Electron Backscatter Diffraction Analysis of Beam Sensitive Samples Using Direct Detection Technology

Matthew M Nowell¹ and René de Kloe²

Electron Backscatter Diffraction (EBSD) is a well-established Scanning Electron Microscopy (SEM)based technique for characterizing the microstructure of crystalline materials with applications on materials including metals, semiconductors, ceramics, and geological materials. However the analytical conditions used for typical EBSD data collection can cause damage to beam sensitive samples, both during the EBSD collection process and during SEM imaging prior to EBSD. The use of direct detection technology enables collection of useable EBSD patterns at conditions suitable for analysis of these types of samples[1].

Traditionally EBSD patterns are obtained using a phosphor screen to convert electrons into light photons. Optical coupling is then used to capture and focus these photons onto an imaging sensor, and read out this signal as a digital image for further processing. Both the phosphor screen and optical coupling have inefficiencies that are avoided with direct detection. There is zero read noise with direct detection, and this enables single electron detection sensitivity and operation at lower beam doses. Beam currents in the 1-15nA range and acceleration voltages in the 15-30kV range are used for typical EBSD analysis. Direct detection can collect EBSD patterns and maps down to beam currents of approximately 10pA and down to acceleration voltages of 3kV.

Direct detection also allows for more efficient collection at lower acceleration voltages. With phosphorbased collection, the efficiency of photon to light conversion decreases with decreasing electron beam energy. Operation at lower acceleration voltages reduces the energy introduced into the sample and helps minimize sample damage in beam sensitive samples. It also reduces the interaction volume of the electron beam within the sample and improves the spatial resolution of EBSD collection. This can be important for analysis of fine-grained samples, where larger step sizes during EBSD mapping would not be sufficient to resolve the microstructure.

Hybrid organic-inorganic perovskite solar cells have been analyzed with direct detection [2-3]. These samples are very challenging due to their beam sensitive nature and general instability over time. EBSD collection, and even SEM imaging, can be difficult on these samples at typical EBSD acquisition conditions. Lower beam doses using direct detection have been successfully used to characterize the microstructure. Results from different methylammonium lead halide films acquired using a Clarity direct detector will be presented, and the role of acquisition parameters discussed.



^{1.} EDAX LLC., Pleasanton, CA USA.

^{2.} EDAX B.V., Tilburg, The Netherlands.

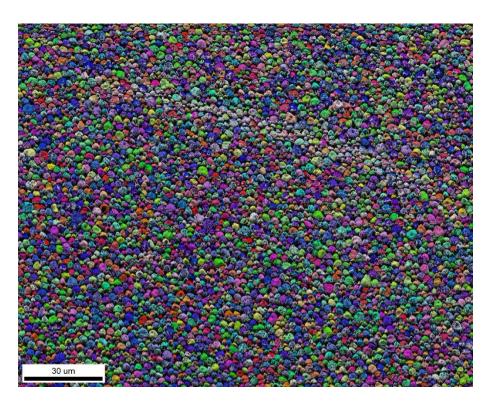


Figure 1. EBSD IPF Orientation Map of $MaPbI_3$ perovskite solar cell measured with Clarity direct detector.

References:

- [1] AJ Wilkinson et al., Physical Review Letters 111 (2013), p. 065506.
- [2] S Jariwala et al., Joule **3** (2019), p. 1.
- [3] G Adhyaksa et al., Advanced Materials **30** (2018), p. 1804792.