Research Article

A Different, Nondestructive Method of Investigating In Situ Degradation in Hybrid Perovskite Solar Cells

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One of the most significant impediments to the upscaling of hybrid organic-inorganic perovskite (HOIP) solar cells is poor stability. So, to effectively establish precautionary strategies, it is necessary to have a comprehensive grasp of the mechanisms that contribute to their degradation. Conventional characterization techniques used for in situ degradation assessment of perovskite solar cells either track just universal parameters, which yields poor insights about localized degradation processes, or the technique itself induces degradation, which may confound results. Developed in this study is a nondestructive technique of analyzing in situ active layer degradation in HOIP solar cells’ active layer utilizing a combination of optical modelling and ageing device’s reflectance tracking. The optical dielectric functions of the solar cell’s functional layers were modelled using the layers’ transmitted and reflected light spectra. The transfer matrix approach was used to fit the reflectance spectrum of entire solar cell layer stacks utilizing the computed dielectric functions for individual materials and accounting for the possibility of lead iodide gradient layer formation in the model. Leveraging no other characterization method, the presence of a lead iodide gradient layer was identified within the solar cell layer stack, an unmistakable indicator of methyl ammonium lead iodide perovskite (MAPI) degradation, probing with only photons.

1. Introduction

Hybrid organic-inorganic perovskite- (HOIP-) based solar cells have experienced tremendous growth in energy conversion efficiencies for lab-scale cells over the last decade and are now close to the stage of getting commercialized. According to the US Energy Office, in order to be commercialized, HOIP-based solar cells must overcome hurdles such as long-term stability and durability, scalability in terms of module efficiency, and, last but not least, manufacturing yield and process control [1]. Studies are presently concentrating on researching and comprehending degradation processes as well as methods to mitigate them. According to established experimental data in literature, HOIP irreversibly degrades by decomposing into solid inorganic salt, lead (II) iodide (PbI₂), and volatile organic molecules [2–6]. Since HOIPs are ionic materials, ionic migration and chemical reactions with the adjacent layers accelerate their degradation. This is also exacerbated by moisture, heat, electric fields, and high-energy electromagnetic waves [7–12].
1.1. Degradation Study of HOIPs. Researchers have innovatively applied various conventional characterization techniques to conduct in situ characterization on HOIP films to gain a thorough understanding of their degradation mechanisms. Song et al. used high-resolution time-resolved mass spectrometry (MS) to analyze the volatile degradation products from perovskite films in their study of photodecomposition in HOIPs [9]. Schlipf et al. applied the grazing incidence neutron scattering (GISANS) for an in situ study of the degradation of HOIP films exposed to moisture [5]. With a focus on the nonvolatile degradation products, Leguy et al. employed a combination of time-resolved X-ray diffraction XRD and ellipsometry to study the evolution of moisture-induced degradation in methylammonium iodide-(MAPI-) based perovskite films [13].

In situ degradation studies of the HOIP active layer in solar cells using conventional methods present a problem since it is sandwiched by charge transport layers and electrodes. To investigate how the active layer has altered chemically over time or how the environment has impacted it, the majority of studies frequently use a postmortem analysis approach. For instance, Guo et al. characterized the active layer following degradation using time-of-flight secondary ion mass spectrometry (ToF-SIMS), which required peeling off the top electrode [3]. To investigate metal electrode-driven MAPI degradation in solar cells, Han et al. used a similar postmortem methodology employing SEM and XRD to characterize how different environmental ageing conditions affected the degradation of the active layer [6]. However, the postmortem approach provides a limited perspective because it only considers the initial and postaging states and ignores the intermediate stages of degradation. In other words, such type of investigation displays the end-of-life for the specimen.

Another issue with traditional characterization methods is that they may damage the HOIP crystals, making interpretation of results difficult and ambiguous, especially when used in situ. Ran et al. demonstrated that when exposed to electron beams, one of the most commonly used tools in degradation characterization techniques, HOIPs suffer from ionization and displacement damages, as well as from electrostatic charging [14]. Furthermore, the Meng et al. study emphasized the negative effects of high vacuum on HOIPs, which is a necessary condition for some of these characterization equipment [15]. As a result, noninvasive characterization methods that (i) provide quantitative degradation information about the active layer of potentially even encapsulated HOIP-based solar cells and (ii) allow for continuous characterization without introducing measurement-induced artifacts are the necessity of the hour.

1.2. Optical Constants. Differences in a material’s physical, chemical, and mechanical properties can influence how it interacts with or responds to electromagnetic waves such as light. This behavior is captured by the material’s optical constants, expressed by the complex refractive index \( \varepsilon(\lambda) = n - i k \) and the complex dielectric function \( \varepsilon(\lambda) = \varepsilon' - i \varepsilon'' \). For thin films, the optical constants can be determined from the experimental spectroscopic ellipsometry and spectrophotometry through a process of iterative model-calculation and fitting [16–18]. Applying this method, Leguy et al. were able to determine the optical constants of MAPI thin film from spectroscopic ellipsometry [19]. Ball et al. demonstrated that the device properties obtained from an optically modelled HOIP device stack showed good agreement to experimentally determined data [17]. Hoppe et al. showed that optical properties can be determined even for buried layers inside the complete solar cell stack [18, 20]. They furthermore demonstrated that the output power can be optimized by optical modelling for single junction as well as for tandem solar cells [18, 21, 22].

In this work, optical modelling of reflectance data with the help of the transfer matrix method (TMM) and in combination with an effective medium approach (EMA) was used as a noninvasive method for tracking the evolution of active layer degradation in an inverted HOIP solar cell. This method allowed us to determine both the volume fraction of PbI₂, an undeniable by-product of degradation in the active layer, as well as the vertical distribution of PbI₂ over the active layer depth.

2. Experimental

2.1. Materials. Prepatterened ITO (25.3 * 25.3 * 1.1 mm, 10 ohm/sq) glass substrates were sourced from Xinyan Tech, Hong Kong. Lead (II) iodide (99.999%, trace metal basis) (PbI₂) and methylamine iodide (MAI) (low water content) were purchased from TCI Deutschland. Phenyl-C70 butyric acid methyl ester (PC₇₁BM) was purchased from Solenne BV, United Kingdom. The PEDOT:PSS (VP AI 4083) formulation was acquired from Heraeus, Germany, and bathocuproine from Alfa Aesar, Germany. Aluminum (99.999% pure) pellets were sourced from Kurt J. Lesker Company, UK.

2.2. Solution Preparation. The perovskite precursor was prepared by dissolving 461 mg of lead iodide (PbI₂) and 153 mg of methylamine iodide (MAI) (low water content) resulting in a 1:1 molar ratio per 1 mL of DMF and DMSO mixed (4:1 v/v) solvent and stirred for 30 mins at 70°C inside the glovebox. In order to have a particle-free film, the solution was filtered with a 0.45 micron PTFE filter before spin cast. For the electron transport layer (ETL), 20 mg PC₇₁BM ([6, 6]-phenyl-C71-butyric acid methyl ester) was dissolved in 1 mL dichlorobenzene, resulting in a 2 wt.-% solution.

2.3. Layer Stack. The layer stack of a complete device-inverted solar cell is glass/ITO/PEDOT:PSS/MAPI/PCBM/BCP/Aluminum. To obtain the optical constant models of the individual layers, their films were prepared on bare glass.

2.4. Device Fabrication. The prepatterened ITO substrates as well as the glass substrates were cleaned in an ultrasonic bath successively using acetone and isopropanol at 40°C for 15 min each. The HTL, PEDOT:PSS (VP AI 4083) was spin cast from aqueous dispersion on the ITO via a 0.45 micron polyether sulfone (PES) filter at 3000 rpm for 40 s and was immediately transferred to a hot plate. The films were annealed for 30 mins at 150°C on the hotplate in the air.
Subsequently, while in the glovebox, the perovskite precursor was spin-coated on the PEDOT:PSS layer at 3000 rpm for 35 s, and by the 25th sec, 300 μL of chlorobenzene was dripped on the wet MAPI film to enhance crystallization of the MAPI. The film was then transferred to a preheated hot-plate at 100°C and annealed for 10 min. Thereafter, the PCBM solution was spin cast on the cooled perovskite layer at 1000 rpm for only 30 seconds and then was dried for 10 mins at 100°C to remove remaining solvent molecules. The substrates were patterned by gently scratching the edges to reveal the ITO before it was transferred to physical vapor deposition (PVD), the metal evaporation chamber, without any exposure to the ambient. At a pressure of about 10⁻⁶ mbar, a 5 nm thick film of BCP was thermally evaporated on the PCBM layer through a shadow mask, which was directly followed by the deposition of a 300 nm Al electrode without breaking the vacuum. Thus, resulting in a 0.42 cm² active area of the solar cells, which were not encapsulated. Films of each functional layer were fabricated on glass individually using the same parameters as above.

2.5. Ageing. The solar cells were aged under ambient conditions in a drawer, at a relative humidity of 45%-50% and at a temperature of 23 to 25°C. The reflectance of the solar cells was then measured at 10 min intervals during ageing until complete degradation.

2.6. Optical Spectroscopy. Reflection (R) and transmission (T) spectra were simultaneously recorded with two Avantes AvaSpec-ULS3648-USB2-UA-25 fiber spectrometers over a wavelength range from 300 to 1100 nm with integration time of 13 milliseconds and 100-times averaging.

2.7. XRD Characterization. X-ray diffraction (XRD) was performed with the diffractometer Panalytical X’Pert Pro MPD (Netherlands) equipped with the following: copper line fine focus X-ray source with Cu Kα1 + Kα2 radiation, parallel mirror with 1/32” divergence slit, 10 mm mask, 0.04 rad Soller, and 1.4 mm anti-scatter slit for the incoming beam. The diffracted beam section involves a programmable anti-scatter slit with 1/32”, Soller 0.04 rad, and Panalytical PIXCEL line detector with 255 channels in scanning mode (1D). The 2 theta scan range was from 10° to 52° with a step size of 0.026° and a scan time of 11 min. The diffractometer is a vertical theta-theta diffractometer, where the sample is horizontally positioned and not rotated during measurement. Phase identification and quantification were done with the program Malvern Panalytical High Score Plus, Version 4.9 and pdf2 database from 2001 from ICDD and COD database from 2021.

2.8. Optical Modelling. Dielectric functions were modelled for all materials based on transmittance and reflectance spectra within CODE by WTheiss Hardware and Software, USA. Based on the established and Kramers-Kronig consistent complex dielectric functions, respectively, complex optical constants, the reflectance of the whole solar cell layer stack was reproduced. The connection between the dielectric function and the complex refractive index \( n = n + ik \) is given as follows: \( \varepsilon' = n^2 - k^2 \) and \( \varepsilon'' = 2nk \).

The model of the dielectric function is designed around its relationship with the electric field vector of the probing light wave that interacts with the material. As a result, a material’s unique properties, such as vibrations of the electronic system and atomic cores, as well as contributions from free charge carriers, can be characterized by a couple of frequency-dependent oscillators [23]. Kim oscillators and a continuous dielectric background were employed to represent the dielectric function in this work. A Kim oscillator permits a smooth transition between a Gaussian and Lorentzian line profile and is an expansion of the simple harmonic oscillator model for vibrational modes [24].

3. Results and Discussion

3.1. Optical Constant of the Solar Cell’s Layers. Figure 1(a) depicts the sequential arrangement of the functional layers of the inverted planar MAPI solar cell investigated in this study. The structure consists of a glass/ITO substrate, with PEDOT:PSS serving as the hole transport layer. This is followed by the MAPI photoactive layer, onto which the PCBM electron transport layer is placed. Next, there is the BCP buffer layer, and finally, the aluminum back electrode. While the study is focused on the degradation of the sandwiched photoactive MAPI layer, the optical constant model of all the layers, including the glass substrate, is needed to optically model the solar cell. The optical constant model of the 1.1 mm thick quartz glass substrate was first determined by fitting the modelled transmittance and reflectance from the dielectric function model consisting of several oscillators to the measured reflectance and transmittance. A good fit was obtained for the glass substrate as shown in figure S1a in the supplementary. The optical constants of the individual layers that make up the solar cell (as shown in Figure 1(a)) were determined by first preparing their thin films on bare 1.1 mm thick quartz glass, and then measuring their reflectance and transmittance. Afterwards, their measured optical data was fitted to obtain their optical constant model. Good fits for the reflectance and transmittance data of the individual layers were obtained as can be seen in figure S1b-f. We also compared the film thicknesses obtained via optical modelling to the experimentally determined thicknesses in Table 1. The good agreement between these values partly attests to the accuracy of the optical constant models obtained for the layers. The refractive index \( n \) and extinction coefficient \( k \) of these layers are shown in Figures 1(c) and 1(f), respectively.

The transmittance and reflectance spectra of the MAPI photoactive layer, measured from a MAPI thin film on a glass substrate, as well as the respective fits from the model dielectric function, are shown in Figure 1(b). The obtained extinction functions \( n \) and \( k \) (shown in Figures 1(c) and 1(d)) were compared to existing works in literature [17, 25]. Indeed, the results are in good agreement with the optical constants obtained for single crystal MAPI, which is a clear indication that the polycrystalline film of MAPI is pure and does not contain PbI₂.

Based on the chemistry of MAPI perovskite degradation and published experimental data that shows that solid PbI₂.
Figure 1: Continued.
Figure 1: Continued.
is the remnant, MAPI irreversibly decomposes into PbI₂ and volatile organic compounds [6, 15, 26]. Thus, obtaining the dielectric model for the pure PbI₂ film is necessary in this study. In order to confirm the degradation remnant of MAPI perovskite, a pristine MAPI film was aged in ambient conditions (45-50% RH; 24-25°C) for 14 days, until it turned from a dark brown film to a nonphoto active yellowish film indicating complete degradation to PbI₂. To assess the degree of degradation, this film was characterized and compared to pristine PbI₂. The complex dielectric function models for MAPI, degraded MAPI, and pristine PbI₂ films on glass were obtained as earlier described by fitting their measured reflectance and transmittance as shown in figure S2.

Table 1: The measured thicknesses compared to the thickness obtained from optical modelling of the thin films deposited on a 1.1 mm glass substrate.

<table>
<thead>
<tr>
<th>Layer</th>
<th>Measured thickness (in nm)</th>
<th>Thickness (in nm) by optical modelling</th>
</tr>
</thead>
<tbody>
<tr>
<td>ITO</td>
<td>180.0 ± 25*</td>
<td>179.2</td>
</tr>
<tr>
<td>PEDOT:PSS</td>
<td>68.8 ± 5.6</td>
<td>67.5</td>
</tr>
<tr>
<td>MAPI</td>
<td>323.5 ± 85</td>
<td>307.4</td>
</tr>
<tr>
<td>PCBM</td>
<td>62.4 ± 14.9</td>
<td>53.6</td>
</tr>
<tr>
<td>BCP</td>
<td>21.5*</td>
<td>21.4</td>
</tr>
<tr>
<td>Al</td>
<td>314.6*</td>
<td>320.4</td>
</tr>
</tbody>
</table>

The films were measured using laser microscopy except *provided by the supplier and *measured by quartz crystal in situ film thickness monitor.

Figures 2(a) and 2(b) depict the obtained refractive index (n) and extinction coefficient (k) values of fresh MAPI, degraded MAPI, and pristine PbI₂. These figures demonstrate that the degraded MAPI exhibits characteristics that are not only similar to those of pure PbI₂ but also share similarities with the fresh MAPI film. The photoluminescence plots of the films, as depicted in Figure 2(c), indicate that the degraded MAPI film is composed of a mixture of pure MAPI and PbI₂ phases. This is evident from the photoluminescence peaks observed around 770 nm and 580 nm in the degraded film, which correspond to both MAPI and PbI₂, respectively. The X-ray diffraction (XRD) findings, depicted in Figure 2(d), suggest that the degraded MAPI film exhibits the presence of PbI₂ and exhibits a spectral resemblance to the unaltered PbI₂. However, it also reveals the existence of an additional phase. By employing Toraya’s direct derivation approach for phase quantification [27], the analysis revealed that the degraded methylammonium lead iodide (MAPI) sample comprises 57.8% of hexagonal PbI₂ and 42.2% of the hydrated MAPI phase (see figure S4), and the pristine PbI₂ film is simply hexagonal PbI₂ phase (see figure S3).

Another noteworthy result derived from Figure 2(b) is that the nonzero values of the extinction coefficient below the bandgap of the damaged MAPI can be ascribed to the heightened roughness of the MAPI film during degradation, as indicated by Shirayama et al. [28]. Therefore, it may be inferred that the extinction coefficient exhibits aberrations resulting from light scattering effects as the wavelength increases. In their study, Leguy et al. also made the observation that damaged MAPI films display an elevated level of surface roughness. This suggests that the optical constants
Figure 2: Continued.
acquired for the degraded MAPI correspond to a combination of phases, which includes embedded pinholes, as well as PbI₂ and hydrated MAPI. The optical properties of the degraded methylammonium lead iodide (MAPI) material would be employed instead of those of the pristine lead iodide material in the subsequent optical model for the degradation of solar cells, to simplify the analysis.

3.2. Solar Cell Optical Model. An all-encompassing optical model for the solar cell was established by including a complete array of prior determined dielectric function models of the functional layers. The reflectance of the entire solar cell was simulated and then compared to the measured reflectance of the solar cell as can be seen in Figure 3(a). The consistency between the optically simulated and measured reflectance of the solar cell shows that despite the optical constants, the models of each layer being generated from the data collected from the films on glass are identical to that of the solar cell. The obtained optical model of the solar was subsequently utilized to study degradation. For the degradation study, the reflectance of a fresh solar cell without encapsulation was intermittently measured until complete degradation took place, as evidenced by the transformation of the black photoactive layer into a yellowish color, as shown in Figure 3(b).

3.3. Morphology of Degradation in MAPI Solar Cell. Based on the chemical reaction depicted in equation (1), it has established that MAPI perovskites undergo degradation when exposed to various factors such as light, heat, and moisture ingress [29]. Both methylamine and hydrogen iodide exhibit volatility, resulting in the formation of solid PbI₂ as the left-oversolid product. Based on existing scholarly research, it has been observed that under appropriate circumstances, the degradation process can potentially be reversed, provided that the volatile products are effectively retained and not lost [30].

\[
\text{CH}_3\text{NH}_3\text{PbI}_3(s) \xrightarrow{T,hf,H_2O} \text{CH}_3\text{NH}_2\uparrow + \text{HI}\uparrow + \text{PbI}_2(s). \tag{1}
\]

Nevertheless, in a MAPI thin film, the first formation of solid PbI₂ occurs as distinct crystals that advance both horizontally and vertically within the film’s thickness, rather than forming a completely continuous film at the interface between the perovskite and air. The aforementioned characteristics were observed in the scanning electron microscope (SEM) image, shown in figure S5, of the degrading MAPI perovskite, which shows discrete growth of bright PbI₂ crystals. In the context of the MAPI solar cell layer stack, it is reasonable to anticipate a larger degree of reversibility due to the retention of the volatile degradation products, as the MAPI layer is positioned between the electron transport layer (ETL) and the hole transport layers (HTLs). However, as in the case of this study, it should be noted that methylamine and hydrogen iodide, being volatile and soluble in PEDOT:PSS, have the ability to diffuse out of a fully assembled solar cell through this layer. Furthermore, the presence of the PCBM layer, characterized by its soft and disordered nature, is expected to play a substantial role as a loss pathway for these volatile products, resulting in the retention of PbI₂. The phenomenon of solar cell’s active layer degradation is found to propagate in two directions: laterally over the surface and vertically through the active layer.
layer thickness. This can be confirmed by the presence of the overlapping patches in the photoluminescence and electroluminescence imaging of the deteriorating solar cell, as depicted in figure S6. In essence, degradation of the MAPI active layer is characterized by the absence of a closely uniform film of PbI₂. Instead, there is a distribution of
discrete PbI$_2$ crystals observed both on the surface of MAPI and throughout the thickness of the film.

3.4. In Situ Degradation Study. Considering the morphological characteristics of MAPI degradation and acknowledging the nonintermixing nature of MAPI and PbI$_2$ at the atomic level, the optical behavior of the mixture may be effectively described using the effective medium technique. The Bruggeman approach, equation (2), is employed to model the active layer as an effective medium approximation layer. In this representation, the matrix is constituted by MAPI, while the embedded particle is represented by PbI$_2$. Using this approach, the volume percentage of PbI$_2$ in MAPI can be obtained at each specific time during degradation via the measured reflectance. In the equation, $\varepsilon_M$ is the dielectric function of the matrix, $\varepsilon$ is the dielectric function of the particle, $\varepsilon_{\text{eff}}$ is the effective medium dielectric function, and $f$ is the volume fraction (i.e., volume of embedded particle ÷ total volume).

$$\left(1 - f\right)\varepsilon_M - \varepsilon_{\text{eff}} + f\frac{\varepsilon - \varepsilon_{\text{eff}}}{\varepsilon + 2\varepsilon_{\text{eff}}} = 0. \quad (2)$$

To address the variability of degradation product across the active layer thickness, a subdivision of the active layer was implemented. Each sublayer was then treated as an effective medium approximation layer to analyze the vertical distribution of lead iodide throughout the active layer thickness, as depicted schematically in Figure 3(c). The determination of the volume fraction and vertical distribution of PbI$_2$ was achieved through the application of a concentration gradient layer, which allows for the fitting of the measured reflectance using the earlier defined MAPI solar cell optical model.

The quantification of the volume percentage of PbI$_2$ at any specific moment during degradation is achieved by fitting the reflectance spectra of complete solar cells, which were measured at a given time, to the simulated reflectance of the optical model of the solar cell. In figure S7(a to f) of the supplementary, it shows the experimental reflectance spectrum of a solar cell, as well as the reflectance obtained from fitting the optical model. It is important to note that the model’s fitting process allowed for the adjustment of only the concentration gradient and volume percentage of PbI$_2$ as free parameters. The observed discrepancies particularly around 700 nm to 800 nm observed between the measured reflectance and the fitted reflectance can be attributed to the increase in roughness effects of the active layers as it degrades. It is important to note that the actual measurement is conducted on a spot with dimensions of approximately 5 millimeters by 5 millimeters.

Figure 3(d) shows the obtained concentration gradients of PbI$_2$ within the active layer of the solar cell, acquired at various ageing times. The y-axis shows the volume fraction of PbI$_2$ in MAPI with zero meaning 100% MAPI and 1 implying 100% degraded. The x-axis shows the depth across the active layer thickness, with 0 nm corresponding to the interface between PEDOT:PSS and MAPI to the point of maximal depth, which corresponds to the interface between MAPI and PCBM. Accelerated degradation was observed on the MAPI/PCBM interface which is evident from the increased volume percentage of PbI$_2$ at greater depths within the active layer as the ageing time progresses. This can be attributed to the absence of encapsulation for the solar cell, resulting in heightened degradation at the interface between the solar cell and air. This degradation is primarily caused by the intrusion of moisture and the subsequent loss of volatile organic compounds.

4. Conclusion

In summary, we successfully employed reflectance measurements and optical modelling to quantitatively track the formation of PbI$_2$, serving as an indicator of MAPI degradation, within a complete HOIP solar cell. The understanding of the discrete crystallization pattern of the degradation product enabled us to employ the effective medium approach and the sublayering technique in our optical modelling to ascertain the evolution of PbI$_2$ within the active layer of MAPI. In accordance with the understood degradation chemistry of MAPI, our study revealed that the degradation process is particularly pronounced at the interface between MAPI and PCBM due to its interaction with ambient moisture and the subsequent loss of organic molecules, namely, hydrogen iodide (HI) and methylamine, within the unencapsulated solar cell. This observation can be attributed to the direct interface between the MAPI and PCBM, which is characterized by the latter’s porous nature. This study showcases the efficacy of optical modelling as a noninvasive approach for examining the degradation of the active layer in solar cells based on highly sensitive hybrid organic-inorganic perovskite (HOIP) materials.

Data Availability

All data generated or analyzed during this study is included in this published article (and its supplementary information file).

Conflicts of Interest

The authors declare no conflict of interest.

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Supplementary Materials

Supplementary material for this article is available on the journal’s online library. The supplementary material includes the following: the fits of the measured optical
reflectance and transmittance of the layers making up the MAPI solar cell; XRD scan fits for phase identification; morphology of degradation in MAPI (via SEM, ELI, and PLI imaging); and fits of the solar cell’s reflectance as it degrades. (Supplementary Materials)

References


