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An in situ-EDXRD study of reactively co-sputtered Cu(In,Ga)S2 layers

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Reactive and non-reactive magnetron sputtering is widely used in the photovoltaic industry but mostly for the deposition of electronically inactive layers (front and back contacts). Recently, it was shown that $CuInS_2$ thin films prepared by co-sputtering from a Cu and an In target in an Ar/H_2S atmosphere yield solar cell efficiencies of more than 11%. However, the co-sputtering process showed certain instabilities which have to be studied in detail. Therefore, we investigated the growth process of reactively sputtered $Cu(In,Ga)S_2$ (CIGS) layers *in-situ* by means of time resolved energy-dispersive X-ray diffraction (EDXDR). For that purpose our sputtering chamber was mounted on a goniometer at the synchrotron radiation source HASYLAB (Hamburg). A white X-ray beam in the energy range of about 6 to 60 keV combined with an energy-dispersive Ge detector was used to monitor the crystal growth. By analyzing both, diffraction peaks of the grown phases as well as fluorescence peaks, the film growth and the phase formation was monitored time resolved.
Several CIGS layers have been deposited at varying substrate temperatures and different sputtering rates from the CuGa and In targets. Special attention was payed to the influence of the substrate type (Si/SiO_2/Mo, Si/SiO_2TiN, Si/SiO_2) and an applied substrate bias voltage. In the EDXRD spectra, directly after the start of the sputter deposition, a rising diffraction signal attributed to the (112) lattice planes of CIGS is observed. The energetic position of this peak changes significantly during the experiments. This can be attributed to different Ga contents in the growing CIGS film. Especially interesting is the nucleation stage of the layers. Depending on the substrate temperature and the substrate type the condensation of In, monitored by the InK_α fluorescence signal, starts immediately after the start of sputtering or is delayed by several minutes in dependence of deposition temperature and substrate surface. In the case of the delayed condensation of In, the formation of pure $CuGaS_2$ on the substrate is observed, which is later converted into $Cu(In,Ga)S_2$ with increasing In content, indicated by a change of the peak position.

Keywords
reactive Magnetron Sputtering, in-situ EDXRD
Chalkopyrite Films, Thin Film Solar Cells