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Report:

Tin (Sn) has been comprehensively studied at high pressure and high temperature. Its phase diagram has been explored using synchrotron x-ray diffraction techniques to probe the crystallographic structures, using the diamond anvil cell to generate static high pressure. A transformation from the ambient β -Sn structure to the high pressure body-centered tetragonal (bct) polymorph is known to take place at 9.8 GPa. The phase boundary has a negative Clapeyron slope up to the β -bct-liquid triple point at 3.8 GPa and 580 K. While the static high pressure phase diagram is well established, the structural observation of each phase and their corresponding phase transition boundaries undergoing rapid dynamic compression has been a challenge. Shock compression experiments using sound velocity measurements have shown that transitions are observed on compression at ~1 GPa higher, and ~2 GPa lower on release, than the static phase boundary. The shock compression experiments have been limited to ambient temperature. With the upgraded ID09 of the EB-ESRF, fine details in the XRD spectra could validate the phase transition mechanism. By pre-heating the targets various compression paths can be explored and enable looking at the liquid-solid phase transition, thus giving access to the first study of the recrystallization mechanisms in tin under shock compression.



Figure 1: Integrated x-ray diffraction pattern (black points) with a LeBail refinement (red line) at 8 ns delay, taken at $\lambda = 0.8266 \text{ Å}$. The low-pressure β phase and the high pressure γ phase are both distinctively observed.

The target assembly and the compression schemes were designed so as to obtain very reproducible single-shot laser-pump/X-ray probe data with a 0.1 ns resolution time delay, to follow different compression paths covering a large domain of the Sn phase diagram below 15 GPa and to perform accurate XRD measurements. The sample consisted of a 15 μ m thick foil of poly-crystalline bismuth (Goodfellow, 99,9% purity) glued to a 100 μ m thick sapphire on the laser side and to a 100 μ m thick silica window on the other side. The sample assembly was mechanically pressed in the sample holder with a calibrated force to approximately 1 μ m in thickness and the epoxy glue layer was then UV cured. The targets have thus been designed to operate in a so-called confined geometry which allows to achieve a stable warm and dense state of a 2-3ns lifetime and then to probe the shock release. Three starting temperatures, 300 K, 420 K and 500 K, of the compression path were studied by pre-heating the targets with a transportable resistive heater. Figure 1 shows the quality of the obtained signal, allowing to distinguish unambiguously between the two phases.



Figure 2: Pressure evolution in the sample for the different phases as a function of time delay. The experimental results are compared with the result of an hydrodynamic simulation using the ESTHER software.

Using the equation of state of the different phases, one has access to the pressure of the sample at a microscopic level thanks to the XRD. An example of result of the evolution of pressure as a function of time delay is presented in figure 2. Due to the large thickness of the sample, an homogeneous state is never achieved and the low-pressure β phase is always observed even at the peak compression. Pressure evolution from the γ -phase is in good agreement with hydrodynamic simulations.

Interestingly, the γ -phase is observed at much lower pressure than expected, both during the compression and the release. This could be observed thanks to the high quality of the diffraction signal which allowed to detect that a small amount of the sample had already transited towards the high-pressure phase.

Additional work is now ongoing to look at the results obtained at high temperature.