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FIB-SEM and HRTEM investigations of microstructure of chalcopyrite CuAlS₂ thin films

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We investigate the microstructure of chalcopyrite CuAlS₂ thin films deposited on silicon (111) substrate. The polycrystalline films with different preferred grain orientations are grown by thermal evaporation from pure powder of synthesis deposited in elemental sources. The CuAlS₂ films were analyzed using focused ion beam scanning electron microscopy (FIB-SEM) and high-resolution transmission electron microscopy (HRTEM). FIB-SEM cross-section images reveal that the irregular-shaped particles are embedded in the film and that the surface region and the bulk are structurally similar, with no ordered defect chalcopyrite structure. However, their composition is slightly different, indicating that they can have different point defects. Microstructure properties of the films using HRTEM highlight grains in (112) textured films with sharp contrasts at the grain boundaries, whereas grains in (220/204) textured films give only weak contrasts indicating a preferential population of electronically rather inactive grain boundaries. Non-chalcopyrite ordering of the metal atoms in CuAlS₂ is observed by HRTEM, which is identified as a CuAu-type ordering. Sharp spots in electron diffraction patterns show the ordered Cu and Al atomically planes alternating along the [001] direction over a long range. The CuAu-ordered structure coexists with the chalcopyrite ordered structure, in well agreement with theoretical prediction. This study shows that dual beam FIB-SEM technique turns out to be an easy, less time consuming and useful method to characterize the crystallites of CuAlS₂ films in cross-section view, compared with the results obtained by HRTEM.

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