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Authors: Anna Frank, Rasa Changizi, Christina Scheu

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Challenges in TEM sample preparation of solvothermally grown $CuInS_2$ films

Anna Frank[a], Rasa Changizi[a], Christina Scheu[ab]*

[a] Max-Planck-Institut für Eisenforschung GmbH Düsseldorf, Nanoanalytics and Interfaces, Max-Planck-Straße 1, 40237 Düsseldorf.
[b] RWTH Aachen, Kopernikusstraße, Aachen.

[*] corresponding author: c.scheu@mpie.de

Highlights

- Various TEM sample preparation techniques have been applied to $CuInS_2$ films
- FIB preparation shows strong influence on the chemical composition analysis of $CuInS_2$
- Scratch samples allow accurate determination of the elemental ratios
- Cross sectional preparation makes precise film thickness measurement possible
- Only combination of preparation techniques enable full characterization of $CuInS_2$

Abstract

Transmission electron microscopy (TEM) is a widely used tool to characterize materials. The required samples need to be electron transparent which should be achieved without changing the microstructure. This work describes different TEM sample preparation techniques of nanostructured $CuInS_2$ thin films on fluorine-doped tin oxide substrates, synthesized solvothermally using L-cysteine as sulfur source. Focused ion beam lamellae, conventional cross section samples and scratch samples have been prepared and investigated. It was possible to prepare appropriate samples with each technique, however, each technique brings with it certain advantages and disadvantages. FIB preparation of solvothermally synthesized $CuInS_2$ suffers from two main drawbacks. First, the whole $CuInS_2$ layer displays a strongly increased Cu content caused by Cu migration and preferential removal of In. Further, electron diffraction shows the formation of an additional CuS phase after Ga^+ bombardment. Second, diffraction analysis is complicated by a strong contribution of crystalline Pt introduced during the FIB preparation and penetrating into the porous film surface. The conventional cross sectional $CuInS_2$ sample also shows a Cu signal enhancement which is caused by contribution of the brass tube material used for embedding. Additionally, Cu particles have been observed inside the $CuInS_2$ which have been sputtered on the film during preparation. Only the scratch samples allow an almost artefact-free and reliable elemental quantification using energy-dispersive X-ray spectroscopy. However, scratch samples suffer from the drawback that it is not possible to determine the layer thickness, which is possible for both cross sectional preparation techniques. Consequently, it is

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