's Electrostatic Discharge chamber at room temperature [6, 20]. This chamber consists of a parallel plate capacitor inside a high vacuum chamber which is pumped down to a pressure of 10^{-5} mbar while testing. The assembly (shown in Figure 3) consists of a polycarbonate base plate (I), a high voltage electrode plate (H) housing six polished copper electrodes (G), the samples (F), sample plates (E), and another polycarbonate insulating plate (B). The optional cryogenic (cooling) reservoir is also shown (C) along with a thermally conductive, electrically insulative layer (D).

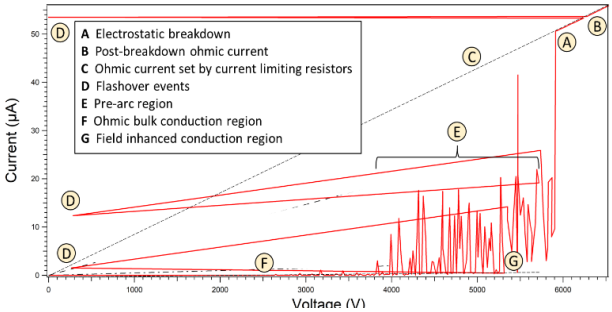

Figure 4- IV curve illustrating various features of a breakdown test.

These layers are held together by four compression screws (A) to apply the correct amount of pressure to the samples.

A voltage was applied across the samples, increasing at a rate of 20 V per 4 sec until breakdown was observed as an abrupt increase in conductivity [1]. After testing, the data was processed using IGOR data analysis software.

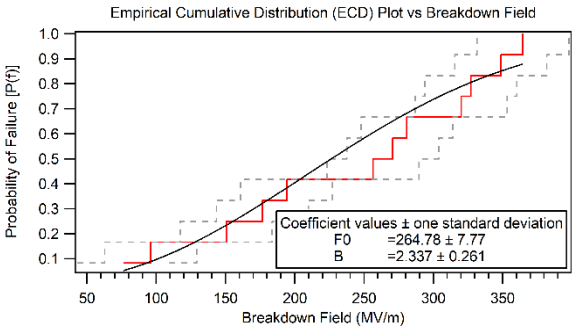

Figure 5- Weibull analysis plot for fully baked LDPE data.

Results

Table 1 summarizes the results of 175 breakdown tests for three types of materials (LDPE, PEEK, and Nylon 66) studied under five exposure configurations of varying duration. The data from the five-month atmospheric exposure group is in the process of being analyzed and so is not included in the table or graphs. The table lists the average relative humidity during different exposures and the effective exposure time

Table 1. Breakdown signatures for samples exposed to increasing humidity conditions

Material	Test Exposure	Relative Humidity (%)	Exposure Time (days)	Effective Exposure Time[a] (days)	Number of Tests per Run	Breakdown Field Strength (MV/m)	Number Pre-arc Discharges per Test	Number Flashovers per Test
						(Mean ± SD or SDOM) [% Change][b]		
PEEK	Fully Baked	0%	0	0	12	(189 ± 12) [0%]	(16 ± 6) [0%]	(0.18 ± 0.12) [0%]
	Humid 2 Days	(90 ± 10)%	2.5 ± 0.3	11 ± 2	12	(210 ± 11) [11%]	(19 ± 3) [19%]	(0 ± 0) [-100%]
	Humid 2 Weeks	(90 ± 10)%	14 ± 0.3	63 ± 7	12	(200 ± 9) [6%]	(16 ± 1) [0%]	(0.11 ± 0.10) [-39%]
	Atm. 1 Month	(20 ± 5)%	33 ± 2	33 ± 9	12	(247 ± 5) [31%]	(31 ± 5) [94%]	(0.25 ± 0.19) [39%]
	Unbaked	(20 ± 5)%	∞	∞	11	(246 ± 6) [30%]	(43 ± 7) [169%]	(0.08 ± 0.09) [-56%]
LDPE	Fully Baked	0%	0	0	12	(239 ± 28) [0%]	(42 ± 13) [0%]	(0.5 ± 0.35) [0%]
	Humid 2 Days	(90 ± 10)%	2.5 ± 0.3	11 ± 2	12	(291 ± 9) [22%]	(55 ± 7) [31%]	(0.17 ± 0.12) [-66%]
	Humid 2 Weeks	(90 ± 10)%	18.5 ± 0.5	83 ± 9	12	(315 ± 7) [32%]	(59 ± 11) [40%]	(0.17 ± 0.12) [-66%]
	Atm. 1 Month	(20 ± 5)%	34.5 ± 0.5	35 ± 9	12	(299 ± 8) [25%]	(47 ± 8) [12%]	(0.08 ± 0.09) [-84%]
	Dry N_2 100 Days	(1 ± 1)%	100 ± 2	5 ± 5	12	(249 ± 20) [3%]	NA	NA
	Unbaked	(20 ± 5)%	∞	∞	12	NA	NA	(0.08 ± 0.09) [-84%]
Nylon	Fully Baked	0%	0	0	12	(242 ± 10) [0%]	(75 ± 13) [0%]	(0.16 ± 0.12) [0%]
	Humid 2 Days	(90 ± 10)%	2.0 ± 0.3	9 ± 2	12	(194 ± 19) [-20%]	(99 ± 8) [32%]	(0.16 ± 0.12) [0%]
	Humid 2 Weeks	(90 ± 10)%	17.5 ± 0.5	63 ± 7	9	(218 ± 11) [-10%]	(60 ± 11) [-20%]	(0 ± 0) [-100%]
	Atm. 1 Month	(20 ± 5)%	36± 3	36 ± 9	12	(192 ± 12) [-21%]	(66 ± 12) [-12%]	(0.50 ± 0.30) [213%]
	Unbaked	(20 ± 5)%	∞	∞	11	(166 ± 12) [-31%]	(31 ± 8) [-59%]	(0 ± 0) [-100%]

[a] Calculated as exposure time multiplied by [%RH / Ambient %RH]. [b] Precent change from value of fully baked samples.

calculated as exposure time multiplied by [%RH / Ambient %RH]. Also listed are the average breakdown field strength with estimated uncertainty (SDOM) and the percent change from value of fully baked samples which result from the humidity exposure. There are similar entries for number of observed pre-breakdown discharges per test and the number of observed flashovers events per test; the quoted uncertainties are the standard deviations for the individual tests of that run.

Figures 6(a-c) show curves for each of the three materials of the average measured values as a function of effective exposure time, respectively, for: (a) electrostatic field strength, (b) average number of pre-breakdown discharges per test, and (c) average number of flashover events per test. Uncertainties for selected points are shown as error bars.

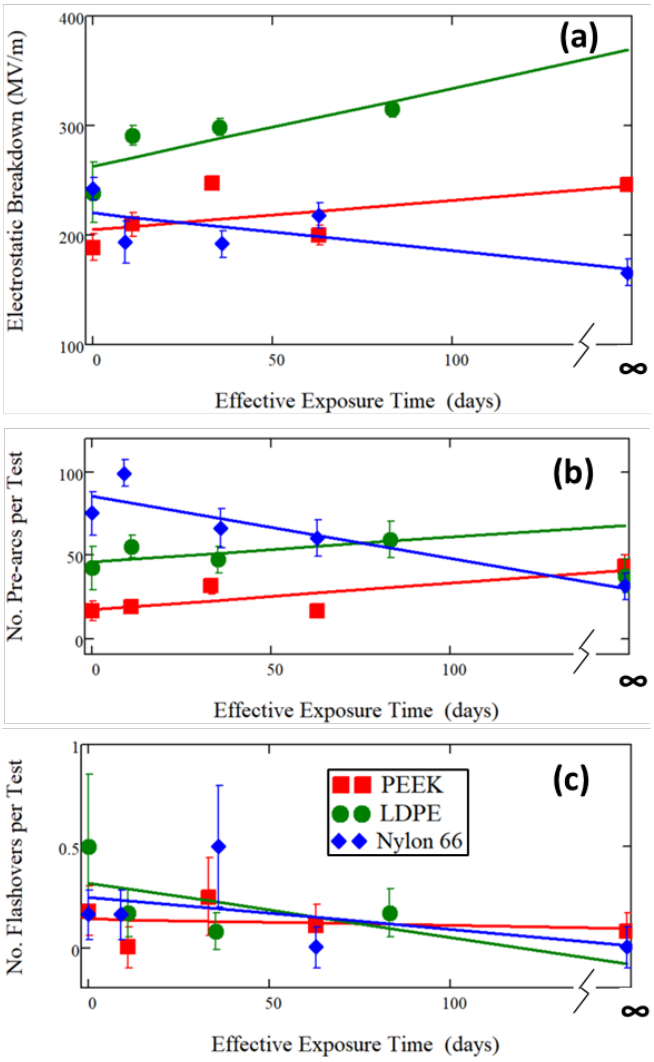

Figure 6-Signatures of breakdown as functions of effective exposure time for PEEK, LDPE, and Nylon 66. Lines are linear fits to the data. (a) Electrostatic breakdown potential, (b) number of pre-arcs per test, and (c) number of flashover events per test.

Linear fits of the breakdown field strength data versus effective elapsed time in Fig. 6 (a) have slopes of +0.72±0.17 MV/m-day for LDPE, +0.27±0.11 MV/m-day for PEEK, and -0.34±0.11 MV/m-day for Nylon 66, respectively. Linear fits of the pre-arc data in Fig. 6 (b) have slopes of +0.15±0.05 pre-arcs/day for LDPE, +0.16±0.04 pre-arcs/day for PEEK, and -0.37±0.06 pre-arcs/day for Nylon 66, respectively. The observed slopes suggest a trend of positive changes in electrostatic breakdown potential and number of pre-arcs per test for more hydrophobic materials (LDPE and PEEK) and negative slopes for more hydrophilic materials (Nylon 66). Note that these slopes are in terms of effective exposure days; hence, they only provide an approximate rate of change per day at an ambient relative humidity of about 20±5%. More sophisticated analysis of the distributions of electrostatic breakdown using Weibull analysis [1] is in progress. See Fig. 6 for a representative linearized Weibull plot.

There is a strong correlation between the observed trends (slopes) for electrostatic breakdown potential and number of pre-arcs per test as functions of effective exposure time when compared for each material. This correlation supports the contention that the distributions of the number of pre-arcs are correlated with those of electrostatic breakdown potential [6, 21]. The numbers of flashover events for all three materials are found to be nearly statistically independent

of effective exposure time; hence they are perhaps better measures of their lack of dependence on effective exposure time.

Conclusions and Future Work

This project quantified the effects of sample bakeout and subsequent exposure to humid conditions on electrostatic breakdown testing. Electrostatic breakdown strength was seen to change approximately linearly with effective exposure time; more hydrophobic LDPE and PEEK breakdown strength increased with effective exposure time, while more hydrophilic Nylon 66 decreased.

The results of this study showing up to ~50% differences in breakdown field strength due to bakeout reinforce the need to tailor tests to conditions for the intended applications. For example, electrostatic breakdown potential measurements for terrestrial applications in ambient conditions should be performed on unbaked samples, while measurements for space applications should be done on well baked samples [8].

Results show that recovery time for baked samples to revert to unbaked values was quite long. The observed magnitudes of the changes in the samples studied suggest that samples need be exposed to low humidity ambient atmospheric conditions for ~50 days or more to see ~10% change in measured electrostatic breakdown strength. Thus, one can conclude that while bakeout of test samples is necessary for applications like the space environment where the samples will be dry [8], short-term exposure of many days to ambient atmospheric conditions will not dramatically affect the breakdown test results.

Similar trends were observed for the number of pre-breakdown discharges versus effective exposure time. This includes agreement of both the sign of the change and the relative magnitudes of the slopes for all three materials studied. This agreement provides strong corroborative evidence for the contention that the distribution of the pre-breakdown discharges versus applied field is highly correlated with the form of the distribution of breakdown events and can potentially provide a more efficient means for measuring breakdown distributions.

The very low frequency of flashover events confirm that the test system used here is well designed and avoids an extraneous issue that sometimes plagues breakdown tests.

Future work for this subject includes adding the data from the five-month atmospheric exposure group with analysis to Table 1 and Fig. 6. and completing full Weibull analysis for the data. We will investigate whether the size and shape of arc damage sites was affected by exposure to humidity [22]. It was observed that overall LDPE exhibited more visible arc sites than the other materials. Also planned is testing more materials to further span ranges of hydrophobicity and using humidity exposure via more controlled methods.

Acknowledgement

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References

[1] Allen Anderson, and J.R. Dennison, 2017, "An Enhanced Operational Definition of Dielectric Breakdown for DC Voltage Step-up Tests," in Electrical Insulation and Dielectric Phenomena, 2017. CEIDP 2017. Annual Report Conf. on Electrical Insulation and Dielectric

Phenomena, p. 433-436.

[2] D. Hastings and H. Garrett, *Spacecraft-environment interactions*: Cambridge University Press, 2004.

[3] J.R. Dennison, "Dynamic Interplay Between Spacecraft Charging, Space Environment Interactions, and Evolving Materials," IEEE Transactions on Plasma Science, vol. 43, no. 9, pp. 2933-2940, Sept. 2015.

[4] W.Y. Chang, J.R. Dennison, Jason Kite and R.E. Davies, "Effects of Evolving Surface Contamination on Spacecraft Charging," Paper AIAA-2000-0868, Proceedings of the 38th American Institute of Aeronautics and Astronautics Meeting on Aerospace Sciences, Reno, NV, 2000.

[5] Gilbert Teyssedre, Christian Laurent, "Advances in High-Field Insulating Polymeric Materials Over the Past 50 Years," IEEE Electrical Insulation Magazine, vol. 29, no. 5, pp. 26-36, 2013.

[6] Allen Anderson, "The Role of Recoverable and Non-Recoverable Defects in DC Electrical Aging of Highly Disordered Insulating Materials," PhD Dissertation, Utah State University, 2018.

[7] T. T. N. Vu, G. Teyssedre, B. Vissouvanadin et al., "Electric field profile measurement and modeling in multi-dielectrics for HVDC application." pp. 413-416, Jun. 2013.

[8] Allen Anderson, K. Moser, and J. Dennison, "Perspectives on the Distributions of ESD Breakdowns for Spacecraft Charging Applications," IEEE Trans. on Plasma Science 45, pp. 2031-2035, 2016.

[9] K.-Y. Law, "Definitions for Hydrophilicity, Hydrophobicity, and Superhydrophobicity: Getting the Basics Right," *J. Phys. Chem. Lett.* vol. 5, no. 4, pp. 686-688, 2014

[10] R.Y.M. Huang, P. Shao, C.M. Burns, X. Feng, "Sulfonation of Poly(Ether Ether Ketone)(PEEK): Kinetic Study and Characterization," *J. Appl. Polymer Sci.,* vol. 82, pp. 2651–2660, 2001.

[11] W. Wang, C.J. Luo, J. Huang, and M. Edirisinghe, "PEEK surface modification by fast ambient-temperature sulfonation for bone implant applications," *J. R. Soc. Interface*, vol. 16: 20180955, 2019.

[12] Brian Wood, David King, J.R. Dennison, "Time-Evolved Constant Voltage Conductivity Measurements of Common Spaceborne Polymeric Materials," Proceedings of the 15th Spacecraft Charging Technology Conference, Jun. 2018.

[13] Vinay Ambegaokar, B. I. Halperin, J. S. Langer, "Hopping Conductivity in Disordered Systems," Physical Review B, vol. 4, no. 8, pp. 2612-2620, Oct. 1971.

[14] Megan L. DeWaal, Joshua Boman, J.R. Dennison, "The Effects of Surface Contaminants on Electrostatic Breakdown Testing," American Physical Society Four Corner Section Meeting, University of Colorado Boulder, Boulder, CO, Oct. 2021.

[15] X. Li, C. H.Tung, K. L. Pey, "The Nature of Dielectric Breakdown," Applied Physics

Letters, Lett. 93, 072903, Aug. 2008.

[16] Megan Loveland DeWaal and JR Dennison, “Effects of Volatile Compounds on Breakdown Field Strength Measurements of Polymers,” Presentation 1C6, *Proceedings of the IEEE Conference on Electrical Insulation and Dielectric Phenomena(CEIDP 2022)*, 210-214, 2022.

[17] H. Craig Miller, “Surface Flashover of Insulators,” IEEE Transactions on Electrical Insulation, vol. 24, no. 5, pp. 765-786, Oct. 1989.

[18] Artemov V, Uykur E, Kapralov P, Kiselev A, Stevenson K, Ouerdane H, et al. “Anomalously High Proton Conduction of Interfacial Water,” ChemRxiv, Cambridge Open Engage, 2020.

[19] Joshua Boman, Megan Loveland, and JR Dennison, “Monitoring Laboratory Conditions with an Inexpensive Custom Data Acquisition System”, American Physical Society Four Corners Meeting, Embry-Riddle Aeronautical University, Prescott, AZ, October 11-12, 2019.

[20] Allen Anderson, J.R. Dennison, A. M. Sim, and C. Sim, 2015, "Measurements of Endurance Time for Electrostatic Discharge of Spacecraft Materials: A Defect-Driven Dynamic Model," IEEE Trans. on Plasma Science 43, 2941-2953.

[21] A. Andersen and JR Dennison, “Pre-breakdown Arcing as a Proxy for DC Dielectric Breakdown Testing of Polymeric Insulators,” *Proc. 2015 IEEE Conf. on Electrical Insulation and Dielectric Phenomena—(CEIDP 2015)*, pp. 574-577, 2015.

[22] Sam Hansen (with JR Dennison and Allen Andersen), “Electrostatic Discharge Breakdown Analysis,” Senior Thesis, Utah State University, Logan, UT, March 2015.