Air Processed Cs₂AgBiBr₆ Lead-free Double Perovskite High-mobility Thin Film Field-effect Transistors

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Abstract

This study focuses on the fabrication and characterization of $\text{Cs}_2\text{AgBiBr}_6$ double perovskite thin film for field-effect transistor (FET) applications. The $\text{Cs}_2\text{AgBiBr}_6$ thin films were fabricated using a solution process technique and the observed XRD patterns demonstrate no diffraction peaks of secondary phases, which confirms the phase-pure crystalline nature. The average grain sizes of the spin-deposited film were also calculated by analysing the statistics of grain size in SEM image and was found to be around 412 (± 44) nm the larger grain size was also confirmed by the XRD measurements. FETs with different channel lengths of $\text{Cs}_2\text{AgBiBr}_6$ thin films were fabricated on an electrode deposited heavily doped p-type Si substrate with a 300 nm thermally grown SiO_2 dielectric under ideal conditions in air processing under ambient pressure and temperature. The $\text{Cs}_2\text{AgBiBr}_6$ FETs showed a p-type nature with a positive threshold voltage. The on current, threshold voltage and hole-mobility of the FETs decreased with increasing channel length. A high average hole mobility of $0.29 \text{ cm}^2\text{s}^{-1}\text{V}^{-1}$ was obtained for the FETs with a channel length of 30 μm , and the hole mobility was reduced by an order of magnitude ($0.012 \text{ cm}^2\text{s}^{-1}\text{V}^{-1}$) when the channel length was doubled. The on current and hole-mobility of $\text{Cs}_2\text{AgBiBr}_6$ FETs followed a power fit, which confirmed the dominance of channel length in electrostatic gating in $\text{Cs}_2\text{AgBiBr}_6$ FETs. A very high-hole mobility observed in or FET that could be attributed to the much larger grain size of $\text{Cs}_2\text{AgBiBr}_6$ film made in this work.

Introduction

In recent years, Perovskite thin film field effect transistors (FETs) have been extensively studied to perform many applications such as photo FET,¹ light emitting FET,² photo detectors³ and ferroelectric RAM⁴. Despite of the fact that a huge number of Perovskite materials were studied for solar cell applications, only quite a few numbers of Perovskite materials have been studied for FET repeatedly. Hybrid organic-inorganic halide Perovskites like methyl ammonium lead iodide (MAPbI_3) have been the most dominant semiconducting channel materials in the studies of Perovskite thin film FETs.⁵ Double inorganic double perovskite material, $\text{Cs}_2\text{AgBiBr}_6$ is considered to be one of the promising alternative non-toxic and highly stable materials in the family of perovskites exhibiting a long charge carrier lifetime, high charge carrier mobility in single crystals, and effective charge carrier masses comparable to those of lead-based perovskite semiconductors.⁶⁻⁸ Recent theoretical investigations suggest that the $\text{Cs}_2\text{AgBiBr}_6$ material could perform much better than the reported works for PV and photocatalytic applications.⁹⁻¹¹ $\text{Cs}_2\text{AgBiBr}_6$ displays a superior stability towards ambient environments and has an indirect band gap between 1.9 and 2.3 eV. Despite the eco-friendliness and long-term stability $\text{Cs}_2\text{AgBiBr}_6$ possess, the large indirect bandgap and poor light absorption ability of the material have restricted their applicability in solar cell applications. The highest reported efficiency of $\text{Cs}_2\text{AgBiBr}_6$ based solar cells to date is 4.3%, whereas the highest efficiency reported for Methyl Ammonium Lead Iodide based solar cell is around 25.8%.^{12,13} Nevertheless, despite the high trap densities in $\text{Cs}_2\text{AgBiBr}_6$, due to their long charge-carrier lifetimes (larger than a microsecond) owing to the shallow nature of the majority of traps, diffusion

lengths exceeding 1 μm and long-term environmental stability than organic-inorganic lead-halide perovskites, make it a promising material for a wide range of applications such as light-emitting diodes (LEDs), radiation detectors, photodetectors, photocatalysts, sensors and in neuromorphic computing.^{14–17}

This study focuses on investigating the structural phase purity and morphological formation of solution processed $\text{Cs}_2\text{AgBiBr}_6$ based lead-free double perovskite thin films. Although double Perovskite of $\text{Cs}_2\text{AgBiBr}_6$ has been intensively studied for solar cells application,¹⁸ it has not been explored in FET application other than the recent study by Li Z et al.¹⁹ As the charge transport in a Perovskite thin film channel is studied based on the existence of grains and grain boundaries of interlayers^{14,19}, very recently, Li Z et al., carried out research to study the role of grain boundaries on charge carrier and ion transport in $\text{Cs}_2\text{AgBiBr}_6$ Perovskite thin films through FET.¹⁹ By tailoring the grain size within the range of 70 nm to 110 nm, they studied the role of the grain boundaries in ion transportation with a support of cathodoluminescence imaging. In this work, we employ $\text{Cs}_2\text{AgBiBr}_6$ material with larger grain size as semiconducting channel in a FET. We report the first $\text{Cs}_2\text{AgBiBr}_6$ FET ever fabricated in air processing under ambient conditions. The gating mechanism of the fabricated $\text{Cs}_2\text{AgBiBr}_6$ FETs is discussed by analysing the electronic properties of FETs by varying channel lengths. The $\text{Cs}_2\text{AgBiBr}_6$ film was fabricated with a grain size of 400 nm and its electronic properties were evaluated. As the Perovskite FETs are being studied to evaluate the mobility of the materials^{20,21}, the hole mobility of the fabricated $\text{Cs}_2\text{AgBiBr}_6$ was calculated in this work. The average hole mobility of $\text{Cs}_2\text{AgBiBr}_6$ channel in this work is found to be remarkably high value of $0.29 \text{ cm}^2\text{s}^{-1}\text{V}^{-1}$ while the one and only previously reported all inorganic $\text{Cs}_2\text{AgBiBr}_6$ was in the order of $10^{-3} \text{ cm}^2\text{s}^{-1}\text{V}^{-1}$. We conclude that the enhanced mobility could be attributed to the larger grain size and high-quality thin film by recrystallization of $\text{Cs}_2\text{AgBiBr}_6$ crystals during the thin film deposition.

Results And Discussions

The $\text{Cs}_2\text{AgBiBr}_6$ thin film was fabricated via a two-step route. Firstly, the $\text{Cs}_2\text{AgBiBr}_6$ crystals were grown as described in the methods section (the grown crystals are illustrated in Fig. 1 (a)) and then the precursor for spin coating was prepared by dissolving the crystals in DMSO. Then the thin films were fabricated by spin coating and subsequently calcinated at 280C for 10 min (Fig. 1 (b)). The $\text{Cs}_2\text{AgBiBr}_6$ FET devices with bottom gate bottom contact (BGBC) device structure was fabricated as shown in figure 1 (c). The BGBC structure was used in our study as it has shown better device performance and one of the most studied device structure.^{14,19,30}

Prior to device fabrication, XRD measurements was carried out to confirm the structural and phase purity of the spin-coat deposited $\text{Cs}_2\text{AgBiBr}_6$ double perovskite thin film and the obtained result has been depicted in Fig. 2 (a). As to avoid two major unwanted intermediate phases of $\text{Cs}_3\text{Bi}_2\text{Br}_9$ (reflection at 12.8° and 30.9°) and AgBr compounds (reflection at 44.2°), the film was annealed at 280°C for 5 minutes

which was found to be the best condition (285°C) for the formation of phase-pure double perovskite structure during the film formation.²⁶ From the observed XRD results, all the obtained major peaks located at 13.63°, 15.73°, 22.34°, 27.41°, 31.77°, 35.56°, 39.23°, 45.56°, and 56.53° attribute to the reflections of Cs₂AgBiBr₆ having the plane values of (002), (200), (220), (222), (400), (331), (224), (044), and (444) where all the peaks correlate with the standard JCPDS (File number: 01-084-8699) data and are also in good agreement with the previous reports on double perovskite Cs₂AgBiBr₆ materials.^{20,21,27} There were no specific reflections of secondary residual reflections of Cs₃Bi₂Br₉ or AgBr confirms the phase-pure crystalline nature for the prepared Cs₂AgBiBr₆ thin film as depicted in the inset of Fig. 2 (a). In addition, the average crystallite size for the prepared Cs₂AgBiBr₆ sample was calculated using Debye-Scherrer formula from the predominating peak signal found at $2\theta = 31.77^\circ$ related to the (400) plane. The observed full width at half maximum (FWHM) of the spin-coated Cs₂AgBiBr₆ film found to be very lower for the applied higher annealing temperature, and the grain size was measured as 434 nm. We also calculated the grain size using SEM image. Fig. 2 (b) depicts the polygonal grain structured surface morphological appearance for the spin-deposited Cs₂AgBiBr₆ double perovskite thin film observed from the SEM analysis. The average grain size of the film was also calculated by analysing the statistics of grain size in SEM image using Imagej software. Accordingly, the calculated result benchmarks the average grain size from the surface morphology was found to be 412 (± 44) nm which is in agreement with the larger grain size calculated from the XRD spectra. The grain size is much less than the shortest channel length (30 μm) of the FET we tested. Moreover, the significant larger grain size achieved up to micro-size (average ~ 412 nm) could be majorly due to the structural reconstruction of Cs₂AgBiBr₆ grains by the aforementioned higher annealing temperature ($\sim 285^\circ\text{C}$) process.²⁸ Remarkably, the achieved uniform and larger grain size all over the surface of the thin films with reduced grain boundaries found to be the key factor in determining efficient charge-carrier and ion transport in various semiconducting device applications^{19,28,29}.

The transfer characteristics of the fabricated Cs₂AgBiBr₆ thin film FETs with channel lengths of 30, 40, 50 and 60 μm were studied within the gate voltage (V_{gs}) range of -20 V to 40 V under the applied constant source drain (V_{ds}) voltage of -40 V. The transfer curves with logarithmic and linear current axis are shown in Fig. 3 (a) and (b) respectively. From the transfer curves we analysed the electronic properties of the Cs₂AgBiBr₆ material and the role of channel length in gating. The change in on current, threshold voltage and hole mobility were used for our discussion in this work. The maximum observed current from the transfer characteristics curve in the Fig. 3 (a) is identified as the on current of the Cs₂AgBiBr₆ FET devices. The minimum necessary gate voltage to turn on the Cs₂AgBiBr₆ FET device is the threshold voltage V_{th} . The intercept of the extension line drawn in the linear region of I_{ds} vs V_{gs} curve and V_{gs} axis in the transfer characteristics curve in Fig. 3 (b) gives the threshold voltage of Cs₂AgBiBr₆ FET. The hole mobility (μ_{h}) values were calculated using the following equation,^{19,31}

$$\mu_h = \frac{2L}{wC} \left(\frac{d(\sqrt{I_{ds}})}{dV_{gs}} \right)^2$$

where L is the channel length (30 to 60 μm), w is channel width (2 mm), C is the effective capacitance of the dielectric material which is 300 nm thickness of SiO_2 ($7.23 \times 10^{-9} \text{ F cm}^{-2}$) and

$\frac{d(\sqrt{I_{ds}})}{dV_{gs}}$ is the slope of the $\sqrt{I_{ds}}$ vs V_{gs}

graph in the linear region

The variations of on current, threshold voltage and hole mobility of $\text{Cs}_2\text{AgBiBr}_6$ FET along with the channel lengths of 30 μm , 40 μm , 50 μm and 60 μm are shown in Fig. 4. The variation of on current values for the $\text{Cs}_2\text{AgBiBr}_6$ FETs with varying channel lengths are shown in Fig. 4 (a). For 30 μm long $\text{Cs}_2\text{AgBiBr}_6$ channel FET the on current is about 5 μA and it logarithmically decreases with the increasing channel length. For 60 μm the average on current reaches the value of 0.5 μA which is an order of magnitude lower than the on current through the 30 μm channel. The values of the threshold voltages of the $\text{Cs}_2\text{AgBiBr}_6$ FETs with different channel lengths are shown in Fig. 4 (b). There is a clear indication that the threshold voltages decrease as the channel lengths increase from 30 μm to 60 μm . Likewise, in on current and threshold voltage, the hole-mobility of the $\text{Cs}_2\text{AgBiBr}_6$ channel also reduces with the increment of the $\text{Cs}_2\text{AgBiBr}_6$ channel lengths as well (Fig. 4(c)). A high hole mobility of $0.29 \text{ cm}^2\text{s}^{-1}\text{V}^{-1}$ was obtained for the FETs with channel length of 30 μm , and the hole mobility reduced by an order of magnitude ($0.012 \text{ cm}^2\text{s}^{-1}\text{V}^{-1}$) when the channel length increased to 60 μm . High on current in the shortest channel length FET can be explained by the shortest resistive path that the charge carriers had to travel through. Decreasing on current with increasing channel length is an indication of increasing channel resistance with channel length. When the channel length is doubled (30 to 60 μm), the on current decreased by an order of magnitude (Figure 4(a)).

Discussion

To study the role of channel length in electrical conduction and gating, we fitted the on current and mobility vs channel length curves with Origin 8.5 software. The on current (I_{on}) vs channel length (L) curve was found to follow a power relationship of $I_{on} = aL^b$ with an R^2 value greater than 0.97. The fitting equation shows that the value of 'b' is -2.975 (± 0.27) indicates that the on current is proportional to the third power of the channel length L. Meanwhile, the hole mobility of the FETs also showed a power fit (with an R^2 value of greater than 0.94) with a 'b' value of -4.59 (± 0.42) indicates that the mobility is nearly proportional to the fifth power of the channel length (Fig. 4 (c)). The power fit of both on current and mobility confirms the strong dependence of electrical conductance and hole mobility in the channel

length. Changing the channel length to study the charge transport of $(\text{C}_6\text{H}_5\text{C}_2\text{H}_4\text{NH}_3)_2\text{SnI}_4$ perovskite channel was previously done by Matsushima et al.³² They used a sequence of channel lengths varying from 45 to 750 nm to study the intrinsic charge transport properties of $(\text{C}_6\text{H}_5\text{C}_2\text{H}_4\text{NH}_3)_2\text{SnI}_4$ perovskites. They observed hole and electron mobilities to increase with increasing channel length and to saturate after a particular channel length is reached. The increased mobility with increasing channel length is an indication of scatter free conduction and gating in the material.³² However, as per our observation, in the $\text{Cs}_2\text{AgBiBr}_6$ perovskite FETs, the mobility decreased with increasing channel length. Hence, it is confirmed that the gating of these $\text{Cs}_2\text{AgBiBr}_6$ FETs is not dominated by the field modulated charge carriers in the channel. The strong dependence of on current in the channel length also confirms that the gating or conduction does not depend on the Schottky junctions formed at the electrode-channel junction.

It should be noted that the electrical conductance in the $\text{Cs}_2\text{AgBiBr}_6$ thin film is dominated by the grain boundaries.¹⁹ From the power fits of on current and hole mobility, and high gating in the longest channel FETs, one can confirm that the role of grain boundaries in the charge transfer and gating in $\text{Cs}_2\text{AgBiBr}_6$ FETs. It is noted that the threshold voltage of the $\text{Cs}_2\text{AgBiBr}_6$ FETs linearly decreased with increasing channel length and always was positive within the channel length range of 30 to 60 μm . Positive threshold voltage also confirms that the majority charge carrier of the $\text{Cs}_2\text{AgBiBr}_6$ FET is holes when the gate bias is not applied.^{24,25,33,34} The negative shift in threshold voltage with increasing channel length is a significance of reduced effective hole concentration with increasing channel length which is opposite to the observations by Matsushima et al on $(\text{C}_6\text{H}_5\text{C}_2\text{H}_4\text{NH}_3)_2\text{SnI}_4$ perovskite FETs.³² This also shows that the electron and hole transport can be hindered by the grain boundaries during the transport. It is also noted that the grain boundary is an important contributor to the recombination losses.¹⁷ Hence, it can be concluded that the grain boundaries are creating blockades during the charge transfer through the channel. The longer the channel, more grain boundaries have to be tunnelled and hence the conductivity gets heavily decreased. Moreover, the applied gate voltage hugely influences the barrier height at the grain boundaries and hence the charge carrier tunnelling across the grain boundaries. Hence, we can conclude that the grain boundaries are the conduction and gating hotspots in the $\text{Cs}_2\text{AgBiBr}_6$ FETs.

This is an initial state of $\text{Cs}_2\text{AgBiBr}_6$ FET studies and it is important to have a collation with the one and only published report on $\text{Cs}_2\text{AgBiBr}_6$ FET. The characteristics of the device made in this study is compared with the previous work on $\text{Cs}_2\text{AgBiBr}_6$ FET in Table 1.¹⁹ The hole mobility of the $\text{Cs}_2\text{AgBiBr}_6$ channel is low as $1.5 \times 10^3 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ in the that study. In our FETs, we found a very high average hole mobility of $0.29 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ which is two order of magnitude higher than the previously reported work. This could be attributed to the much larger grain size of our $\text{Cs}_2\text{AgBiBr}_6$ film, which is about four times bigger than the previous work. As reported in few solar cell works, the film quality and grain size also play a vital role in the power conversion efficiencies.⁷ Though we used the air processing method to fabricate the device, the basic properties like majority carrier and ground state conduction characteristics of our device

do not differ notably from other study, where N₂ environment was used in fabrication with a channel length of 20 μm and width of 1 mm.

Table 01
Comparison of figure of merits of our Cs₂AgBiBr₆ FET with the existing literature

	In the Literature ¹⁹	In our work
Device structure	BGBC	BGBC
Fabrication environment	N ₂	Air
Channel dimension	20 μm × 1 mm	30 μm × 2 mm
Grain size	~110 nm	~412 nm
Hole mobility (at 300 K)	15 ×10 ⁻⁴ cm ² V ⁻¹ s ⁻¹	0.29 cm ² V ⁻¹ s ⁻¹
V _{ds}	-60 V	-40 V
ON current	5.5 ×10 ⁻⁷ A	4.12×10 ⁻⁶ A

Conclusions

A FET with a lead free Cs₂AgBiBr₆ perovskite thin film channel was successfully demonstrated. The Cs₂AgBiBr₆ thin film fabricated by recrystallizing the pre-prepared Cs₂AgBiBr₆ solution followed by annealing at 285°C enhanced the grain growth and resulted with a maximum grain size of 412 (±44) nm. The channel length of the FET strongly influenced the on current, threshold voltage and hole mobility of the FETs. All three parameters were reduced with increasing channel length. The strong dependency of on current and mobility on the channel length confirmed the dominance of grain boundaries in the electrostatic gating in Cs₂AgBiBr₆ thin film FETs. The lower mobility and on-current in the longer channel FET can be attributed to the increased charge scattering at the effectively larger number of grain boundaries of the longest Cs₂AgBiBr₆ thin film. The maximum mobility of 0.29 (±00.07) cm²V⁻¹s⁻¹ observed in the FETs with 30 μm channel was two orders of magnitude higher than the previously reported hole mobility of the same material due to the much larger grain size.

Experimental Methods

Synthesis of Cs₂AgBiBr₆

The Cs₂AgBiBr₆ double perovskite crystals were synthesized through the solution-based process as reported elsewhere^{22,23}. The detailed process are as follows: 2 mmol of CsBr (426 mg, 99.5%, Alfa Aesar), 1mmol of AgBr (186 mg, 99.9%, Alfa Aesar) and 1 mmol of BiBr₃ (446 mg, 99.99%, Alfa Aesar)

metal salts were sequentially added and dissolved in 9 ml of hydrobromic acid (HBr, 48%, Merck) under magnetic stirring. Then, the as-prepared solution, moved to round bottom flask and placed in a silicon oil bath under reflux reaction. The silicon oil gradually heated up to 120 ° and held for 2 hr with gentle stirring of the solution. Thereafter, the solution smoothly cooled down to room temperature with the rate of 5°/h. Finally, the well-grown (as shown in Fig. 1 (a)) Cs₂AgBiBr₆ double perovskite crystals were collected by filtering and washed three times with absolute ethanol and dried overnight in vacuum oven. For further using, these crystals could be grounded to obtain the powder form using pestle and mortar.

Fabrication of Cs₂AgBiBr₆ FET

The Cs₂AgBiBr₆ FETs were fabricated on a 300 nm SiO₂ grown heavily doped Si (SiO₂/Si) substrate (Ossila Ltd, United Kingdom). The source and drain electrodes were deposited on top of the SiO₂ dielectric layer by using successive thermal evaporation of 5 nm Chrome and 50 nm gold, with the channel lengths of 30 μm, 40 μm, 50 μm and 60 μm using Edwards E306 thermal evaporator under high vacuum of 10⁻⁵ mTorr.^{24,25} The synthesized Cs₂AgBiBr₆ crystals were dissolved in 250 μl of dimethyl sulfoxide (DMSO) (100ml, anhydrous, ≥99.9%). The precursor solution was magnetically stirred on a hotplate at 70°C for 60 min to get the homogenous precursor solution. The electrode deposited SiO₂/Si substrate was cleaned by two sonication processes in acetone and 2-propanol (isopropyl alcohol) for 20 minutes each round and dried with nitrogen flow. 50 μl of Cs₂AgBiBr₆ precursor solution was spin coated on the substrate at 1500 rpm for 60 seconds. After spinning, the material on the electrodes were carefully removed by cotton buds and followed by subsequent annealing at 250°C for 5 minutes for the formation of phase pure double perovskite crystal structure.

Cs₂AgBiBr₆ material characterization

The deposited thin-film X-ray diffraction data for Cs₂AgBiBr₆ were recorded using a Bruker D8 XRD machine. To avoid the crystal interference of the highly crystalline Si substrate, we used a soda lime glass with 1 mm thickness as the substrate for XRD measurements (Fig. 1(b)). SEM images for the film deposited on the SiO₂/Si substrate were obtained using a Carl Zeiss instrument with an acceleration voltage of 2 kV for the top-view images and for studying the elemental composition using EDX spectrum (20 kV). All of the samples were pre-sputtered with carbon, and mirror and through-the-lens detectors were employed.

Cs₂AgBiBr₆ FET characterization

The Cs₂AgBiBr₆ thin film FETs with different channel lengths were electrically characterized by using the computer interfaced Keithley 2602 under pulse mode measurement. The circuit connections along with the schematic of the FET are illustrated in Fig. 1 (c). The electrical contacts with the devices were made by using gold wirers and a portable probe station with spring-loaded contacts. All the electrical measurements were measured under ambient conditions. The transfer curves were measured by sweeping the gate voltage from -20 V to 40 V by keeping the V_{ds} as constant at -40 V.

Declarations

Author Contribution

G.A, F.H.G & T. M proposed the idea. F.H.G & S.P prepared, characterized and analyzed the prepared material and thin film samples. G.A & T. M fabricated the FET device. G.A, F.H.G, T. M & S.P prepared the figures and tables. T.M & S.P prepared the manuscript with the support of G.A & F.H.G. The entire project was supervised, administered and shaped by D.V, P.R & T.M.

Conflicts of interest

There are no conflicts to declare.

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Figures

Figure 1

Photograph of (a) grown Cs₂AgBiBr₆ crystal in a conical flask and (b) Cs₂AgBiBr₆ thin film deposited glass surface. (c) Schematic of the Cs₂AgBiBr₆ FET fabricated in our work and circuit connections made for electrical characterizations

Figure 2

(a) XRD pattern (Inset: digital photograph of the spin-deposited Cs₂AgBiBr₆ thin film), and (b) SEM image of the prepared Cs₂AgBiBr₆ lead-free double perovskite thin films.

Figure 3

Transfer characteristic curves of Cs₂AgBiBr₆ FET with channel lengths of 30 μm, 40 μm, 50 μm and 60 μm (a) semi-log scale and (b) linear scale

Figure 4

(a) On current (b) threshold voltage and (c) hole mobility of the Cs₂AgBiBr₆ Perovskite FETs with different channel lengths of 30 μm, 40 μm, 50 μm and 60 μm.