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PMMACDICOMPOSITE ASXRAYSENSOR

Strictly as per the compliance and regulations of :

PMMA-CdI_2 Composite as X-Ray Sensor

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I. Introduction

In recent years there is continuously increasing digitalization in every field affecting human life. In diagnostics, use of X-ray computed tomography (CT) or computerized axial tomography scan (CAT scan) and digital X-ray imaging are now routine activities [1-4]. These techniques require the digital data which can be processed by a computer. Normally this data is obtained by X-ray detector. In earlier versions of such machines, cesium iodide crystals were used as scintillation detectors. Later in 1980s use of high pressure Xenon gas ion chambers were used [5]. In modern machines scintillation materials like rare earth oxide ceramics are in use. These materials absorb X-rays and re-emit the absorbed energy in the form of light. Light signals are then converted into electrical signals by photodiode and processed. However there is a possibility of direct conversion of X-ray signals into electrical signals with help of solid state X-ray detectors. These are basically semiconductor materials like silicon or germanium doped with lithium. X-rays passing through these materials are absorbed and are converted to electron-hole pairs to create electrical signals. For proper sensitivity, operations of such detectors require liquid nitrogen temperature. This helps in minimizing thermally generated electron hole pairs. In 1990s many solid state detector materials were explored like alpha-selenium, poly cadmium zinc telluride, mercuric iodide [6], lead iodide etc. Basis for a good solid state detector material is based on the following major properties:-

1. Material should have high band gap. This helps in minimizing thermally generated noise.
2. Constituent atoms should have high atomic number. This is for maximum absorption of X-ray energy.
3. Mobility-life time product of the material should be high. This helps in better charge collection.
4. Operating electric field should be low. It helps in keeping electronics involved simple.
5. Operating temperature likely to be room temperature for ease in operation.
6. Response time should be small for faster data processing.
7. Fabrication of detector should be simple with flexibility of design, shape and size.
8. Highly stable or have low degradation.

Every solid state detector material has some the above properties but lacks in some other properties. One of the important issues is flexibility in design. X-Rays are high energy radiations. When they interact with these materials they are partially reflected and partially refracted through the material. Limited portion is absorbed by the material. If detector is properly shaped, it is possible to maximize the absorption of X-rays. Single crystalline material gives limited flexibility in designing the shape of detector. Keeping this in mind, it was 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 polymer (poly-methyl methacrylate). 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 (3.3-3.1e.V.)[7]. this is especially helpful in reducing the dark current due to thermal agitation at room temperature. Poly-methyl methacrylate (PMMA) is well known polymer. It is selected due to its following properties [8-10].

1. Low linear mould shrinkage (0.003-0.0065 cm/cm)
2. Good mechanical properties {Hardness (63-97 Rockwell,M) and Tensile strength (47-79 MPa)

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3. Most resistant to direct sunshine exposure.
4. Low water absorption (0.3-2%) makes it very suitable for electrical device making.
5. Very low electrical conductivity (10^{-14}-$10^{-15}\Omega^{-1}.cm^{-1}$)
6. High dielectric strength (17.7-60kV/mm)

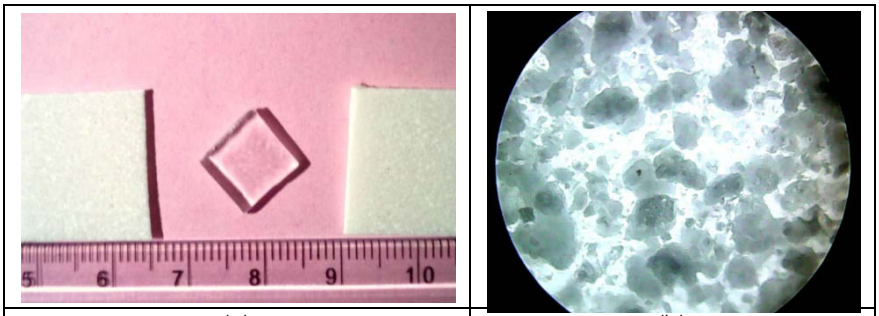
II. Experimental

Cadmium iodide (99%, Loba Chemie) is used as base material. White crystalline powder is fine grinded using agate mortar and pestle to a fine powder. No additive is used during this process. Due care is taken to keep the powder dry. 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. No further hot pressing is done to prevent any mechanical damaging to the sheets. After this fine sheets of PMMA-CdI_2 composite were obtained. They were cut in square sizes of 1cmX1cm for further study. Sheets, without air bubble are 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. Coated sheets were subject to electrical studies for determination

1. Electronic band gap determination
2. Voltage- Current variation study under
   a. Dark conditions (No radiations)
   b. In presence of Low intensity Gamma Radiations(Cs-137 source 67k Bq)
   c. Under X-Rays (Cu-Target)

III. Experimental Results and Discussions

Composite sheets are found to be whitish in colour (Fig. 1(a)). These sheets are nearly 1mm in thickness and are flexible. When observed under low power optical microscope, small air bubbles are found to be trapped in some these sheets. Optical micrograph (Fig. 1(b)), taken under polarized white light shows the formation of crystallites of cdI_2. Crystallite size is measured to be of the order of micrometer. It can be seen that small crystallites are suspended in PMMA matrix. Sheets with three concentrations (by mass) were prepared i.e. 9.71%, 13.95% and 19.09%. Micrographs of these sheets clearly indicate that PMMA gaps decreases with increase in concentration and increasing the filler contacts between cadmium iodide crystallites.



(a) (b)

Fig. 1 : Optical photographs of sheets. (a) Sheets with cdI_2 (Side Sheets) Central sheet is of Pure PMMA
(b) Transmission optical micrograph of PMMA-CdI_2 Sheet (Magnification 100)

Electrically these sheets have very high resistance (3-4 X $10^{11}\Omega$) at room temperature. Two probe methods are used to study their electrical conductivity. Composite sheets with silver paste on both sides were griped by two spring loaded electrodes. This assembly is kept in a PID controlled oven. Regulated power supply is used to provide voltage and current is measured by picoammmeter which is calibrated by Kethley 6485. All measurements for band gap determination are done at low voltage (18V). Temperature is kept below 60^0C, as glass softening temperature of PMMA is 100^0C. When subjected to change in temperature these sheets show semiconducting behavior (Fig. 2).

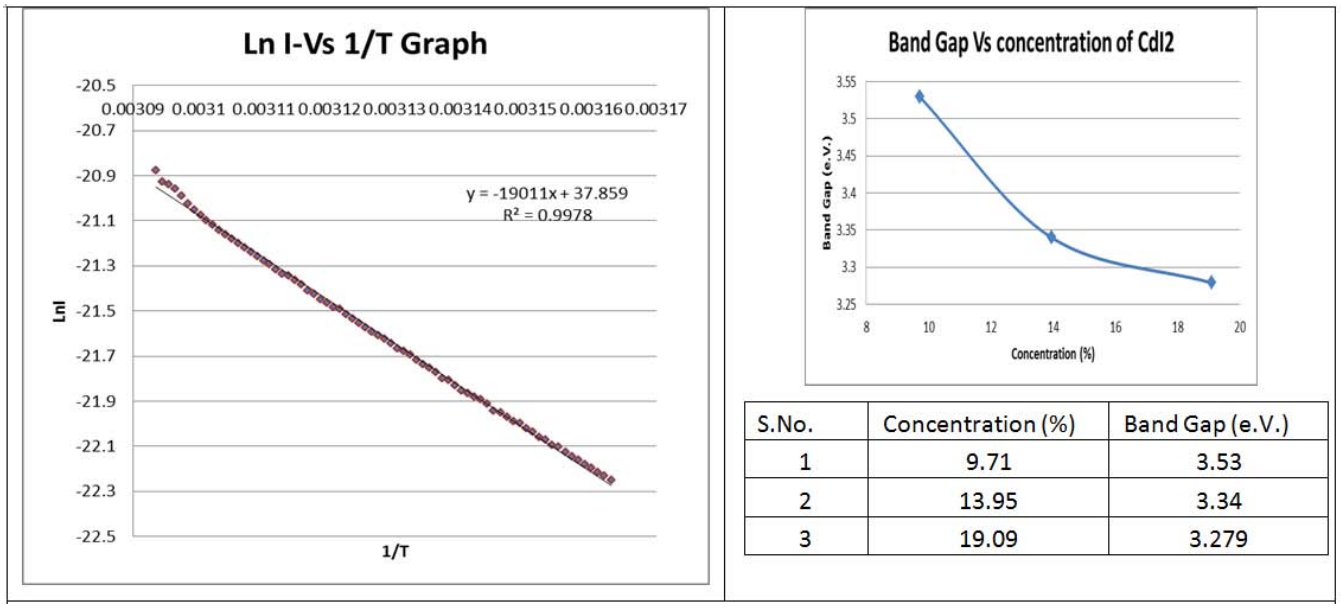
Conductivity of such hybrid structures can be given by combining Mott Variable Range Hopping equation (For PMMA) and basic Arrhenius Equation for semiconducting materials (CdI_2).

$$\sigma = \sigma_m \exp^{-(\frac{T_0}{T})^{1/4}} + \sigma_a \exp^{-(\frac{E_g}{2kT})}$$

Here σ_m is mott conductivity coefficient and T_0 constant, σ_a is Arrhenius conductivity coefficient, T is absolute temperature, E_g is band gap and k is Boltzmann constant. Electrical conductivity of PMMA is negligible (10^{-14}-$10^{-15}\Omega^{-1}.cm^{-1}$) in comparison to the conductivity of cadmium iodide (2.5 X 10^{-9} $\Omega^{-1}cm^{-1}$ [11]). Cadmium iodide is a layered compound. I-Cd-I layers are piled one over the other. There is strong electrovalent bond between cadmium and iodine atoms. However two layers of I-Cd-I are attached to each other by weak vander Waal's bonds. These weak bonds are

responsible for various polytypes structures [12, 13] of the compound as well as high electrical conductivity in comparison to PMMA.

The term $\sigma_m \exp^{-(\frac{T_0}{T})^{1/4}}$ is negligibly small as compared to the term $\sigma_a \exp^{-(\frac{E_g}{2kT})}$. This means that conductivity variation with temperature will show Arrhenius behavior. Same is observed experimentally.



S.No.	Concentration (%)	Band Gap (e.V.)
1	9.71	3.53
2	13.95	3.34
3	19.09	3.279

Fig. 2 : LnI Vs 1/T variation is straight line indicating the semiconducting behavior of the material. Energy band is found to be decreasing with increasing concentration. Voltage is kept 18V

It is found that number of charge carriers passes through the sheets increases with rise in temperature. This can be understood as the polymeric chains of PMMA 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 molecules 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 X 10^4 V/m sufficient enough to cause the drift. (Fig.3)

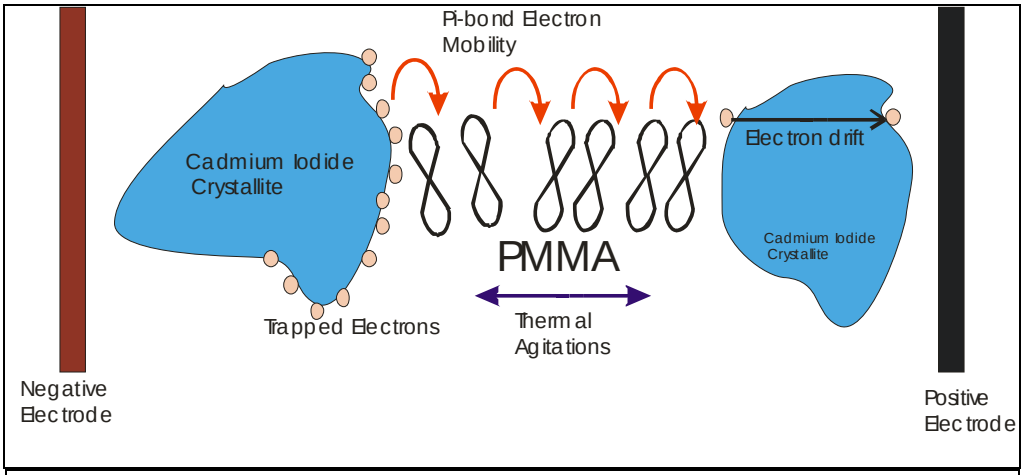


Fig. 3 : Schematic diagram showing the phonon assisted hopping mechanism of charge transmission through the PMMA-CdI_2 composite sheets

Also when exposed to gamma radiations, voltage vs current variations shows a very small change in the variations of current with voltage. Similar variations are observed in all the sheets with different concentrations. It is found that there is small increase in the resistance of the sheet in presence of the gamma

radiations. When gamma source is removed and again the variation of voltage and current is taken, small increase is further observed in the resistance. (Fig4). Following inferences can be drawn from the study:

1. These sheets are almost transparent to the low intensity Gamma radiations as very small changes are observed. This is primarily due to less thickness of sheets (thickness of 1mm).
2. Although interaction of Gamma radiations is limited, yet they are able to create some damage to the bonds and cause some additional trap centers.

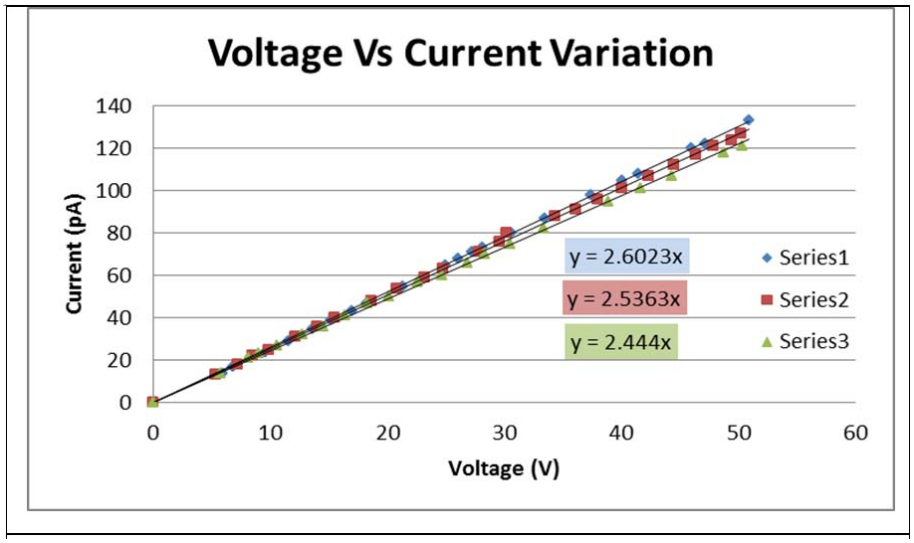


Fig.4 : Voltage Vs Current variation for Raw samples ((Series1) resistance= $3.84X10^{11}\Omega$), During exposure with gamma rays ((Series 2) resistance=$3.94\ X10^{11}\Omega$) and after exposure ((Series 3) resistance is$4.09\ X10^{11}\Omega$)

Sheets were exposed to X-rays (Cu target 30KV, 10 mA plate current) parallel to electrode plates (Fig.5). X-ray intensity to which sheets were exposed is of the order of $10^{-2}W/cm^2$. It was found that photo current increases by a factor of 230.7 in comparison to the dark current (Fig.6 (a)).

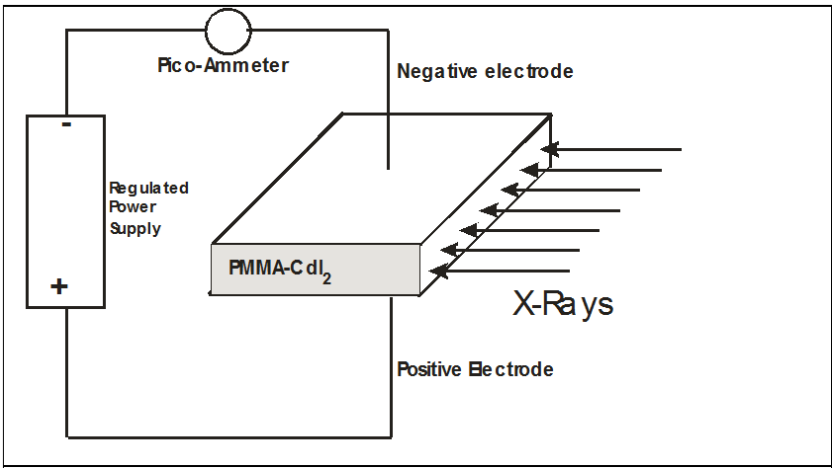


Fig 5 : Circuit diagram to measure photo amplification

Under the X-rays illumination photocurrent generated (Ip) is directly proportional to five main parameters

1. Radiation term eI_x (e=Electronic Charge And I_x=Radiation intensity),
2. Performance factor of material $\eta\tau\mu$ (η=efficiency of conversion of radiations into charge carriers, τ=carrier life time i.e. average life time of carrier before recombination and μ=charge mobility),
3. Electric field applied (E),
4. Dimensional parameters like area to volume ratio of crystallite and
5. Temperature of material.

Material used as a sensor should have very low response time. To evaluate response time switching studies were conducted on these films (Fig.6(b)). Switching study revels following important findings:

1. Rise time and fall time is nearly 2 second for 10 second pulse on these films.
2. Photocurrent does not stabilize very fast. It shows

some zig-zag variation with time as seen in Fig.6 (b). This variation is expected as charge trapping occurs on the surfaces of cadmium iodide crystallite. At higher temperature stability is higher. This is due to quick release of trapped charges under thermal agitation. Such materials still require improvements before they can be used as X-ray Imaging Detectors [14, 15]. Similar results were obtained in all the sheets.

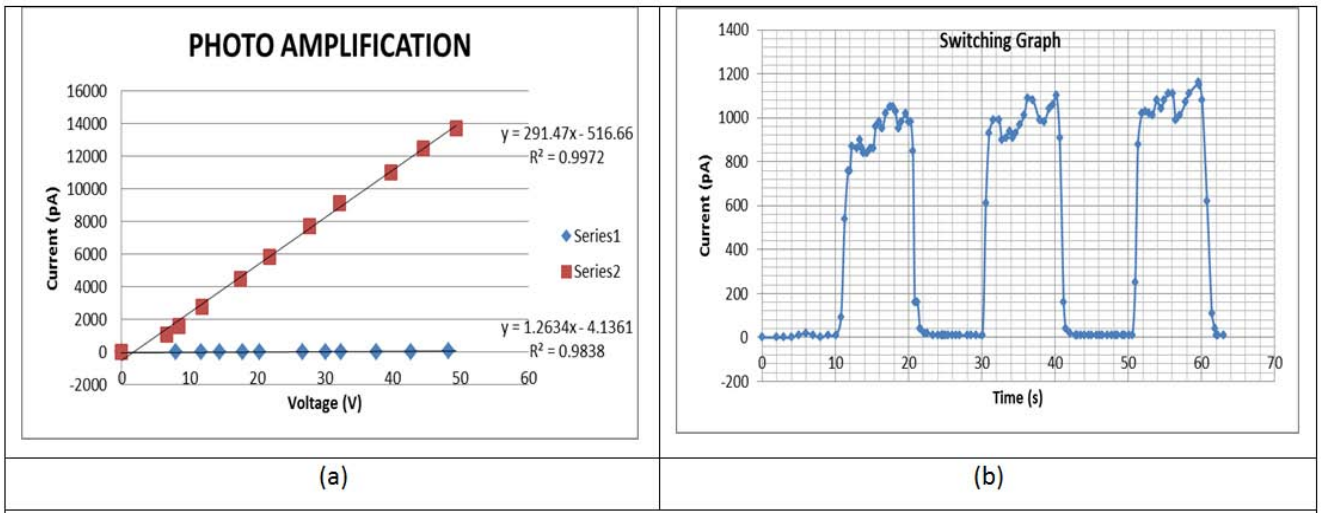


Fig. 6 : (a) Photo current (series2) is 230.7 times the dark current (Series 1) at Room Temperature (32^{0}C) (b) Photo current switching curve

IV. Conclusions

PMMA-cadmium iodide is a good X-ray sensor as photocurrent amplification is quite high (of the order of 230.7 for 19.09% concentrated composite). Although these sensors are not good for fast data switching as rise time and fall time is high. However at temperature close to 40-50^{0}C stability of photocurrent is high. Such sheets are however transparent to low energy gamma rays.

V. Acknowledgment

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