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CHEOPS – Production technology to achieve low Cost and Highly Efficient phOtovoltaic Perovskite Solar cells

Deliverable D2.2

Report on best encapsulation processes compatible with perovskite PV technology

WP2 – Upscaling and stability

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Contents

Acronyms

ALD	Atomic Layer Deposition
CHOSE	Università degli Studi di Roma Tor Vergata *(CHEOPS Beneficiary 4)*
CSEM	Centre Suisse d'Electronique et de Microtechnique *(CHEOPS Beneficiary 1)*
DH	Damp Heat
Fraunhofer	Fraunhofer Institute for Applied Polymer Research *(CHEOPS Beneficiary 6)*
HTL	Hole Transport Layer
IEC	International Electrotechnical Commission
OLED	Organic Light Emitting Diode
OPV	Organic Photovoltaics
PK	Perovskite
PV	Photovoltaic
UV	Ultraviolet
WP	Work Package

Executive Summary

This deliverable presents several encapsulation processes which were evaluated to allow PK based PV devices to pass the established stability tests.

Fraunhofer developed general processes for glass-to-glass encapsulation. Thin film encapsulation by ALD will also be considered by Fraunhofer IAP in the future to enhance the stability for larger modules, as it has produced interesting results for OLED and OPV. CSEM supported this development with its know-how on encapsulation polymers traditionally used in Si-technology. CHOSE developed an encapsulation using an adhesive tape.

All these encapsulated devices were tested under normal conditions at different values of storage temperatures and humidity. For encapsulation Fraunhofer IAP used a semi-automated encapsulation machine at Fraunhofer IAP's pilot line. The compatibility of the proposed encapsulation processes with the layers to be encapsulated also had to be carefully considered. For that the consortium discussed the requirements of the different lab and pilot PK based PV modules.

With these results it can be affirmed that the degradation is not determined by the encapsulation process. This is believed to be an important conclusion in regards to the stability of PK based PV devices.

Lastly, the basic understanding of encapsulation processes of perovskite solar cells, possible encapsulation processes and proposals of adhesives and tapes for glass-to-glass encapsulation were discussed.

The next step in the process will be the transfer of the encapsulation processes to larger scale modules measuring 15 x 15 cm.

1 General processes in an encapsulated device

Encapsulation is a method that determines the reliability of a component. In the field of organic electronics in particular, there is a wealth of knowledge on this topic, which can be applied directly to the encapsulation of PK based PV cells.

In this report, several encapsulation processes are evaluated to allow perovskite solar cells to pass the established stability tests. Fraunhofer IAP was responsible for the development and optimization of the encapsulation processes of PK based PV cells. Fraunhofer IAP, CSEM and CHOSE also developed their own special encapsulation methods for PK based PV cells up to 5x5cm.

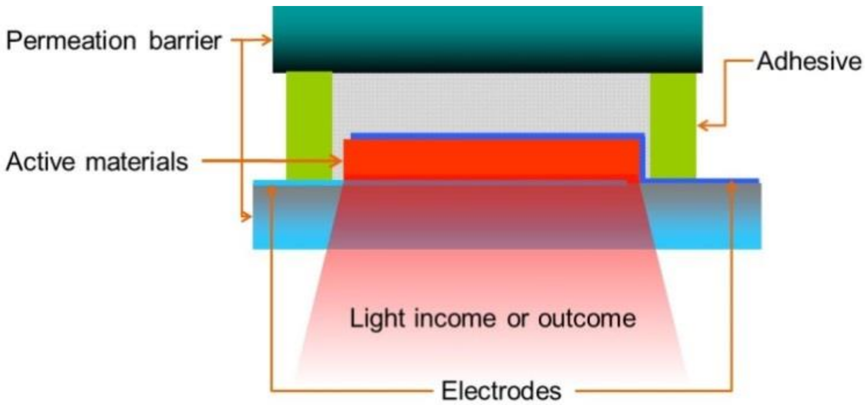

Figure 1. General set-up of an encapsulated device.

The model above (Figure 1) shows the critical parts in an encapsulated device. This included the gluing together of the surfaces of cover glass and, the studying of the adhesive behaviour of the adhesives themselves and the investigation of the processing conditions of the whole encapsulation process. All three factors influence each other.

1.1 Degradation mechanism

The degradation mechanisms are determined by different processes on each one of the device and corresponding layers. We are distinguishing the following layers and materials:

- Active Layer
- Electrodes
- Adhesives

1.1.1 Active Layer - Perovskite Materials

These layers are known to react with oxygen and/or water. Additionally, the stability is influenced by temperature and light and under maximum power-point conditions (MPP).

1.1.2 Electrodes

In these cells, calcium, aluminium and silver are mostly used as contacts and they, except calcium, are mostly stable under environmental conditions. Calcium is also used as a low work function electrode and it is known to react with water and oxygen in the following manner:

$2\,Ca + O_2 \rightarrow 2\,CaO$
$Ca + 2\,H_2O \rightarrow Ca(OH)_2 + H_2$
$CaO + H_2O \rightarrow Ca(OH)_2$

In order to prolong the lifetime of the solar cells, the stacking layers mainly need to be protected from oxygen and water. We discuss the most important mechanisms below.

1.1.3 Permeation through the adhesive

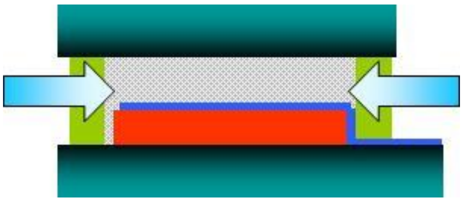

Figure 2. Important pathways for degradation of sealed perovskite device (adhesive).

With the assumption that the encapsulation adhesives have a thickness of about 10 µm and a width of 1 mm the adhesive-specific permeation rates are:

$7*10^{-5}$ g m/ (m^2 d) for H_2O
0.2 cm^3 m/ (m^2 bar) for O_2

These properties can be determined through calcium mirror tests. In this way, it is possible to:

- Evaluate the permeation through substrates, caps and adhesives
- Test if sample is completely sealed by the degradation of calcium
- Control the diffusion of water through the barrier layer and/or adhesive by restricting the filtering path
- Measure degradation of metallic calcium and the rate via light transmission when it forms $Ca(OH_2)$
- Optical transmission is correlated to layer thickness via calibration
- Transmission rate as function of time, temperature and humidity
- Lower test limit $5*10^{-6}$ g/(m²d)
- Test conditions depending on demands:
    - 23 °C @ 50% RH
    - 38 °C @ 90% RH,
    - 60 °C @ 90% RH,
    - 85 °C @ 85% RH.

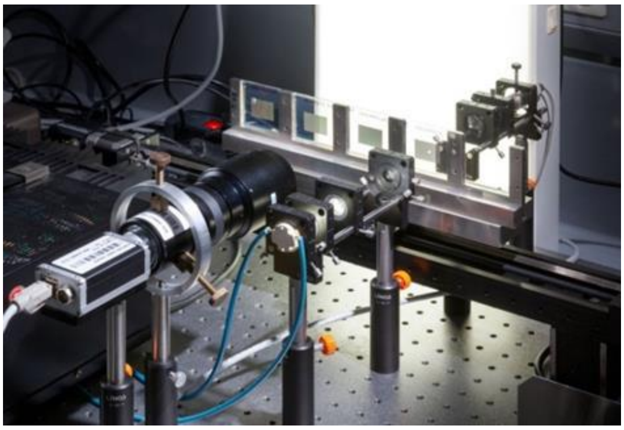

Figure 3. Calcium mirror set-up @ Fraunhofer IAP.

A typical investigation shows the reduction of the calcium mirror thickness.

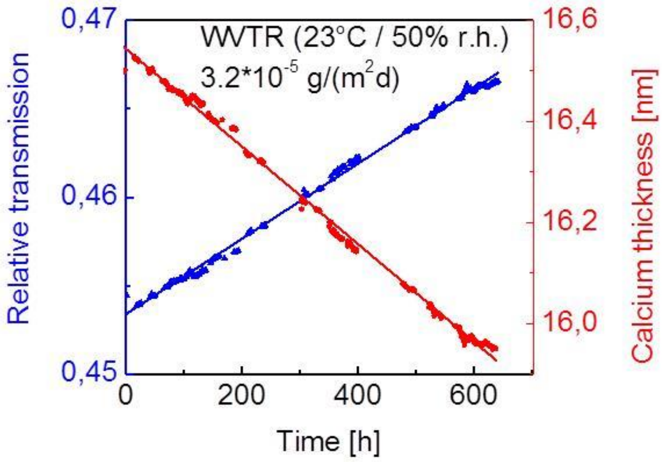

Figure 4. Determination of the water vapour transmission rate.

1.1.4 Permeation through Interfaces

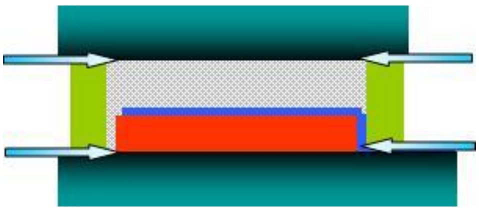

Figure 5. Important pathways for degradation of sealed perovskite device (Interface).

High energy surfaces, metals or oxides, in contact with the resin tend to bind, forming adsorbates and working as permeation channels. To prevent this from happening, the contact surfaces need to be cleaned before encapsulation.

1.1.5 Reverse Permeation

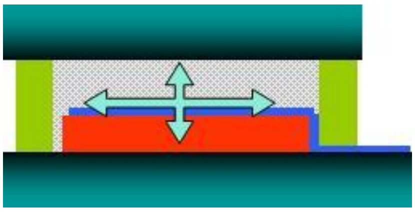

Figure 6. Degradation of sealed perovskite device (capsulated oxygen and moisture).

The typical water content of different materials of the following materials is: e.g. PET: 0.5 %, PC: 0.35%, PI: 2.9 %. Reducing the amount of water contained within the space of PK layers via encapsulation materials is critical for long term lifetime of an encapsulated device.

2 Encapsulation results

2.1 Encapsulation by Fraunhofer

Encapsulation adhesives need to have good barrier properties, optimized with respect to thickness and layer width.

For this purpose, various adhesives were examined by Fraunhofer IAP for their suitability.

- Araldite Araldite® 2011, thermal curing for full encapsulation
  - only stable up to 65 °C / 50 % RH
- ACW Glue AC A 1430, http://www.addisoncw.com/assets/Product-Data-Sheets/aca1430b.pdf
  - for edge encapsulation stable up 85 °C / 85 % RH
- Double adhesive tape from 3M or other supplier (CHOSE)
  - for full encapsulation stability is under investigations
- DELO 116135
  - for edge encapsulation stable up 85 °C / 85 % RH

Fraunhofer IAP preferred the DELO 116135 for further experiments.

The following steps were performed for encapsulation:

- Placing the cover substrates in UV press (window side)

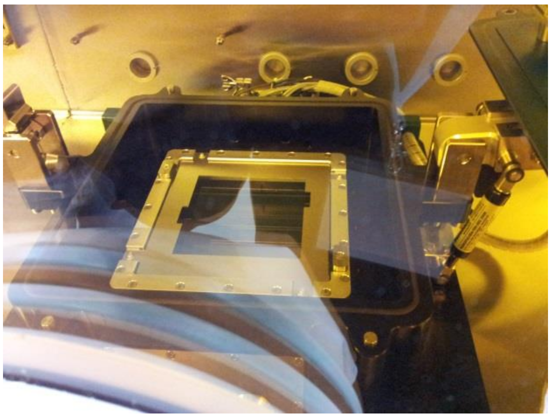

Figure 7. UV-press chamber (Fraunhofer IAP, pilot line).

- Optimization of the central position with the help of a glued frame
- Place the device with the calcium mirror or with the active device (with the device on top) in the middle position of the chuck (all other positions are taped)
- Application of the adhesive with the dispenser on cover substrates at marked points

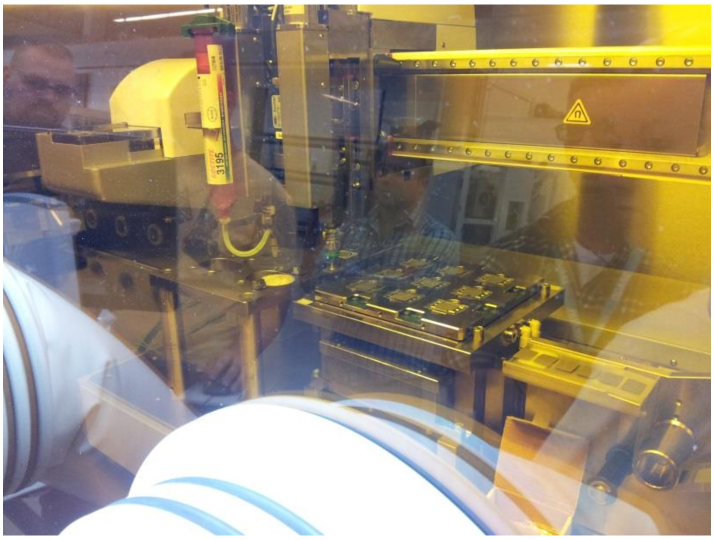

Figure 8. Dispenser for adhesive preparation (Fraunhofer IAP, pilot line).

- Assembly of cover substrate and substrate at low pressure to degas adhesive and avoid additional gas inclusions (gap height 1.9 mm / pre-pressure 500 mbar)
- Curing for 30 seconds at 1000 W with UV light (about 100-200 mW / cm² at 380-420 nm); Recommendation of Delo is about 50 mW / cm²
- Heat for 30 minutes at 80 ° C on hotplate

The compatibility of the adhesive with the different components in the active device was investigated in two ways. First, calcium mirrors were prepared, which were glued over the entire surface with the adhesive. The distance between active area and the edge of the glass was 1 mm or 2 mm, respectively. The adhesive thickness was adjusted to 15 µm by means of spacers. Second, the samples were stored under ambient conditions (23 °C/ 50 % relative humidity) and at elevated temperature and humidity (60 °C / 90 % relative humidity). The characterization was carried out by an optical measurement of the calcium, which allows a conclusion on the layer thickness and thus the degradation, also a possible defect formation was followed microscopically.

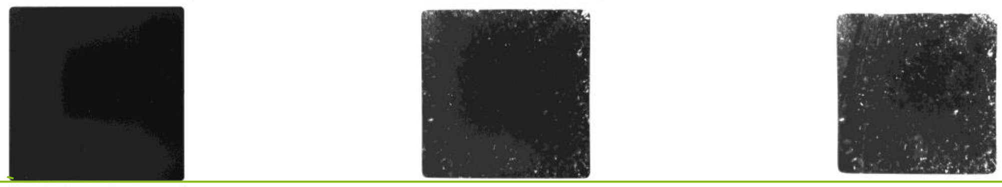

Figure 9. From left to right: Calcium-Mirror after 0 h, 72 h, 144 h (sample 160401117SK, <u>1 mm edge</u>, storage at 60 °C und 90 % rel. humidity).

Figure 10. From left to right: Calcium-Mirror after 0 h, 72 h, 144 h (sample 160401112SK, <u>2 mm edge</u>, storage at 60 °C und 90 % rel. humidity).

Figure 11. From left to right: Calcium-Mirror after 0 h, 72 h, 144 h (sample 160401116SK, 1 mm edge, storage at 23 °C und 50 % rel. humidity).

After storage at elevated temperature, pixel shrinkage is already visible after 140 hours, which is more pronounced for a narrow edge of 1 mm than for the wider edge of 2 mm. In the same period, no degradation of calcium is visible when stored under ambient conditions. Even after one month of storage time, no pixel shrinkage can be detected when stored under ambient conditions.

To test the compatibility of the adhesive with the active materials, with no possible external degradation effects, the encapsulated samples were first left in the glove box for 72 hours before being exposed to the test conditions.

2.2 Encapsulation by CHOSE

CHOSE used the following encapsulation process: Chose developed a lamination of the PK module using the Kapton adhesive tape deposited on the active area of the device. At first, light curable glue is deposited on the whole active area of the PK module. After that the positioning of a protective glass takes place as well as the curing of the light-curable glue under a class A simulator at 1 sun for 30 seconds followed by the deposition of a UV-curable glue on the edge of the protective glass. The encapsulation processes are finished by curing of the UV glue under UV irradiation for 40 seconds masking the active area of the PK module using a black adhesive.

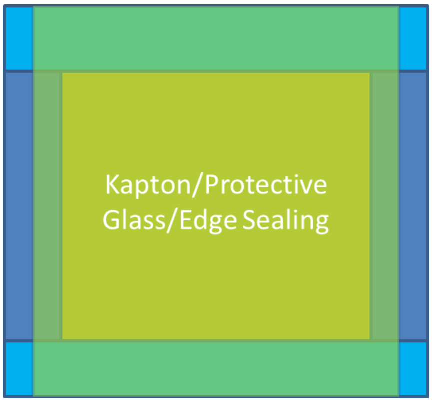

Figure 12. Sketch of the sealing procedure used by CHOSE.

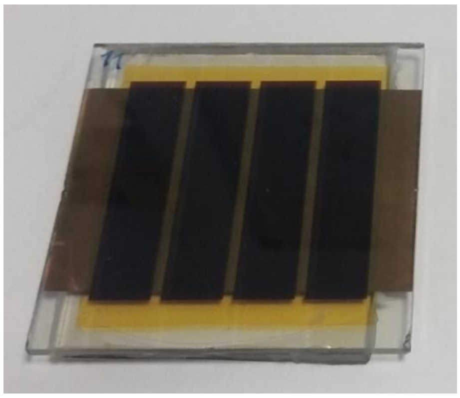

Figure 13. photograph of the encapsulated cells.

2.3 Encapsulation by CSEM

CSEM developed a glass/glass encapsulation scheme using a butyl rubber edge sealant. This encapsulation process can be used for both single-junction perovskite cells/modules and perovskite/silicon tandem cells. The water tightness of this encapsulation was optimized, leading to a highly reliable encapsulation for standardized reliability testing, as defined in D2.1 / T2.1.3. Importantly, appropriate processing conditions were found to avoid inducing damage to the perovskite cell during encapsulation.

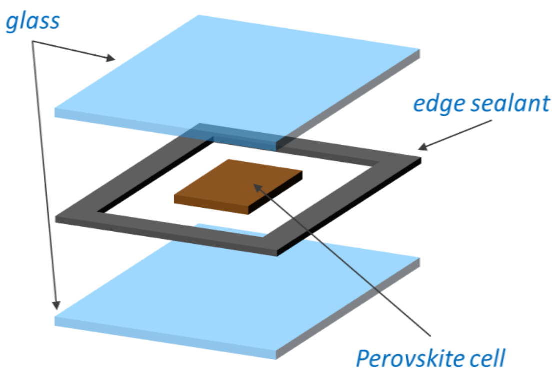

Figure 15. Sketch of the sealing procedure used by CSEM.

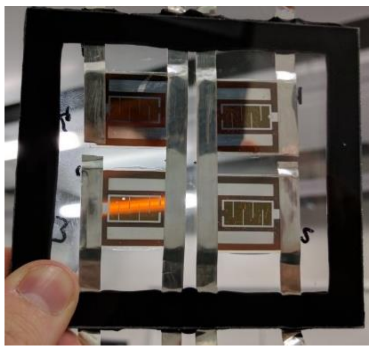

Figure 14. Photograph of the encapsulated cells.

3 Stability

3.1 Measurements at Fraunhofer IAP

Additionally, in the search for highly stable devices, Fraunhofer IAP has experimented with a perovskite mix that contains a mix of rubidium and caesium, together with the more common methylammonium and formamidinium. Long term testing of these devices has shown that they maintain more than 80 % of their initial performance after 70 days. These devices were kept on the dark, at open circuit condition, room temperature and inert conditions. The decline of performance over time is shown in the image below.

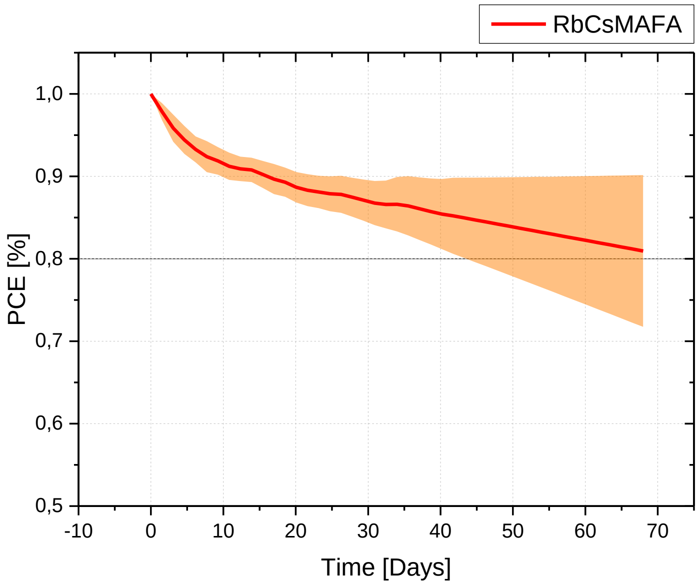

Figure 16. Degradation of perovskite solar cells made of RbCsMAFA of Fraunhofer IAP.

It should be noted that these devices were built in a p-i-n structure containing PEDOT:PSS as hole transport layer (HTL). It is known that this material is not stable on the long term due to its water retention.

3.2 Measurements at CHOSE

The results of CHOSE showed the degradation at 30 °C and 40 % relative humidity under light exposure at MPP conditions. The efficiency decreased under these conditions from 13 % to 9.1 % after 350 h.

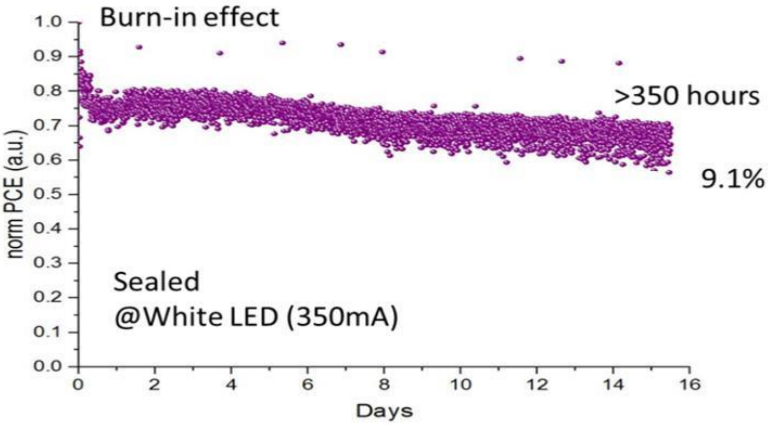

Figure 17. Degradation of perovskite mini module of CHOSE.

3.3 Measurement at CSEM

First results of damp heat (DH) degradation tests (1000 h at 85% relative humidity/85°C) on PK devices encapsulated with this process are shown. These results show that after 1000 hours of DH the cells degrade by less than 10% relative, which is still far from IEC standard, but still encouraging as the selected process is common in the PV industry and holds great promises for future upscaling.

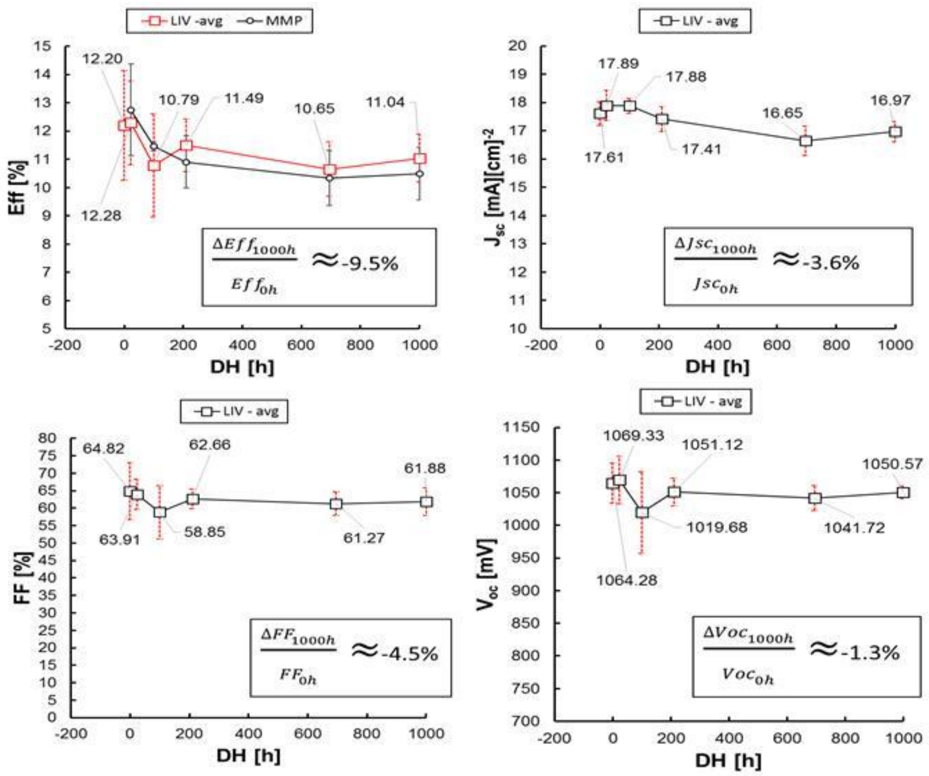

Figure 18. Degradation of perovskite mini module of CSEM.