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FABRICATION AND TIME DEGRADATION STUDY OF MERCURIC IODIDE (RED) SINGLE CRYSTAL X-RAY DETECTOR

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Mercuric iodide is well known semiconductor solid state X-ray detector. Stability of Mercuric Iodide detector is one of the major problems to its practical applications. In the present work, single crystal of mercuric iodide was grown from a well purified material and detector is fabricated on a fused quartz base with platinum electrodes. Degradation of detector is studied and analyzed.

Keywords: WIDE BAND SEMICONDUCTOR, MERCURIC IODIDE, PHOTO-CONDUCTION, X-RAY SENSOR, DEGRADATION AND STABILITY.

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1. INTRODUCTION

Many solid state X-ray detector materials are available today like α -selenium, poly-cadmium zinc telluride, mercuric iodide, lead iodide etc. Out of these, mercuric iodide is most important material, due to its following promising properties (a) It is wide band gap semiconductor (having band gap of 2.13 eV [1]) which leads to reduction in density of thermally generated free carriers as compared to density of X-ray generated carriers. This helps in keeping low dark current in comparison to photocurrent. (b) Due to high atomic number (Hg-80u and I-53u) of its constituents, it has high photon absorption coefficient, (c) Mobility-life-time product of the material is quite high i.e. $10^{-5}\text{cm}^2/\text{V}$, which ensures the better charge collection [2], (d) Operational electric field is fairly low $\approx 10^4 \text{ V/cm}$ which removes the requirement for high voltage power supply. (e) Material processing temperature is low i.e. 100°C therefore, it's processing is easy. However, the material has certain problems too i.e. material has high surface reactivity which drastically affects its stability [3], material poses serious problem in controlling stoichiometry and physical defects and device polarization minimizes the reproducibility of the results for I-V characteristics. Many attempts have been made in past [4-20] to study the properties of mercuric iodide and using it as an X-ray detector probe.

2. EXPERIMENTATION

Mercuric iodide (99% pure) from Qualigen (India) is taken as a starting material which is a red powder almost insoluble in water. It shows limited solubility in ethanol and other organic solvent. As declared by the manufacturer, it contains some water soluble impurities and small amount of ash. First of all chemical is purified with distilled water and dried properly in vacuum. Thereafter, it is distilled repeatedly through a set-up as shown in Fig. 1.

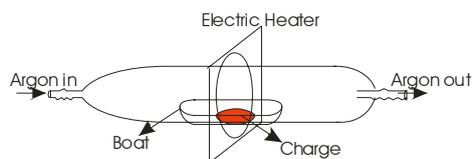


Fig. 1 – Picture of set up used for purification

Material is taken in a glass boat and with help of electric heater, initial warming is done with 99% pure argon flowing over it which helps in degassing the charge. After 10-15 minutes, temperature is raised to 150°C and argon flow is stopped. Outlet of argon is closed, however inlet is kept open which ensures the least mechanical disturbances and helps in crystallization.



a



b

Fig. 2 – Pictures of set -up (a) and crystals (b)

After half an hour thin crystals of big size are found depositing on the walls of the tube and boat. Fig. 2 (a) shows the picture of real set-up and Fig.2 (b) shows the crystal formations on the boat. Crystals are carefully collected and redistilled four times. Fig. 3 shows crystals having dendrite growth which is attributed to the fast growth rate. On reducing the growth rate it is found that crystals are very small in size and it poses difficulty in separating them from the residual charge.



Fig. 3 – Dendrite crystal growth features

Single crystals of mercuric iodide were grown using slow evaporation technique at room temperature (25-35degree Celsius) and purified mercuric iodide material was taken and dissolved in absolute ethanol (Merk India). Mixture is stirred well for 15 minute, using magnetic stirrer and is then allowed to settle down for 10 minutes. Un-dissolved material is separated from the solution by filtering it through Wattman filter paper (No.1). Since

solubility of the compound is poor, the solution is further centrifuged to remove any small crystallite from the solution, before keeping it in a chamber (Fig. 4). For growth of crystals in dark, petridish was covered with black paper foil and stainless steel metallic cover top. This helps in cutting down electromagnetic radiations. Crystal starts growing within two to three weeks to the size of few mm. Crystals which were grown were removed from the petridish with help of thin and soft plastic sheets. Crystals were kept on absorbent paper to remove extra solution from the crystals which were dried in air.

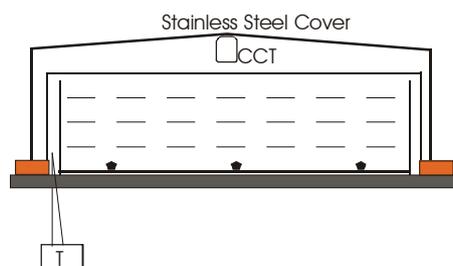


Fig. 4 – Experimental setup for solution growth

Few crystals were selected on the basis of their good morphological shapes and subjected to X-Ray diffraction studies. Crystals with minimum arcing and streaking on the Laue X-Ray photographs (Fig. 5) were then selected for making probe.



Fig. 5 – X-Ray Laue Photograph (Transmission)



Fig. 6 – Crystal with platinum electrodes

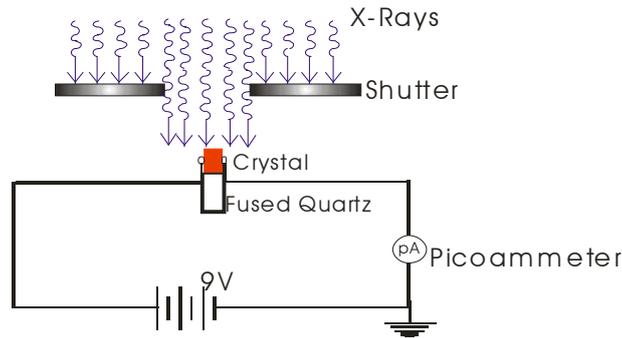


Fig.7 – Sample probe under X-Ray exposure

Two platinum electrodes were made using thin wire (0.5mm) by twisting and rounding it. Crystal and electrodes were cleaned by ethanol and the crystal is fixed between two electrodes and thick film of mercuric iodide is grown over it under low pressure conditions (0.001 mm Hg) as shown in Fig. 6. Sample probe is kept under continuous X-rays (30 KV, 10 mA, Cu-Target and Keithley Digital pico-ammeter is used to read dark and photocurrents as shown in Fig. 7. It was found that under X-ray exposure, photocurrent increases substantially.

3. RESULTS AND DISCUSSION

Probe was kept constantly under the electric field provided by the D.C. source (9V) except for some time (few minute) during which the source e.m.f was measured. Ratio of photocurrent (in presence of X-Rays) and dark current were recorded for nearly a month time for one of the probe. It is found that the variation is nearly logarithmic in nature.

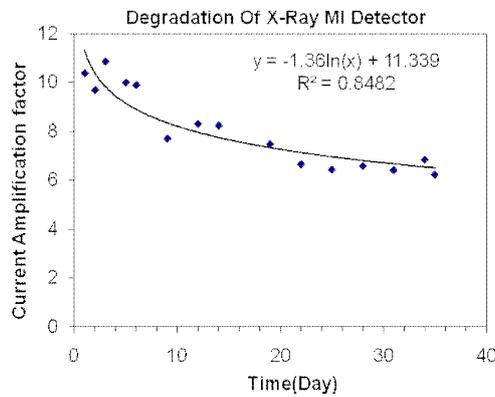


Fig. 8 – Variation of photocurrent with dark current

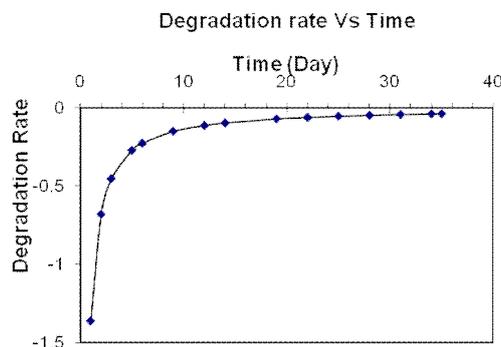


Fig. 9 – Rate of Degradation of detector performance

Probe was developed taking all precautions regarding its chemical purity, almost in-reactive electrodes and high resistive base material. However the initial instability is observed for nearly 20 days as shown in Fig. 8 which almost reduces to expectable limit as shown in Fig. 9. Thereafter, it is attributed to the crystal defects and stresses which creep in during the crystallization and handing of the crystals for probe formation. Constant electric field helps in settling down of these defects and brings stability.

4. CONCLUSION

Mercuric iodide single crystal probe shows high current amplification factor even after 20 days and it remains constant needs thereafter and hence is a promising candidate for X-ray detector.

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