Advanced x-ray characterization of halide perovskites at EMIL: Progress and Planning

R.G. Wilks,[1,2] C. Hartmann,[1,2] N. Llobera-Vila,[1,2] E. Handick,[1,2] R. Félix,[1,2] S. Gupta,[3] G. Hodes,[3] D. Cahen,[3] M. Bär[1,2]

[1]Renewable Energy, Helmholtz-Zentrum Berlin für Materialien und Energie GmbH (HZB), 14109 Berlin, Germany
[2]Energy Materials In-Situ Laboratory Berlin (EMIL), Helmholtz-Zentrum Berlin für Materialien und Energie GmbH (HZB), 12489 Berlin, Germany
[3]Department of Materials and Interfaces, Weizmann Institute of Science (WIS), Rehovot, 7610001, Israel

Using a variety of lab- and synchrotron-based x-ray spectroscopic techniques, the "Interface Design" group at the HZB has been actively studying the electronic and chemical structures of mixed halide perovskites in recent years. In the last year, the group has also moved toward the study and synthesis of (wide-gap) Pb-free, Sn-based perovskite materials. Cells using Sn-based absorbers have thus far exhibited only a relatively low efficiency, possibly due to the easy oxidization from Sn^{+2} to Sn^{+4} producing deep defects in the absorber material. Adding SnF_2 during preparation can inhibit this oxidation; however, the mechanism through which SnF_2 acts and its impact on the electronic structure is not fully understood. To identify the roles of SnF_2 treatment, halide composition, and deposition route, thin films of spin-coated $CsSnBr_3$/compact- TiO_2/FTO/glass (WIS) and UHV prepared $CsSnCl_3$/Mo/glass (HZB), were characterized by hard x-ray photoelectron spectroscopy (HAXPES). The vacuum deposition of $CsSnX_3$ films was performed at the Energy Materials In-Situ Laboratory Berlin (EMIL), which connects various large-scale and laboratory-scale deposition tools to a wide-energy range synchrotron beamline, allowing advanced characterization using soft and hard x-ray spectroscopies of materials produced in the system while avoiding environmental exposure – a key capability when studying Sn-based materials. A detailed study of vacuum-deposited $SnCl_2$ precursor layers of different thicknesses by lab-based x-ray photoelectron and Auger electron spectroscopy reveals significant changes in the chemical environment of Sn and Cl along the layer profile. Further studies will be used to monitor in situ perovskite formation, similar to what was previously performed with $CH_3NH_3PbI_{3-x}Cl_x$; where temperature-dependent HAXPES measurements of precursor materials could monitor the film formation and directly observe the conversion to perovskite.
We will report on our past and recent spectroscopic studies of halide perovskites, present the current status (and challenges) of preparing Pb-free, Sn-based perovskite materials at EMIL and suggest future steps.