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Polycrystalline lead iodide films produced by solution evaporation and tested in the mammography X-ray energy range

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ABSTRACT

Lead iodide polycrystalline films have been deposited on corning glass substrates using solution evaporation in oven. Films 6 μm-thick were obtained with full coverage of the substrates as verified by scanning electron microscopy. Some pin-holes were observable. X-ray diffraction revealed a crystalline structure corresponding to the 4 H–PbI_2 polytype formation. Polarized Raman scattering experiments indicated a lamellar structure. Anisotropy was also investigated using depolarization ratio calculations. The optical and electrical properties of the samples were investigated using photoluminescence and dark conductivity as a function of temperature, respectively. Activation energies of 0.10 up to 0.89 eV were related to two main electrical transport mechanisms. Films were also exposed to X-ray irradiation in the mammography X-ray energy range. The detector produced was also exposed to X-ray from 5 mR up to 1450 mR. A linear response was observed as a function of dose with a slope of 0.52 nA/mm^2 per mR.
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1. Introduction

Lead iodide (PbI_2) is a compound semiconductor material with high atomic number (82 and 53 for Pb and I, respectively) and a mass density of 6.2 g/cm^3 what leads to a high photon stopping power for ionizing radiation [1, 2]. It provides lamellar structure (I–Pb–I). The forces within a lamellar structure are ionic, giving a strong binding between anion and cation layers, whereas the iodine–iodine interaction across the layer are due to weak van der Waal's interactions [3]. Periodic stacking order alternation in the direction of the *c*-axis results in the formation of polytypes in PbI_2. 2H–PbI_2 and 4H–PbI_2 are the two simplest polytypes of PbI_2 [4]. They belong to the space groups D_{3d}^3 and C_{6v}^4, respectively [5]. The Ramsdell notation is used to describe the situation presented above [6]. In 2H notation, the number 2 refers to the sequence of two layers of iodine atoms per crystal unit cell and the letter H refers to the Hexagonal Bravais lattice that results from the stacking arrangement, thus 4 H–PbI_2 refers to the sequence of four layers of iodine atoms per crystal unit cell [5,7]. The direct band gap of PbI_2 is larger than 2 eV [2,8], what should enable the material to work as radiation detector with low noise at room temperature [9]. Due to its properties, this material has been presented in the past as a promising candidate for applications in room temperature ionizing radiation detection using the direct detection method [10]. This is more interesting than the indirect detection method because a lower dose would be used to obtain the same final quality of a digital image [11]. However, until the present moment there is no large-scale commercially available equipment assembled based solely on the direct detection method using PbI_2. One of the bottlenecks is the difficulty for producing other than a-Se based high quality direct sensing materials deposited in film form, over large areas, during low deposition times and with good photo-to-dark current ratio at a reasonable price for industrial applications [12].

In fact, the film form of PbI_2 has been the subject of some works published by researchers in the whole world. Alternative deposition methods have been used for PbI_2 thin deposition as spray pyrolysis [13], close space [14], solution growth [15] and thermal evaporation [2,16] leading to different structures, thickness and electrical conductivity of the final samples. In this contribution, the solution evaporation (SE) deposition technique was used as a possible alternative method for the fabrication of PbI_2 polycrystalline films with potential application as X-ray detector device. The deposition method presents two intrinsic advantages: i) it can be easily expanded for large area substrates, as desired for flat panel medical detector systems and ii) it presents a relative low deposition time with an average deposition rate above 30 Å/s. We report the fabrication and characterization of PbI_2 films using SE as an alternative deposition technique. The structural and electrical properties and the response of the films to X-ray

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exposure in the range of mammography diagnosis are presented and discussed.

2. Materials and methods

A PbI_2 powder commercialized by Aldrich (nominal purity of 99.999%) was dissolved in N.N-dimethylformamide (DMF-C_3H_7ON) with a concentration of 150 mg ml^{-1} for preparation of the starting solution. Corning glass substrates were inserted into Petri dishes, and they were then carefully covered by the solution. Total volumes from 200 μl to 400 μl were used in each deposition. The system was inserted into a furnace at 90 °C for half hour in ambient atmosphere. DMF evaporates leaving behind a PbI_2 film. Note that a higher concentration solution of PbI_2 in DMF (462 mg ml^{-1}) was already used to insert PbI_2 into the TiO_2 nanopores as a route to high-performance perovskite-sensitized solar cells [17,18].

Thickness and surface morphology of the samples were investigated by scanning electron microscopy (SEM). X-ray diffraction experiments (XRD) were performed for crystal quality analysis. The data were recorded using a Cu k_α radiation (0.15405 nm) from a Siemens D5005 diffractometer (40 kV and 40 mA). The 2θ angle scanning was performed from 10° up to 70° with a step of 0.02°. Raman scattering experiments were performed at room temperature with illumination at 647.1 nm and analysis from 10 cm^{-1} up to 250 cm^{-1}. The laser beam radiation lies well above the absorption edge of PbI_2. Photoluminescence (PL) measurements were performed at 15 K in the range from 450 nm up to 750 nm using a HeCd laser excitation (325 nm) with slit gap of 100 μm. Dark current was recorded as a function of temperature in the range from 5 °C to 85 °C using a Hewlett Packard (4140B) pA meter/DC voltage source. Heating of the samples was carried using a Tektronix (P2511G programmable power supply) by Joule effect, while liquid nitrogen was used to cool down the sample. For electrical measurements in the dark and under X-ray exposure, detectors were assembled in coplanar configuration with graphite electrodes deposited on the surface of the samples. Graphite was chosen because it makes ohmic contact with PbI_2 [19].

The photocurrent was measured as a function of irradiation in the X-ray medical exam energy range using a General Electric Senographe 500T mammography X-ray source with a molybdenum (Mo) anode (k_α radiation of 17.5 keV) using 0.5 mm Al filtration. An equivalent X-ray energy beam of 14 keV was obtained using tube potential of 30 kVp. A Radcal 9015 dosimeter and a Radical 10 × 5–6 ionization chamber from Radcal Corporation, Monrovia, CA were used for exposure measurements. A Keithley model 610 C electrometer was used to obtain the electrical signal response. The distance between X-ray source and PbI_2 detector was kept at 45 cm.

3. Results and discussions

An optical micrograph of the surface of the resulting film is shown in Fig. 1(a). The surface presents a homogeneous distribution of dots. Note in the upper left part of the figure that a nucleation seems to occur with radial direction. Increasing the amount of solution on top of the substrates leads to a final film were these structures are even more pronounced, as can be seen at the right side of Fig. 1(b). To illustrate the effect of prolonged evaporation time, a third experiment was performed: a solution was prepared with a concentration of 40 mg ml^{-1} and the solution was inserted in the beaker and left to evaporate for 2 weeks in the dark, in a box with ambient conditions. This led to the three dimensional needles observed in Fig. 1(c).

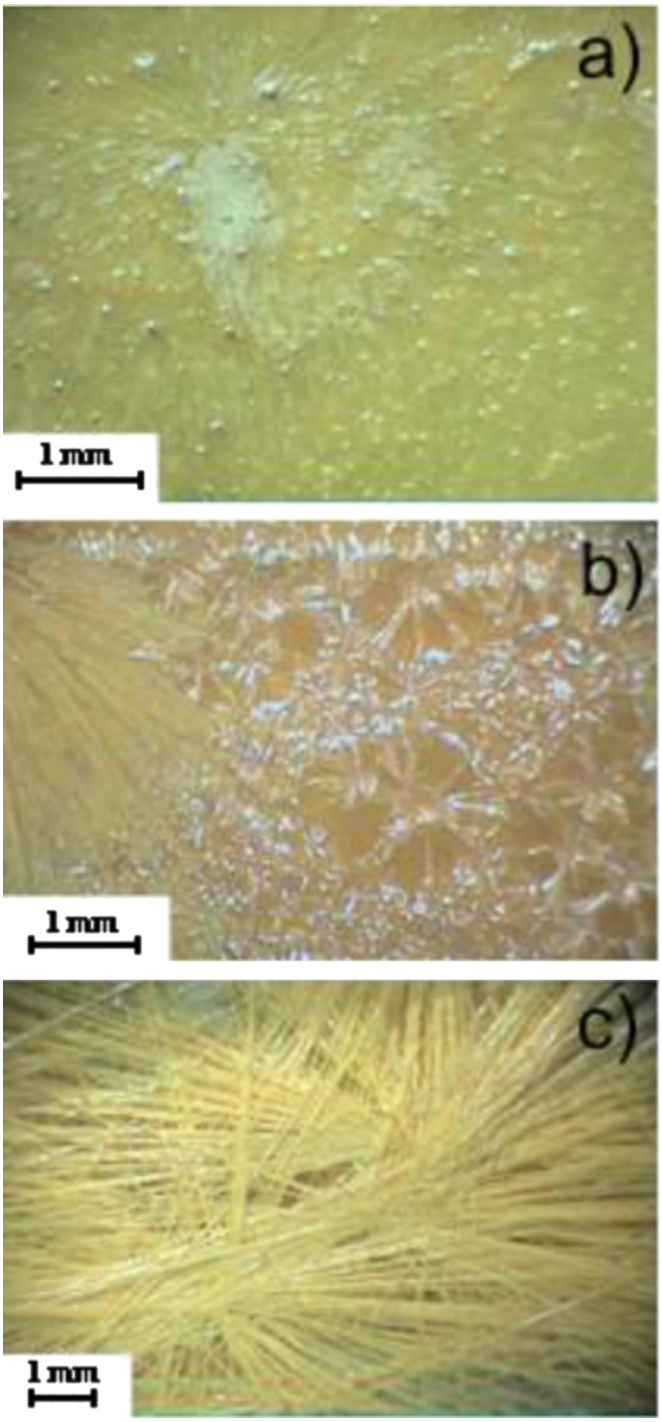

Fig. 1. Optical micrograph of final samples prepared using solvent evaporation: (a) 150 mg ml^{-1} (PbI_2 in DMF) evaporated for half hour in oven at 90 °C, total volume of 200 μl; (b) same as in (a), but a larger volume of solution of 400 μl was used and (c) 40 mg ml^{-1} (PbI_2 in DMF) evaporated in ambient conditions for 2 weeks, total volume of 50 ml.

In this paper we are mainly focused on the more homogeneous material in film form of Fig. 1(a). Nevertheless, it should be mentioned that the needle-shaped crystalline structures (as presented in Fig. 1(b) and (c)) were already widely discussed in the literature. The crystallization of an active compound from solution is used in the pharmaceutical industry for separation or purification. In this case, the ability to develop crystallization processes to avoid needle shapes is of significant interest and desired for pharmaceutical industrial lines [20]. Furthermore, biological

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