The Role of Static Charge in Ultra-Low Alpha Particle Emissivity Measurements

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***Abstract*—Electrically-insulating samples placed on the grounded sample tray in ionization detectors perturb the electric field within the detector. The resulting alpha particle emissivity of the samples is reduced depending on the magnitude and polarity of the surface voltage. Data are shown for samples with positive and negative surface charge, as well as methods to both measure and eliminate the effects of the surface charge.**

***Index Terms*—Alpha particles, dielectric, electrical insulator, ionization detectors, low background, surface charge.**

I. Introduction

RECENTLY, several publications have described the use and operating principles of XIA's new ultra-low background ionization counter, the UltraLo-1800, in making alpha particle measurements for samples with emissivity down to $\sim$ 0.3α/khr-cm^2 [1]–[3]. The efficiency associated with performing these measurements quickly involves the exceedingly low counter "background" which is achieved using active signal discrimination techniques.

Since the samples sit on the electrically grounded cathode, and reside *within* the active counter volume, electrically-insulating samples can distort the uniform electric field within the counter. This is especially true when the samples have a buildup of static charge on their surface. The effect of this is to *reduce* the reported alpha particle emissivity since some of the electrons from the ionization of the counter gas have trajectories that fall outside of the anode and are therefore not detected as alpha particles originating from the sample. For ultra-low emissivity samples, it is not immediately evident from the measured alpha particle emission rate, or from the energy spectra, that the samples possess static charge on their surface. Therefore it is essential to understand the influence of static charge, of *both* polarities, and to find effective measures to eliminate it from non-conducting samples.

This issue does not exist with commercially available gas proportional counters since the samples reside underneath the grounded cathodes. These cathodes act as Faraday shields [4] and therefore shield the electric field in the active volume of the counter from stray electric fields caused by the static charge on the surface of the samples.

Examples of electrically-insulating samples pertinent to the semiconductor industry include underfills, epoxy resins, glasses, oxides, and polyimides. Alpha particles emitted from these materials, if in close proximity to transistors, can cause single event upsets, so it is essential to develop methods of making accurate alpha particle emissivity measurements of these materials.

In this paper we first describe the generation of static charge on electrical insulators with reference to the triboelectric series. Experiments are described in the next section where we examine the effect that a glass substrate, whose surface has been charged to a positive high voltage, has on the energy spectrum of a monoenergetic radioactive source placed on top, and of the natural radiation from the glass, itself. We demonstrate that it can take up to several weeks for the glass to discharge by itself, by examining the alpha particle emissivity from the glass over time. Methods are described, and evidence given, on ways to discharge the glass. Next we describe the effect of negative surface charge by using a Teflon substrate. We introduce the use of a commercially-available non-contact volt meter that can be used to determine whether the surface on an electrically insulating sample is charged or not. We next discuss the results of a finite element model applied to examine how samples whose surfaces are charged to either positive or negative voltage perturb the electric field in the active region of the ionization counter. The results of the model can easily explain the loss of detection efficiency caused by charged substrates.

II. Static Charge Generation of Insulators

The triboelectric series shows an ordering of the electron affinity of a group of commonly used materials. When two materials on the list are placed in contact with one another, electrons are transferred from the material with the smaller electron affinity to the one with the larger electron affinity. Higher (static) voltages are achieved when materials with widely different electron affinities come in contact with one another. So, from the chart shown in Fig. 1, glass obtains a positive charge when rubbed with Teflon, and conversely, Teflon obtains a negative charge when rubbed with glass [5].

A major concern for insulating materials whose alpha particle emissivity is to be measured, is that the material preparation, storage, or even handling could cause static charge to build up on the top surface to be measured. This could present a measurement problem, especially qualifying the alpha particle emis-

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Fig. 1. Tribolectric series on common materials, from [5].

sivity of electrically insulating materials, such as underfill and various dielectrics, in the ultra-low emissivity (ULA) category of $\varepsilon < 2\alpha/\text{khr-cm}^2$, necessary for the production of state-of-the art semiconductor devices.

III. Experimental Details

Experiments described in this paper- exploring the effects of positive static charge on the surface of samples were observed using a 300 mm diameter, 680 μm thick glass wafer from Swift. The glass had a moderately high alpha particle emissivity, $\sim 150\alpha/\text{khr-cm}^2$, and was easily charged to a potential of several kV by rubbing it with a piece of Teflon. In order to facilitate making alpha particle emissivity measurements on both an XIA UltraLo-1800 ionization detector and a model 1950 Alpha Sciences gas proportional counter [6], the glass wafer was diced into a square of area 446 cm^2. The same piece of glass and the same alpha particle detectors were used for all of the experiments with positive static charge.

For experiments examining the effects of negative static charge on the surface of a sample, we obtained a 0.032" thick Teflon sheet, and cut it into a 300 mm diameter sample. This sample was charged to negative voltages of several -kV by rubbing it with a glass beaker, or a glass wafer.

For all experiments described in this paper, we modified the XIA UltraLo-1800 ionization detector by adding an 1800 cm^2 liner on top of the grounded cathode sample tray. The liner was a 0.032" thick aluminum plate coated with an ultra-low alpha particle emissivity electrically-conducting polymer [7]. The alpha particle emissivity from this liner was $\sim 0.5\alpha/\text{khr-cm}^2$, approximately 3X lower than the default stainless steel tray whose alpha particle emissivity was $\sim 1.5\alpha/\text{khr-cm}^2$.

In a first experiment, a low activity ($\sim 8.5\,\text{E}-5\ \mu\text{Ci}$) ^{230}Th radioactive source[1] was used to demonstrate the effects of positive static charge on the surface of the glass sample. For reference, this nuclide emits monoenergetic alpha particles at energies of 4.688 MeV [8]. The alpha particles were collected on the XIA counter for 1 hr with the source placed on the center of the tray liner (no glass) and, separately, with the source on the center of the glass that had been charged by rubbing it with Teflon.

In a second experiment, we examined the energy spectrum and alpha particle emissivity from the *natural* radiation of the glass itself, as a function of the positive static charge delivered to the top surface of the glass. In the first case, we ensured that the glass had no static charge (see discussion in Section VII) and in the second case, we charged the surface of the glass, again, by rubbing it with a sheet of Teflon.

In a set of the third experiments, we charged the glass using the Teflon as described earlier, and measured the alpha particle emissivity for a period of several days. Then, we opened the counter and attempted to discharge the glass using a variety of methods including: 1) leaving the ^{230}Th source on top of the glass for three days with the counter high voltage turned off, 2) using a high activity ^{241}Am source in close proximity to the glass with the sample tray extended in air, and 3) using a piezoelectric antistatic gun (see discussion in Section VII). Then we resumed measuring the alpha particle emissivity for a period several days to two weeks to observe the effect of the perturbation.

In the last experiment with the glass sample, we measured the alpha particle emissivity of the glass in the Alpha Sciences gas proportional counter, in both the charged and uncharged conditions, to compare to the results from the second and third experiments, and to establish the "nominal" alpha particle emissivity from the sample.

In an experiment similar to that described above on the glass sample, we charged the Teflon sample and placed the low activity ^{230}Th source on top and in the center of the disk and observed both the count rate and energy spectrum and compared them to the case where the source was measured without the Teflon.

Since the alpha particle emissivity of the Teflon sample (and most plastics, in general) was very low, we could not examine the time evolution of the alpha particle emissivity directly quickly, due to poor statistical uncertainties, as we had done with the charged glass sample. Instead, we charged the Teflon sample and placed the 446 cm^2 glass piece on and in the center of the Teflon disk. The glass obtained a negative charge on its top surface when it was in contact with the Teflon. This enabled us to use the natural radioactivity of the glass in a negative charged condition to examine how the emissivity of a sample was affected by negative surface charge.

IV. Experimental Results

Figs. 2 and 3 show alpha particle energy spectra from the ^{230}Th source for the case where the source was placed on the counter tray liner and on the charged glass, respectively. In Fig. 2, the low energy tail in the spectrum is due to alpha particles that are emitted at shallow angles. By contrast, comparing the energy spectra from Fig. 3 to Fig. 2, we can immediately see the influence of the static charge on the glass which causes 1) the distortion of the energy spectrum at the lower energies, and 2) many fewer detected alpha particles. Note that the vertical scales are identical on Fig. 2 and Fig. 3, and that the

[1]From Eckert and Ziegler

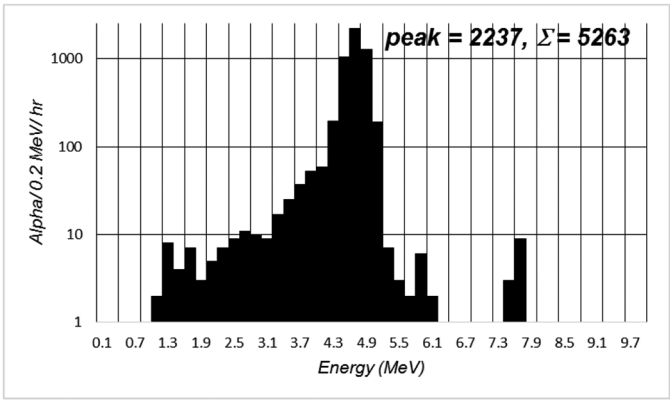

Fig. 2. Energy spectra of ^{230}Th source placed on the tray liner.

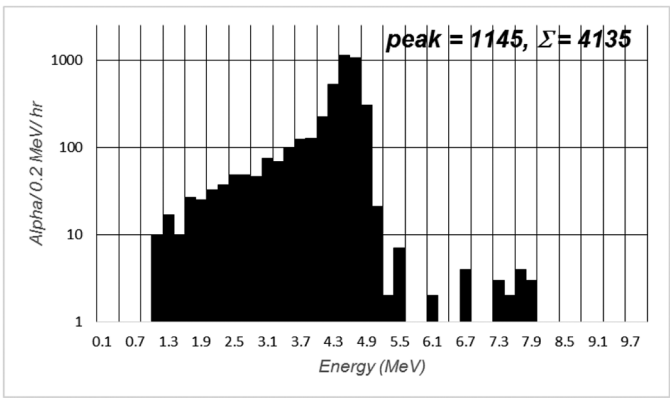

Fig. 3. Energy spectra of ^{230}Th source placed on the charged glass.

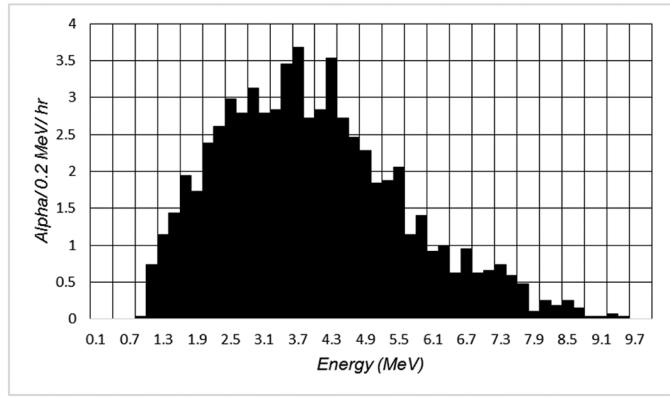

Fig. 4. Energy spectrum of the uncharged 446 cm^2 glass sample.

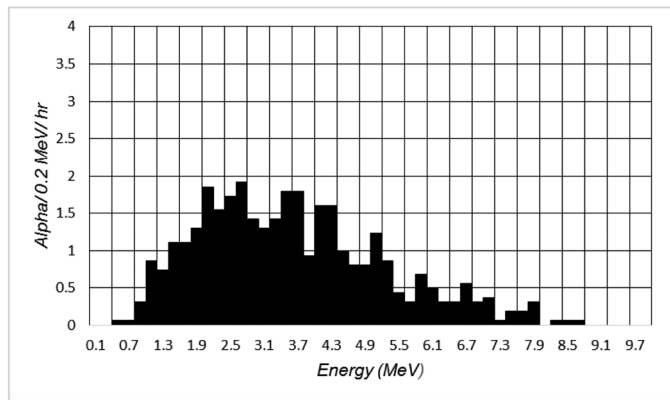

Fig. 5. Energy spectrum of the charged 446 cm^2 glass sample.

semi-log plot exaggerates the low energy distortion shown in Fig. 3. The number of alpha particles in the peak and total number of alpha particles are shown in the figures. The alpha particles near 7.7 MeV (from ^{214}Po) are due to radon adsorbed on the samples for the short period of time that the counter was open to change samples. These alpha particles decay during the first 4 hours [3].

Additionally, although not shown here, the average rise time of the detected signals was much longer in the charged glass case, indicating a longer drift time for the electrons transiting the counter when compared to the nominal operating conditions (i.e., no static charge on the sample). This is consistent with the reduction of the electric field in the active region of the counter, caused by the positive surface charge on the sample.

The energy spectra from the natural radiation in the glass are shown in Fig. 4 and Fig. 5, on a linear scale, for the no static charge and charged cases, respectively. The shape of the energy spectra are representative of a thick ^{232}Th source. In this experiment, the alpha particle emissivity of the *charged* glass sample was $\sim 1/2$ of that measured on the uncharged sample. This underscores the importance of ensuring that insulating samples are fully discharged prior to making measurements in detectors that are sensitive to these effects, such as the XIA UltraLo-1800. Of course the reduction in the observed emissivity depends on the magnitude of the static charge on the surface. As in the first experiment, the average energy of the alpha particles emitted from the charged glass is lower compared to the uncharged case.

Fig. 6 shows the time evolution of the alpha particle emissivity of the charged glass which shows an increase over time. In this experiment, the sample was counted for a period of eight days. Then the counter was opened and the ^{230}Th radioactive source was placed next to the glass sample, the counter's high voltage was turned off, and the counter was purged to remove the air and moisture from the brief period of time when the counter was opened. After three days, the counter was opened, the source was removed and the counter was again purged. Then the sample was counted for another four days. A best-fit linear function is also shown in Fig. 6, both before the source was placed on the sample, and after, which seems to describe the increase in the sample's emissivity over time. The source apparently had no effect on the natural discharge rate of the glass sample.

An additional experiment was performed on the charged glass by measuring its emissivity for an initial period of seven days. Then the counter was opened and with the sample tray extended in air, a $\sim$ 0.1 μCi ^{241}Am source was placed just above and pointed at the glass sample at nine locations in a 3×3 pattern for 5 minutes at each location. Next, the source was removed, the counter purged, and the sample counted for another two weeks. Fig. 7 shows the time evolution of the alpha particle emissivity

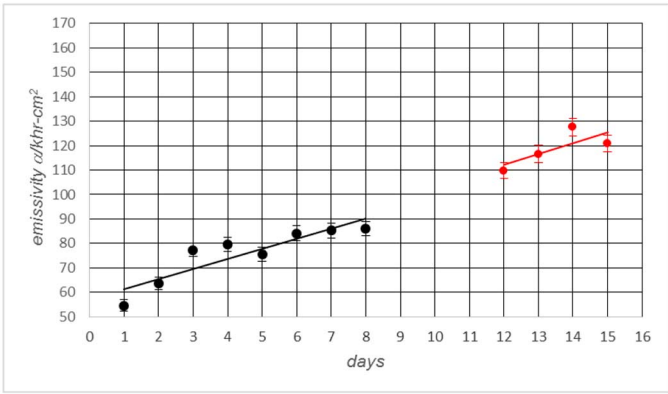

Fig. 6. The alpha particle emissivity of the charged glass measured for 8 days, then for 4 days after a 3 day period where the ^{230}Th source was left in the counter (with the high voltage off) to dissipate the static charge.

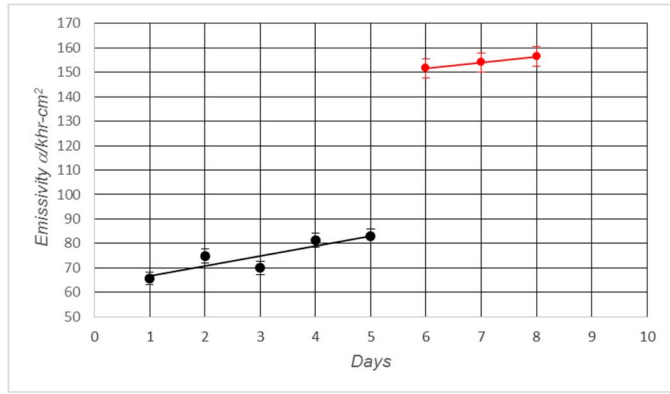

Fig. 8. The alpha particle emissivity of the charged glass measured for 5 days, then for 3 days after a piezoelectric antistatic tool was used to discharge the sample.

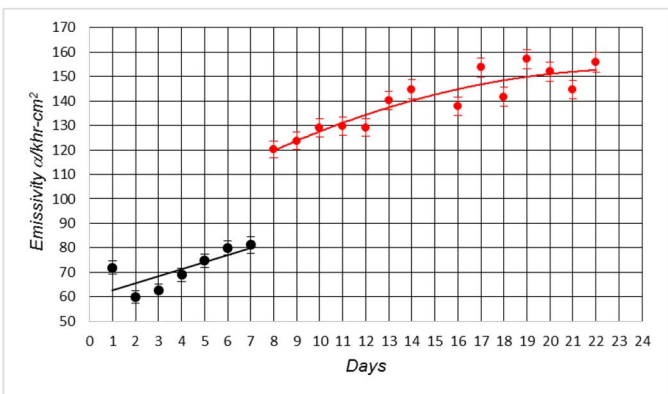

Fig. 7. The alpha particle emissivity of the charged glass measured for 7 days, then for 14 days after a ~ 0.1 μCi ^{241}Am source was placed in 9 locations in a 3×3 pattern for 5 minutes each.

of this sample for the duration of this experiment. The black circles represent the emissivity before the source was placed above the sample, and the red circles, after the source was removed.

In stark contrast to the data shown in Fig. 6, the ^{241}Am source was only partially effective at reducing the static charge on the surface of this glass sample as can be seen by the discontinuity of the emissivity just after the source was removed. Radioactive sources can be effective at eliminating static charge by ionizing the air surrounding the area. Even with the reduction of the static charge, it still took nearly three weeks for the emissivity to asymptotically approach the uncharged value.

In a final experiment on the charged glass, the emissivity was measured for a period of five days, then the counter was opened, and a piezoelectric antistatic tool was used at the four corners of the glass sample. Releasing the trigger on the antistatic tool is supposed to release negative ions (squeezing the trigger releases positive ions). The trigger was squeezed when the tool was pointed away from the glass sample, and the trigger was released over a corner of the sample. This procedure was repeated in each of the four corners of the charged glass sample. Fig. 8 shows the time evolution of the alpha particle emissivity of this sample for the duration of this experiment. The black circles represent the emissivity before the antistatic tool was used, and the red circles, after the tool was used. It seems clear that the antistatic tool was effective at eliminating the positive static charge on the glass sample because the alpha particle emissivity approached the value obtained for the uncharged glass.

In the last experiment with the glass sample, we measured the alpha particle emissivity in our Alpha Sciences gas proportional counter. As expected, since the grounded, aluminized mylar cathode window shielded the active volume from any effects of static charge below the cathode, we observed nearly the same alpha particle emissivity of the glass in the uncharged and charged states.

The alpha particle energy spectrum resulting from placing the ^{230}Th source on top and centered on the charged 300 mm Teflon disk, looks qualitatively similar to that shown in Fig. 3. The energy spectrum was distorted towards the low energy side; in other words there were more low energy alpha particles detected that when the source was placed on the empty tray liner. The distribution of rise times in this case was markedly different compared to when the ^{230}Th source was placed on top of the charged glass as shown in Fig. 9. In this figure, the black dots represent alpha particles, red dots, "mid-air" events, and the blue dots, "slow/high" events. The figure shows the distribution of collected events when the ^{230}Th source was placed on the charged glass (left), tray liner (middle) and charged Teflon (right).

The *average* rise time and *range* of rise times is much larger for the case where the ^{230}Th source was placed on the charged glass (left) compared to when it was placed on the charged Teflon (right). For alpha particles in the 1–10 MeV range, the distribution of rise times, *with little moisture in the counter*, is in the range $60\ \mu\text{s} < \text{Tr} < 85\ \mu\text{s}$. Moisture or solvent vapor in the counter increases the electron's drift velocities and results in transit times $< 60\ \mu$s. Events with rise times less than 60 μs, or greater than 85 μs are rejected by the pulse shape discrimination algorithm. Additionally, as described in [3], alpha particles emitted from radon sources *within* the counter are rejected in the sorting of events due to their smaller-than expected rise times. Events with rise time $< 60\ \mu$s are referred to as "mid air events," since their origin might come from the middle of the counter, and events with rise time $> 85\ \mu$s are labelled "slow/high." The charged glass (Teflon) reduces (enhances) the electric field in the active region of the counter which is the reason why there are

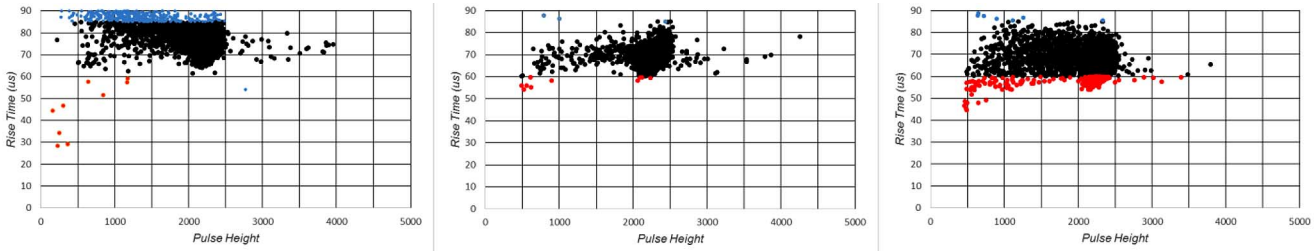

Fig. 9. The distribution of collected events when the ^{230}Th was placed on the charged glass (left), tray liner (middle), and charged Teflon (right).

many slow/high (mid air events) compared to the case where the source was placed directly on the tray liner, as shown in Fig. 9. Any event with a rise time not in the 60 μs $<$ Tr $<$ 85 μs range is rejected and leads to a loss of detection efficiency.

V. Modeling Electric Fields in the XIA UltraLo-1800

A two dimensional equivalent geometry of the UltraLo-1800 ionization detector was set up using COMSOL Multiphysics–Electromagnetics finite element analysis package [9]. The goal was to study the electric field pattern as various charged substrates were placed inside the detector just on top of its grounded cathode. The electrostatic field pattern for a non-conductive media was obtained by solving for the distributed electric potential governed by Gauss' law.

Fig. 10 shows the key elements of the detector in the model. The half width of the chamber is 266 mm, and the separation between the anode and cathode is 150 mm in height. The right hand side wall represents a symmetric boundary condition as the chamber is 2 $\times$ 266 mm wide. The top horizontal anode plane is given a potential of 1 kV and is located 150 mm away from the ground plane. The half-width of a 1 mm thick sample (either glass or Teflon) is considered to be 100 mm. The sidewalls of the counter are a graded voltage divider to provide a large area of uniform electric field within the active volume of the counter. This is modeled as follows: the left hand side wall is represented by a 12 mm thick acrylic lined with 1 mm thick Kapton. The acrylic boundary is given a charge-free boundary condition. For completeness, 10 $\times$ 1 mm copper strips are distributed on top of the Kapton. Copper would play a role if an analysis to include conductive media was carried out. For each material the corresponding relative permittivity is shown in Fig. 10. Observe that for vacuum or gas filled space the relative permittivity e is unity.

Several test cases were studied. The first reference case (not shown) was a sample consisting of a glass substrate with no surface charge applied to it. The electric field pattern showed a uniform pattern with linear drop in electric potential from 1 kV to 0 V (ground plane).

The second case introduced a $+1$ kV voltage to the glass sample and the results are shown in Fig. 11. The color legend was preselected to encompass the maximum $+1$ kV (red) and a minimum -1 kV (blue) potential field as shown below.

The potential *gradient* between anode surface and glass sample is greatly reduced, thus reducing the corresponding field strength necessary to influence the electron's trajectory in this zone (red color). This helps to explain why the efficiency

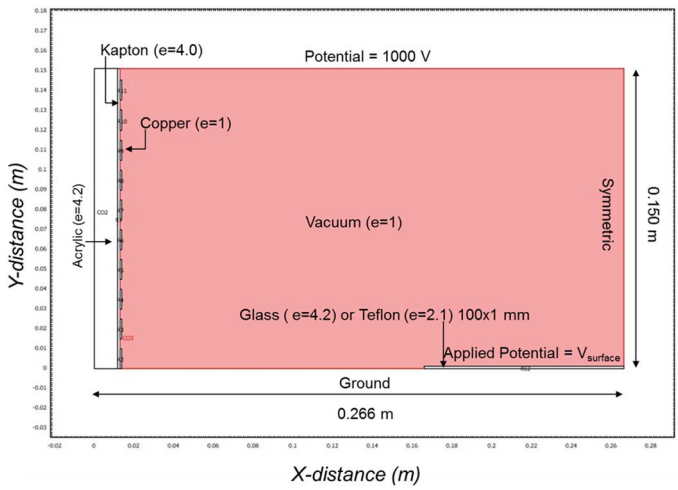

Fig. 10. Electrostatic model of the XIA UltraLo-1800.

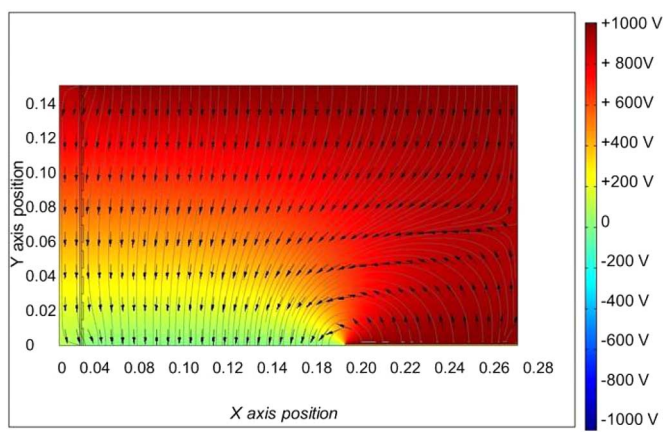

Fig. 11. The results of an electrostatic model of the XIA UltraLo-1800 ionization counter with a 200 mm $\times$ 200 mm glass sample charged to $+1$ kV.

is reduced when the glass sample was charged (some electrons turn around in the electric field and are not drifting to the anode), and why the drift velocity was retarded resulting in long rise times (slow/high events).

The third case studied a Teflon sample with -1 kV equivalent negative charge on its top surface and the results are shown in Fig. 12. This resulted in the potential gradient doubled compared to the first reference case between the anode and the Teflon surfaces which could greatly influence the electron trajectories. The effect of the increased electric field is to reduce

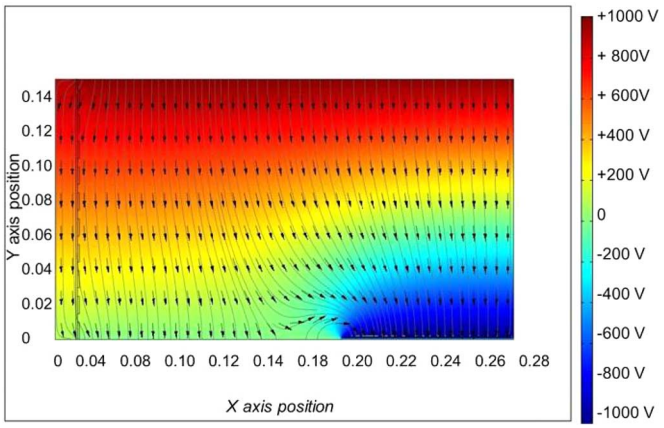

Fig. 12. The results of an electrostatic model of the XIA UltraLo-1800 ionization counter with a 200 mm $\times$ 200 mm Teflon sample charged to -1 kV.

the transit time of many events, thus the increase in the number of "mid air" events as shown in Fig. 9.

VI. Measuring the Static Charge on Samples

We used a Trek model 523 hand-held non contacting electrostatic voltmeter to measure the static charge induced on the glass, Teflon, and other electrical insulators [10]. The meter confirmed that we indeed induced a positive voltage when we rubbed the glass with Teflon, and a negative voltage when we rubbed Teflon with glass (as expected based on the triboelectric series). As a check that the meter was operating correctly, we used the meter to measure the voltage on the XIA counter tray, the tray liner, and several ground points on an AC power bus and measured essentially no voltage. We have found this meter to be useful to determine whether or not the surface on other electrically-insulating samples was charged naturally (from handling, or storage), and therefore whether the samples needed to be discharged prior to making alpha particle emissivity measurements. For example, we recently used the meter to see if a 300 mm wafer containing diced chip carcasses, adhered to dicing tape, had any surface charge (it did not).

We have found that it was possible to charge the glass sample, when placed on the XIA tray liner, to a voltage up to about $+1.5$ kV by rubbing it with Teflon in a circular motion for about 1 min. Additionally, we have charged the Teflon to voltages ~ -1 kV by rubbing the Teflon with a pyrex beaker or a glass wafer. The meter is also useful to show that the surface charge on some materials, especially the Teflon, may not be uniform across the entire surface.

Finally, we found that it was very easy to inadvertently charge insulating samples. For example, a 300 mm diameter glass wafer that had been stored in a wafer box, and placed on plastic, had a static charge of approximately 4 kV near the wafer center, and that it was possible to charge it to $>$ 20 kV (limit of the meter), by rubbing it for a few seconds with a nitrile glove. These were the same gloves typically used when handling ULA materials to place them into and remove them from the alpha particle detectors.

VII. Methods to Reduce the Static Charge

Moderate activity (few μCi) ^{210}Po sources, marketed as "Staticmaster" devices have traditionally been used to reduce or eliminate state electricity in record albums [11]. The alpha particles emitted from the source ionize the surrounding air. The devices are effective in neutralizing static charges of *either* polarity. Note that an activity of 1 μCi emits alpha particles at a rate of $1.3\text{E}8\alpha/\text{hr}$ which is *8 orders of magnitude greater* than the alpha emission from a 300 mm diameter wafer corresponding to an alpha particle emissivity of $2\alpha/\text{khr-cm}^2$. However, the use of such a "hot" source on or near an ultra-low emissivity, non-conducting sample within a ULA particle detector may not be appropriate.

We have shown that leaving a low activity alpha particle source in the detector next to the non-conducting sample, *with the ionization detector's high voltage off*, for a period of days is not an effective means to discharge a sample. This is probably due to the ultra-low alpha particle emission rate from this source.

The use of a piezoelectric antistatic gun to neutralize the static charge can be quite effective [12]. These static guns are a modern alternative to the use of radioactive sources like the Staticmaster. The Zerostat model of the antistatic gun has a removable light emitting diode (LED) on the cap to ensure that the device is functional. Squeezing the trigger releases positively-charged ions and releasing the trigger releases negatively-charged ions. This device can be used to eliminate the static charge or to induce charge on the surface.

Alternatively, we have had success at eliminating the effect of the static electricity on the surface of the glass by using three different methods [13]. 1) Similar to methods used in scanning microscopy, we have sputtered an ultra-thin metal (10 nm Molybdenum) layer on top of a 300 mm diameter glass wafer and grounded it to the sample tray. The Mo layer acts to prevent the formation of static charge on the surface. 2) We have experimented with using a grounded high transmission electroplated metal grid on top of the charged glass samples, which, like the cathode in the Alpha Sciences proportional detector, shields the active volume of the ionization detector from the static charge on the sample's surface. 3) Static electricity on our glass samples has been eliminated by spraying them with STP Son-of-a-Gun [14] protectant, wiping vigorously, and then grounding the samples. Each of these methods has some drawbacks. Sputtering metal onto the sample, and the STP treatment involve extra sample handling and preparation. Use of the metal mesh involves procuring a high-transmission, ultra-low emissivity material, and correcting the alpha particle emissivity from the sample (with the mesh) for the mesh's emissivity and transmission. Alternatively, one could use an ultrathin, grounded, metalized mylar film over the insulating sample. As with the mesh, one would need to correct for the emissivity of the film. Finally, the alpha particles from the sample would lose energy through the film. Alpha particles emerging from the sample at 1.5 MeV would lose about 0.5 MeV going through a 2 mm thick mylar film [15]. For this reason, we recommend the use of the grid placed over the samples.

VIII. Conclusion

We have shown that the effects of static electricity on the *surface* of electrically insulating samples can cause the alpha particle emissivity of samples being measured to be underestimated. By using a monoenergetic radioactive alpha particle source, the effects of static charge on a neighboring non conducting sample has been shown to cause a distortion of the energy spectra, at lower energies, as well as a reduction of the measured count rate. The same effects were demonstrated using the natural radioactivity of the *charged* glass sample itself. We observed that the initial alpha particle emissivity of the charged glass was about 1/2; the value of the same *uncharged* glass and that the glass discharged over the course of about three weeks.

Using a commercially-available electrostatic meter, we were able to measure the polarity and magnitude of static charge induced on moderately active glass samples as well as that induced on a piece of Teflon. This meter, as well as direct alpha particle emissivity measurements, confirmed that some new techniques were effective at discharging the induced static electricity on these samples. Other techniques were demonstrated to *eliminate* the effect of the static charge on the alpha particle emissivity measurements.

Considering that the anode on the XIA UltraLo-1800 detector has a bias of $\sim$ 1 kV at a distance 6 in (15 cm) above the cathode, the static voltage induced on the surface of the glass or Teflon is considerable and easily perturbs the nominally weak (66.7 V/cm) electric field of the counter. This was confirmed in our finite element modeling effort. Given the polarity, and magnitude of the static charge induced on the surface of non-conducting samples, the electrons generated by the ionization of the argon gas near the sample may drift towards the sample, in the wrong direction (for the case of the charged glass), or towards the anode at increased velocity (for the case of the charged Teflon). This qualitatively explains our results which showed a marked *reduction* of reported alpha particle emissivity for both cases.

We have demonstrated that the loss of detection efficiency for non-conducting samples depends on polarity and magnitude of the surface charge on the samples. It is expected that the loss of efficiency would also depend on other factors like sample size, homogeneity or distribution of the surface charge, sample thickness, and perhaps even the liner material on the sample tray. With the caveats presented in this paper, great care should be taken in making alpha particle emissivity measurements on insulating samples with ionization counters.

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