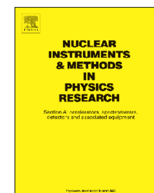
Nuclear Instruments and Methods in Physics Research A 736 (2014) 128–134

Contents lists available at ScienceDirect

Nuclear Instruments and Methods in Physics Research A

journal homepage: www.elsevier.com/locate/nima



Structural characterization of “as-deposited” cesium iodide films studied by X-ray diffraction and transmission electron microscopy techniques

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ARTICLE INFO

Article history:
Received 18 November 2012
Received in revised form
29 October 2013
Accepted 30 October 2013
Available online 11 November 2013

Keywords:
Cesium iodide
X-ray diffraction
Crystallite size
Transmission electron microscope
Grain size

ABSTRACT

In the present work, cesium iodide (CsI) thin films of different thicknesses have been prepared by thermal evaporation technique. The crystallite size and grain size of these films are compared by using X-ray diffraction (XRD) profile analysis as well as by transmission electron microscopy (TEM) counting, respectively. These two methods provide less deviation between crystallite size and grain size in the case of thin CsI films of 4 nm, but there is comparatively large difference in case of thicker CsI films (20 nm, 100 nm and 500 nm). It indicates that dislocations are arranged in a configuration which causes small orientational difference between two adjacent coherent regions. The crystallite size obtained from XRD corresponds to the size of the coherent scattering region, whereas in TEM micrograph, single grain may correspond to many such coherent scattering regions. Other physical parameters such as strain, stress and deformation energy density are also estimated precisely for the prominent XRD peaks of thicker CsI films in the range $2\theta = 20^\circ - 80^\circ$ by using a modified Williamson–Hall (W–H) analysis assuming uniform deformation model (UDM), uniform deformation stress model (UDSM) and uniform deformation energy density model (UDEDM).

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1. Introduction

Alkali halide materials are of technological importance due to their excellent electron-emitting properties in the ultraviolet (UV), vacuum ultraviolet (VUV), extreme ultraviolet (EUV) and X-ray energy ranges. These materials are currently employed in vacuum and gas-based photon detectors [1,2], detection of scintillation light [3], medical imaging [4], positron emission tomography [5] as well as a protective layer in visible-sensitive photon detectors [6]. Among alkali-halide photocathodes, CsI is the best choice, owing to its high quantum efficiency (QE) in the VUV wavelength range [7,8]. CsI films are also used to enhance the field emission (FE) sources which have potential applications including display devices [9], X-ray tubes [10], charged particle accelerators [11] and high power microwave devices [12]. Shiffler et al. [13] have reported a reduction in outgassing and improved emission uniformity after CsI coatings on carbon fibers. Even two orders of magnitude reduction in turn-on voltage were successfully achieved by means of CsI coating on carbon fiber-based FE devices by the same group [14]. Due to the importance of CsI photocathodes, several thin film preparation methods, such as thermal evaporation [15,16], ion beam sputtering [17], e-gun evaporation [18], spray pyrolysis [19], pulsed laser deposition [20], are used to study the various physical and chemical properties of CsI. However, it has been observed that the thermal evaporation is the best choice forming a stoichiometric Cs:I ratio [21] as well as the highest absolute quantum efficiency (QE) compared to other preparation techniques [17–20]. Even with its enormous applications in a variety of fields discussed above, very few of the earlier studies in this field deal with characterization of CsI film structure [22–27]. X-ray diffraction (XRD) and transmission electron microscopy (TEM) are the two important techniques which are commonly used for the structural characterization.

The XRD peak profile analysis endeavors to characterize microstructural features of the sample from the shape and breadth of Bragg's diffraction peaks, which arise due to finite crystallite size and microstrain. As broadening due to finite crystallite size and microstrain occurs together, various analytical method, such as Variance method [28], Warren–Averbach method [29] and Williamson–Hall analysis [30], have been adopted to separate both effects. Among all available methods, Williamson–Hall is a simplified approach to deconvolute strain and finite size induced broadening by plotting the total breadths of the reciprocal lattice point against their distance from origin [31]. On the contrary, Variance and Warren–Averbach methods are more complex to analyze and their application is restricted to materials having high

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http://dx.doi.org/10.1016/j.nima.2013.10.075

symmetry or which exhibit a high degree of preferred orientation. Therefore, in present paper, we emphasized on W–H method to study the variation of crystallite size with thickness of the films and to separate the strain and finite size induced broadening.

In Williamson–Hall method, broadening in Bragg's peak is assumed to be the sum of peak broadening due to finite crystallite size and induced strain. If strain is assumed to be uniform in all crystallographic directions then W–H model turns to uniform deformation model (UDM). In UDM, all the material properties are independent of the direction along which they are measured. Further, in uniform deformation stress model (UDSM) the strain is assumed to have a linear proportionality with stress according to Hook's law. UDSM is an approximation which is valid only for the small strain present in the crystal. Another model, uniform deformation energy density model (UDEDM) is used to determine the energy density of a crystal. In this approach the crystals are assumed to have a homogeneous and isotropic distribution. However, this assumption does not hold good and constants of proportionality associated with stress–strain relation are no longer independent when stress energy density is considered.

The present paper accounts for the surface characterization of as-deposited CsI thin films of different thicknesses prepared by thermal evaporation technique. The characterization of crystalline materials mainly comprises the description of grain size and internal stress or strain due to various lattice defects [32]. Usually the size obtained by XRD corresponds to the average of the smallest undistorted region in the material whereas TEM counting is related to regions separated by continuous boundaries in the TEM micrograph. To distinguish the two sizes, we will use terms such as crystallite size for XRD and grain size for TEM results. A comparative evaluation of the mean grain size of as-deposited CsI thin films obtained from direct TEM measurement, as well as the crystallite size obtained from Williamson–Hall method using XRD measurement, is studied. In addition, the strain associated with the as-deposited CsI films due to lattice deformation is estimated by a modified form of Williamson–Hall approach namely uniform deformation model (UDM). The other modified models such as UDSM and UDEDM are also used to provide an idea of the stress as well as the uniform deformation energy density.

2. Experimental details

The experimental setup for CsI consists of a high vacuum evaporation chamber which includes an oil-free Pfeiffer-made pumping unit equipped with a turbo-molecular pump having a pumping speed of 510 l/s for N_2 and a diaphragm pump. Base pressure of this vacuum chamber is of the order of 3×10^{-7} Torr. Small pieces of CsI crystal were placed in a tantalum boat inside the chamber and carefully heated to allow out-gassing from the surface of the crystal, if any, under a shutter. After proper out-gassing and melting of CsI crystals, thin films of different thicknesses were deposited on polished aluminum (Al) substrates and formvar coated copper (Cu) grids. Before deposition, typical composition of different residual gases including water vapor inside the chamber was monitored through a residual gas analyzer (SRS RGA 300 unit) as shown in Fig. 1. It has been confirmed that the amount of water vapor inside the vacuum chamber was under controlled manner. During the film deposition, the rate of evaporation was about 1–2 nm per second and the boat and substrate were kept at a distance of about 20 cm. The thickness of the film was controlled by a quartz crystal thickness monitor (Sycon STM100).

After film deposition, the vacuum chamber was purged with nitrogen (N_2) gas in order to avoid the effect of humidity on the prepared CsI samples. Immediately after the chamber opening under constant flow of N_2, as-deposited CsI thin films were extracted and placed in a vacuum desiccator. Further, CsI films deposited on formvar coated copper grid were used for TEM measurement while those deposited on Al substrate for XRD measurement.

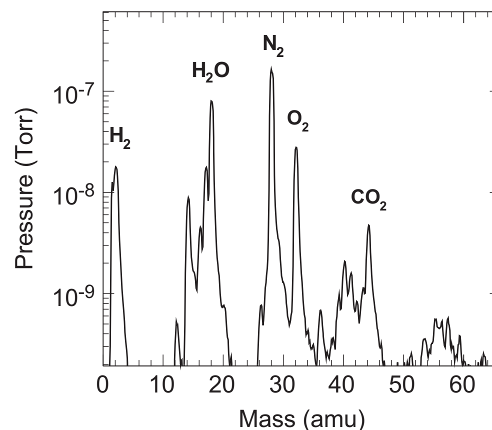

Fig. 1. Residual gas composition inside the vacuum chamber.

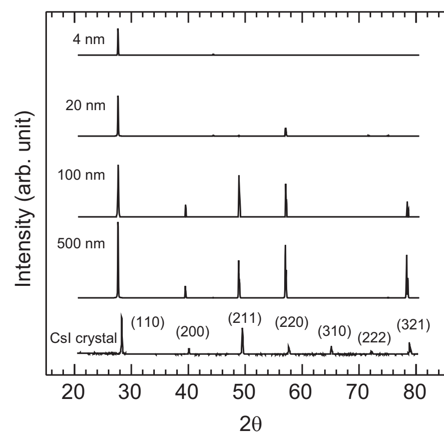

Fig. 2. X-ray diffraction pattern of CsI thin films of different thicknesses, deposited on aluminum substrate and of CsI crystal.

The structural measurements were performed by X-ray diffraction (XRD) technique in the Bragg–Brentano parafocussing geometry using PANalytical XPert PRO XRD system. The incident beam optics consists of a CuK_α radiation source ($\lambda = 1.5406$ Å) and a nickel (Ni) filter. XRD measurements have been performed in continuous scan mode in the range $2\theta = 20^\circ - 80^\circ$. The diffracted beam optics consists of a 0.04 rad solar slit and a scintillator detector. Similarly, transmission electron microscopy (TEM) measurements were done by means of FEI Tecnai 20G^2 operating at 200 KV voltage for the examination of structure and grain size of CsI films.

3. Results and discussion

3.1. Crystallite size and strain by XRD analysis

XRD patterns of cesium iodide thin films with different thicknesses prepared by thermal evaporation technique are shown in Fig. 2. No extra diffraction peaks corresponding to Cs, Cs_2O, $CsIO_3$ or other CsI phases are detected indicating that pure CsI is of polycrystalline, stoichiometric nature. Further, the XRD result of raw CsI crystal used for thermal evaporation is shown for comparison. The XRD scan exhibits a number of intense and sharp peaks

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