



	Experiment title: Ms-scale time-resolved X-ray diffraction of Tin (Sn) at high pressure and high temperature	Experiment number: HC4467
Beamline: ID24	Date of experiment: from: 16/06/2021 to: 21/06/2021	Date of report: 24/08/2021
Shifts: 18	Local contact(s): Matteo Levantino	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Charles Pépin*, CEA; Florent Occelli*, CEA; Ramesh André*, CEA ; Paul Loubeyre*, CEA		

Report:

Scientific background

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. Intermediate strain rates can now be achieved with a dDAC and coupled to x-ray diffraction, allowing studying the compression rate dependency of the phase transition. Moreover, working with a DAC allows preheating the sample and thus exploring the high temperature part of the phase diagram to study the dependency of the recrystallization of tin with the compression rate.

Experimental technique

A dynamic diamond anvil cell set up developed in our laboratory shown in fig. 1 has been installed on the beamline. The delay generator was synced with the internal clock of the ESRF. XRD measurements have been performed at ~ 19 keV. Three different diamond anvil cells had been prepared in advance in our laboratory. The cells had been prepared so as to be easily reloaded with new samples on site.

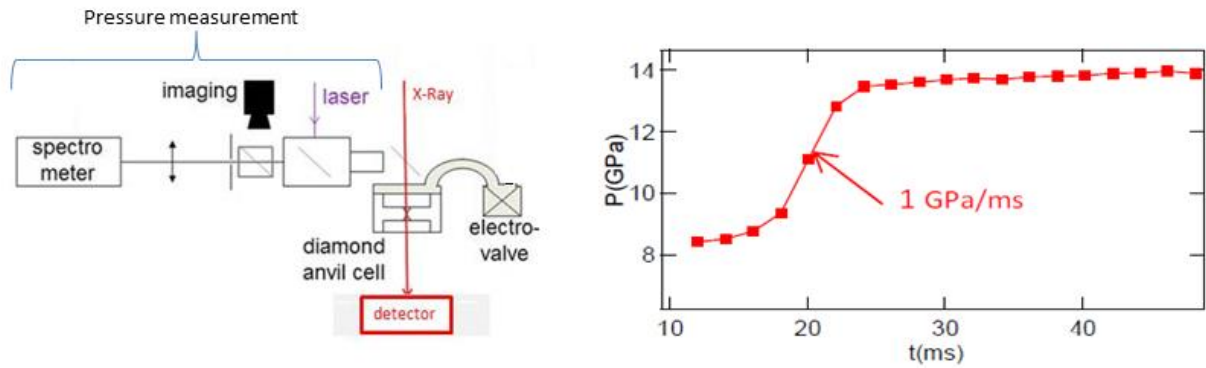


Figure 1: Schematic view of set-up and of the ramp compression over time

Results

11 different Sn-samples have been successfully loaded during the beamtime. Preheating of the sample was performed at 400, 450 and 550 K. As seen in figure 2 and 3, the phase transitions going from beta-Sn to gamma-Sn and from liquid to gamma-Sn can be well identified.

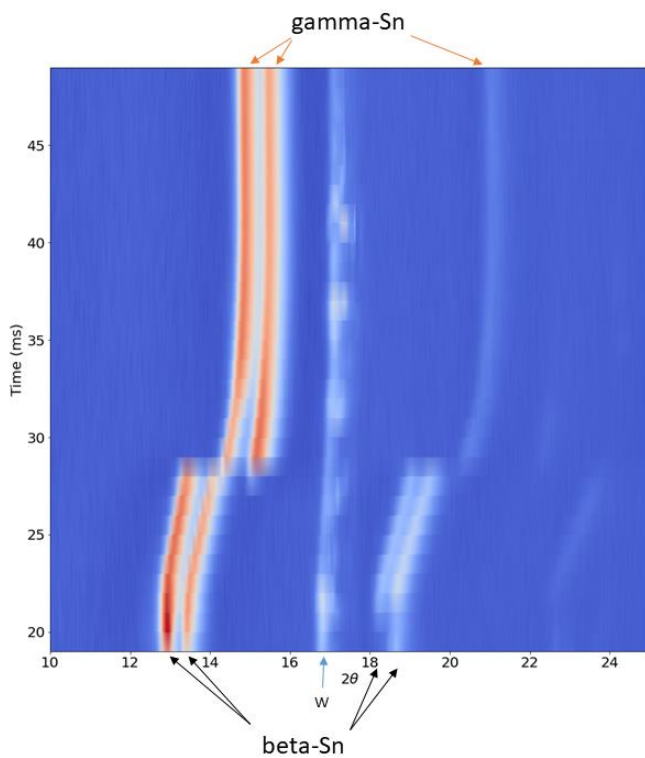


Figure 2: Dynamic compression of beta-Sn at 300 K

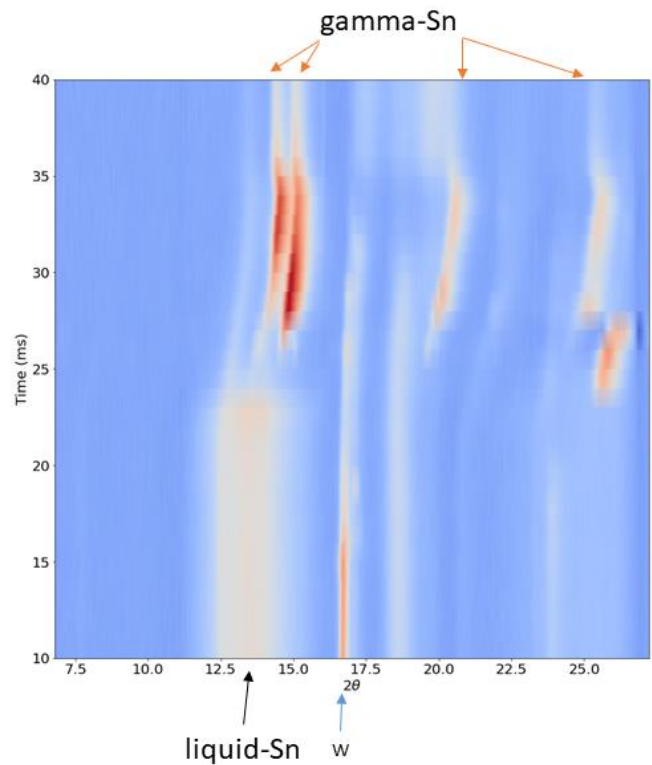


Figure 3: Dynamic compression of liquid-Sn at 550 K

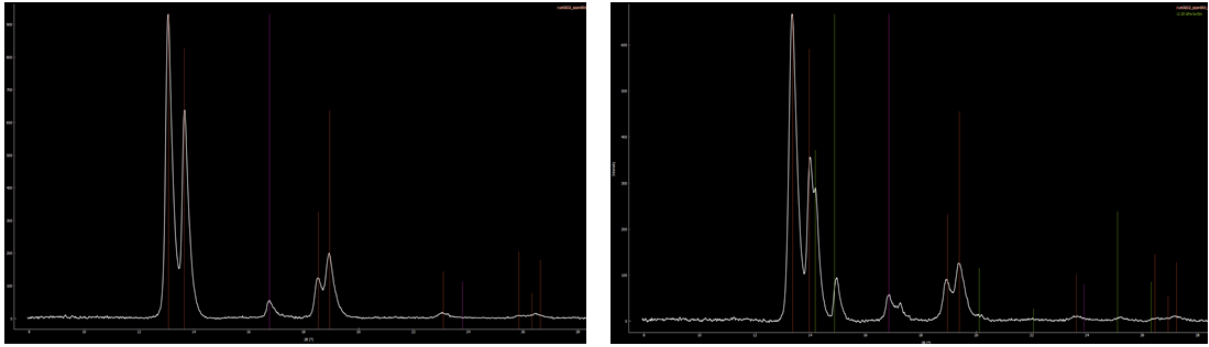


Figure 4: Example of diffractograms for the starting material and at the beta-gamma phase transition

Figure 4 shows an example of diffractograms obtained with the jungfrau detector. Some additional work is now needed to precisely determine the pressure during the dynamic compressions of the samples.