ESRF	**Experiment title:** Russian grant: Synthesis of new high-temperature conductors based on hydrates at high pressures and study their structure and properties.	**Experiment number**: HC-2601
Beamline: ID27	**Date of experiment**: from: 11.12.2015 to: 13.12.2015	**Date of report**: 28.02.2018
Shifts: 6	**Local contact(s)**: Mohamed Mezouar.	*Received at ESRF:*

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Report:

Series of X-ray diffraction experiments with Sn (tin) samples in H2 medium at high pressures in the range of 118-225 GPa was carried out at the ID27 ESRF (European Synchrotron Center) station. X-ray difraction were taken on a narrow-focus (FWHM = 1.7 × 2.3 μm) high-energy monochromatic beam (33 eV, λ = 0.3738Å) using a two-dimensional MarCCD 165 detector at the distance of 192.55 mm, with an exposure of 30-90 s. Two-dimensional X-ray diffraction images were analyzed and integrated into one-dimensional radiographs in the Dioptas program (ver.0.2.4) [1]. A full-profile analysis and calculation of the parameters of the unit cells was performed in the JANA2006 program [2] using the LeBail method [3].

Figures 1a and b show the evolution of X-ray diffraction patterns from Sn under pressure in two diamond anvils cells with low (DAC # 2) and high (DAC # 1) hydrogen content, respectively. Comparison of the diffraction data suggests that the formation of tin hydride occurs under pressure in the DAC # 1 chamber (Fig. 2). The test sample in the cell with a low hydrogen content can be considered pure Sn.

The X-ray diffraction patterns of Sn (DAC # 2) at pressures up to 158 GPa are indicated in the body-centered cubic Sn-bcc (body centered cubic) cell with the space group Im-3m (Sn-x = 0, y = 0, z = 0) (Fig. 3a). At a pressure of 158 GPa, a weak reflex appears at 2Θ = 10.36 ° (d = 2.07Å), the intensity of which increases over the entire pressure range up to 225 GPa.

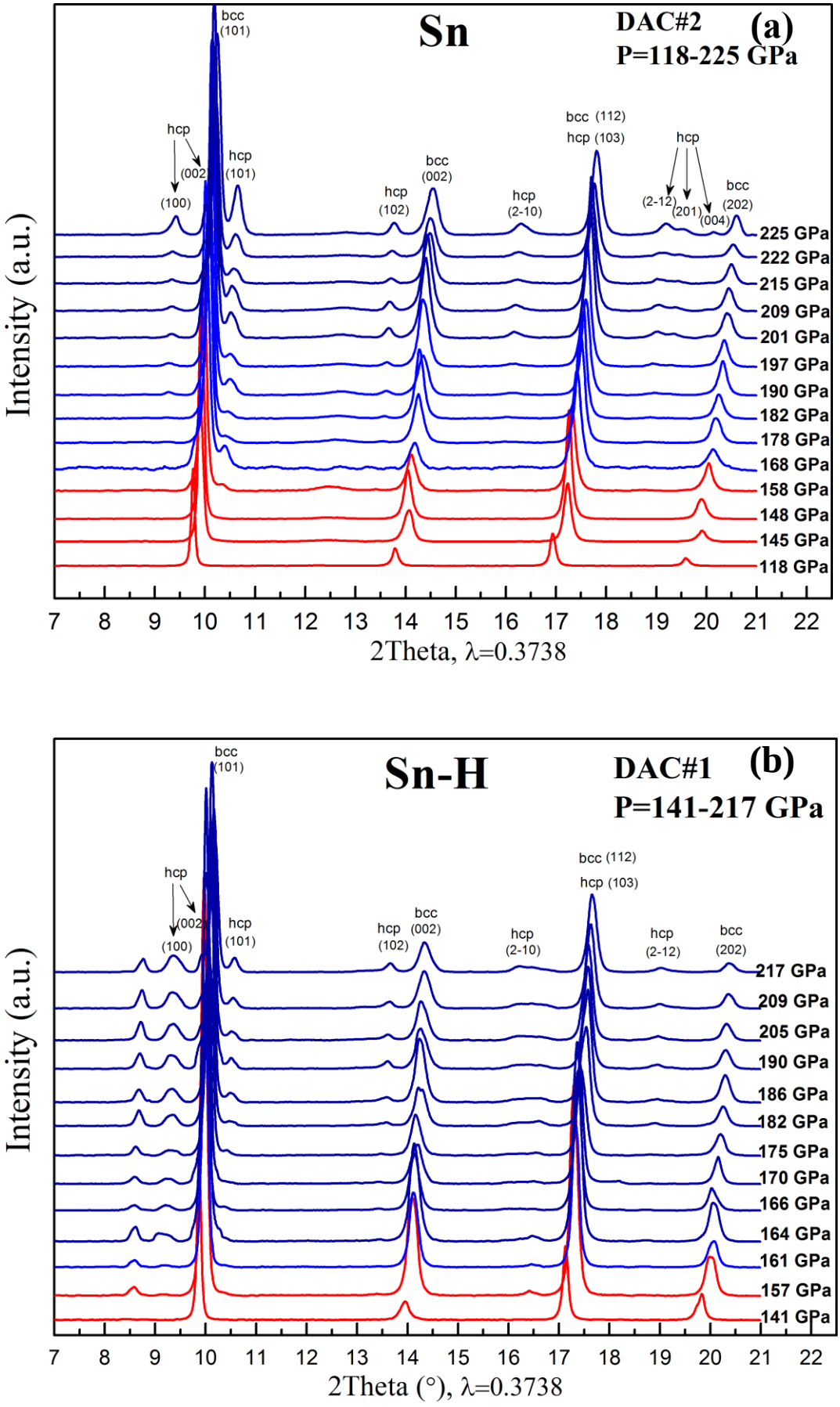

Fig. 1. X-ray diffraction patterns taken at different pressures from samples of Sn in H2 in DAC # 2 (a) and DAC # 1 (b).

At 190 GPa, reflexes appear at the angles 2Θ = 9.25 ° (d = 2.32 Å) and 2Θ = 13.60 ° (d = 1.58 Å) on X-ray diffraction patterns, and at a range of reflexes 2Θ = 9.98 ° (d = 2.15 Å) at 16 GPa, 16.13 ° (d = 1.33Å), 19.02 ° (d = 1.13Å), 19.02 (d = 1.11Å). All additional reflexes are indicated in the structure with a hexagonal dense packing of Sn-hcp (hexagonal closed packed) (prg P63 / mmc, Sn-x = 1/3, y = 2/3, z = 1/4), which previously was calculated and detected in Sn at pressures up to 194 GPa [4]. The results of the refinement (Fig. 3b) demonstrate good convergence of the experimental and model diffraction patterns and confirm the coexistence of two Sn phases with a cubic and hexagonal structure in the pressure range 160-225 GPa (Fig. 3b). The calculated parameters of elementary cells for the sample Sn at different pressures and the graph of the dependence of the volume of unit cells on pressure are given in Table 1 and in Fig. 4. accordingly.

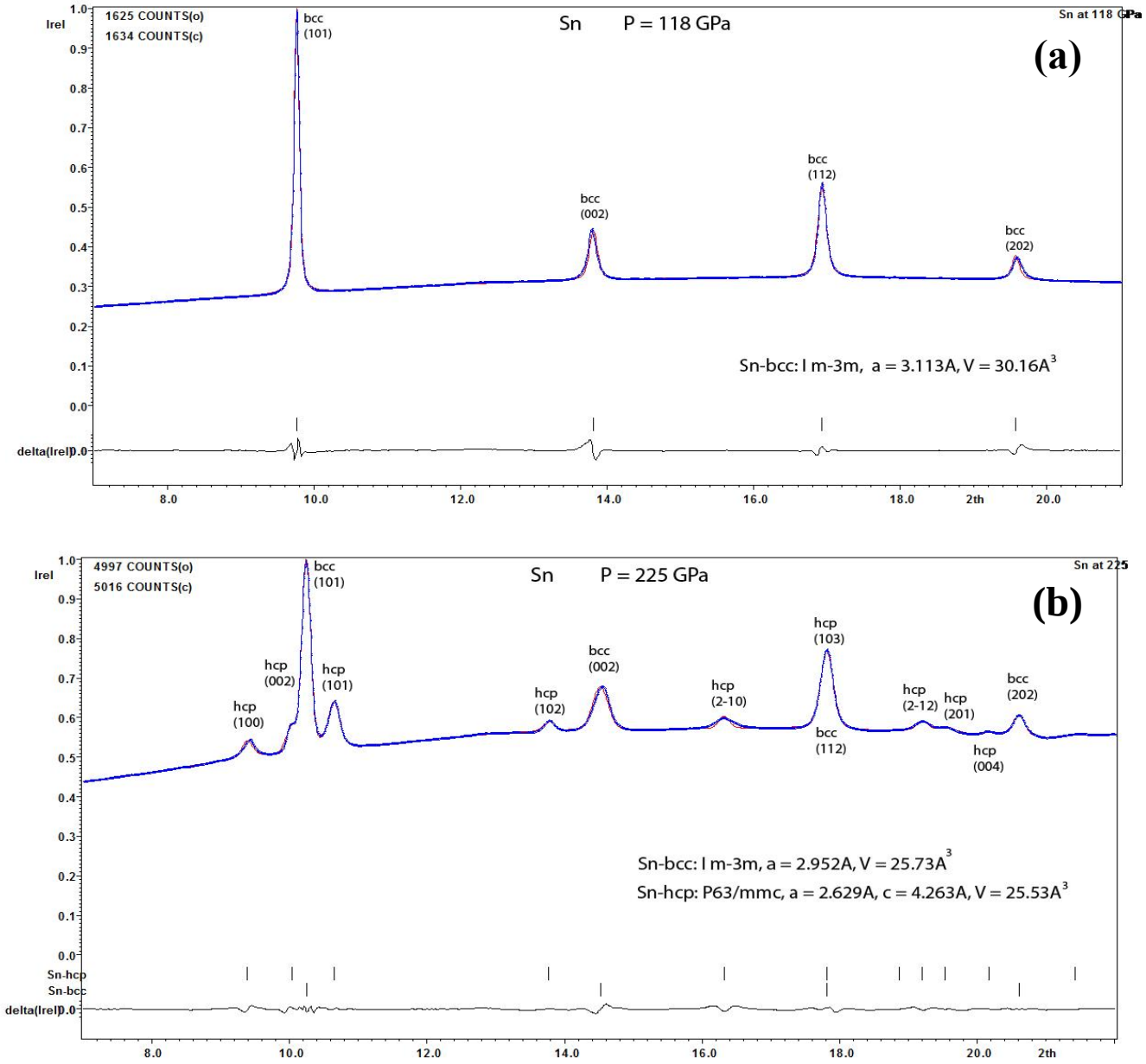

Fig. 3. Refinement by the LeBail method of the Sn structure in the DAC # 2 (a) -cubic body-centered Sn-bcc structure at 118 GPa, (b) in the cubic bulk-centered Sn-bcc structure and the Sn-hcp hexagonal structure at 225 GPa.

X-ray diffraction patterns of Sn-H in DAC # 1 (Fig. 1b) to 157 GPa are indexed in a cubic body-centered cell. (Im-3m, a = 3.0742 (4) Å, V = 29.053 (4) Å3) At 157 GPa, the first appears a reflex with 2Θ = 8.6 ° (d = 2.5 Å), absent on the X-ray diffraction patterns of tin in DAC # 2. At pressures above 164 GPa, a pronounced bifurcated reflex to 2Θ = 9.3 ° appears. The remaining reflexes corresponding to the hexagonal phase of Sn-hcp appear from 180 GPa. Figure 3b shows a two-dimensional diffraction pattern and an integrated x-ray pattern of the Sn-H sample from the DAC # 1 chamber at 217 GPa. The presence of an additional peak in the near field of angles and a slightly different character of the evolution of X-ray diffraction patterns under pressure can indicate the formation of SnH4 tin hydride with increased unit cell parameters. In the future, decoding and refinement of the structure of tin hydride is planned to continue using ab initio calculations and the Rietveld method.

Table 1. Calculated parameters of elementary cells of cubic (bcc) and hexagonal (hcp) phases of Sn under pressure in chamber DAC # 2.

Pressure GPa	*Sn-bcc*		*Sn-hcp*		
	a_{bcc}, Å	V_{bcc}, Å^3	a_{hcp}, Å	c_{hcp}, Å	V_{hcp}, Å^3
118	3.1126(4)	30.156(4)	--	--	--
145	3.0490(4)	28.345(4)	--	--	--
148	3.0491(6)	28.348(5)	--	--	--
158	3.0394(6)	28.078(5)	2.727(7)	4.290(3)	27.627(6)
168	3.0205(6)	27.557(5)	2.722(3)	4.301(2)	27.602(6)
182	3.0069(6)	27.188(4)	2.700(7)	4.272(2)	26.979(4)
190	2.9963(4)	26.903(5)	2.677(2)	4.313(1)	26.774(6)
197	2.9896(4)	26.720(4)	2.681(5)	4.255(3)	26.503(4)
201	2.9786(4)	26.426(4)	2.660(1)	4.285(1)	26.263(4)
209	2.9757(4)	26.351(4)	2.656(1)	4.283(1)	26.178(4)
215	2.9629(5)	26.012(5)	2.641(1)	4.280(1)	25.866(5)
222	2.9599(4)	25.932(4)	2.638(1)	4.274(1)	25.766(4)
225	2.9522(6)	25.730(5)	2.630(7)	4.263(1)	25.533(6)

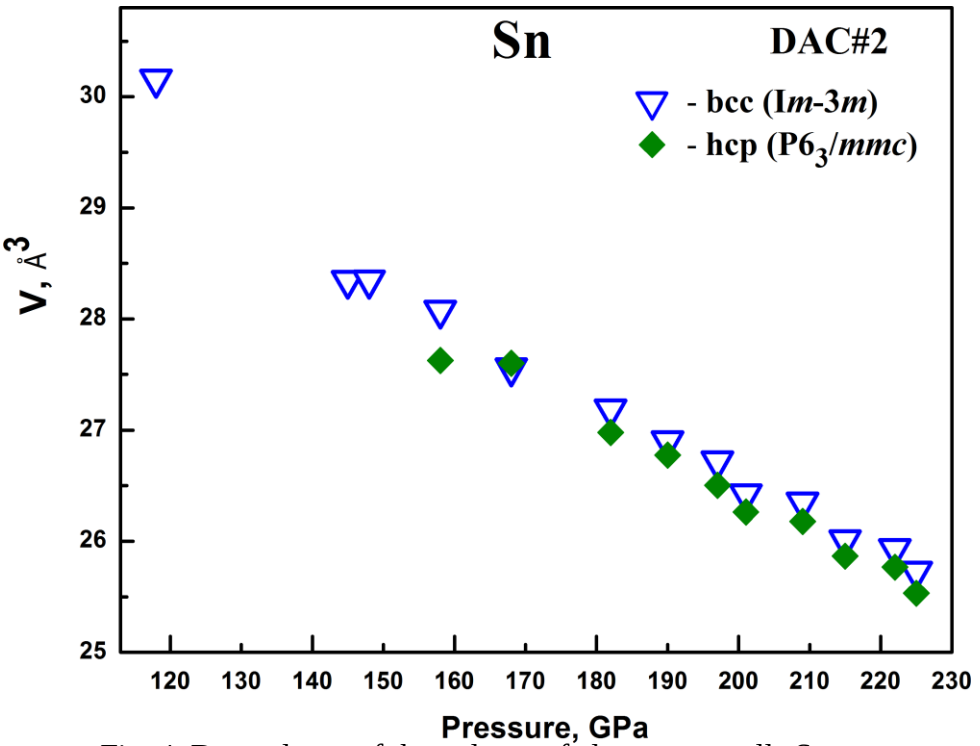

Fig. 4. Dependence of the volume of elementary cells Sn on pressure (DAC # 2).

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