Abstract

Hybrid organic–inorganic perovskites enable the production of semiconductor devices at low cost from solution processing. Their remarkable structural versatility offers unique and diverse physical properties, leading to their incorporation in a wide variety of applications. One major limitation is the significant negative environmental impact associated with developing perovskite devices; common solvents used in perovskite film deposition are highly toxic, which represents a barrier to the transfer to an industrial setting of the perovskite technology. Here we report on the fabrication and characterisation of the first laser printed organic–inorganic perovskite films. The method is solvent-free, scalable and low-cost, allowing fast deposition over large areas and with minimal material waste. We show that the laser printed perovskite films are crystalline and exhibit electrical properties on par with single crystals, despite the fact that the microstructure consists of randomly oriented crystallites. The toner used during printing is designed for optimal film transfer and the vertical separation of its components results in a segregation of the perovskite film in the middle of the stack, therefore also encapsulating the perovskite layer, a process that yields a remarkable resilience to defect formation upon environmental exposure.

1. Introduction

Metal-halide perovskites are organic–inorganic hybrid materials that, in a very short period of time, have established themselves as serious contenders for next-generation electronic applications. Their extraordinary optoelectronic properties, coupled with facile processing, have transformed the way we think about contemporary photonic and optoelectronic applications. Devices based on metal-halide perovskites impact almost every aspect of semiconductor research and reached competitive performance in photovoltaic cells [1–4], light-emitting devices [5–7], photodetectors [8–10], transistors [11–15], lasers [16, 17], and sensors [18, 19]. It is beyond doubt that these materials can enable an astounding array of new products that will soon become a part of our lives. Along with robust and stable electronic performance, the ability to produce uniform films over large areas and at low cost is a pre-requisite for large-scale deployment of optoelectronic applications based on perovskites. The next step in transitioning to the marketplace is the development of manufacturing protocols that are scalable, sustainable, low-cost, environmentally friendly, and preserve material quality. Solution deposition facilitates low-cost processing and warrants compatibility with many substrates, but it comes with many challenges: film properties are highly dependent on precursor composition, solution handling history, coating method, substrate type and post-processing steps [14, 20–23]. In addition, the toxicity of the solvents represents a barrier to the industrialization of the perovskite technology [3, 24]. Several groups have focused on replacing hazardous solvents with ‘green’ solvents [25, 26], or eliminating them completely in solvent-free methods [27, 28]. While these approaches reduce the
negative environmental impact, they necessitate a rather complex infrastructure and have limited scalability. Here, we introduce a solvent-free processing technique for metal-halide perovskite thin films which is cost effective, environmentally friendly and compatible with arbitrary substrates. This method relies on laser printing of the electrically active layer and allows high throughput, fast, large-area coatings of perovskite films. Laser printing has been previously used for depositing organic semiconductor films [29], as well as patterning device electrodes [30]. In this work, we exploit it for the first time for depositing metal halide perovskites. We fabricated thin films of CH$_3$NH$_3$PbI$_3$ on flexible substrates and evaluated their structural, electrical, and electronic properties. The films were deposited from a toner consisting of multiple components, in which small amounts of charge and rheology control agents were introduced to ensure optimal transfer. We found that a spatial de-mixing of the toner constituents occurs during printing, followed by vertical phase separation, with the perovskite layer segregated to the middle of the film. The perovskite film is crystalline and adopts the known tetragonal structure with randomly oriented crystallites, typical of solution-printed perovskite films [31]. Electrical characterization using space-charge limited current measurements yielded properties on par with single-crystal perovskites, with an average resistivity of $\rho = 1.6 \times 10^{-4}$ $\Omega m$ and a maximum charge carrier mobility of $\mu = 20$ cm$^2$ V$^{-1}$ s$^{-1}$, but unfortunately large variations in performance have been obtained across large areas. These results confirm that laser-printing can yield high quality films, as suggested by its sub-band gap optical absorption edge (Urbach energy) spectrum. The current-voltage curves exhibit no hysteresis and the films are stable under ambient operation over extended time periods (10 h), as a result of the non-perovskite toner components encapsulating the perovskite layer in the laser-printed stack. Our work introduces an exciting new avenue for the fabrication of stable perovskite devices that is scalable and devoid of hazardous solvents.

2. Experimental details

2.1. Toner fabrication

Toner fabrication starts with the synthesis of the metal halide perovskite powder. To achieve this, we followed established protocols for perovskite synthesis [32]. First, 3.9 g of a 3:1 molar ratio of CH$_3$NH$_3$I (98%, Aldrich): PbI$_2$ (99.999% trace metal basis, Sigma-Aldrich) was dissolved in 100 ml γ-butyrolactone and the solution was stirred using a magnetic stir bar at 65 °C overnight on a hot plate placed in a nitrogen filled glovebox. Next, the solution was heated at 100 °C for 6 d, until the entire solvent evaporated and the perovskite crystals formed. The crystals were then re-dissolved in 100 ml γ-butyrolactone in the glovebox. Carnauba wax (2 g, Alfa Aesar 31 117) was emulsified in 100 ml of γ-butyrolactone by stirring the mixture in a foil-covered beaker inside of a fume hood and gradually raising the temperature of the mixture to 150 °C over a period of 100 min. Once the wax had melted, the temperature was reduced to 65 °C and the perovskite solution was removed from the glovebox and added along with 900 mg hexamethyldisilazane (HMDS)-treated fumed silica. The solvent was evaporated by gradually increasing the hotplate temperature over 11 d to 195 °C, then keeping this temperature constant for 16 h in the fume hood. The resulting wax/perovskite/silica, i.e. the toner mixture, was melted off the sides of the glass using a heat gun, solidified, and ground using a mortar and pestle (figure 1(a)). Next, the powder was ball milled using stainless steel ball bearings. This step is critical since a small particle size is necessary both to ensure a good transfer during printing, and to generate high resolution of the printed pattern [29]. During the processing and mixing, the total mass of the wax/perovskite/silica mixture decreased to 2.76 g. To give the mixture a positive triboelectric charge, 145 mg of Bontron N07 A charge control agent (CCA) was added to the mixture (5% wt), and then the wax/perovskite/silica/CCA mixture was milled without ball bearings. At the end of this step, we collected the toner consisting of a mixture of perovskite and non-perovskite components, into a container with a lid, and transferred it into the printer cartridge.

2.2. Printing perovskite layer

The printing procedure was similar to that reported for organic semiconductors [29], with the various steps being optimized for the new electronic material. In short, the toner developed as described in the previous section and consisting of various components in the form of a fine powder was loaded into the modified cartridge of a Brother 2770 office laser printer, which was connected to a computer to feed the desired pattern. Following the typical protocol of the printing process, the organic photoconductor (OPC) drum of the laser printer gets charged first, and the laser discharges selected areas to define the pattern imported from the computer. The discharged pattern on the OPC then attracts the positive triboelectrically charged CCA present in the toner powder. Next, a polyethylene terephthalate (PET) (Apollo) sheet was fed to the printer and the pattern consisting of the toner powder was transferred to this substrate, which is oppositely charged (figure 1(b)). In the last step, the heated printer roller fused the toner to the substrate (figure 1(c)). We found...
that in order to obtain a continuous film, the substrate had to be passed through the printer several times (typically 10) in the same orientation.

2.3. Structural characterization of the laser printed perovskite

A Zeiss Gemini 300 field emission scanning electron microscope (FE-SEM) was used to obtain the SEM images and perform the energy dispersive X-ray spectroscopy (EDS) analysis. For SEM surface (profile) imaging the electron acceleration voltage was 10 kV (2 kV) and for EDS analysis the electron acceleration voltage was 5 kV. Height profiles of the sample were obtained in air using an Asylum Research MFP-3D Bio atomic force microscope (AFM). An Asylum Research AC160TSA-R3 silicon cantilever (force constant: 26 N m$^{-1}$, resonance frequency: 200–400 kHz) was used in tapping mode with a feedback setpoint of 800 mV, and 20 $\mu$m $\times$ 20 $\mu$m scans were performed at a rate of 0.5 Hz.

Grazing incidence wide-angle X-ray scattering (GIWAXS) measurements were performed at the Complex Materials Scattering (CMS) beamline of the National Synchrotron Light Source II (NSLS-II), Brookhaven National Laboratory. The X-ray beam with an energy of 13.5 keV was used in transmission mode and scattering images were collected on a custom-made Pilatus-800 K detector placed 372 mm from the sample center using exposure times of 5 s. Background subtraction was performed on 2D GIWAXS images plotted as a function of azimuthal angle, and on integrated traces and vertical ($q_x = 0$ Å$^{-1}$) and horizontal ($q_z = 0$ Å$^{-1}$) line cuts.

2.4. Fabrication of perovskite films by spin coating

Perovskite films of similar composition were fabricated by spin coating for reference. These films were deposited on Si wafers terminated with a 200 nm layer of thermally grown SiO$_2$ that were cleaned via sequential steps of a 10 min soak in acetone at 85 °C, a 10 min soak in isopropyl alcohol at 85 °C, and a 10 min UV-Ozone oven treatment, followed by a deionized water rinse and drying via nitrogen gas stream and a 5 min annealing on a hotplate at 155 °C. Ti/Au contacts were deposited by e-beam evaporation via a shadow mask. Perovskite precursor was made by mixing a 3:1 molar ratio of CH$_3$NH$_3$I (98%, Aldrich): PbI$_2$ (99.999% trace metal basis, Sigma-Aldrich) in N,N-dimethylformamide (anhydrous, 99.8%, Sigma-Aldrich) and mixing the solution at 85 °C overnight in a nitrogen glove box using a magnetic stir bar. The perovskite precursor was deposited onto the cleaned substrates via spin coating using a 10 s ramp and a speed of 3000 RPM. Films were thermally annealed at 115 °C on a hotplate for 90 min.

2.5. Electrical characterization of the laser-printed perovskite film

To test the electrical properties of the laser-printed perovskite films, we fabricated samples using both a top- and a bottom-contact architecture. The electrodes consisted of 100 nm Au thermally deposited through a shadow mask at a rate of 2 Å s$^{-1}$. The channel length varied between 30 $\mu$m and 100 $\mu$m and the channel width varied between 200 $\mu$m and 1000 $\mu$m. Electrical tests were performed in a probe station in air and dark using an Agilent 4155 C semiconductor parameter analyzer. Over 50 samples of each type have been characterized. The averages for the resistivity and mobility were evaluated based on the measurements acquired on 10 films obtained from different printing runs, with 5 measurements taken on different locations on each film.

2.6. Photothermal deflection spectroscopy (PDS)

PDS measurements were performed on a laser-printed perovskite sample and a reference spin-coated sample. Under inert conditions, the samples were submerged in a signal enhancing liquid (Fluorinert FC-770). The samples were excited using a tunable, chopped, monochromatic light source (150 W Xenon short arc lamp...
with a Cornerstone monochromator) and probed using a laser beam (635 nm diode laser from Thorlabs) propagating parallel to the surface of the sample. The heat generated through the absorption of light changes the refractive index of Fluorinert, resulting in the deflection of the laser beam. This deflection was measured using a position sensitive detector (Thorlabs, PDP90 A) and a lock-in amplifier (Amatec SR7230) and directly correlated to the absorption of the film.

3. Results

The surface morphology of the laser-printed perovskite films was examined using SEM, results are shown in figure 2(a). The surface exhibited inhomogeneous features, including domains of various size and shape, as well as plateaus and fissures, yielding differences in height. These films appear to be less uniform and to exhibit a surface topology significantly rougher than that obtained in MAPbI$_3$ films processed by other methods, e.g. spin coating [12, 33], chemical vapor deposition (CVD) [34], or sequential pulsed laser deposition and sublimation (PLD-S) [35]. To examine how the different toner components were distributed within the laser printed film, SEM imaging and EDS analysis was performed on a cross section of the film (figures 2(b) and 1, supplementary material (available at stacks.iop.org/JPhysMaterials/3/034010/mmedia)). These measurements revealed that a vertical phase separation between the perovskite and the non-perovskite toner components occurs, as indicated by the different colors included in the figure (Note: the colors are just a guide for the eyes). The perovskite layer existed between two layers of non-perovskite toner components. As the film was printed via 10 successive passes, the encapsulation of the perovskite layer most likely occurred post-deposition, a process catalyzed by the heat generated by the fuser roller (which operates at a temperature of 170 °C–190 °C). Thermal annealing enables this separation of materials by melting the wax within the toner and allowing it to separate from the perovskite, together with the other toner components (the CCA and fumed silica). We suspect that this encapsulation explains why the laser printed film does not undergo the typical degradation pathways demonstrated in the discoloring of MAPbI$_3$ thin-film samples [36], rather the film retains the initial dark color even under prolonged light exposure for times exceeding 3 weeks.

The roughness of the printed perovskite films was evaluated from atomic force microscopy (figure 2(c)) and we obtained an RMS value of $r = 257 \pm 74$ nm, a value that is significantly higher than the typical roughness of perovskite films created via other methods, such as PLD-S (43.1 nm) [35] or spin coating (12 nm) [33]. The large variation in the surface roughness results from the non-homogenous nature of the surface in different spots of the film. AFM images taken on several different spots are shown in figure 2, supplementary material. Nevertheless, in our samples the roughness corresponds to the top toner layer, and not the perovskite film buried underneath it. While we were not able to access directly the roughness of the perovskite film, the SEM images in figure 2(b) suggest that it is significantly flatter than the top surface. The average height of the laser printed stack was also measured using AFM and a value of $6.6 \mu m \pm 1.4 \mu m$ was obtained, hence, based on the information presented in figure 2(b), the perovskite layer has a thickness of approximately 0.5 $\mu m$. The thick films result from the fact that we had to perform 10 passes to obtain a continuous film and, with improving the printing process, we anticipate that we will be able to reduce the film thickness and create continuous domains.

To characterize the solid-state structure of the printed perovskite films, grazing incidence wide-angle X-ray scattering (GIWAXS) was collected in transmission mode for printed samples on PET substrates, for which the 2D scattering pattern is shown in figure 3(a). The 2D GIWAXS images were not directly background subtracted as the PET substrate is biaxially oriented, resulting in a scattering background with anisotropic intensities whose subtraction would depend on rotation of the background image to match that of the sample image. The reflections associated with the PET substrate can be observed at approximately...
Structural characterization of a printed perovskite film. (a) 2D-GIWAXS image of printed perovskite film on PET. (b) Stacked traces of the calculated powder pattern of the tetragonal phase of MAPbI$_3$ (grey), diffraction trace obtained by azimuthally integrating a 2D GIWAXS image of the printed perovskite film (black), in-plane line cut obtained at $q_{xy}=0\ \text{Å}^{-1}$ (blue) and out-of-plane line cut obtained at $q_z=0\ \text{Å}^{-1}$ (red) of the same 2D GIWAXS image. (c) A 2D-GIWAXS image of the printed perovskite film on PET plotted as a function of azimuthal angle. (d) Azimuthal line-cut at $q=1.00\pm 0.01\ \text{Å}^{-1}$, corresponding to the (002) and (110) reflections of the MAPbI$_3$ tetragonal phase. Dashed lines indicate regions without data given gaps in the detector panel.

Figure 3. Structural characterization of a printed perovskite film. (a) 2D-GIWAXS image of printed perovskite film on PET. (b) Stacked traces of the calculated powder pattern of the tetragonal phase of MAPbI$_3$ (grey), diffraction trace obtained by azimuthally integrating a 2D GIWAXS image of the printed perovskite film (black), in-plane line cut obtained at $q_{xy}=0\ \text{Å}^{-1}$ (blue) and out-of-plane line cut obtained at $q_z=0\ \text{Å}^{-1}$ (red) of the same 2D GIWAXS image. (c) A 2D-GIWAXS image of the printed perovskite film on PET plotted as a function of azimuthal angle. (d) Azimuthal line-cut at $q=1.00\pm 0.01\ \text{Å}^{-1}$, corresponding to the (002) and (110) reflections of the MAPbI$_3$ tetragonal phase. Dashed lines indicate regions without data given gaps in the detector panel.

Two-point I–V measurements taken on the printed perovskite film are shown in figure 4. Two distinct regimes can be distinguished, given by the value of the slope n of the curve $I \propto V^n$ which is indicated in the

$q = 1.17\ \text{Å}^{-1}$; other reflections are associated with the perovskite thin film. To confirm the structure of the perovskite thin film, as well as quantify the distribution in orientation of crystallites, azimuthal integration and both in- and out-of-plane line cuts were obtained; these were then background subtracted after the corresponding background files were properly shifted in azimuthal angle to match the sample files. These background subtracted images and traces are shown in figure 3(b). The calculated powder pattern from the tetragonal phase of MAPbI$_3$ is shown in grey, with the azimuthally integrated trace, in-plane and out-of-plane traces stacked above, respectively.

The printed perovskite accesses the known tetragonal phase, and as shown in both the in- and out-of-plane line cuts, the reflections in its X-ray traces match those in the calculated pattern. That all reflections are present in both the in- and out-of-plane line cuts indicate that the film comprises crystallites that are randomly oriented. To further highlight the distribution of crystallite orientations accessed via printing, the 2D GIWAXS image was plotted as a function of azimuthal angle and background subtracted, shown in figure 3(c). Reflections associated with the tetragonal phase of the perovskite are seen at all azimuthal angles. We took a line-cut at $q=1.00\pm 0.01\ \text{Å}^{-1}$, corresponding to the (002) and (110) peaks of the perovskite tetragonal phase, and plotted this as a function of azimuthal angle in figure 3(d), which shows diffraction peak intensity over the range of angles, indicating a distribution of crystallite orientations. Intensity variation with angle may result not only from the difference in structure factor for the (002) and (110) peaks, but also the distribution of perovskite crystallite sizes. Collectively, our structural analysis by GIWAXS indicates the printed perovskite adopts the known tetragonal phase upon printing, with small crystallites that are randomly oriented with respect to the PET substrate.

Two-point I–V measurements taken on the printed perovskite film are shown in figure 4. Two distinct regimes can be distinguished, given by the value of the slope n of the curve $I \propto V^n$ which is indicated in the
inset. The linear dependence of the current (I) on voltage (V) at low applied bias follows the expression:

\[ I = \frac{1}{R} \cdot V \]  

where R is the resistance and suggests (although does not confirm) unipolar injection with ohmic contacts. From these measurements, the average resistivity of the printed perovskite layer was found to be \( \rho = 2.6 \cdot 10^7 \pm 2.0 \cdot 10^7 \Omega\)m in the case of the top contact samples and \( \rho = 5.5 \cdot 10^6 \pm 5.4 \cdot 10^6 \Omega\)m for the bottom contact samples. The high variance in the resistivity values comes from the inhomogeneous nature of the printed films, voids, and thickness variation: small fluctuations in the toner stoichiometry along the printer roller can lead to different local compositions of the resulting film and yield variations in the electrical properties. Further optimization of the toner formulation and printing process will most likely increase the film uniformity and reduce batch to batch variations. Nevertheless, these resistivities are similar to those determined in MAPbI\(_3\) single crystals, confirming that a high quality perovskite layer can be obtained by laser printing [37]. It is notable that these excellent electrical properties were obtained in spite of the large contact resistance that is expected due to the presence of the non-perovskite toner components between the electrodes and the perovskite film.

The charge carrier mobility (\( \mu \)) was extracted from the high voltage regime of the measurements presented in figure 4, i.e. the space-charge limited current (SCLC) measurements. Here, the quadratic dependence of the current on voltage confirms the validity of the Mott-Gurney model (equation (2)) in this voltage range (6 V to 10 V):

\[ J = \frac{9}{8} \varepsilon_0 \varepsilon_r \mu V^2 \]  

Here, \( J \) is current density, \( I \) represents the current, \( A \) is the surface area, \( \varepsilon_0 \) is the permittivity of free space, \( \varepsilon_r \) is the relative permittivity of the material, \( V \) is the applied bias, and \( L \) is the separation between electrodes. The charge carrier mobility extracted using equation (2) was \( \mu = 13.1 \pm 0.4 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1} \), with a maximum of \( \mu = 20 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1} \). These mobilities are on the same order as the SCLC mobilities found in single crystal samples of MAPbI\(_3\) [38], and higher than the mobilities obtained in field-effect transistor devices [11–14, 39]. Based on the energy level alignments, this represents a hole mobility. Nevertheless, we have shown in the past that Au contacts promote ambipolar transport in this material [14], a result which confirmed that, similar to the case of other systems [40], the energy band diagram does not guarantee the nature of charge injection due to the complex, convoluted phenomena occurring at metal/semiconductor interface. This suggests that the mobility value using SCLC measurements might be overestimated.

In figure 5 we show the evolution of the resistivity as a function of time; the measurements were taken in ambient and dark and no bias was applied between measurements. It can be observed that the electrical
properties remain constant, with a slight increase (<10%) in resistivity recorded after ten hours. This stability is remarkable given that no post-processing step was necessary, and results from the fact that during laser printing the phase separation process results in encapsulation of the perovskite layer between the non-perovskite component, which prevents the interaction with air. This result represents a significant milestone since the fast degradation of solution-deposited perovskite layers under environmental device operation due to interaction with moisture, oxygen, etc, is a serious barrier to advancing this technology from laboratory-scale to industrial-scale applications [41]. Although notable improvements have been achieved through material and device design [24, 42], the devices have not yet reached the stability requirements for mass deployment.

To evaluate the optical properties of the laser printed perovskite film, we performed photothermal deflection spectroscopy (PDS) measurements (figure 6). Since the laser-printed perovskite film also contains
other light absorbing materials, a foil with all non-perovskite film components was prepared and was used as a reference to isolate the contribution of the perovskite part. We found that the laser-printed perovskite shows the typical MAPbI₃ absorption onset [20, 43], and a calculated Urbach energy (E_U) of 23.8 ± 1.6 meV. This value is only slightly larger than the Urbach energy measured for a spin-coated film (21.2 ± 0.8 meV), which confirms the excellent electronic quality and the low degree of energetic disorder of the laser printed perovskite. In addition to offering a low-hazard solution for manufacturing large-area modules, laser printing also addresses another major challenge in perovskite processing, namely film patterning. Patterning is critical in reducing the crosstalk between devices, but conventional protocols cannot be adopted for devices based on metal halide perovskite semiconductors because these materials are soluble in water, which is an essential reagent in photolithography, and will therefore be destroyed during the fabrication process. This solvent-free method also eliminates the challenges faced by complex multi-layer architectures, where layer degradation due to the interactions with the solvent of consecutive layers is very common. The success of this technology and its transfer from laboratory-scale fabrication to industrial applications hinges on the ability to improve the film uniformity over large areas. Therefore, future efforts will focus on elucidating the processes governing the crystal formation during laser printing and gaining a better control of film formation by optimizing the toner composition (nature and quantity of components, method of mixing and grinding). Post-processing, which was very efficient in improving the properties of solution processed perovskite films, will also be explored, while being conscious to adopt only environmentally friendly methods.

4. Conclusions

In conclusion, we deposited high quality metal halide perovskite layers by laser printing and evaluated their electrical properties. This solvent-free processing method is environmentally friendly and yields films with low energetic disorder despite the microstructure consisting of randomly oriented crystallites. Charge transport in such films is approaching the intrinsic limit, as extracted from single crystal measurements, with negligible hysteresis in the current-voltage characteristics, and high environmental stability. Our results introduce a new method for perovskite semiconductor processing, namely laser printing; exploiting this ubiquitous technology could radically transform the manufacturing of perovskite devices, enabling high volume production at low-cost and with minimum negative effect on the environment. Nonetheless, maintaining film uniformity over large areas and being able to etch the encapsulating layers consisting of non-perovskite materials are challenges that should be addressed before this method can be implemented into large-area device processing.

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