ESRF	**Experiment title:** Russian Grant Proposal: Synchrotron Mössbauer spectroscopy and X-ray structure studies of high temperature hydride superconductors at extreme conditions.	**Experiment number**: HC-2902
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Report:

The search for high-temperature superconductivity is one of the major challenges in condensed matter physics and solid-state chemistry. The poly-hydrides of metals, including Sn, are promising candidates for superconductors, which can be obtained at very high pressures of the megabar range.

We performed several x-ray investigations of Sn-hydrides and Sn as reference sample at pressure range of 140-217 GPa using synchrotron X-ray diffraction technique at ID27 ESRF.

The Sn-hydrides were synthesized by two ways: (a) from Sn (foil) in H_2 medium under high pressure at room temperature and at laser heating as well; (b) from preliminary synthesized SnH_4 gas (stannane) which was cryogenically loaded into diamond anvil cell and compressed to very high pressure at room temperature. We made at least four experiments in which we observed similar changes of XRD patterns at pressures about 160 GPa related to formation of Sn-hydride. All XRD patterns from Sn-hydrides are very similar and have characteristic reflection corresponding interplanar distance d~2.5Å.

We assume that Bragg peaks of the experimental XRD patterns from SnH_4 (stannane) at pressures above 160 GPa are best indexed to face-centered cubic lattice (see examples in Fig.1(b-d). Systematic extinctions may correspond one of the F-centered space groups without glide planes (*Fm-3m*, *F-43m*, *Fm-3*, *F432*, *F23*). We consider fcc-structure (*Fm-3m*) or its orthorhombic distortion in *Fmmm* structure to be more probable. All XRD patterns also contain at least one broadened peak at 2Th=9.5° (another one at 10.2° can be from Pt electrode) which is not indexed in fcc-structure. It may indicate the presence of the second

intermediate phase of Sn-hydride (hcp-phase for instance) or some low-symmetry distortions of the fcc-stucture.

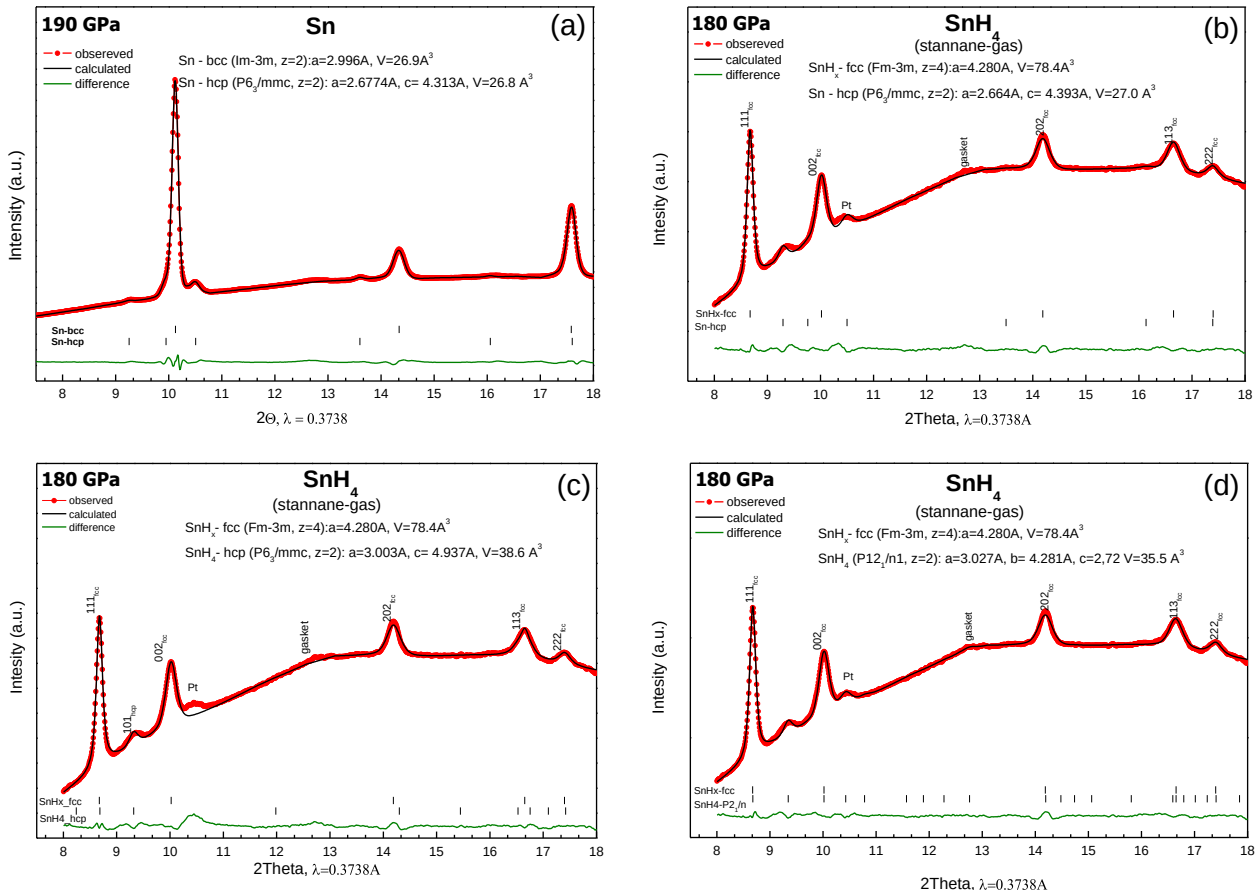

Fig.1 Le Bail refinements of XRD patterns for Sn (reference sample) at 190 GPa (a) and for SnH_4 (stannane-gas) at 185 GPa in different strutural models (b-d): (b) – Sn-hcp+SnHx-fcc, (c) – SnHx-fcc + SnH_4-hcp, (d) – SnHx-fcc + SnH4-P$12_1/n1$ (monoclinic distorted fcc) (d).

Synchrotron Mossbauer NFS (Nuclear Forward Scattering) studies of $Sn^{119}H_4$ at high pressures, cryogenic temperatures and in strong magnetic fields were recorded at the station ID18 on the synchrotron ESRF (European Synchrotron Radiation Facility, Grenoble, France) operating in a 16-band mode. A helium cryostat was used, with a superconducting magnet in which the high-pressure chamber was cooled to a temperature of 4 K. The characteristic rate of accumulation of the NFS spectra was about 20 counts / sec, which allowed the removal of qualitative NFS spectra within 40-60 minutes. A study was carried out in two diamond anvils cells in which the SnH4 sample was at a pressure of 120 and 160 GPa. The pressure was determined outside the cryostat at room temperature by broadening the Raman peak of the first order from the diamond anvil. The chamber was then placed in a cryostat and cooled to 4 K. After that, the spectra were measured at different magnetic field values and with increasing temperature. The obtained NFS spectra were processed using the MOTIF program developed by Yu. Shvydko [1]. To monitor the field under the same conditions of temperature and field, spectra were measured on the tin foil as a reference sample.

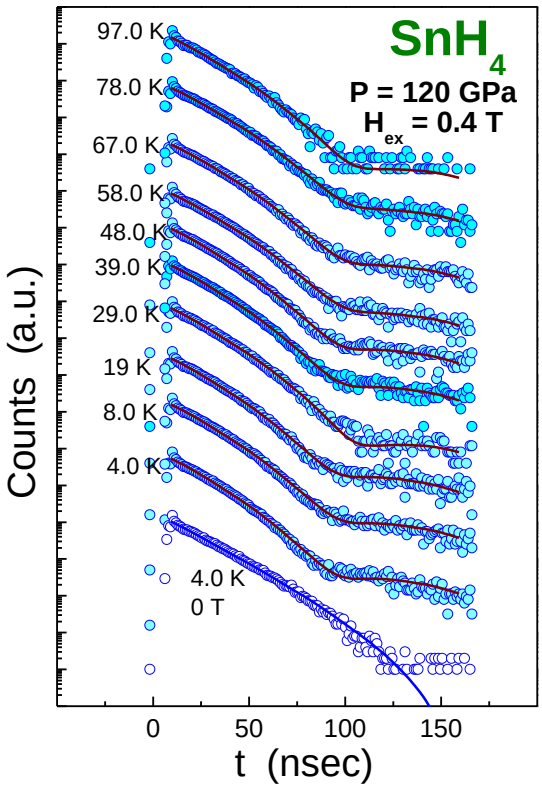

Figure 1. Evolution of NFS spectra with increasing temperature from the sample of Sn^{119} H4 at a pressure of 120 GPa. Symbols are experimental points, solid lines are approximations according to the MOTIF program [1]. Approximation was carried out according to the model of the 1st magnetic sextet. For comparison, the spectrum measured with zero field is given.

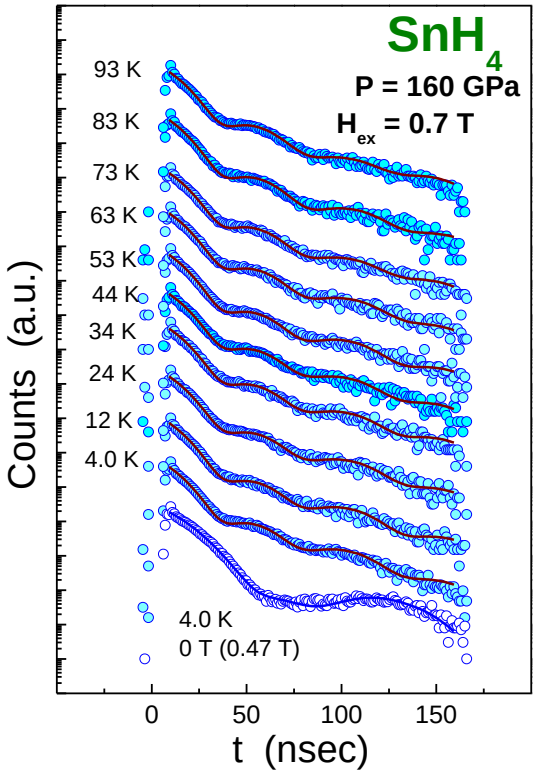

Figure 2. Evolution of NFS spectra with increasing temperature from the sample $Sn^{119}H_4$ at a pressure of 160 GPa. Symbols are experimental points, solid lines are approximations according to the MOTIF program [1]. Approximation was carried out according to the model of 2 magnetic sextets. For comparison, the spectrum measured with zero field is given.

[1]. Shvyd'ko, Y.V., Phys. Rev. B, 1999. **59**: p. 9132.