SEARCH FOR IMPROVED SURFACE TREATMENT PROCEDURES
IN FABRICATION OF HgI_2 X-RAY SPECTROMETERS*

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ABSTRACT

Update

The influence of various fabrication parameters on the surface quality of HgI_2 x-ray spectrometers was studied in detail. Exposure of etched HgI_2 to ambient atmosphere for ~ 24 hours may reduce the electron surface recombination velocity by almost an order of magnitude. Reduction of the etching solution temperature (KI in water) to about 0°C and an increase of the KI concentration to ~ 20 wt.% are also important.

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ABSTRACT

The influence of various fabrication parameters on the surface quality of HgI_2 x-ray spectrometers was studied in detail. Exposure of etched HgI_2 to ambient atmosphere for ~ 24 hours may reduce the electron surface recombination velocity by almost an order of magnitude. Reduction of the etching solution temperature (KI in water) to about 0°C and an increase of the KI concentration to ~ 20 wt% are also important.

1. INTRODUCTION

Recent work¹ at our Laboratories has shown that surface effects are extremely important for the performance of HgI_2 x-ray spectrometers. The absorption depth of x-rays in HgI_2 is only about several microns or less. Therefore, the collection of the free charge carriers generated by the absorbed x-ray photons is limited by both

surface recombination and bulk trapping. The generated free charge collection efficiency η is given by:¹

$$\eta = (1 + s/\mu E)^{-1} (\mu E \tau / L) \{1 - \exp(-L/\mu E \tau)\}, \quad (1)$$

Where L = detector thickness ($L \gg$ photon absorption depth)

E = electric field

μ = relevant carrier mobility (i.e., hole or electron mobility for a positive or a negative polarity to the irradiated electrode, respectively)

τ = same carrier type bulk trapping time

s = carrier surface recombination velocity

When bulk trapping is small compared to surface recombination effects ($L/\tau \gg s$), the half maximum charge collection field E_{hmc} (the field which gives $\eta = 1/2$) is given by $E_{hmc} = s/\mu$, which is independent of the detector thickness L . On the other hand, when surface recombination is small ($s \gg L/\tau$), E_{hmc} is given by $E_{hmc} = (0.63/\mu\tau)L$, which is proportional to the detector thickness. By studying the charge collection vs. voltage plots for various detector thicknesses, it was then possible to estimate the relative roles of surface recombination and bulk trapping for both holes and electrons.¹ With the detectors used, hole surface recombination and hole bulk trapping were of comparable importance. In contrast, electron surface recombination dominated over electron bulk trapping.

As far as our experience stands, this property of electrons in HgI_2 is valid in all detector quality crystals. X-ray detectors are always operated with a negative polarity to the irradiated electrode. Since electron bulk trapping may be neglected, improvement of detector performances could be achieved by finding surface preparation procedures which will reduce the surface recombination velocities. We have recently started a research project directed to that end, the first stages of which are reported here.

2. EXPERIMENTS

All HgI_2 detectors studied were fabricated from crystals grown from the vapour by the temperature oscillation method.^{2,3} A typical volume of a grown crystal⁴ was about $1\text{-}8\text{ cm}^3$. The basic detector handling and preparation procedures include cutting, thickness measurement, surface treatment, contact application, wiring, and contact passivation. In each of these steps, there is a chance of causing surface damage that could increase the surface recombination velocity. The preparation procedure detail is as follows. Samples having an area of about 30 mm^2 and thickness of $0.2\text{ -}2\text{ mm}$ were cleaved with special cleaving equipment⁵ along the crystallographic C plane.⁶ The thickness is then established from the measured attenuation of ^{241}Am radiation (59.6 keV emission) upon passing through the sample. The sample thickness is derived from a known calibration plot. Accuracy of this non-destructive measure is $\sim 1\%$. The samples are then etched in a 7 wt% KI solution in water for about two minutes. After etching, aquadag contacts (Acheson Inc.) of area $\sim 20\text{ mm}^2$ are painted on both sides of the HgI_2 slices. Platinum wires, 0.05 mm in diameter, are used as electrical leads. The contacts are then covered with a Humiseal protective coating (Columbia Technical Corporation). After curing, this compound serves for contact passivation and adds to the mechanical stability of the electrical leads.

In this paper we report on the effect of variations in various parameters on the electron surface recombination velocity. We have changed the etchant concentration, the etchant temperature, and the time of exposure to ambient atmosphere between etching and contact application. Results of more work that is currently being carried out will be reported when sufficient evidence is accumulated.

Evaluation of the electron surface recombination of each surface was done as follows. The HgI_2 detector was placed in an aluminum can with the surface to be studied facing a 0.25-mm-thick beryllium window. The bias across the detector

(the studied side being negative) was provided by a Fluke high voltage power supply. The detectors were exposed to the 5.9-keV x-ray emission of a 1 mCi ^{55}Fe source. The radiation penetration depth into HgI_2 ⁷ is only about 2-3 μm , which is always much shorter than the typical detector thickness. The charge transferred by a current pulse, which follows any photon absorption in the HgI_2 , is pre-amplified (Seforad SR-105 charge-sensitive preamplifier), further amplified (Tennelec TC-205A linear amplifier), and eventually fed into a multichannel analyzer (Elscint meda). The number of current pulses of a certain charge vs charge spectra, measured for a known period of time (typically 2 sec), were stored in the multichannel analyzer's memory.

The measuring procedure was as follows. A voltage V was applied across the HgI_2 detector, then a shutter which blocked the radiation of the ^{55}Fe source from reaching the detector was immediately removed. The multichannel analyzer recorded and stored the accumulated data. Four spectra were made for each voltage, 2 sec long, and then both radiation and voltage were turned off. The next set of measurements to be taken at higher voltage was made only after waiting for about 10 minutes. This procedure was found to be effective to minimize polarization effects.⁸

The charge Q collected for each spectrum is given (in channel number units) by

$$Q = \sum_i N_i \quad (2)$$

where

i = channel number, and N_i = the counts in channel i.

The charge collection efficiency η is given by $\eta = Q/Q_0$, where Q_0 is the saturated charge collected at very high voltages.

3. RESULTS AND DISCUSSION

The first data to be shown are variations in the exposure time of the etched surfaces to ambient atmosphere. In Figure 1, we show electron collection efficiency vs. electric field curves obtained for the two sides of the same detector. The contact to side A was applied within 15 minutes after an ordinary etching procedure. The contact to side B was applied 20 hours later, during which time the sample was simply lying on the table. It is clearly seen that charge collection saturation is obtained at much lower fields for side B. It thus appears that surface exposure to ambient atmosphere improves the surface properties. The electron surface recombination velocities may be derived from the half maximum collection field E_{hmc} by the relation $E_{hmc} = s/\mu$. Taking $\mu \simeq 100 \text{ cm}^2/\text{V sec}$, we get $s_A = 6 \times 10^4 \text{ cm/sec}$ and $s_B = 8.5 \times 10^3 \text{ cm/sec}$. Results of a series of such measurements with various detectors and different waiting times are summarized in Figure 2. Here we plotted the surface recombination velocity ratio s_B/s_A vs. time, where s_A and s_B are the surface recombination velocities of the first and second prepared contacts, respectively, and the time is the period between the two contact applications. It is seen always that $s_B < s_A$. Also, as time increases, the surface recombination velocity s_B becomes smaller relative to s_A . No change in surface recombination is observed after contact preparation. The above results, as seen in Figures 1 and 2, are quite surprising. Usually etched surfaces appear smooth and shiny. After several days they tend to become matte in appearance or even a yellowish layer develops on the surfaces. Apparently some interaction takes place between the HgI_2 surface and atmosphere which helps to reduce the surface recombination velocity. The exact nature of this process, i.e., the actual atmospheric constituents responsible and the influence of other physical parameters such as light or temperature, is yet to be studied. Nevertheless, the results suggest at least a one-day wait is necessary between etching and contact application to improve HgI_2 x-ray spectrometers.

We next considered the influence of etchant temperature. In Figure 3, we plotted the electron surface recombination velocities obtained for various detector surfaces after etching with 7 wt% KI solution in water vs. solution temperature. The etching time was always 2 minutes; the first contact has always been prepared 1 hour after etching, and the second one 80 minutes later. The lowest surface recombination velocities were obtained for etchant temperatures around 0°C - 10°C (the etchant freezes below $\sim 0^{\circ}\text{C}$). As temperature is increased, the surface recombination velocity also increases. Increase of temperature above $\sim 50^{\circ}\text{C}$ damages the sample bulk. The time between preparation of the two surfaces is too short to draw definite conclusions. Still, a tendency is seen that the surface recombination velocity of the second prepared surface is smaller than that of one prepared first, as seen in Figures 1 and 2. The reason for the temperature effect could be related to the etching rate, which increases at higher temperatures, and is to be studied further. Still, a practical conclusion suggests that HgI_2 etching would be carried out with etchant temperature between 0° to 10°C .

The final parameter we examined was the KI concentration in the etching solution. In Figure 4, we plotted the electron surface recombination velocities obtained for various detector surfaces after etching with a KI solution in water at $\sim 5^{\circ}\text{C}$ vs. KI concentration up to 20 wt%. The etching time was always 2 minutes; the first contact has always been prepared ~ 1 hour after etching, and the second one ~ 1 hour later. No considerable influence of the KI concentration is seen. Perhaps higher KI concentrations reduce the influence of exposure to ambient atmosphere to reduce the surface recombination velocity. A practical recommendation would be to use for etching a 20 wt% KI solution in water. However, this should be approached with caution.

In the above experiments we have shown that variations of parameters involved in the fabrication of HgI_2 x-ray spectrometers change the surface quality, hence

the detector performances. Much more work is needed to critically cover all detector fabrication factors, such as ambient atmosphere conditions, etching agents, and contact materials.

4. SUMMARY

Electron surface recombination in HgI_2 was found sensitive to the sample handling and detector preparation procedures. We studied the influence of changes in the etchant concentration, the etchant temperature, and the time of sample exposure to ambient atmosphere between etching and contact application. The practical recommendations which arise are: sample etching should be made with ~ 20 wt% KI solution in water at a temperature between 0°C to 10°C , and the contact application should be made about 24 hours after etching.

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FIGURE CAPTIONS

Figure 1. Electron collection efficiency vs. electric field for the two surfaces of the same HgI_2 sample.

Side (a) - Surface under a contact applied immediately after etching in 7 wt% KI solution in water

Side (b) - Opposite surface when the contact was applied 20 hours after etching.

Figure 2. Surface recombination velocity ratio s_B/s_A vs. waiting time between first and second contact preparations.

Figure 3. Electrons surface recombination velocities in various detectors vs. etchant temperature.

Different symbols stand for different HgI_2 detectors. Full and open symbols indicate results for first and second prepared contracts, respectively. Waiting times between contact preparations was 80 minutes.

Figure 4. Electron surface recombination velocity in various detectors vs. KI concentration in water used to etch the HgI_2 . Different symbols stand for different detectors. Full and open symbols indicate results for first and second prepared contacts, respectively. Waiting time between contact preparations ~ 1 hour; etchant temperature $\sim 5^\circ\text{C}$.

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Figure 1

S-2112-TP

FIG 1

Figure 2

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Figure 3

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Figure 4

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