ESRF	**Experiment title:** **Melting Studies of Sn at High Pressure using Laser-Heated Diamond Anvil Cells and X-ray Diffraction**	**Experiment number**: HS-3834
Beamline: ID-27	**Date of experiment**: from: 17/07/2009 to: 21/07/2009 from 30/10/2009 to: 31/10/2009	**Date of report**: 01/03/2010 (second report)
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Report:

We investigated the melting line of Sn through laser heated diamond anvil cell (DAC) experiments conducted at ID27 of the European Synchrotron Radiation Facility in July and October 2009. Sn powder samples were pre-pressed into foils and small pieces of these were loaded into membrane DACs together with NaCl as pressure medium. The gasket material employed was Re (pre-indented to 30 μm) and single bevelled diamond anvils with 150 / 300 μm culets were used. NaCl had been dried in a furnace for several days before being used in experiments and all loadings were carried out in glove boxes to avoid contamination / oxidation of the sample and pressure medium. Samples were first compressed to a desired pressure at room temperature (using the NaCl pressure medium diffraction lines as pressure gauge) before being laser heated on both sides through YAG lasers. YAG lasers were slightly defocused to provide a uniform hot spot on both sides of the sample. Simultanous x-ray diffraction patterns and thermal emission spectra were then recorded every few seconds from a 2 μm x 3 μm area at the centre of the hot spot while the laser power is gradually increased. Melting was identified by the appearance of liquid $S(Q)$ scattering in the

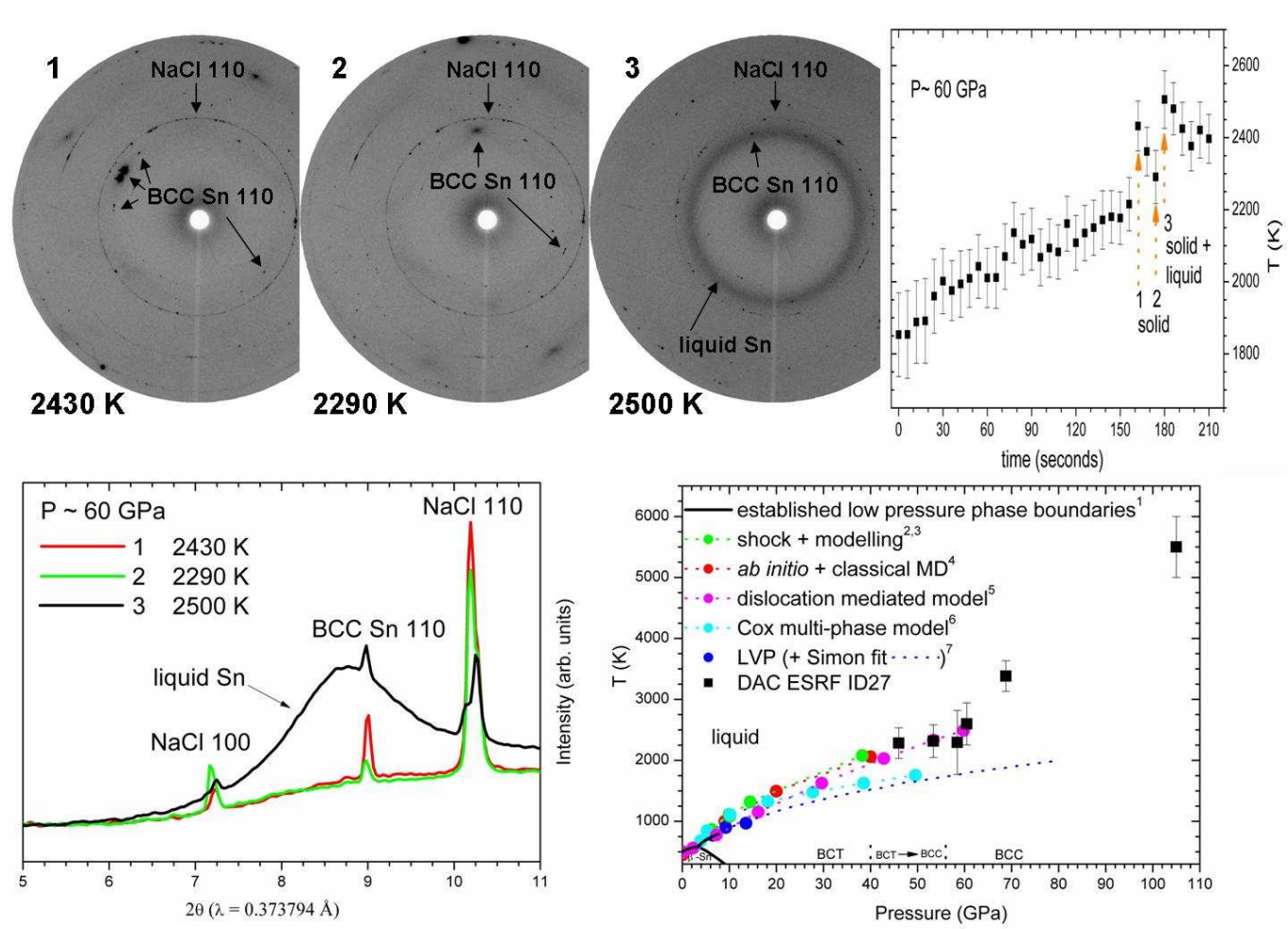

Figure 1 Top left: *Sequence of raw x-ray patterns recorded from centre of laser hot spot on Sn sample held in DAC at ~60 GPa, laser power continuously increases.* ***Top right:*** *Planck fits to thermal emission spectra recorded from same area of sample, temperatures corresponding to raw x-ray patterns shown on left indicated.* ***Bottom left:*** *integrated intensities corresponding to raw x-ray patterns – the appearance of liquid scattering features is evident in both raw and integrated data.* ***Bottom right:*** *Sn melting line data obtained in the present study plotted together with data from previous investigations.*

recorded diffraction patterns. Such a sequence of raw diffraction patterns and associated temperature ramp (Planck fits to corresponding thermal emission spectra) are shown in the top panel of figure 1, recorded at ~ 60 GPa as the heating laser power is gradually increased. The appearance of liquid scattering features at ~2500 K is clearly visible in both the raw x-ray diffraction patterns (top left panel of figure 1) and the integrated patterns (bottom left of figure 1). The raw x-ray patterns show rapid re-crystallisation of BCC Sn as the temperature nears the melting point, as is expected at the crystal – liquid equilibrium. The patterns also showed that no chemical reaction of the sample with the sourrounding medium or impurities (e.g. O) had occured as all diffraction lines before and after laser heating can be assigned exclusively to either NaCl or Sn.

Our melting points of Sn obtained according to the above method are plotted together with some other previous data in the bottom right panel of figure 1.[1] In the range 50 – 60 GPa our data agree well with simple

extrapolations of the Sn melting lines reported from shock investigations (in combination with modelling[2] and temperature measurements[3]) and from a combination of *ab initio* and classical MD[4] simulations. The melting line obtained from the dislocation mediated model[5] also intersects our data in this range. Melting lines obtained from the Cox multi phase model[6] and those from a fit by a Simon law to low pressure large volume press (LVP) data[7] predict melting points which are much lower than those obtained in the present study.

A question remains, however, in how far the BCT – BCC transition (reported between 40 – 56 GPa at room temperature[8, 9]) influences the shape of the melting line. The Cox model does not account for this transition and a Simon fit to the LVP (BCT) data is only appropriate up to the BCT – BCC – liquid triple point. Similarly, although the other data in figure 1 agrees better with our reported melting points none of these data account for the BCT – BCC transition (and associated BCT – BCC – liquid triple point) either. This might explain the discrepancies between the low pressure LVP data and these data sets. A change in the slope of the melting line at the BCT – BCC – liquid triple point might also explain why our higher pressure data (~70 and ~100 GPa) indicates a much steeper melting slope (of BCC Sn) than that predicted by extrapolating (any of) the existing data sets. In the data presented we only observe melting from BCC Sn suggesting that the BCT – BCC – liquid triple point is below ~40 GPa. We have some lower pressure data where we did not observe melting (the temperatures did not go high enough) and we are currently carefully re-examining all these diffraction patterns to see if they can provide some information about the BCT – BCC transition at elevated temperatures.

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