Report on EXAFS measurements at the Dubble beamline (ESRF)

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Goal of experiment

The goal of the experiments was to investigate the local environment and the temperature-induced modifications of Sn atoms in metastable $Ge_{1-x}Sn_x$ thin films, using Sn K-edge XAS. This study can shed light on the underlying physical mechanisms of temperature-induced relaxation processes such as Sn segregation and agglomeration observed in metastable GeSn films and to microscopically understand the way energetic interactions may modify the structural and compositional properties of these films.

Samples

1) Amorphous GeSn layers of 80 nm with 4.5% and 11.0% Sn were fabricated on silicon substrates and crystallized at 500˚C. Some samples were doped with trace amounts of Ga (~ 10^{18} atoms/cm^3).

2) Crystalline GeSn layers of 30 nm and 100 nm with 5.7% and 4.3% Sn, respectively, were epitaxially grown on germanium substrates by molecular beam epitaxy. The samples received different temperature treatments between 400˚C to 700˚C.

3) Metallic Sn (foil) and SnO (powder) were used as a reference.

Preliminary results

The low Sn concentrations (8×10^{15} – 3×10^{16} Sn atoms/cm^2) together with the low photon flux on the sample at these high energies (~30 keV) complicated the measurements. Additionally we encountered problems such as X-ray scattering, diffraction from the crystalline substrate and a high background signal. All measurements were performed in grazing incidence to increase the exposure of the GeSn thin layer to the X-ray beam and to reduce the background signal arising from the substrate. Notwithstanding these experimental challenges we were able to measure 9 (7 samples + 2 reference samples) samples of our 27 samples.

We were able to detect fluorescence from the Sn K-edge, and scattering from nearest neighbors in the GeSn layers.

We have measured amorphous and crystalline GeSn layers and can see a clear difference in EXAFS signal, which follows from a difference in lattice spacing between the amorphous and crystalline phase. Furthermore we observe the formation of Sn clusters in samples with 11.0% Sn content after annealing

at 500 ˚C. For samples with 4.5% Sn there was no observation of Sn clustering before and after annealing.

The introduction of trace amounts of Ga (~ 10^{18} atoms/cm^3) during deposition has significant influence on the crystallization process: the presence of Ga leads to polycrystalline GeSn instead of single crystalline GeSn. The XAFS measurements show that the presence of Ga does not lead to the formation of Sn clusters before and after annealing.

Conclusion

Our measurements were successful and demonstrate for the first time the possibilities of XAFS measurements for the characterization of the local environment of Sn in metastable GeSn thin films. Having proven the feasibility of such experiments, the study can be extended to a broader set of relevant $Ge_{1-x}Sn_x$ thin film systems, providing perspective of future Sn K-edge XAFS measurements at DUBBLE.