



Comparison of X-Ray Switching Studies on Pure, Doped and Composites of CdI₂

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ABSTRACT

X-ray detectors are most important component of any digital imaging system which requires good spatial resolution, low radiation and digital storage of data. In present days mostly charge-coupled devices (CCD) or complementary metal oxide semiconductor (CMOS) are used in imaging devices. In order to determine the response time of the detector at room temperature, we performed the switching studies on CdI₂ composites, pure and doped cadmium iodide.

KEYWORDS : PMMA, Composites, High energy radiation, Cadmium Iodide, X-rays

INTRODUCTION

Medical diagnostic techniques are developing very fast. X-ray computed tomography (CT) or x-ray computerized axil tomography are few important diagnostic techniques. These are basically digital automated techniques in which X-ray signals are converted into digital electronic signals and analyzed by well-developed signal processing software. [1-4]. In the core of these machines, there exists a detector which converts the electromagnetic wave signal into electric signal. There many types of detectors in use e.g. Xenon gas ion chamber [5], rare earth oxides, iodides of heavy metals like mercury [6], lead, cadmium [7] etc. There is an increasing demand for more advanced and smart materials for X-ray detection. One of the requirements for such detector materials is to have flexible design. Single crystalline material gives limited flexibility in designing the shape of detector. Keeping this in mind, it was also planned to develop composites detectors. To have better spectrum of physical and chemical properties it was planned to blend an inorganic material (cadmium iodide) with organic polymers [8] (poly-methyl methacrylate, poly-styrene, polycarbonate and silicon rubber). Cadmium iodide molecule contains elements having high atomic masses (cadmium-112.4u and iodine-126.9u). This helps in good absorption of X-rays. Further electronic band gap of cadmium iodide is high (3.3-3.1 eV) [9]. This is especially helpful in reducing the dark current due to thermal agitation at room temperature. Poly-methyl methacrylate (PMMA) is well known polymer [10-12].

EXPERIMENT

Cadmium iodide (99% LobaChemie) is used as base material. Purification is done through zone refining method where the main chamber and the boat were made from corning glass. Although cadmium iodide is most anhydrous of metal halides, it does contain a small amount of water vapors, causing oxide formation and consequent sticking of ingot to the container walls. Further in order to minimize reaction of cadmium iodide with gases in the surrounding pure argon is used. It suppresses the evaporation and prevents oxidation. The flow of gas was allowed initial at slow rate. During this period heaters are not given any voltage. Slowly air is flushed out of the glass tube. After sufficient time (10-15 min), voltage to the heater is slowly raised till a fine molten zone is formed. After 9-10 passes visible impurities could be seen accumulated at the end of ingot [Fig. 1]. The intermediate portion of the ingot consists of highly pure material in single crystal form.



Fig 1: Boat with ingot of Cadmium Iodide

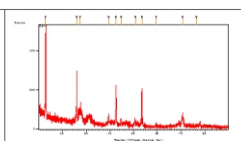


Fig 2: XRD Peaks of Cadmium Iodide

For preparation of composite material following four polymers were selected as matrix materials. (1) Poly-methyl-methacrylate (2) Polycarbonate (3) Poly-Styrene and (4) Silicon rubber. Polymer as matrix material and cadmium iodide is taken in same ratio i.e. 50% of polymer and 50% of cadmium iodide by weight. PMMA granules were dissolved in chloroform (99% Fisher Scientific India) and mixed with fine powder of cadmium iodide. Uniform mixture is then kept to settle down for 48 hours in vibration free atmosphere. After this fine sheets of PMMA-CdI₂ composite were obtained. They were cut in square sizes of 1 cm x 1 cm for further study. Sheets, without air bubble were used for the study. Sheets were polished for smooth surfaces and coated with silver paste. Coated sheets were kept for drying for nearly two hours. Poly carbonate granules are heated and when material becomes soft, cadmium iodide fine powder is mixed and stirred well. Material is cooled slowly to avoid any cracks in the material. When cooled to room temperature, material is cut into small bars of 1 cm x 1/2 cm for X-ray switching and band gap determination experiments. Poly-Styrene is easily soluble in acetone. Hard styrene granules are dissolved in sufficient amount of acetone. Slurry formed is vigorously mixed with fine powder of cadmium iodide. Material is then left to settle down for 24-48 hr. Material then is cut into small plates for the electric measurement study.

Silicon rubber (1010) is mixed with curing agent in ration (100:3) and is mixed well with the fine powder of cadmium iodide. Material is left to settle down for 24 hr. Fine flexible sheet of cadmium iodide composite is obtained. Sheet is cut into small pieces for the electric studies. Electrode on the sheets is made by silver paste. Fine coating of silver paste is left for air drying for 1-2 hr. Sheets were subjected to visible microscopic studies to find-out any possibility of cracks etc. Only those samples were put to studies which are free from any crack of silver electrode or otherwise.

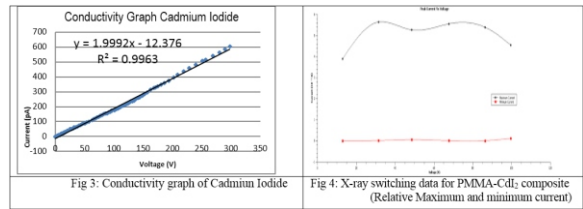
Cadmium iodide is a semiconductor material. One of the important electrical properties of the material is its electronic band gap. To find out electronic band gap of the sample, we used the process of determining electrical conductivity at various temperatures. Sample is kept in oven, which is electrically heated using PID control. We used two probe methods to find out electrical conductivity.

EXPERIMENTAL RESULTS AND ANALYSIS

X-Ray diffraction and particle size are determined by the Scherrer equation, the average crystallite size was found to be 0.0347 μm. Single crystals were subjected to measurement of electric conductivity. Crystal show almost linear variation of V-I curve at constant temperature [Fig 3].

During the switching studies, crystal surface was repeatedly illuminated by white X-rays generated from Cu-target X-ray tube operated at accelerating voltage of 30KeV and at plate current of 10mA. Frequency of switching is of the order of low frequency 0.5Hz. Crystal area of nearly 3mm² is exposed in the middle of the crystal. Electric field is applied perpendicular to the direction of the X-ray beam. Magnitude of electric field is varied in the range of few volt/mm. Current is collected by electrodes and measured by pico-ammeter.

The prepared composite detector is exposed to X-rays in repeated manner. Current through the detector is recorded with time. X-ray switching studies on PMMA-CdI₂ composites [Fig.4] were conducted at various voltages.



DISCUSSION

X-ray sensing results of various cadmium iodide crystals and composites are summarized in table 1 given below.

Table 1 Comparative results of cadmium iodide crystals

Material	I_{max}/I_{min} (at voltage 50v)	Range I_{max} (A)	Range I_{min} (A)	FFT Distri- bution	Error pattern of differential
Pure Cadmium Iodide (Melt)	4.6	10^{-10}	10^{-11}	Symm etric	Low at low frequency and High at high frequency
Pure Cadmium Iodide (solution)	1.5	10^{-10}	10^{-10}	Symm etric	Low at low frequency and High at high frequency
Bromine doped Cadmium Iodide	1.1	10^{-10}	10^{-10}	Symm etric	Low at low frequency and High at high frequency
Lead doped cadmium iodide	1.1	10^{-8}	10^{-8}	Highly symm etrical	Low at low frequency and Moderate at high frequency
PMMA-Cadmium Composite	142.8	10^{-11}	10^{-13}	Highly Symm etric	Low at low frequency and High at high frequency
PS-Cadmium Composite	4.3	10^{-10}	10^{-11}	Symm etric	Low at low frequency and High at high frequency
PC-Cadmium Composite	130	10^{-10}	10^{-11}	Symm etric	Low at low frequency and High at high frequency
SR-Cadmium Composite	1.23 (at 443V)	10^{-10}	10^{-10}	Highly Non- symm etrical	Low at low frequency and very high at high frequency

X-ray sensing with cadmium iodide composites have reveled interesting results. As it is clear from the table 3 given above that, composites have better I_{max}/I_{min} ratio in comparison to the single crystals (pure and doped). It is evident that x-ray efficiency (I_{max}/I_{min}) of the cadmium iodide crystals lies in the range (1.1 to 142.8). It is highest in composites especially in PMMA and PC, where as pure melt grown crystals performed better than doped crystals.

Pure crystals of cadmium iodide have higher I_{max}/I_{min} ratio due to purity. Charge carriers generated by the X-rays are able to reach electrodes basically due to purity. As atomic arrangement is periodic, charges find less resistance to reach electrodes. On other

hand doped crystals develop defects like grain boundaries, which act as trapping sites. This reduces the I_{max}/I_{min} ratio. It can be noticed that in case of lead doped crystals of cadmium iodide both I_{max} and I_{min} are higher. This is due to the maximum strain produced by the lead ions having maximum percentage change in size (Table 2).

Table 2 Relative size of various ions

Ion	Size (pm)	Percentage Change in size
Cd ⁺⁺	109	-
Pb ⁺⁺	133	+22.0
I ⁻	206	-
Br ⁻	182	-11.65

Composite materials are found to be better X-ray sensing materials and also they have good mechanical properties. This brings them in the category of technological advantageous material. This is because they can be shaped according to the requirement. Composites of cadmium iodide have shown semi-conducting behavior with band gap ranging from 3.27-3.53 ev. Higher band gap of these composite semiconductors has advantage to minimize current due to thermal agitation at room temperature. Further small fluctuation in temperature has minimal effect on the variation of photocurrent. Composites of PMMA, PS and PC are mechanically hard. So their mechanical handling is easy. These composites are more rigid and show extremely high stability[13].

Best result is obtained in the PMMA composite.It is found that number of charge carriers passes through the sheets of PMMA and cadmium iodide composites increases with rise in temperature. This can be understood as the polymeric chain and cadmium iodide crystallites act as traps of charge carriers. With rise in temperature, phonon excitation increases. Phonon assisted hopping process helps in releasing the trapped charges. These charges using π -bond electrons move through the polymer moles to the nearby cadmium iodide crystallite. Further these electrons drift under electric field through the cadmium iodide crystallites. Electric field applied to the sheet is of the order of 1×10^4 V/m sufficient enough to cause the drift..Composite of cadmium iodide with polycarbonate also show similar behavior with slightly less I_{max}/I_{min} ratio. However, composites with polystyrene, show limited I_{max}/I_{min} ratio. This is primarily due to:

- (1) Benzyl group presence in this polymer. Due to the benzyl group there exist higher localized electrons in these composites. As a result their conductivity is higher. Dark current through the material is therefore higher in comparison to the photocurrent developed by X-rays.
- (2) Initial material i.e. synthetic rubber used in the process has limited purity (less than 98%). Impurities present in the material are also responsible for higher conductivity of the material.

CONCLUSION

Single crystals of cadmium iodide are good room temperature X-ray sensors with photo multiplication ranging from 1.1-4 at 50V. However, single crystals of cadmium iodide are very soft, slight mechanical disturbance causes damage in their structure and deteriorate X-ray sensing capabilities very fast. Whereas, doped single crystals of cadmium iodide are relatively hard but have higher conductivity. As expected also, the resultant photo current developed in these crystals is relatively small than the dark current.. Hence doping caused decrease in X-ray sensing capabilities. Composite materials are found to be better X-ray sensing materials and also they have good mechanical properties. This brings them in the category of technological advantageous material. They can be shaped according to the requirement. Composites of cadmium iodide have shown semi-conducting behavior with higher band gap which gives advantage to minimize current due to thermal agitation at room temperature. Further small fluctuation in temperature has minimal effect on the variation of photocurrent.Composite specially PMMA minimum degradation with high energy radiation exposure. Rise and fall time of PC

composite are best among these composites. Hence this material can be used for X-ray imaging also.

ACKNOWLEDGMENT

The authors wish to thank director Mr. Brahms Singh, of L'Orgueil Physics Centre (Delhi) for financial support of this project. We are also thankful to Mr.Ganesh for assisting in the lab work

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