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# Temperature induced structural, electrical and optical changes in solution processed perovskite material: Application in photovoltaics



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## ABSTRACT

A solid-state mesoporous titanium dioxide (mTiO<sub>2</sub>) layer based hetero-junction solar cell, employing nanoparticles (NPs) of methyl ammonium lead iodide perovskite (CH<sub>3</sub>NH<sub>3</sub>PbI<sub>3</sub>) as light harvesters has been studied. The optimum performance parameters of CH<sub>3</sub>NH<sub>3</sub>PbI<sub>3</sub> are observed as a function of sintering temperature and confirmed by X-ray diffraction analysis and UV–vis spectrophotometry. A solid-state solar cell with the sandwich structure of mTiO<sub>2</sub>/CH<sub>3</sub>NH<sub>3</sub>PbI<sub>3</sub>/Graphite paste showed a power conversion efficiency of 1.11% tested under standard Air Mass 1.5 Global (1000 W m<sup>-2</sup>, AM1.5G) solar spectrum. Two-diode model is used to explore the performance limiting factors of the developed solid-state perovskite solar cell.

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## 1. Introduction

In the last decade, solar energy has emerged as a potential alternative to the conventional fossil fuel based energy sources. The direct conversion of solar radiation into electrical energy is the most suitable way of utilizing solar energy because of its convenience and diverse applications. Worldwide, silicon based solar photovoltaic cells are efficiently deployed for converting solar energy into electricity [1]. The price of electricity generated from silicon solar cell is quite high and there is an urgent need to reduce the cost of solar cell [2]. In the last decade, dye-sensitized solar cells (DSSCs) invented by Grätzel and O'regan [3] based on nano-crystalline mesoporous titanium dioxide (mTiO<sub>2</sub>) films have attracted much attention as a potential low-cost alternative to single or polycrystalline silicon solar cells. DSSC has reached a conversion efficiency of 13% [4], but also faced challenges in sealing of electrolyte and its stability in outdoor conditions [5]. In order to meet the above challenges, many research groups are working towards the development of a low cost solid state methyl ammonium lead iodide (CH<sub>3</sub>NH<sub>3</sub>PbI<sub>3</sub>) perovskite based solar cells [6–15]. Presently, these solar cells have reached a conversion efficiency of 16% [16] and establishing itself as a cheap alternative to conventional silicon solar cells due to its tunable band gap, high absorption coefficient, high

carrier mobility and good stability [17–19]. In these types of solar cells, perovskite material is coated on a mesoscopic layer of wide band gap oxide semiconductor to absorb the incident photons [6–15,20]. The incident photons excite these molecules to an energy level higher than the conduction band edge of TiO<sub>2</sub> to inject electrons and holes in the respective charge carrier transport materials. These electrons and holes collected at the front and the back electrode respectively constitute the photo-current of the perovskite solar cells [7,8]. The fabrication of perovskite solar cell with liquid electrolyte (iodide/tri-iodide redox couple) is reported by Im et al. [21] with a conversion efficiency of 6.54%, but the performance of these systems degraded rapidly due to the dissolution of perovskite in liquid electrolyte. Laban and Etgar have reported efficiency of 8.4% using solution processed method having Au as a back contact material without the hole transporter [14]. A maximum power conversion efficiency of 10.85% has been achieved without hole transport material by Aharon et al. [22]. In order to avoid the solubility problem and to improve the hole collection ability of solar cells, various solid state hole transport materials i.e. spiro-MeOTAD, P3HT, PTAA etc. [8,23,24] have been explored and bestowed a maximum conversion efficiency of 16% [16]. However, with so much focus on the cell structure, there are only few reports where the detailed crystal structure and its optical properties are discussed [25,26]. In this report an attempt has been made to understand the material aspects of light harvesting CH<sub>3</sub>NH<sub>3</sub>PbI<sub>3</sub> perovskite material and its application in solid state mTiO<sub>2</sub>. Further, two diode model is used to explore the device limiting factors.

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## 2. Material synthesis and characterization

### 2.1. Material synthesis

The perovskite sensitizer  $\text{CH}_3\text{NH}_3\text{PbI}_3$  was prepared according to the reported procedure [6]. A hydroiodic acid (30 ml, 0.227 mol, 57 wt% in water, Aldrich) and methylamine (27.8 ml, 0.273 mol, 40% in methanol, TCI) were stirred for 2 h at 0 °C and vapour evaporated at 50 °C for 1 h to produce  $\text{CH}_3\text{NH}_3\text{I}$ . The precipitate was washed three times with diethyl ether and dried under vacuum and used without further purification as per the process described in Ref. [21].  $\text{CH}_3\text{NH}_3\text{PbI}_3$  is prepared by mixing and stirring of readily synthesized  $\text{CH}_3\text{NH}_3\text{I}$  (0.395 g) and  $\text{PbI}_2$  (1.157 g, 99% Aldrich) in  $\gamma$ -butyrolactone (2 ml, 99% Aldrich) mixture at 60 °C for 24 h. The mesoporous  $\text{TiO}_2$  paste was prepared using the method reported elsewhere [27]. Nanocrystalline  $\text{TiO}_2$  powder ( $\approx 25$  nm, Sigma Aldrich) (5 g) and 2 ml acetic acid was ground in a mortar for 10 min. Subsequently, 2 ml water and 3 ml ethanol were added and ground for 2 min and the process is repeated 10 times. Then the paste was stirred for 2 min, followed by ultrasonication then 25 g terpeneol was added and stirred for 30 min. Then the paste was stirred and ultra-sonicated. Ethyl cellulose (3 g/30 g of 10% solution in ethanol) was added to the paste with continuous stirring and ultra-sonicated for half an hour. Then the remaining ethanol was evaporated with a rotary evaporator. The paste was ball milled for 6 h to give uniform and smooth texture.

The FTO substrates ( $7 \Omega \text{ cm}^{-2}$ ) were immersed in 40 mM  $\text{TiCl}_4$  aqueous solution at 70 °C for 30 min and washed with water and ethanol. Then they were annealed at 500 °C for 30 min to form a dense blocking layer. The mesoporous  $\text{TiO}_2$  paste was then spin coated at 3000 rpm for 30 s and annealed at 500 °C for 30 min. The samples were again immersed in 40 mM  $\text{TiCl}_4$  aqueous solution at 70 °C for 30 min and washed with water and ethanol followed by annealing at 500 °C for 30 min. The prepared  $\text{CH}_3\text{NH}_3\text{PbI}_3$  solution was then spin coated at 2000 rpm for 30 s and sintered at different temperatures under ambient atmospheric conditions. This procedure was repeated for three times to achieve a sufficient thickness of perovskite layer on  $\text{mTiO}_2$  layer. Commercially available graphite paste (Ted Pella) was used on perovskite sensitized  $\text{mTiO}_2$  layer before taking a back contact with platinum (Pt).

### 2.2. Material characterization

The X-ray diffraction studies for structural analysis of  $\text{TiO}_2$  and perovskite has been done by X'Pert Pro diffractometer in the  $2\theta$  range of 5–70°. On the  $\text{mTiO}_2$  coated FTO substrates the perovskite solution was spin-coated at 2000 rpm for 30 s followed by heat treatment at different temperatures in the range of 70 °C to 130 °C for 15 min. The influence of the sintering temperature on the morphology and structure of the perovskite layer coated on the  $\text{mTiO}_2$  were examined by field emission scanning electron microscopy (FE-SEM). The optical properties of perovskite material were studied by Shimadzu UV-visible spectrometry (UV 2600). The perovskite solar cell was fabricated by applying graphite paste on perovskite layer. The arrangement of  $\text{mTiO}_2/\text{CH}_3\text{NH}_3\text{PbI}_3/\text{graphite}$  paste was heated at 77 °C for 10 min for curing graphite paste. Platinum foil is used to take back contact from the graphite layer. The devices have been tested under standard Air Mass 1.5 Global ( $1000 \text{ W m}^{-2}$ , AM1.5G) solar spectrum at 25 °C for photocurrent and voltage measurements by a solar simulator (Photoemission Inc., USA, Class AAA) and an Agilent 2772 source meter. The active area of the device was  $0.3 \times 0.3 \text{ cm}^2$ . The stability tests were conducted according to ISOS-D-1 protocol [28]. To determine the relative stability of fabricated solar cell, the solar cell was tested under ambient relative humidity ( $\approx 50\%$ ) conditions.

## 3. Results and discussion

### 3.1. FE-SEM analysis

Fig. 1(a)–(e) shows the FE-SEM image of the perovskite samples sintered at different temperatures and  $\text{mTiO}_2$  film. The samples annealed at 70 °C showed a non-uniform film morphology with large islands of precursor material ( $\text{PbI}_2$  and  $\text{CH}_3\text{NH}_3\text{I}$ ) (Fig. 1(a)). The FE-SEM images of the perovskite sintered at various temperatures (Fig. 1(a)–(e)) revealed that, the crystallization of the perovskite crystals at 70 °C have not taken place, indicating the presence of remnant precursors. As the sintering temperature is increased to 85 °C there is a noticeable change in the appearance of the film, which shows that the crystallization has started in the perovskite layer (Fig. 1(b)) with diminishing traces of precursors. The film formed upon sintering at 100 °C is quite similar to that of 85 °C (Fig. 1(c)) but a better uniformity and crystalline nature is observed in the film. Also the perovskite crystal interconnection density has improved. After increasing the sintering temperature to 115 °C, the crack formation was observed in the perovskite crystals, which indicates the beginning of the dissociation of the  $\text{CH}_3\text{NH}_3\text{PbI}_3$  perovskite crystals (Fig. 1(d)) [29]. With further increase in the sintering temperature to 130 °C, the densely interconnected network has disappeared and the larger part of  $\text{mTiO}_2$  remains uncovered. The high temperature leaves behind a thin layer of perovskite and this structure is almost similar to that of 70 °C (Fig. 1(e)).

### 3.2. X-ray diffractometer analysis

The obtained XRD pattern was analysed using High Score Plus software. The XRD pattern was used to study the effect of perovskite processing temperature on its lattice parameters. Fig. 2 shows the XRD-spectra of  $\text{mTiO}_2$  layer and perovskite on  $\text{mTiO}_2/\text{FTO}$  sintered at various temperatures (temperature mentioned correspond to the pattern from bottom to top respectively). The instrumental broadening error was corrected, the background was removed and the noise level was minimized before the analysis. The diffraction peaks were observed at  $2\theta$  values of 13.77, 15.21, 29.61, 33.03, 41.59 and 44.23 correspond to the reflection planes of (0 0 2), (1 1 0), (2 2 0), (3 1 0), (2 2 4) and (3 1 4) respectively of the tetragonal perovskite structure. A comparison of XRD patterns of the perovskite materials on  $\text{mTiO}_2/\text{FTO}$  sintered at different temperatures shows that the samples sintered at 70 °C indicates the presence of peaks corresponding to the precursors ( $\text{PbI}_2$  and  $\text{CH}_3\text{NH}_3\text{PbI}_3$ ) and not sufficient to crystallize the precursors into desired perovskite materials, which is observed in Fig. 1(a) of the FE-SEM image. With further increase in the sintering temperature to 85 °C, the peaks corresponding to precursors disappeared and perovskite ( $\text{CH}_3\text{NH}_3\text{PbI}_3$ ) peaks appear in accordance with the literature [14]. This is also confirmed by the presence of grains in FE-SEM image of perovskite materials (Fig. 1(b)). The XRD pattern for the samples sintered at 100 °C shows an increase in the intensity of the (0 0 2) plane as compared to the 85 °C sample (Fig. 2), indicating the formation of large perovskite crystallites and confirmed by FE-SEM image (Fig. 1(c)). In Fig. 2, XRD pattern of the sample sintered at 115 °C shows again a decrease in the intensity of (0 0 2) peak indicating the beginning of dissociation of  $\text{CH}_3\text{NH}_3\text{PbI}_3$  into its precursors and can be correlated with the appearance of cracks in FE-SEM images of the sample (Fig. 1(d)). The dissociation may be attributed to the temperature dependent high mobility of  $\text{CH}_3\text{NH}_3^+$  cation, which cannot be fixed in crystal structure due to the disordered orientation [30]. The intensity of (0 0 2) peak ( $\text{PbI}_2$ ) shows that dissociation of  $\text{CH}_3\text{NH}_3\text{PbI}_3$  into its constituent precursors. The complete dissociation of  $\text{CH}_3\text{NH}_3\text{PbI}_3$  is also reported by other researchers at

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