Large-area through-thickness TEM analysis of thin film solar cell absorbers using double-wedge geometry

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Thin film systems play a key role in many modern technologies, like sensors, coatings, electronics and photovoltaic. Often it is the combination of vertical film structure and lateral homogeneity which determine the properties of a thin film and its usefulness for a specific application. In order to evaluate the vertical and lateral film structure both spatially averaging techniques, like X-ray diffraction, and microscopic techniques are employed. Among the microscopic techniques transmission electron microscopy (TEM) is particularly useful because it can be applied in cross-section geometry allowing to directly image the vertical film structure. Moreover, larger areas of the film can be analyzed in plan-view geometry. By combining both geometries a qualitative picture of the three-dimensional (3D) film structure can often be deduced. However, in cases where the film shows lateral variations on the length scale of micrometers (or above), like in polycrystalline films with relatively large grains, the combination of cross-section and plan-view analysis is not sufficient for deducing the 3D film structure. In these cases a cross-section sample shows a too small electron transparent area to be representative for the vertical film structure.

We have recently devised a new sample preparation technique that overcomes these limitations by a double-wedge sample geometry that probes different depths in the film and separates depth information in a continuous series of thin slices that are spread over a lateral distance [1,2]. Using this sample geometry the thin film structure can be studied at each depth below the surface on large areas by plan-view TEM providing statistically relevant data on the 3D film structure.

Here we apply the technique to CIGSSe thin film solar cells based on the chalcopyrite absorber material Cu(In,Ga)(S,Se)₂. Such solar cells are promising candidates for reducing the costs of photovoltaic because they combine a small material and energy input with a relatively high cell efficiency [3]. Figure 1a shows a typical cross-sectional TEM image of a solar cell structure consisting of a metallic contact layer (marked "1"), a fine-grained intermediate layer (marked "2") and the polycrystalline absorber (marked "3" to "5"). Across the absorber the grain size increases significantly making it difficult to obtain a representative picture of the film structure in cross-section geometry. In the lower part of the absorber (marked "3") pores can be seen which, however, appear enlarged due to preferential thinning during TEM sample preparation. Figure 1b illustrates the double-wedge sample geometry employed for large-area through-thickness TEM analysis (for details on the sample geometry, see [1,2]). Fig. 1c depicts selected plan-view TEM images and diffraction pattern taken along the edge of a double-wedge sample. The images represent different depths in the film as indicated by the corresponding numbers in the cross-section image. In our contribution we will show how the evaluation of many of such plan-view images and large-area diffraction patterns can be used for obtaining statistically relevant information on the 3D structure of CIGSSe thin film solar cells.

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Figure 1. a) Cross-section TEM bright-field image of a CIGSSe solar cell structure. b)~Schematic of the double-wedge sample geometry for large-area plan-view imaging at each depth in the film. The position of the film-substrate interface is indicated by a yellow line. Please notice the very small angle of the front side bevel (~ 0.2°) which corresponds to a "stretching" of the film along the edge by a factor of ~ $300 (= 1 / \tan 0.2^{\circ})$. c) Selected bright-field TEM images and diffraction patterns taken along the edge of a double-wedge sample. The numbers 1 to 5 also shown in figure part a) indicate the depths in the film system represented by the plan-view images.