nanoQI project reports

03/2020 - 08/2023; Multimodal X-ray and Hyperspectral Thin-Film Nano-material Evaluation and Quality Imaging; grant agreement ID: 862055

- Verification of performance of HSI in evaluation of ALD coatings
- Verification of performance of XRR and HSI as Quality control in industrial R2R vacuum coating
- Verification of in-situ observation of perovskite layer curing and quenching and surface imaging on > 20 x 20 cm^2 on moving substrate (0.5 m/min)

Verification of performance of HSI in evaluation of ALD coatings

Please find the full text in the attachment.

This deliverable is part of the public dissemination;

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The HSI system was implemented in the glovebox of the S2S pilot line at Fraunhofer IAP. Different series of oxide depositions (Al_2O_3 , TiO_2) on silicon wafers as reference, glass and PEN substrates on sizes up to 150 mm x 150 mm were prepared to evaluate their thickness, homogeneity and morphology and to elaborate the sensitivity of the setup and compared with SoA offline systems. ALD layers of different composition were prepared on different substrates (Si-wafers as reference, glass, polymer substrates) on sizes up to 150 mm x 150 mm. The spatial homogeneity of these layers was evaluated by HSI and compared to off-line characterization by optical spectroscopy, lab-based XRR (FhG-FEP) and spectroscopic ellipsometry and synchrotron based spatially-resolved XRR (resolution few 100 µm, TUDO). This spatial information from off-line characterization is obtained by the investigation of different sample areas, which was be correlated to the HSI results. Furthermore, a “quasi in-situ” test on Al_2O_3 and TiO_2 with nominal deposition differences of 1 or 2 nm were prepared and measured to get information of the sensitivity of the HSI to thickness changes as low as 1 nm. Another test was prepared with Al_2O_3 deposition with conditions out of the optimum process conditions. Besides the thickness evaluation the samples were investigated with XPS to determine remaining carbon content and sense how sensitive HSI can image insufficient process conditions. In order to integrate the HSI into the daily work at FhG-IAP in processing of flexible electronic stacks several functional layers deposited by spincoating were investigated to which extend complex structures will be sensitive for HSI investigations such as complete OLED layer stacks including the ALD thin film encapsulation layer (together with NEO). Thus, we were able to characterize layers of PEDOT:PSS, an often used charge carrier material in organic electronic devices, as well as PVK (poly N-vinylcarbazol), a functional polymer, which is part of the active layer in OE devices.

Verification of performance of XRR and HSI as Quality control in industrial R2R vacuum coating

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The deliverable deals with the verification of the HSI as a valuable tool for roll-to-roll inline processes. This is demonstrated with the example of different single layers and one double layer system. Details are given in the corresponding chapters. Ground truth data came from different sources, among them also X ray Reflectometry. The general procedure of machine learning is presented in section 2.1.The examples for single layer systems and double layer systems are shown in the sections 2.3 and 2.2, respectively.

In the content of the deliverable various quantitative numbers are given:

1. a set of surface images of a roll need to be taken from a roll
2. a roll section of 100 m x 0.6 m
3. R2R processing at 2 m/min possible
4. 4-layer stack: individual layer thickness and its deviation with sensitivity of ± 1 nm.

Even though these four criteria had not been demonstrated together, the work for this deliverable dealt with all of them. Images of the roll had been taken. Due to aperture restrictions at the coFlex 600, the maximum width of observation was 0.49 m. This value can easily be adapted to 0.6 m in a production machine. More than that, the scaling of resolution with observation width, achievable under normal circumstances can be evaluated based on the project results.

The maximum total length of 275 m was investigated with the HSI system in the roll-to-roll machine. This was done with the partner roll from NORD. The HSI inspection was demonstrated at a maximum line speed of 6.4 m/min. The partners have developed a general understanding of the deformation of the field of view, in dependence of the interaction between the frame rate and line speed. This deformation of the field of view is not limiting the information provided by HSI. However, the understanding is necessary to identify the exact position which a measurement signal is related to.

Considering all the difficulties which had been faced during the installation, the four-layer stack seemed to be too complex for the evaluation approach of NanoQI. More details and a guideline for a modified approach in a possible follow up project are given in section 2.4.

Verification of in-situ observation of perovskite layer curing and quenching and surface imaging on > 20 x 20 cm² on moving substrate (0.5 m/min)

Please find the full text in the attachement.

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This delievrable describes the validation and demonstration of hyperspectral imaging (HSI) and x-ray diffraction (XRD) analytical tools for process and quality control in perovskite solar cell manufacturing. The XRD and HSI hardware and software are used to evaluate characteristic properties of perovskite coatings, such as thickness, roughness and phase composition. Partial least square regression (PLS) quantification models are developed to correlate the in-line HSI signals with the film properties.

Perovskite films are deposited on 30 cm x 40 cm glass substrates by slot die coating of the precursors ink, followed by vacuum quenching and thermal annealing in a semi-automated pilot line. Hyperspectral images are recorded during the film quenching and after a complete curing cycle (quenching and subsequent annealing). XRD spectra are measured after complete curing of the samples on several points of the sample. Thickness and roughness are measured by means of off-line techniques.

By incorporating the perovskite layers characterized with the NanoQI tools in a complete p-i-n device architecture, semitransparent solar cells were fabricated and their photovoltaic parameters were determined with a solar simulator at standard testing conditions.