ESRF	**Experiment title:** In situ study of the size dependence of the phonon density of states in the alfa- and beta-Sn phase in thin films and application to nanoscale superconductivity	**Experiment number**: **HE-1352**
Beamline: ID18	**Date of experiment**: from:10/02/2014 to:11/03/2014	**Date of report**: 15/03/2015
Shifts: 27	**Local contact(s)**: D. G. Merkel	*Received at ESRF:*

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Report:
The aim of the experiment was to study the phase transition from alpha-Sn to beta-Sn in thin films. More specifically, the aim was to investigate the role of the vibrational entropy during the phase transition in Sn. Hereto, alpha-Sn thin films needed to be grown in the UHV setup at the beamline. We had brought our own Knudsencell and crucible with ^{119}Sn, together with a power supply for the Knudsen cell. After practising how the transfer mechanism works of the UHV chamber to move the samples from one chamber to another, the Knudsencell was installed and the setup was baked out. On 17/02/2014 the Knudsencell was degassed to make a sample. However, the power supply could not deliver enough power to reach the desired temperature with the Knudsencell and when changing the power supply, the filament of the Knudsencell was broken. The Leuven Knudsencell was replaced by an ESRF Knudsencell, and a second bake-out was started. After this bake-out, the RHEED gun, Auger electron gun and the Ar sputtering gun were tested (which were needed for cleaning of the InSb substrate and testing the quality of the substrates).

4 alpha-Sn films were grown in total: 40nm, 20nm, 10nm and 5nm alpha-Sn on an InSb substrate. We started with measuring the 40nm sample. However, the alignment of this sample failed (probably due to curvature of the InSb substrate) and we continued with the 20nm sample. This sample was cooled down to 130 K and a reflectivity curve was measured. At this temperature, NIS scans were performed and a time spectrum was measured. From the PDOS that was extracted from the NIS scans, the sample was found to be in the alpha-Sn phase. The sample was gradually heated up and a time spectrum was measured at 310 K, 320 K, 330 K. The sample was then cooled down again to 130 K and NIS scans were measured. The sample was heated up again and a time spectrum was measured at 335 K, 340 K, 350 K, 360 K, 370 K, 390 K and 430 K. Big changes in the time spectrum at 430 K were observed and the sample was cooled down again to 130 K and NIS scans were measured. From the PDOS that was extracted from these NIS scans, the sample was found to be in the beta-Sn phase. The same procedure was repeated for a 10nm alpha-Sn film. For this sample, the transition from the alpha-Sn to the beta-Sn phase took place at 450 K (instead of 430 K for the 20nm sample).

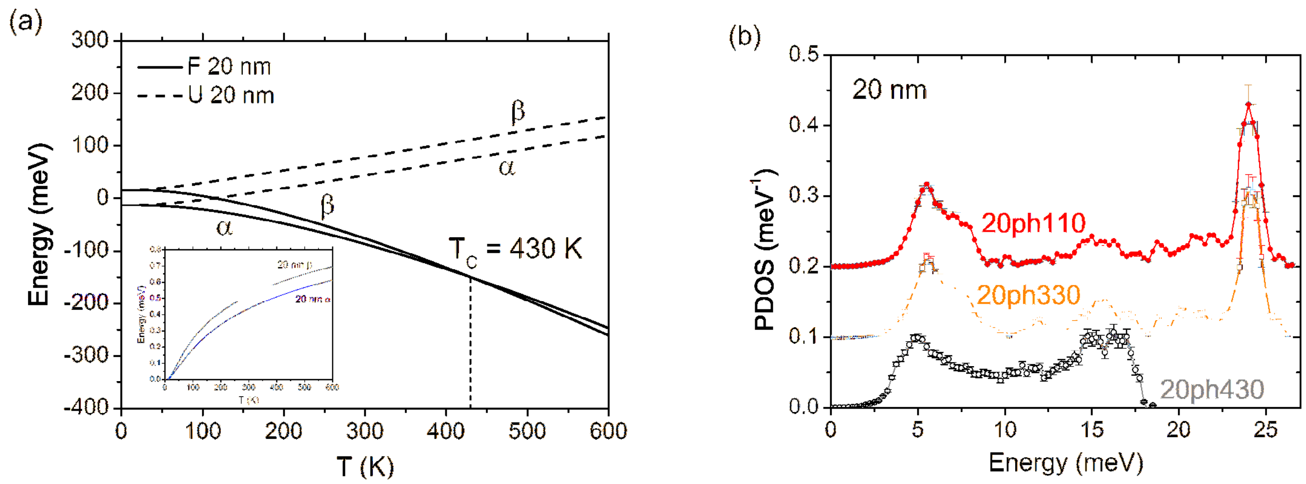

Figure 1: *(a) Internal energy and free energy as a function of temperature (inset: Vibrational entropy as a function of temperature) for the alpha-Sn and beta-Sn phase, obtained by numerical integration of the PDOS; (b) PDOS for the 20nm sample measured after heating the sample up to different temperatures.*

In Fig. 1(b) the phonon densities of states measured for the 20nm sample are shown. By numerically integrating these PDOS, the internal energy and vibrational entropy per atom as a function of temperature can be obtained (see Fig.1(a)). From these quantities, the free energy per atom can be calculated. Also for the 10nm sample, these energy curves were calculated. From these energy curves for both samples, it was concluded that the vibrational entropy plays a major role in the phase transition from alpha-Sn to beta-Sn. However, for alpha-Sn layers stabilized on an InSb substrate, the substrate stabilization energy has to be taken into account, which depends on the film thickness. It is the film thickness that determines at what temperature the phase transition will take place. These results will be the subject of an article which is in preparation.