An Evaluation of An Ultralow Background Alpha-Particle Detector

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***Abstract*—XIA has provided IBM with a prototype ultralow background alpha particle counter for evaluation. Results show a significant decrease in background compared to other commercial counters allowing for rapid measurement of low-emissivity materials.**

***Index Terms*—Alpha-particle detector, electronic signal rejection, ionization counter, low background.**

I. Introduction

A MAJOR contribution to single-event upsets (SEUs) is due to alpha-particle activity in the semiconductor packaging materials. Typically, the alpha-particles are emitted from materials containing trace amounts of Th or U. In addition, naturally occurring isotopes of elements used in semiconductor fabrication, like ^{190}Pt or ^{174}Hf, are themselves alpha-particle emitters [1]; however, the thicknesses of the layers used, along with their long half-lives, result in very low emission rates. On the other hand, Pb is still used in some semiconductor manufacturing, and the emission from ^{210}Pb can lead to substantial alpha-particle emission. In some applications, Pb is being replaced by Sn alloys, and there is concern that the Sn may be contaminated with residual Pb during mining and refining of the Sn alloy. To minimize the alpha-particle component of SEU, it is becoming increasingly important to use very low alpha-particle emissivity materials in all stages of the manufacturing process.

International Sematech issued a roadmap in 2001 calling for an alpha-particle detection limit of 0.1 α/kh-cm^2 in the energy range 1–10 MeV for measurement times of less than 168 h (1 week) with sample sizes < 1500 cm^2, and a cost of less than \$50 000 [2]. Recently, Wrobel *et al.* [1] showed that a contamination of as little as 0.1 PPB of uranium can lead to an alpha-emission rate of this size and that alpha particle emission rates of a few times this magnitude can give SEU rates equivalent to those caused by cosmic neutrons. In addition, Autran *et al.* [3] showed in a recent paper that the alpha-particle emission, inferred by underground SEU experiments on a 65 nm device was about 0.9 α/kh-cm^2. Clearly , there is now a pressing need to resolve alpha-particles in packaging material at the 0.1 α/kh-cm^2 level, which no commercially available detector can currently reach in acceptable counting times.

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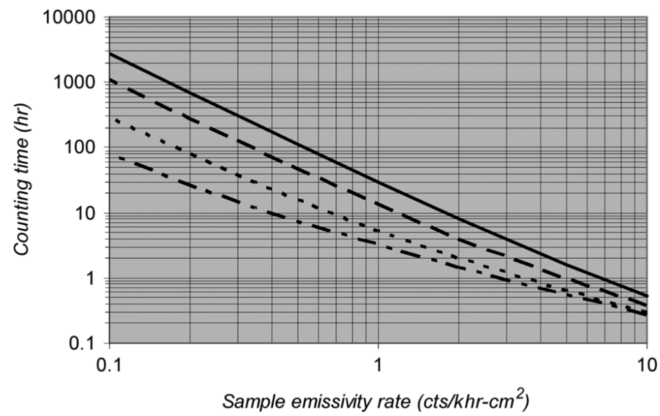

Fig. 1. Counting time required for a 1000 cm^2 sample to yield 90% confidence as a function of sample emissivity for a counter background of 5 cts/h (solid), 2 cts/h (dashed), 0.5 cts/h (dotted), and 0.1 cts/h (dash-dot).

XIA LLC has patented, and recently fabricated, several prototype large sample area, low-background ionization counters intended to address this need [4]. The research laboratory at IBM has been evaluating one of these prototype counters over the course of about 18 months. This paper will outline the counter's salient features, discuss the characterization of the counter, and compare some of our results to those obtained on a commercially available counter.

II. Low Background Ionization Counter

1) Counting Time Versus Background: Counting low-level sample activity in the presence of background in a counter is a difficult endeavor. As the count rate from the sample falls to or below the background rate, the time required to gather the necessary statistics increases dramatically. It has been shown [5] that

$$t = \frac{K^2}{S^2}(S + 2B) \tag{1}$$

where S and B are the sample rate and background rate, respectively, expressed in counts/hour, t is the counting time, in hours, and K is the ratio of S to its standard deviation σ_s. Setting $K = 1.64$, for example, gives results with 90% confidence (from a table of normal errors). From (1), t scales as $1/S$ when $B \ll S$, and t scales as $1/S^2$ when $B \gg S$.

Fig. 1 shows a plot of the measurement time required as a function of sample emissivity at several values of the counter background to achieve $K = 1.64$ for a sample size of 1000 cm^2.

From (1), as shown in Fig. 1, it would take about 2700 h to measure a sample with 0.1 cts/kh-cm^2 emissivity with a counter of background of 5 cts/h at a confidence level of 90%. Counting

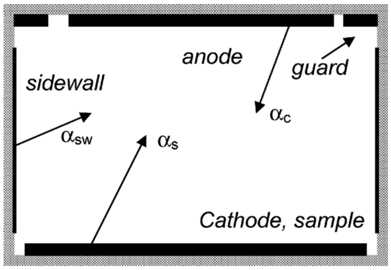

Fig. 2. Schematic cross-sectional view of the ionization counter.

time is reduced by approximately 9× if the counter background is reduced to 0.5 cts/h. For the case of zero background, the counting time would be reduced to only ~27 h, showing the critical importance of lowering backgrounds to effectively count in the 0.1 cts/kh-cm^2 emissivity range.

Using $\mathrm{K} = 1.64$ means that one would measure an emissivity of 0.1 ± 0.064 cts/kh-cm^2, with a 90% probability that the true value lies between 0.036 and 0.164 cts/kh-cm^2. Since the probability of the actual emissivity exceeding 0.30 cts/kh-cm^2 is less than 1%, this result is useful for materials screening, particularly if it can be achieved rapidly. Larger K values would obviously be more desirable, but are impractical in presently available counters, since their large background counting rates make the required counting times unacceptably long.

2) Physics of the XIA Counter: A cross-sectional drawing of the ionization counter and photograph of it are shown in Fig. 2 and Fig. 3, respectively. The anode and guard rings are biased with positive high voltage, and the cathode, which serves as a sample tray, is grounded. The guard ring surrounds and is coplanar with the anode. To shape the electric field within the counter's volume, PCB boards with graded voltage are located on all four side walls. Alpha particles emanating from the sample, sidewall, and ceiling are shown, respectively, as $\alpha_{\rm s}, \alpha_{\rm sw}, \alpha_{\rm c}$ in Fig. 2.

The anode and guard ring on the prototype counter are square, with outer dimensions 42.7 and 52 cm, respectively. There is a gap approximately 0.8 cm between the outer edge of the anode and the inner edge of the guard ring. Each of the four sidewalls has dimension 52.5 cm long × 15 cm high. The cathode dimension is 52 cm × 52 cm.

The counter is filled with pure Ar as the counting gas, (boiloff from liquid Ar). Custom charge-sensitive preamplifiers, connected to the anode and guard ring, integrate the induced charge as the electrons created in the alpha-particles' ionization tracks drift upward in the applied electric field. The signal's rise time is equal to the electron drift time, which is proportional to the drift distance squared, and inversely proportional to the electron drift mobility in Ar and to the applied voltage. The signal's amplitude is linearly related to the electrons' drift distance, and the number of electron/hole pairs in the track [4]–[6]. Alpha particles emanating from the anode (ceiling) and the sample (cathode) can then be easily distinguished, based on both their rise time and signal amplitude, provided that the separation between the cathode and anode is significantly greater than the range of alpha particles in Ar (~5 cm at standard temperature and pressure (STP) for 4.5 MeV α's) [7]. The present counter's height, 15 cm, provides a ratio approximately equal to three.

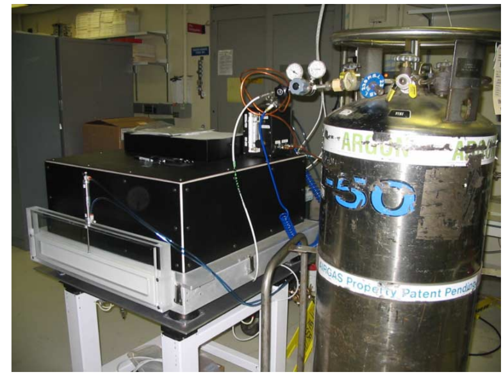

Fig. 3. Photograph of the ionization counter and the dewar supplying Ar.

Alpha particles emanating from the sidewalls of the counter can be rejected since a significant fraction of their ionization charges are collected on the guard electrode, providing a veto signal.

In contrast to the XIA ionization counter, the signal risetime in proportional counters is governed by the arrival of signal charges at the multiplication region. Thus these signals cannot be used to identify the particles' origins because that information is carried in their drift times. In these counters, then, "low" background can be achieved only through the use of ultralow emissivity construction materials.

Fig. 3 shows the liquid argon dewar on the right. The black box on top of the counter contains the high-voltage power supply and signal processors.

3) Electronics, Signal Processing: The anode and guard ring are capacitively coupled through high-gain, low-noise preamplifiers to individual digital signal processors that synchronously digitize, capture, and store signals from both channels whenever a pulse exceeding a user-defined threshold is detected on either. These signals are then fit to determine their rise times and pulse heights [5]. For each event, these fit values are compared to preset thresholds in order to ascribe a physical origin to each event (e.g., sample, sidewall, anode, or noise). The counter's thresholds are set to record alpha particles in the range of 1–10 MeV. To prevent excessive dead times, count rates from radioactive sources are limited to $\sim 2\,\alpha/\mathrm{s}$. The signal processors communicate to the system laptop through a fiber optic USB cable.

4) Graphical User Interface (GUI): Several of the alpha counter's novel capabilities are accessed through a GUI. As an example, one screen is available to sample noise on either the anode or guard ring electrode and replaces the need for both an external oscilloscope and a spectrum analyzer. This capability is useful in diagnosing noise from a variety of sources including ground loops. An example of the functions provided by the GUI is shown in Fig. 4, where the noise from the anode is displayed (top) and the fast Fourier transform (FFT) of the noise is shown on the bottom. The FFT shows several peaks with noise at 120 Hz and ~680 Hz—which are the resonant frequencies of the ac line voltage, and of the counter with gas flowing, respectively.

Another useful GUI function is a real-time display of the classification (alpha, ceiling, sidewall, noise) of an event, displayed on a 2-D scatter plot of anode rise time as a function of anode pulse height. The rise time and the pulse height of the anode and

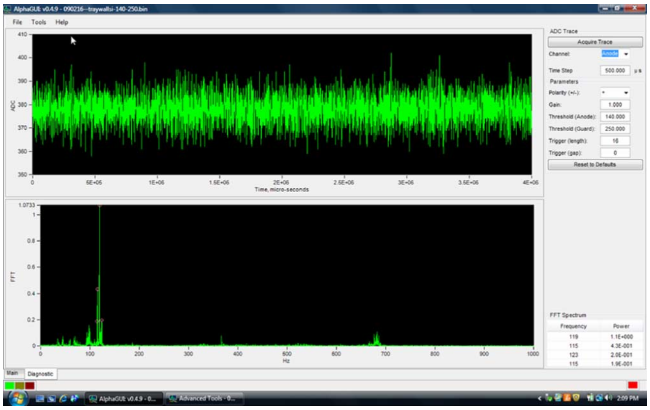

Fig. 4. GUI screen to aid in debugging noise.

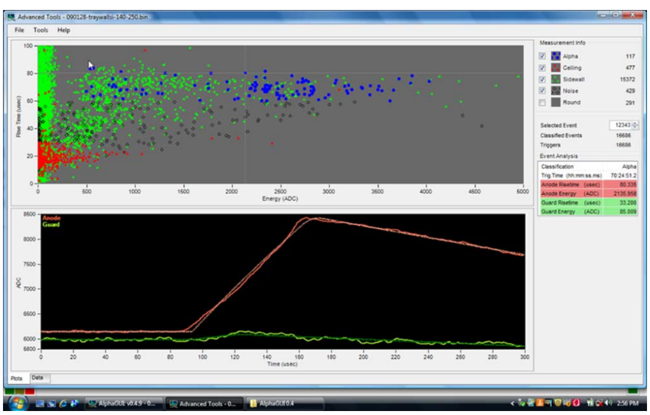

Fig. 5. GUI screen showing the classification of events and signal shapes.

guard signals are used to classify each event. An example of this function is shown in the upper portion of Fig. 5. Each dot represents an event. Dragging the mouse to an event of interest, and clicking on it, shows both the anode (red) and guard (green) signals, and the time-dependent fit to these signals. The rise time and amplitude are determined from the fit (see lower portion of Fig. 5).

5) Detector Signals: Two low-activity alpha-particle sources were used to characterize the ionization counter: an 85 pCi monoenergetic ^{230}Th source[1] at 4.69 MeV and a ^{210}Pb source [8] that provides alpha particles with a continuous energy distribution up to 5.3 MeV. Representative anode and guard ring signals resulting from placing the ^{210}Pb source at the center of the sample tray, the center of one sidewall, and the center of the anode are shown in Figs. 6, 7, and 8, respectively (black is the anode signal, red the guard ring). Fig. 6 (sample) shows a large amplitude, 70 μs risetime, anode signal, and noise on the guard ring. Fig. 7 (sidewall) shows a large guard ring signal, and no anode signal. Fig. 8 (ceiling) shows a small, 15 μs, risetime signal on the anode, as expected, and just noise on the guard ring. While, for sidewall events, the amplitude of the guard ring pulse will clearly vary with the vertical location of the alpha emission, for proper counter operation, that amplitude needs only to be large enough, compared to the noise, to reliably generate a veto signal.

[1]Source Purchased From Analytics, Eckert and Ziegler, 1380 Seaboard Industrial Blvd., Atlanta, GA 30318.

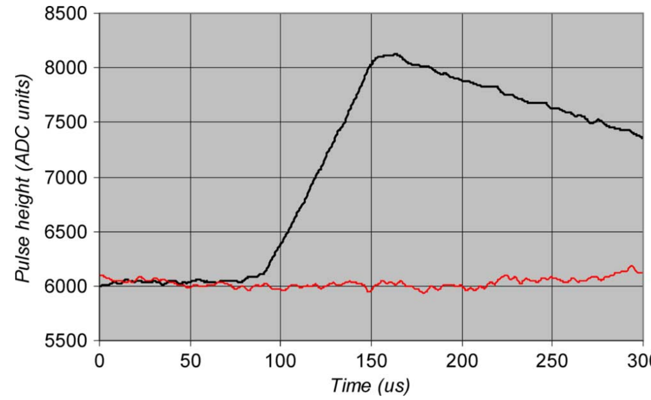

Fig. 6. Source on the sample tray (cathode).

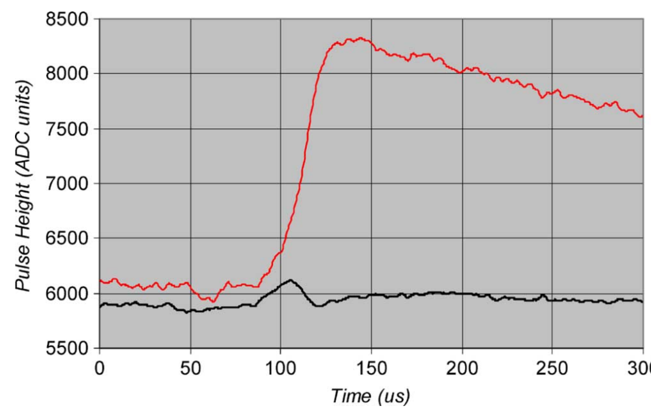

Fig. 7. Source on sidewall.

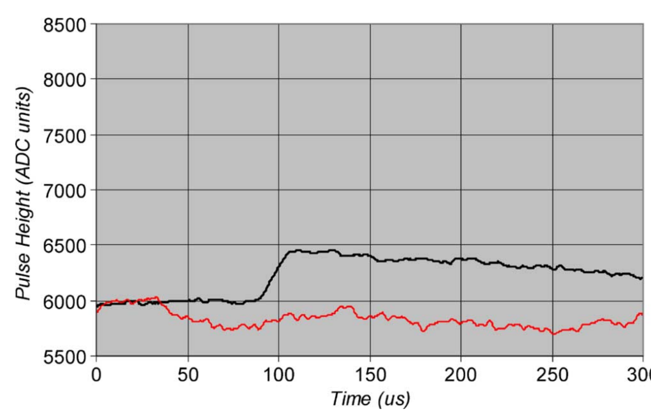

Fig. 8. Source on anode (ceiling).

Since the alpha particles emitted from the sample emerge in all directions, and the energy required to produce an electron/hole pair in argon is ~30 eV, ionization counters typically have poor energy resolution. However, some useful energy information can be obtained to determine the isotope producing the alpha particles in a sample. As an example, Fig. 9 shows the energy spectrum of a ^{210}Pb source obtained using a silicon detector, and Fig. 10 shows the energy spectrum obtained using the XIA counter. The large number of counts below ~1 MeV in Fig. 9 is due to beta particles (β's), which the ionization counter is not sensitive to. The minimum anode pulse height on the XIA counter is 500, corresponding to alphas with energy of ~1 MeV.

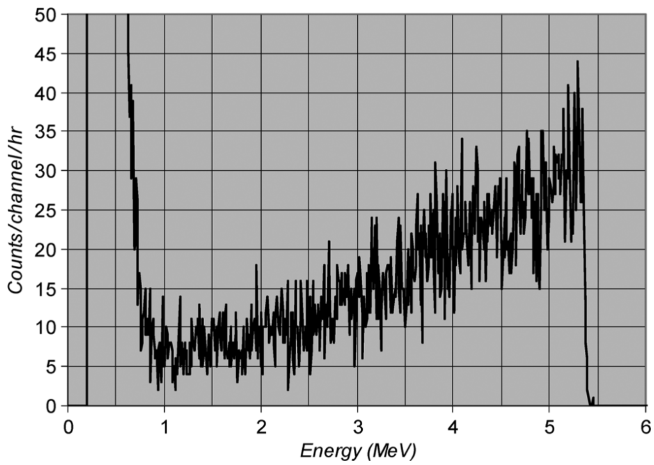

Fig. 9. Energy spectrum of the ^{210}Pb source, measured using a silicon detector.

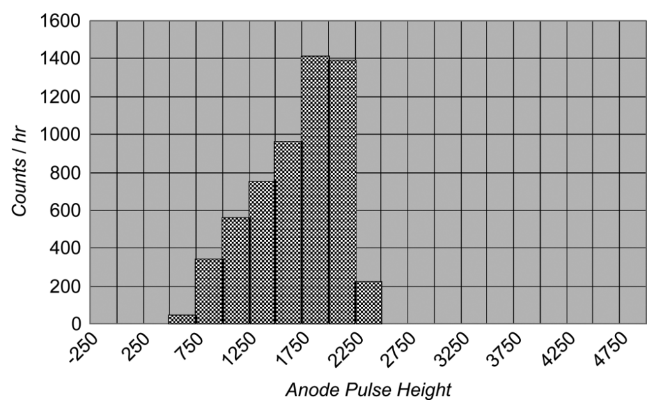

Fig. 10. ^{210}Pb source, energy response of the XIA counter.

6) Effect of Counting Gas (N_2, Ar): In an effort to reduce operational expenses, experiments were performed to look at the performance of the prototype XIA counter using nitrogen gas instead of argon. As mentioned earlier, the source of argon for the XIA counter has been boil-off from a liquid argon dewar which provides a high-purity gas source. A large, 180-liter dewar typically lasts about 3–4 weeks with flow rates of about 4 L/min. A simple cost analysis shows that, in our laboratory, nitrogen costs about seven times less than argon, on a per ft^3 basis.

Argon is typically used as an ionization counter fill gas because of the small energy loss per ion pair formed during ionization in Ar; this is commonly referred to as the "W-value." For the detection of alpha-particles, the W-factor is 26.3 and 36.4 eV/ion pair for argon and nitrogen, respectively [9, p. 130]. Given a fixed cathode-to-anode separation, and electric field, the anode pulse height varies as the inverse of the W-factor- so the pulse height is expected to be reduced by about 28% using nitrogen as a fill gas.

The anode rise-time varies inversely with the electron mobility. At a reduced electric field of ~ 0.1 Vcm^{-1}torr^{-1} (corresponding to an electric field of 1100 V/15 cm, at 760 torr) the electron drift velocities are about 0.2 and 0.32 cm μs^{-1} in argon and nitrogen, respectively [9, p. 182], [10]. Therefore, the anode risetime is expected to be reduced by about 37% using nitrogen, compared to argon as a fill gas.

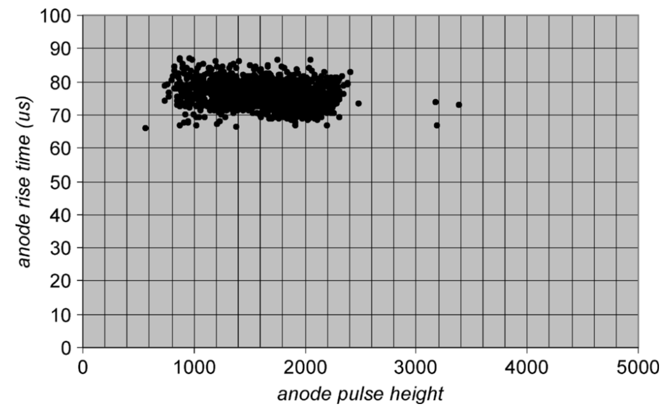

Fig. 11. Anode rise time versus pulse height, argon in counter.

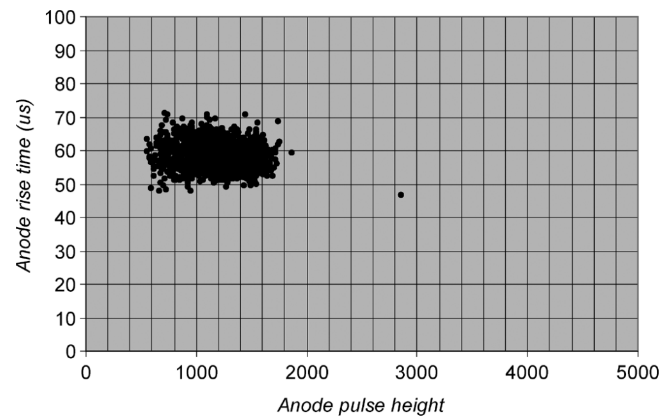

Fig. 12. Anode rise time versus pulse height, nitrogen in counter.

Figs. 11 and 12 show scatter plots of the anode rise time as a function of the anode pulse height for the case where the counter was filled with argon and nitrogen, respectively using the ^{210}Pb source described earlier. As expected, the range of anode rise times and anode pulse heights are significantly reduced when operating the counter with nitrogen compared to argon.

Since a set of parameters stored in a file is used to eliminate most of the extraneous signals (noise, ceiling and sidewall events) based on the anode rise time and pulse height, as well as the guard pulse height, switching from argon to nitrogen requires modifying the parameters to account for the performance differences between the two gases. After making the appropriate parameter changes, the background count rate in the prototype counter was found to be independent of the gas used.

III. Results

1) Background: "Background," in the context of the alpha-particle detector, is that fraction of the count rate, or emissivity, that arises from sources other than the sample. The established method for determining this number is to make a measurement in the absence of a sample. Since most of the background in existing proportional counters comes from alpha-particle emission from the materials *within* the counter, this approach has worked fairly well for activities of a few cts/kh-cm^2 and above. Thus the background level of each counter is unique, reflecting both its individual construction and its usage history, which involve possible radioactive contamination. The counter tray can also become contaminated, but can often be cleaned. Other, less significant, sources of background in proportional counters might emanate from *outside* influences like power line disturbances, microphonics, radon in the counter gas, etc.

When attempting to measure samples whose activities are at or below $\sim$ 1 ct/kh-cm^2, this approach becomes problematic because removing the sample merely exposes a new area (e.g., the sample tray) to the counter that can easily have a higher activity than the sample itself. Ideally, one would like to replace the sample with an equal area of zero emissivity material, but such materials are notoriously difficult both to procure and to maintain pristine, since even short exposures to atmosphere can contaminate them with radon daughters. To address this issue, the XIA counter design assumes that the sample fills the entire 1800 cm^2 active area of the sample tray, so that the tray's emissivity is excluded from the measurement. However, many samples, e.g., processed wafers, are substantially smaller in area. For these samples our approach was to remove the sample, measure the activity of the sample tray, and then proportion the total activity measured with samples on the tray between the samples and tray in direct proportion to their exposed areas. To minimize errors associated with this approach, the tray was lined with a very low activity material whose measured activity is routinely $\leq$ 0.6 $\pm$ 0.1 cts/kh-cm^2. Since the fraction of uncovered tray was often less than 50% the resultant background is significantly lower than other counters used at IBM and is a major step toward the Sematech goal of 0.1 cts/kh-cm^2 [2].

This approach, however, is only valid if the XIA counter's active signal rejection scheme is sensitive only to counts emitted from the active sample area. We will present data later in this section that suggest the presence of another source of background counts that does not scale with exposed tray area. If verified, this term, coupled with the background estimation scheme described above, would lead to the consistent overestimation of alpha activities in the range below a few cts/kh-cm^2.

2) No β-Sensitivity: The ^{210}Pb source emits β's with an endpoint energy of 1.16 MeV (from the decay of ^{210}Pb $\rightarrow^{210}$ Bi). The XIA counter was exposed to these β's, by covering the source with plastic ($\sim$ 100 μm thick), which was thick enough to stop the most energetic alpha particles from ^{210}Po but allow the β's to pass through. No change in the background was observed, showing that the counter is insensitive to β's.

3) Sidewall and Anode Rejection: The ^{210}Pb source was placed on the center of the sidewall, and separately, on the center of the anode. Events were rejected that emanated from the sidewalls, ceiling, and from noise pulses using the same rejection algorithm used for determining the counter background. While the emission rate from this source was about 1650 α/h, no increase in the alpha particle counting rate was observed compared to the background, for either case. In fact the difference between the counting rate with the source on the sidewall (or ceiling) compared to the background was less than about 1/2 σ for an 18-h run. We therefore estimate that both the sidewall and ceiling rejection rates are better than 1 in 3E4, confirming the efficiency of the active signal rejection approach.

4) Detection Efficiency: Using the ^{210}Pb source, the count rate of detected alpha particles was measured as a function of source position from the center of the sample tray. For reference, the anode is 42.7 cm wide. Fig. 13 shows that the count rate falls off, as expected, by about a factor of 2 near the edge of the anode's projection on the tray, where 1/2 of the alphas will move away from the anode. The lines in Fig. 13 are to guide the eye. The counter's efficiency is essentially 100% up to about 17.5 cm from the tray center.

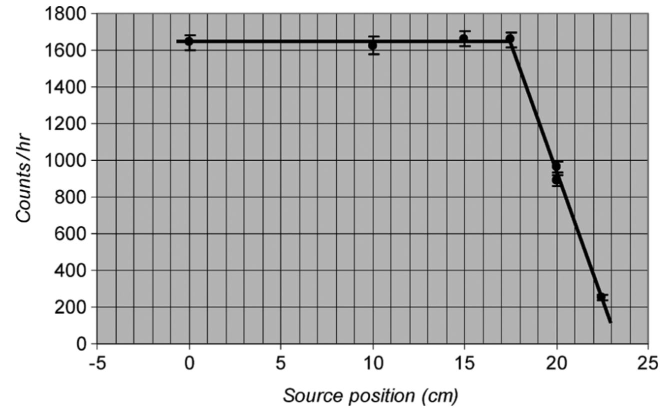

Fig. 13. Count rate versus distance from tray center for ^{210}Pb source.

TABLE I
COUNTING RESULTS ON ONE Sn/0.5% Ag SAMPLE, *MODEL 1950

Counter	Count Time (hrs)	Activity (α/khr-cm^2)
Alpha Sciences*	273	1.8 ± 0.2
Alpha Sciences*	177	1.4 ±0.2
XIA	70	1.9 ± 0.2
XIA	24	1.9 ± 0.3
XIA	24	2.1 ± 0.4

5) Comparison to Commercial Counters: Several low-emissivity samples were measured in both the prototype XIA alpha-particle counter and in commercially available proportional counters (Alpha Sciences, Model 1950) and the results compared. In the data reduction for the XIA counter, as discussed above, the sample tray counting rate that is subtracted from the counting rate with the sample on the tray is proportional to the uncovered fraction of the sample tray. Thus, a 1000cm^2 sample covers 56% of the tray (a typical value); then 44% of the empty tray counting rate is subtracted from the sample counting rate.

In the following results, the 1-sigma errors stated are from counting statistics alone.

A first experiment consisted of making several measurements of the emissivity of a large-area, ($\sim$ 1000 cm^2) flat sheet of Sn/0.5% Ag with Alpha Sciences counters and the XIA counter. Results from these measurements are summarized in Table I. As can be seen, the results agree, within their standard errors, yet the XIA results were obtained in approximately 10% of the time, 1 day versus 10 days.

In a second experiment, four 17.5-cm, square pieces from 300 mm wafers were plated with low-alpha lead. First, several months after plating, the alpha-particle emissions of the four pieces were measured for 164 h in parallel, each in its own Alpha Sciences Model 1950 counter. Then the four wafer pieces were placed in the XIA counter and the alpha-particle emission measured from all four simultaneously for 10 h. The average emissivity found by the Alpha Sciences counter was 4.0 $\pm$ 0.4 cts/kh-cm^2, versus 4.4 $\pm$ 0.6 cts/kh-cm^2 found by the XIA prototype counter, again in agreement within statistics. Total measurement time, however, was reduced from 656 h (total) to 10 h.

In a final experiment to look at the lowest attainable activity levels, an IBM-proprietary ultralow-emissivity material was deposited onto four silicon wafers each with area ~ 400 cm^2. These wafers were measured, in parallel, using four Alpha Sciences counters for 189 h. Then the four wafers were placed in the XIA counter and the alpha-particle emission measured from all four simultaneously for 116 h. The average emissivity of the four wafers when measured by the Alpha Sciences counters was 0.1 ± 0.2 cts/kh-cm^2, versus 0.6 ± 0.1 cts/kh-cm^2 when measured in the XIA prototype counter. Total measurement times were 756 h versus 116 h. We note that the Alpha Sciences error is twice the emissivity value, as would be expected from both (1) and Fig. 1, for such a "short" counting time.

The difference between these two measurements is statistically significant. Further, as noted in our first comment on background measurement, the measured emissivity of the low-emissivity tray liner material was also 0.6 ± 0.1 cts/kh-cm^2 and is considerably larger than the value expected, based on other measurements. Taken together, these results suggest the presence of some as yet unidentified source of alpha activity that is evading the XIA counter's active background rejection scheme. Our current hypothesis is that either radon or cosmic-rays, or both, are producing ionization tracks within the counter's active volume. While the active rejection can eliminate many of these events, those events occurring within an ionization track length of the sample tray's surface produce signals that are identical to tracks emanating from the sample and so cannot be distinguished. Because such events originate in the volume, they would not scale with uncovered tray area and so lead to overestimates of the "background corrected" sample activity.

Thus these comparisons show that measurements from the XIA counter appear to give systematically larger measured emissivities than those measured with commercially available counters for ultra low-emissivity samples and nearly identical results for samples with low emissivities. Experiments are ongoing to resolve this discrepancy.

The published background for Alpha-Sciences proportional counters is $< 5\ \alpha$/kh-cm^2 (1–11 MeV), and 4 α/kh-cm^2 (3–11 MeV) for the Ordella proportional counters [11], [12]. These backgrounds are much larger than the results obtained with the prototype XIA counter, which presently achieves a value somewhere below 0.6 α/kh-cm^2. The active signal rejection in the XIA counter is key to achieving this ultralow background. The influence of noise on the count rate (both with samples and when measuring the background) is eliminated since each signal is analyzed. Being so small, the XIA background needs to be known far less accurately than the proportional counter backgrounds in order to make sample measurements at the same accuracy at ultralow activity levels, which leads to concomitant reductions in background measurement times as well.

IV. Summary and Discussion

A prototype XIA low-background counter was extensively evaluated at IBM. Its active signal rejection by pulse shape analysis effectively eliminates counting the alpha particles emitted from the counter sidewalls and from the anode and is key to providing low counter background. The background count rate is currently very constant at about 0.6 ± 0.1 cts/kh-cm^2 in either argon or nitrogen gas. This is nearly 10 times lower than commercially available proportional counters and allows the XIA counter to make accurate measurements more quickly at very low sample activity levels.

One remaining issue with the XIA counter is the presence of a background counting rate that is not proportional to exposed sample tray area. XIA is currently working both to eliminate possible internal sources of radon and to enhance the software to more reliably recognize and eliminate this class of event. Eliminating these events will reduce uncorrected background contributions and lower measured sample emissivity values accordingly.

IBM will be evaluating the commercial version of the XIA detector when it becomes available. Several operational issues with the XIA prototype have been addressed in a new design which will make the counter more user-friendly. These include more robust communications between the laptop and counter, safety and sample handling improvements, and more robust counter electrode surfaces.

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