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Heather Allen
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PHYS 4900 Report

May 2, 2023

Preparation, Characterization and Electron Yield Analysis on Highly Insulating Granular Particles

Heather Allen, Principal Investigator

JR Dennison, Faculty Mentor

*Materials Physics Group
Physics Department
Utah State University*



Abstract

This study focuses on obtaining reliable electron yield measurements of highly insulating granular particles of various shape, size, and composition. Measurements of this kind have long been considered too difficult to collect on granular samples due to experimental complexities leading to a critical knowledge gap in the fundamental electrostatic behaviors of dust. A significant portion of this study was spent on preparing and characterizing granular samples before any type of measurement took place. Particles of varying sizes ranging from $\sim 1 \mu\text{m}$ to $\sim 100 \mu\text{m}$, shapes including cubical, spherical, and angular, and composition including NaCl, MgO, Al₂O₃ and LHS1, were used to create a multilayered sample with a wide range of particulate coverage on an adhesive substrate that can withstand vacuum conditions. After preparation these particulate samples were characterized via scanning electron microscopy and subsequent electron emission measurements were then compared to measurements of the bulk particulate and substrate materials. Results demonstrated the ability to perform these difficult measurements and how the varying sample properties like roughness and coverage affected yields.

Introduction

Knowing how granular insulating materials will react to different physical situations is of the utmost importance when considering environments with large amounts of dust like the surface of the Moon, Mars or even areas of space with large amounts of cosmic dust. Previous encounters with these environments indicated that the interactions of manmade equipment with dust had the potential to be catastrophic, such as the 1969 Apollo 11 mission when Lunar dust electrostatically adhered to astronaut suits and led to purported health effects or when Harrison Schmitt, a geologist and Apollo 17 astronaut, said “Dust is the No. 1 environmental problem on the moon” [1]. In addition to the implications on astronaut health, dust has the capability to damage optical surfaces or halt mechanical functions in spacecraft if not accounted for in design.

Because of this, there has been a particular interest in the behavior of dust in the space industry, including a massive initiative from NASA to research possible lunar dust mitigation solutions. Even now there is very little experimental data on the charging properties of these particles and the data that does exist from the Apollo era has low confidence in its accuracy and quality [2]. This has led to a critical knowledge gap in the fundamental electrostatic properties and behaviors of granular substances. Part of the experimental complexities halting further research is the erratic behavior loose particles when inside a vacuum chamber and the possibility of dust electrostatically lofting (launching upwards) and causing the sample to possibly self-destruct or impair the apparatus setup making future measurements impossible. This behavior must be absolutely restricted before any type of measurements can be made.

This project aims to not only demonstrate a robust preparation methodology producing samples compatible with vacuum conditions, but also to obtain electron yield data on various granular types, including lunar dust simulants. Four different types of granular types were examined, 2 with cubical particle shapes: Sodium Chloride (NaCl – 250 to 500 μm) and Magnesium Oxide (MgO – 1 to 10 μm), 2 with highly angular shapes: Aluminum Oxide (Al₂O₃ – 1 to 120 μm) and a Lunar Highlands Dust Moon Dirt Simulant (LHS-1 – <0.01 to 1000 μm with mean size at 90 μm). Some of the Al₂O₃ samples had spherical particles. In summary, 3 different shapes (cubical, spherical, and angular), particle sizes ranging from ~ 1 to 500 μm , one sample with a broad distribution of particle sizes, and four different chemical compositions were examined.

Theory

The studied materials have a non-crystalline structure and are very electrically insulative. They build up charge well and are thus difficult to get electrically sensitive surface measurements on. Because of this, charge mitigation is an incredibly central idea when measuring the charging effects of radiation. There are two categories of a charged surface, positive or negative. When a surface is positively charged it has lost electrons, so ‘flooding’ the surface with low energy electrons will return the surface to a neutral charge. When a surface is negatively charged, it has additional embedded electrons in the sample and this charge is much more difficult to neutralize. There are two primary ways to mitigate this charge one is to wait for the electrons to escape on their own (introducing a heat source would speed this process up) and the other is to utilize the photoelectric effect and bombard the surface with photons and ‘knock’ the embedded electrons off the surface.

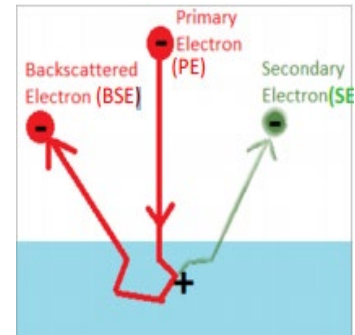


Figure 1: Diagram of incident electron behavior

Utilizing these techniques is essential in electron yield measurements which entails the pulsed bombardment of electrons onto a neutral surface to produce meaningful results. The electron beam irradiating the sample surface contain the primary or incident electrons and this study considers two possible behaviors of these electrons (see figure 1): They can hit the sample surface and leave the material with little energy loss (classified as a ‘backscatter’ electron), or they can interact with the surface through continuous elastic or inelastic collisions. This is how an electron can become embedded in the surface, and if the collision energies are large enough, they can cause an electron from the materials itself to escape at a lower energy which is classified as a ‘secondary’ electron. In reality, both these behaviors occur simultaneously when a surface is bombarded with electrons, but depending on the incident energy net surface interactions may vary

In general, very low and very high beam energies will produce a net negative surface charge, around 40V and below and anything above 3000V. In this middle range of energies then is when there is a possibility of a net positive charge, where the incident energy is at just the right value to knock multiple secondaries off the surface. This net charge is quantified by taking the ratio of the number of electrons coming off the surface to the number of electrons going in. This value is known as the electron yield. The yield varies dramatically between materials, but they almost all follow this general pattern of negative and positive regions and when graphed display an inverted ‘v’ shape on a log-log plot. The secondary electron yields (SEY) also have this ‘v’ shape, and vary significantly on factors such as surface charging and roughness, but backscatter has a logarithmic shape and is not so easily affected like SEY is.

This large variance in electron yield values can be due to several factors, including things like atomic and bonding structures, conductance, or surface roughness. In concept, it is apparent that a rougher surface has more opportunity to recapture escaping electrons so the rougher a surface is the lower the yield value will be. When theorizing on things experimentally difficult to work with like dust it is common to equate it to the charging behavior of a bulk sample with the same composition. However, because of the drastically different surface roughness, it is likely that the two samples have very different yield values (see figure 2).

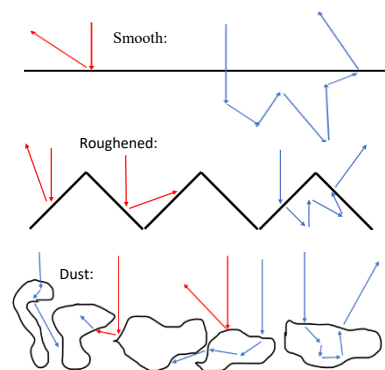


Figure 2: Surface Roughness effects on electron yield.

Because of this possibility, it is important to explicitly study the effects of surface roughness. This experiment will not only focus on obtaining novel electron yield data for granular samples but perform a preliminary comparison of rough (granular) to smooth (crystalline) samples for NaCl, MgO and Al₂O₃ as a contribution to study of surface roughness effects on charging and their contributions to modeling environments like the surface of the Moon.

Methods

The preparation of these granular samples depends on the average particle size. If the average size is above ~100 μm then the dust is simply sifted from above onto an aluminum core carbon adhesive mounted onto a 12 mm diameter steel AFM disc and blown off with nitrogen gas at about ~60 PSI to remove any loose particles. If the average particle size is between 1 to 100 μm then the dust is mixed into a liquid solution, usually deionized water if the particles are not soluble, if they are then typically methanol or isopropyl alcohol will be used. This mixture of suspended dust particles is picked up into a dropper and then placed onto the same type of carbon adhesive and disc as with the larger sized particles (see figure 3). Over time, the particles will adhere to the tape via gravimetric deposition and the liquid will evaporate. After this, the surface will be blown off with nitrogen gas at ~60 PSI to remove loose particles. At this time there is no established method of sample preparation for particle sizes smaller than 1 μm as every attempted method saw particles clumping together.

To verify that these established preparation methods produced uniform, fully covered samples the surfaces were imaged with a scanning electron microscope and then digitally treated so they could then be analyzed by a custom software that calculated the coverage of the surface via pixel greyscale value histograms (see figure 4). These preparation methods proved to be able to produce samples of varying uniform coverage. For an in-depth discussion of the development and verification of the granular sample preparation and characterization, see [3].

Once properly characterized and determined vacuum compatible 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.

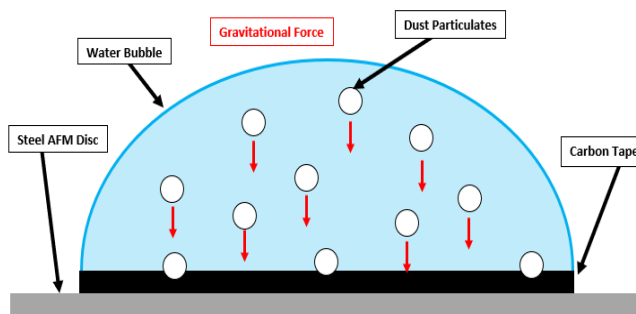


Figure 3: Diagram of sample preparation using gravimetric deposition.

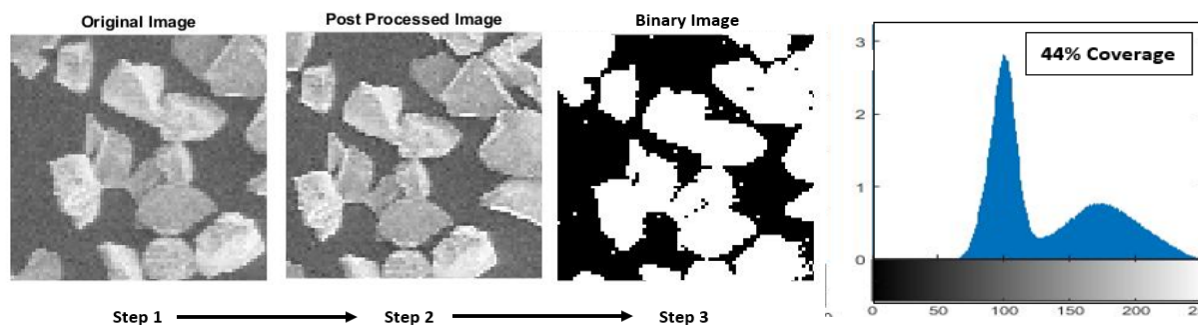


Figure 4: Example of sample characterization software output, including original image, enhanced image, enhanced image greyscale pixel values histogram and its binary image.

After being baked, samples are stored in a nitrogen gas filled container until they are ready for electron yield measurement.

To take electron yield measurements samples are then placed into The Materials Physics Group's specialized electron emission vacuum chamber *Costello*.

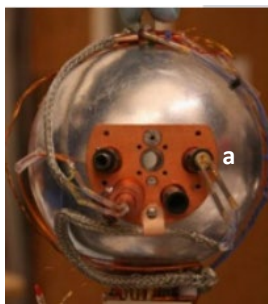
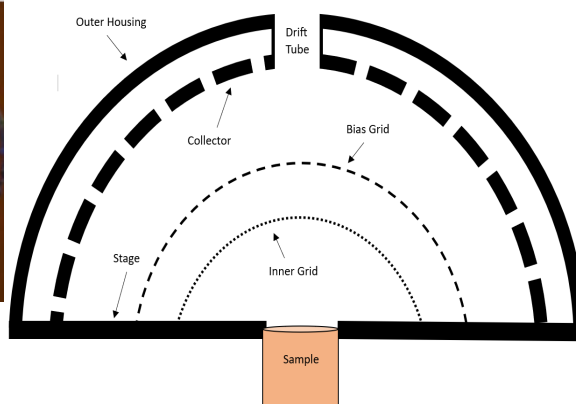


Figure 5: Photo (top) and (right) Diagram of HGRFA components



This chamber simulates the space environment and gets down to 10^{-8} Torr. Once the chamber is ready for measurement, then the Hemispherical Grid Retarding Field Analyzer (HGRFA) is maneuvered over top of the sample and is positioned in front of the STAIB Instruments electron gun where the drift tube ('a' in figure 5) will allow the incident electron beam pulses (10V – 5kV) to hit the sample surface. Due to the shape of the HGRFA, all outgoing electrons from the sample surface will be captured by the various components of the HGRFA. The differentiation between backscatter and secondary electrons lies inside this HGRFA and its multilayered setup.

The inner most layer, the 'inner grid' is a metal grid that shields the sample from unwanted electric field effects, then outside of that is the 'bias grid' held to -50V compared to the collector to repel all low energy electrons and direct them towards the stage which is the metal plate surrounding the sample surface. Any electrons that have enough energy to pass through this bias grid are then detected on the solid metal plate outside of the bias grid known as the 'collector'. All of this surrounded by a metal outer housing held at ground to shield the inside from stray fields.

There are four components measured, the stage, the collector, the sample itself and the bias grid whose electric signals are separately processed in highly sensitive electrometers and fed to the custom data collection software. Of these signals, the collector, sample and stage carry the bulk of the important information of the electrons coming off of the sample.

For each incident beam energy desired, a single pulse is sent in through the drift tube and then charge mitigating techniques are employed to neutralize the surface. Generally, 20 pulses are sent in for each energy level, 10 to measure the total electron yield (backscatter + secondary) and then 10 specifically to measure the backscattered electrons.

Results

In total nine granular samples were tested, three Al_2O_3 spherically shaped dust samples of sizes 1.6 μm , 40 μm , and 120 μm , one angularly shaped particle Al_2O_3 sample, one cubical shaped NaCl sample with a range of 200-500 μm particle sizes, one cubical shaped MgO sample with a range of 1-10 μm particle sizes, one angularly shaped Al_2O_3 sample with particles sized at 67 μm , one angularly shaped LHS-1 sample with a range of <0.1 – 1000 μm particle sizes, one bulk MgO crystal, and one NaCl crystal. In addition to this, the already existing data on polished sapphire (crystal Al_2O_3) was used in comparisons and is displayed alongside the tested samples results.

Each sample had total and backscatter electron yield data collected and during analysis the secondary yield values were calculated and the curve fitting parameters are displayed in Table 1. The listed δ_{max} value is the maximum yield, E_{max} is the incident energy value associated with

Table 1 Key data for SEY fits

Sample Composition	Particle Shape	Particle Size (μm)	SEY Fit				Date of data Collection
			δ_{max}	E_{max}	m	n	
NaCl	Crystal	-	6.9072	793.3	0.1027	2	1.16.23
NaCl	Cubical	25 – 500	1.68	329.6	0.4447	1.1768	11.8.22
MgO	Crystal	-	6.6603	736.9	0.3334	2	2.13.23
MgO	Cubical	1- 10	1.8537	264.9	0.366	1.1839	12.3.22
Al ₂ O ₃	Spherical	1.6	3.8661	1044.1	0.4145	2.2131	4.5.22
Al ₂ O ₃	Spherical	40	5	1000	0.01	2	10.20.21
Al ₂ O ₃	Spherical	120	3.457	1328.3	0.2491	1.8394	11.27.21
Al ₂ O ₃	Angular	67	1.92437	610.079	0.4	1.39	8.2.21
Al ₂ O ₃	Crystal	-	1.92437	610.079	0.094296	1.52848	9.25.19
LHS-1	Angular	<0.1 - 1000	1.58	538.6	0.5146	1.2639	3.2.23

δ_{max} , and then m and n are specific curve fitting parameters. E_{max} is a characteristic value of a material and should remain relatively constant regardless of surface roughness, in contrast δ_{max} , and the two crossover points (when the yield values transition from below 1 to above 1 or vis versa) are expected to depend on things like surface roughness.

Figure 6 is a graph of both the crystal and granular sample of NaCl, comparing total and backscatter data. From this graph it is clear that the TEY crystal data in the energy range of 100 to 800V does not follow the general trend of an inverted ‘v’ shape. There is no confirmed explanation for this, but it is suspected that the actual yield within this range is much higher than what is graphed. This discrepancy is possibly from limitations in the apparatus, or perhaps the analyzing software that is currently not capable of recording yields that high resulting in possibly lost data. It is also likely that some secondary electrons (generally lower energy than backscatter) are higher in energy and being included in the backscatter data due to the unprecedentedly high backscatter values from 400 to 2000V. These reasoning’s are further

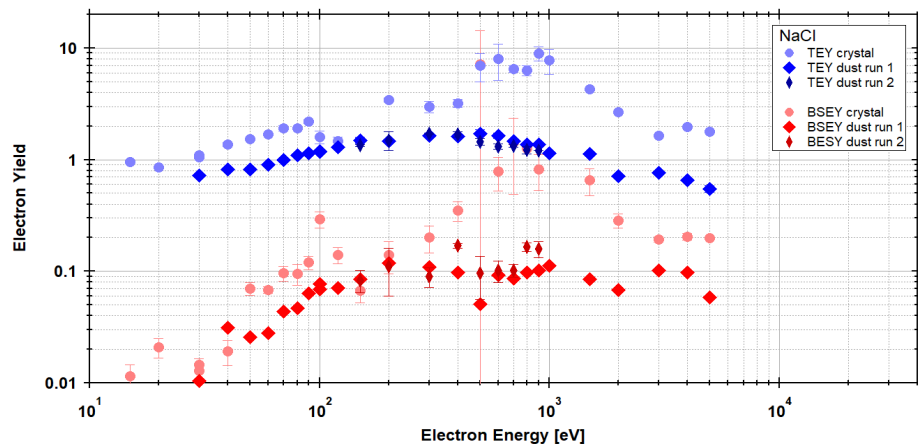


Figure 6: NaCl crystal and dust data for Total electron yield (TEY) and backscatter yield (BSEY)

bolstered by the discrepancy in E_{max} values between the dust and crystal data. As previously mentioned, E_{max} is a characteristic value and should be the same for both samples, because the dust data is much more believable and reproducible (2 runs performed with relatively identical results) E_{max} for NaCl is probably around 400V. If this explanation is correct, then it is possible that the δ_{max} value for the NaCl crystal could be higher than 20, while δ_{max} for the dust sample is just below 2. That is 10x decrease in yield due to surface roughness.

In an effort to obtain empirical evidence of this theorized maximum yield, a yield decay test was performed on the NaCl crystal sample at 400V. This test is essentially the same as electron yield except instead of 10 pulses there are 100 and there is no charge mitigation performed in between them. This purpose of this test is more accurately determine the total yield of a specific value, the first few pulses are the most accurate and as the number of pulses increase the surface will charge up and eventually the signal will go to zero. The results of this test are displayed in figure 7. While no definitive value can be determined by these results, it is clear that the yield at 400V is much higher at 7 or 8 compared to the data points in figure 6 at 3, confirming suspicions of unreliable data. This graph contains much more noise than is standard for this kind of test, indicating a large amount of noise within the setup. Indeed the noise is visible when the raw data is charted for each component at every single pulse shown in figure 8. From this graph it is apparent that the stage signal is the source of the large amounts of noise and is likely the source of the poor quality data in figure 6.

The MgO crystal showed similar signs of the high yields and poor data for NaCl, as is seen in figure 9 comparing a well-defined data set for the MgO dust sample to several data sets on the

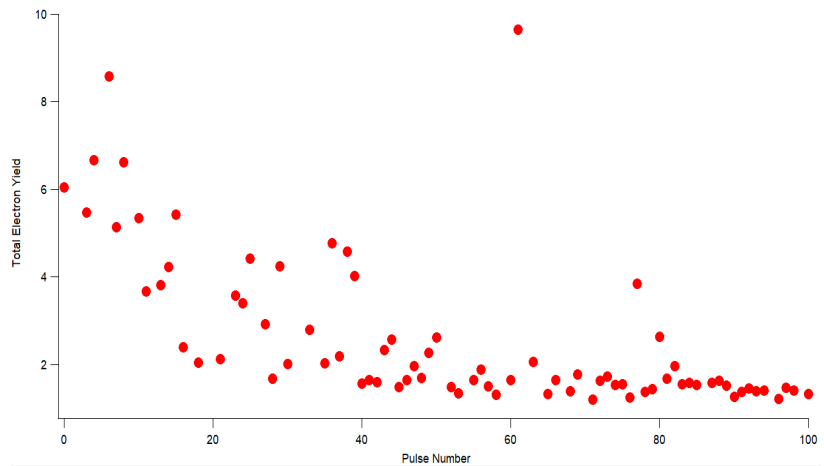


Figure 7: NaCl crystal yield decay data for Total electron yield (TEY) at 400V

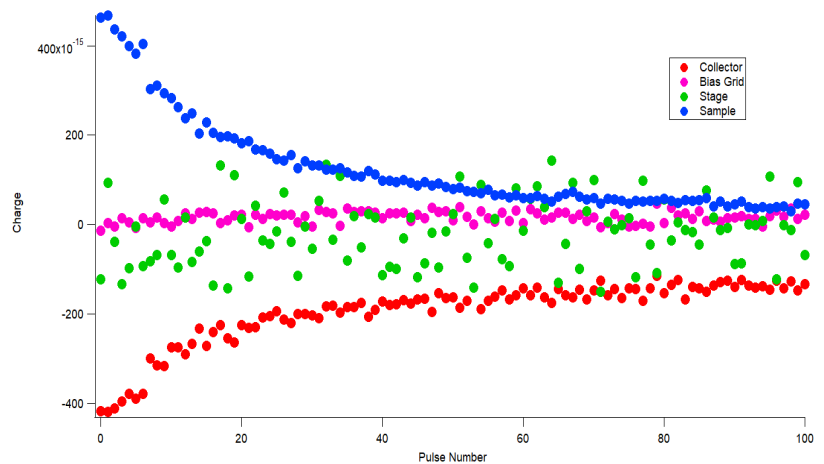


Figure 8: NaCl crystal yield decay raw charge data of each of the components

crystal. Unfortunately a yield decay test was not performed on this sample, but several more data collection runs were performed to compensate similarly revealing a much higher δ_{max} . The δ_{max} for the MgO crystal is roughly around 6, and δ_{max} for the dust sample is 2. Though not as dramatic a suppression as NaCl, it is still 3x smaller than the crystal value.

Note that in figure 6, figure 9, and all the following electron yield graphs there is an upward trend towards 1 in the lower energy range. This trend seemingly violates the inverted

'v' shape a graph should have, but it is not unexpected. In fact this trend can be seen at any energy level, but it is more likely at the lower energies due to the smaller energy electrons being repelled more easily by a negative surface charge. This trend towards 1 away from the two crossover points means that the sample surface is charged up enough to deflect incoming electrons onto the collector or stage without ever actually hitting the sample. So even with charge mitigation techniques being employed, the sample surface is not capable of producing viable yield data at that time. When many data points are at or near 1 it suggests that the quality of the data is low, and little to no charging information can be surmised about the sample material itself.

Next are the results for the Al₂O₃ samples, for more digestible results, figure 10 compared to crystal data to Al₂O₃ spherical and angular particle results. It is obvious that the data acquired for all the spherically shaped particles is abnormal. Especially for the 40 micron alumina data set which has a few perplexing outliers in the data and graph characteristics shown in table 1. The peak energy values do not correspond to the crystal and angularly shaped samples of the same composition and the traditional "inverted v" shape is less visibly identifiable. It is likely that much data is unreliable, even if it is not because the data is unexpected it should be retaken a few times to ensure precision. Because the date of data collection is so much earlier than the majority of the other samples, was collected by somebody else, and only one run was collected there could be many explanations for the poor quality data from a learning curve of the user or an equipment issue that was later fixed but the data was never retaken. In contrast, the angularly shaped particle sample's

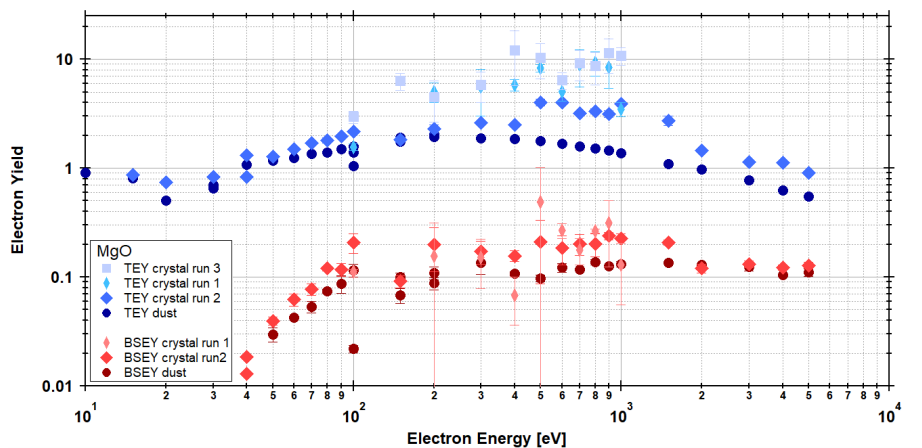


Figure 9: MgO crystal and dust data for Total electron yield (TEY) and backscatter yield (BSEY)

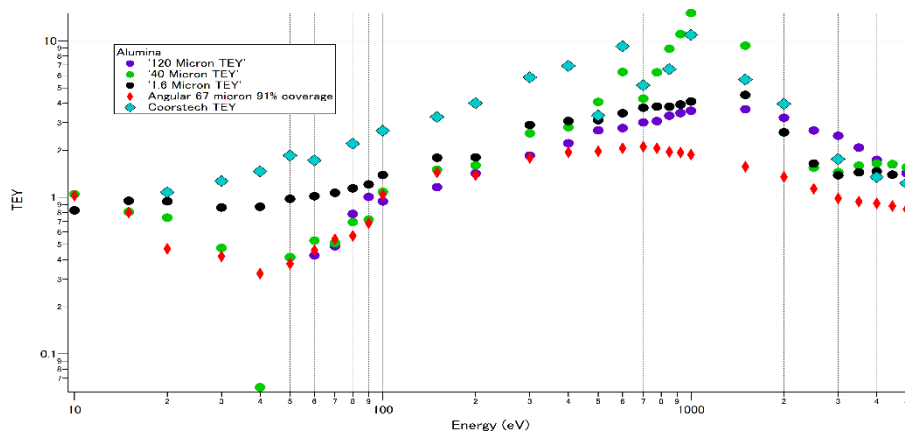


Figure 10: Al₂O₃ crystal, angular and spherical (120, 40 and 1.6 microns) dust data for total electron yield

data agrees extremely well with the crystal data, seeing only a dampening in the peak yield value just as was expected, likely due to surface roughness.

The lunar dust simulant sample contains a large amount of Al_2O_3 and so the yield data collected for LHS-1 is compared to the

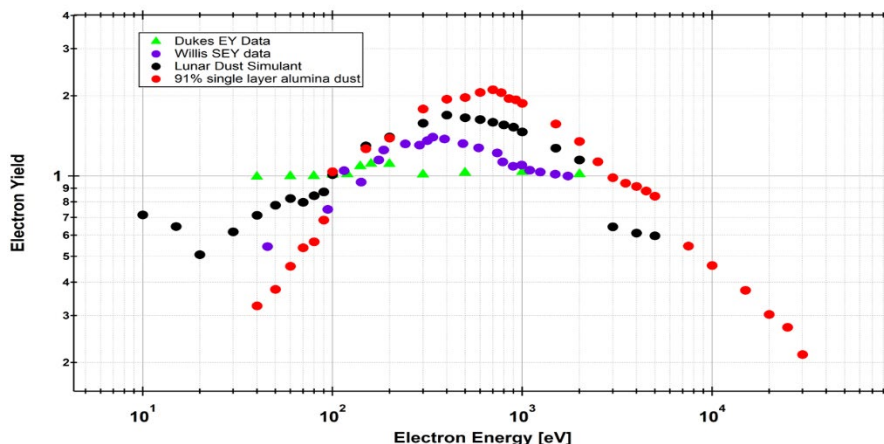


Figure 11: Al_2O_3 angular dust data, LHS-1 dust data and Actual Lunar Dust data [4, 5,6,7]

angular alumina dust data from above in figure 11. As is evident from the comparison, they have similar energy and yield maximums to each other, but are not exactly the same just as would be expected for similar composition dust samples. Figure 11 additionally compares this lunar simulant data to actual lunar dust electron yield data collected in 1973 and 2013 on actual lunar dust shown in green and purple [4, 5, 6, 7]. Not only is the LHS-1 data curve much more defined, every data point of the actual lunar dust data is either at or around 1 indicating that charging of the dust severely affected data collection and resulted in poor quality data. Thus, this new lunar simulant data is among the best experimental data that can approximate electron yield values of lunar dust.

Conclusions and Future Work

This project is far from complete. From data comparisons of NaCl and MgO crystals to their dust sample counterparts it is apparent that surface roughness has significant effects on the electron yield, but there is very little quantifiable data to extrapolate from these results. Future work on surface roughness will aim to quantify roughness and try to correlate that to yield dampening effects measured. This will be done for various different materials in an effort to determine the uniformity of roughness effects between different materials.

Additionally, the work done on dust samples is among the first of its kind, and there is much work to be done on these kinds of samples. Smaller particles will charge differently and thus interact with their environment differently, so determining the electron yield is of particularly high interest, especially in the space industry with cosmic, lunar or martian dust. Future work will involve taking data on simulants for these various materials and possibly on actual samples. Other materials will also be examined to expand this burgeoning database of dust yields compared to bulk sample data.

So far cubical, spherical, and highly angularly shaped dust grain samples have been tested, with the most data being from spherical particles. Future work will expand on grain geometry with a wider variance in size and composition. In a few weeks data will be collected on spherically shaped stainless steel and copper dust, as well as alumina dust particles coated in copper.

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