

Systematic Study of Space-Grade Encapsulants and Barrier Layers for durability of MAPbI₃

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Abstract—Here we present our ongoing and preliminary results into the durability of NH₃CH₃PbI₃ (MAPI) films undergoing a high humidity test as outlined in the AIAA-S111 standard for qualification of space solar cells. The samples were encapsulated in a space qualified 2-part silicone (DC-93500) and coverglass lamination as a protectant and select cohorts were also coated with SiO₂ barrier layers and a silane surface modifier. A first study using a 2-part epoxy encapsulant was used to model the diffusion behavior of moisture through an encapsulant to determine feasibility and indicated an approximate border removal area for robustness. The ongoing test utilizes transmission spectra as a proxy for material dissociation. Few samples show significant degradation up to 880 hours, typically the encapsulated samples as produced show the least, the samples with the silane surface modifier show the most. These data are being processed and quantitative statistics will be shown.

Index Terms—Space photovoltaics

I. INTRODUCTION AND BACKGROUND

The advent of highly efficient, perovskite-structured, thin film solar cells has created significant interest among photovoltaics researchers. The limitations of these devices include an extreme sensitivity to moisture. Although this sensitivity is the subject of intense research, NASA has recently begun to examine the potential utility of these materials for space-based photovoltaics.

In the environment of space there is no moisture or oxygen to react with the perovskite material and cause degradation to the binary constituents. However in order to be qualified for space applications, a series of tests outlined in the AIAA-S111 standard is typically followed and among these tests is an intense humidity test that prescribes soaking the solar cells at 95% relative humidity (RH) at 45° C for 1500 hours. These conditions would destroy most champion devices described in the literature without moisture barriers [1] [2], [3].

Recent effort shown at previous IEEE PVSC demonstrated the ability for ultrathin organofunctional silanes to passivate CIGS from moisture-induced degradation under the conditions of damp heat from IEC 61646 [4]. We hypothesized that encapsulated materials would restrict moisture ingress to

the samples by diffusion-limited processes but that further addition of barrier layers and passivation layers would improve performance significantly. We have developed MAPbI₃ films on glass and performed a durability test based on AIAA-S111 tests. Devices were fully encapsulated using a space-grade silicone encapsulant and the effects of a SiO₂ barrier layer of varying thickness was studied as well as a self assembled ultrathin silane passifier. We will target a 1500 hour experiment and collect information on bare film degradation under these varying protective layers.

II. EXPERIMENTAL

The first structure (Bordered in Blue in Figure 1) is the basic perovskite thin film structure, one encapsulated and one un-encapsulated. The second structure (Bordered in Orange in Figure 1) is a perovskite thin film coated in a 50 nm layer of SiO₂, one encapsulated and one un-encapsulated. The Third structure (Bordered in Green in Figure 1) is a perovskite thin film coated in 50 nm of SiO₂ that is in turn coated in a self assembled layer of the APTES ligand, one encapsulated and one un-encapsulated.

The perovskite ink used was obtained from Sigma-Aldrich and consisted of MAI and PbI₂ dissolved in DMF in the ratio 1:1:1 respectively. The APTES solution used was VM-652 and consists of (3-Aminopropyl)triethoxysilane suspended in a solvent. The encapsulant DC-93500 is a two-part silicone, mixed thoroughly in a 10:1 ratio and then degassed. All of the following steps were performed in a nitrogen purged glove box:

- 1) Drop cast 30 μ l of ink onto a 1" by 1" glass slide
- 2) Spin at 2000 RPM for 90 seconds
- 3) Anneal on a hotplate at 100° Celsius for 10 minutes. The grey film transitions to black seconds after being placed onto the hotplate
- 4) Deposit 50nm to 150 nm of SiO₂ atop the edged perovskite
- 5) Drop cast 2ml of the APTES-Solvent solution while spinning at 200 RPM for 5 seconds then ramping up to 2000 RPM for 30 seconds

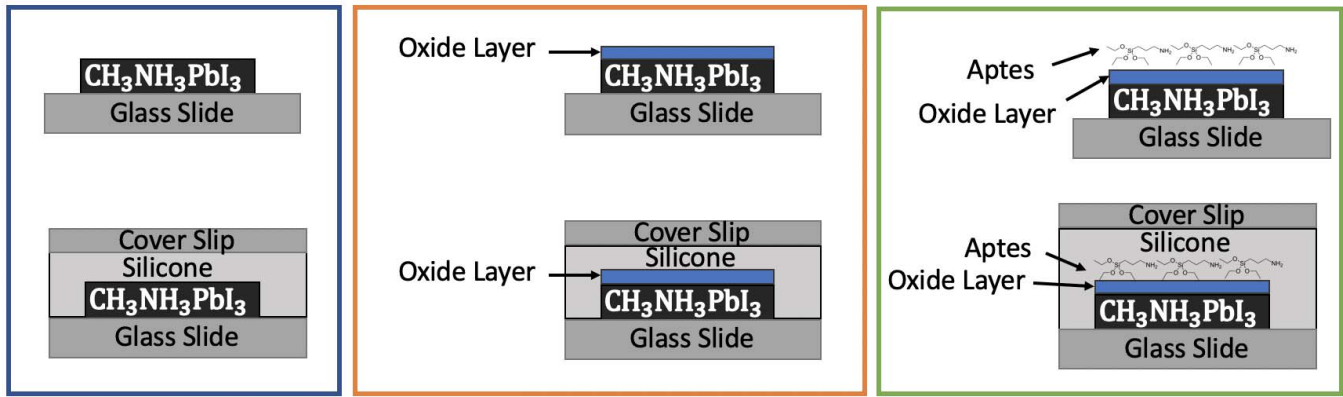


Fig. 1. Illustration of the sample architecture including the lamination and barrier layers. Blue Border: Basic MAPI structure. Orange Border: MAPI + Oxide structure. Green Border: MAPI + Oxide + APTES structure.

- 6) This deposits a 2nm - 4nm (3 to 7 layers) APTES film atop the perovskite

Samples were exposed to high humidity conditions using an environmental test chamber (ETC) programmed to maintain an ambient environment of $95 \pm 5\%$ RH and $28 \pm 2^\circ$ C. The samples were periodically removed from the ETC and characterized by collection transmission spectra and optical imaging. Transmission spectra were collected using a Lambda 950 spectrophotometer with a spot size of about 1 cm^2 . Optical imaging were collected using a Nikon DSLR.

III. DATA ANALYSIS

The glass layers are considered to be impermeable to humidity, therefore the moisture is constrained to diffusion through the sides of the encapsulant. Thus the concentration of moisture, inwards from any edge towards the center of the sample can be treated as a boundless 1-dimensional diffusion problem. The concentration gradient as a function of time, $n(x, t)$, of a diffusive species in an unbounded, 1-dimensional system is, from Fick's law, given by:

$$\frac{n(x, t)}{n_0} = \text{erfc}\left(\frac{x}{2\sqrt{Dt}}\right) \quad (1)$$

where n_0 is the local saturation concentration of the species, x is the displacement from 0, the saturation location, D is the species diffusion constant in the medium and t is time. The complementary error function $\text{erfc}(x, t)$ constrains the shape profile of the relative concentration gradient as a function of time and can therefore be used to fit an estimate of the water diffusion constant in the encapsulant in order to predict behavior over time, as shown in Figure 2.

Figure 2 was created from proof of concept data of perovskite encapsulated with a 2-part epoxy. Utilizing a reaction-diffusion model we estimated the moisture diffusivity in the encapsulant and an estimated critical relative moisture concentration of 0.2, based upon the work by Yang et al. [5]. Therefore the extrapolation of moisture gradient to 1500 hours indicates that a sub-critical moisture concentration to preclude degradation of the perovskite could be found beyond a approximately 1.2 cm border. Note that this measurement is

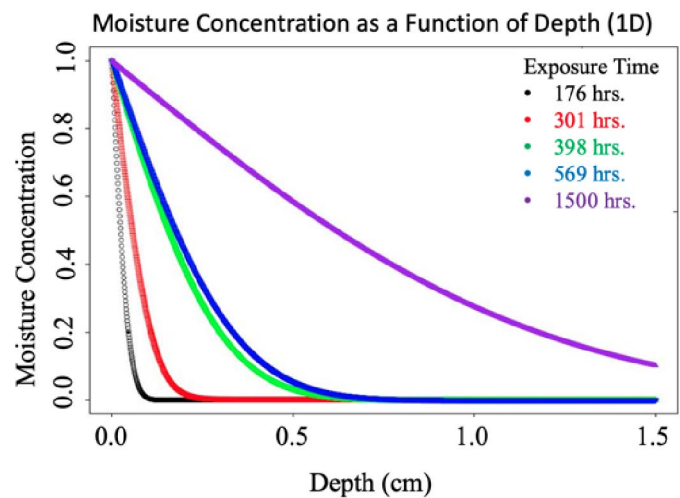


Fig. 2. The fitted curves using equation 1 for epoxy-encapsulated samples as a determination of a typical diffusion constant for moisture in the encapsulant. The purple curve representing 1500 hours is an extrapolation of the data up to that time to model the border region for chemical equilibrium required to pass the AIAA-S111 high humidity test.

found for a 2-part epoxy and the DC-93500 has a significantly lower moisture diffusivity, reducing this border, but this finding suggests that very large solar arrays comprised of perovskite absorbers could be fabricated to pass AIAA-S111 humidity testing.

We have acquired transmission spectra periodically across 880 hours of high humidity testing. Very little degradation is observed over time as evidenced by minimal changes in the transmission spectra. A representative sample is shown in figure 3 as a series of overlaid transmission curves for a sample containing no barrier coating. As can be observed there is minimal change in the transmission spectra. Further a representative sample containing an SiO_2 barrier and APTES surface modifier is shown in Figures 4 and 5, respectively. The APTES samples indicate, contrary to hypothesis, the most significant degradation, which is attributed to the process variability of silane deposition from a non-anhydrous solution. This additional moisture is likely the culprit for the emergence of the PbI_2 absorption band in the figure.

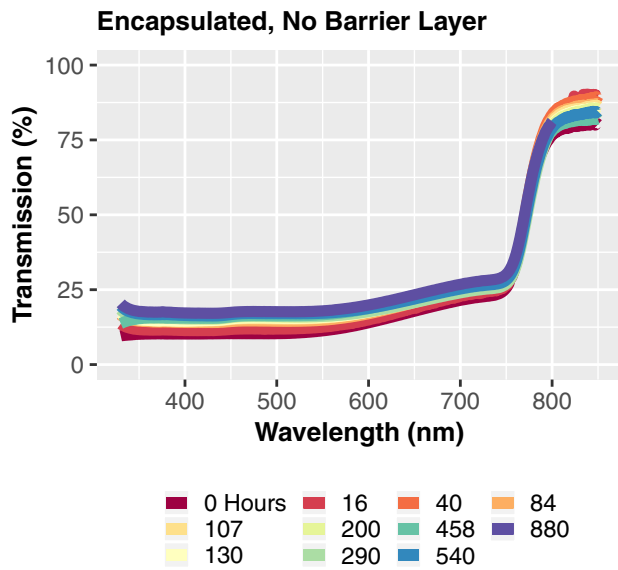


Fig. 3. A series of transmission spectra over time for perovskite films containing no barrier layers and encapsulated in space qualified, two-part silicone.

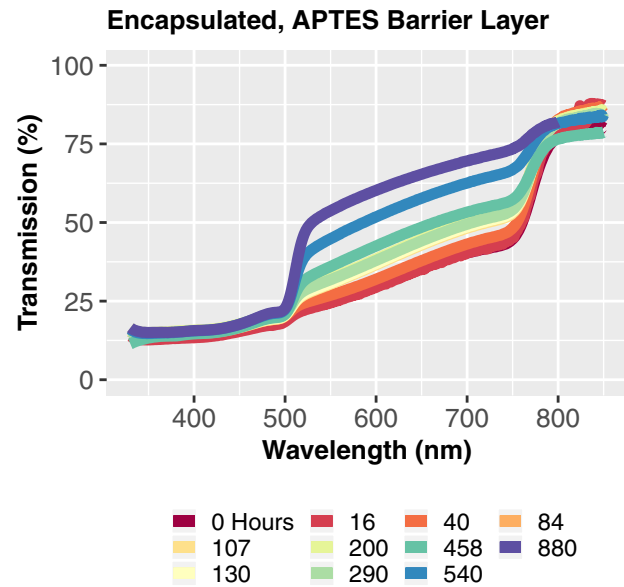


Fig. 5. A series of transmission spectra over time for perovskite films coated with a 50nm SiO₂ barrier layer.

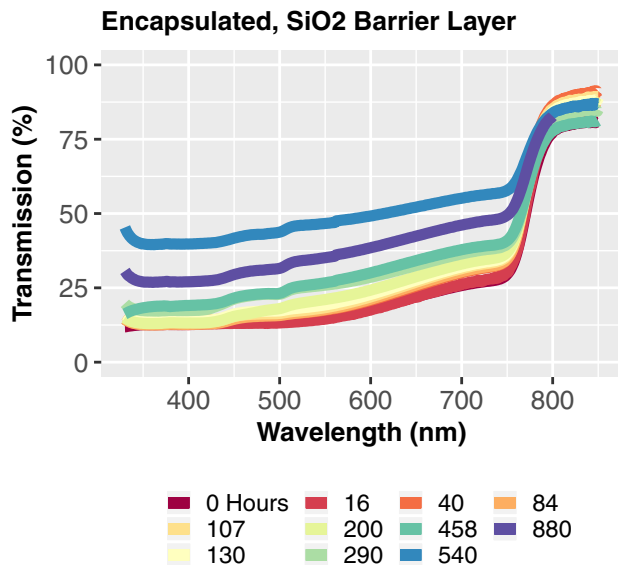


Fig. 4. A series of transmission spectra over time for perovskite films coated with a 50nm SiO₂ + APTES barrier layer.

IV. CONCLUSIONS

The preliminary data we have collected thus far suggest that perovskite material is durable enough to pass a space qualification humidity test when properly encapsulated and given a sacrificial boundary. We see very limited amounts of degradation in the material itself although recognize that morphology and reaction kinetics can be vastly different when the material is deposited in a typical device stack. Further the device performance is a more sensitive probe of degradation than transmission measurements, however this early-stage

data are encouraging when compared to the work of others performing similar measurements. Due to Covid-19 no further data has been collected on this project at the time of the conference.

ACKNOWLEDGMENT

The authors acknowledge the Space Technology Mission Directorate's Center Innovation Fund and Early Career Innovation Programs for support of this work. The authors acknowledge the Materials for Optoelectronic Research and Education (MORE) Center at Case Western Reserve University, and the Ohio Third Frontier program for establishing this core facility in 2010.

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