ESRF	**Experiment title: Single Crystal X-Ray Diffraction Investigation of the Heterogenous High Tc La-H Mixtures**	**Experiment number**: HC-4357
Beamline: ID11	**Date of experiment**: from: 09.07.2021 to: 12.07.2021	**Date of report**: 06.09.2021
Shifts: 9	**Local contact(s)**: Eleanor Lawrence bright	*Received at ESRF:*

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Report:

Objectives

This proposal aimed at exploiting the submicron and ultra-high flux X-ray beam provided by the ID11 beamline to 1) produce a high spatial resolution XRD map of laser-heated La-H samples between 130 and 180 GPa, 2) identify the position of the best La-H crystallites of unknown phases and 3) unambiguously solve their crystal structure by collecting high-quality single crystal X-ray diffraction data. This study, solely possible on the ID11 beamline, is pivotal for the understanding of the exceptional T_c measured from La-H mixtures.

Results

BX90 diamond anvil cells with 80 μm culets were prepared. Lanthanum pieces in paraffin oil (C_nH_{2n+2}) were loaded in diamond anvil cells. Due to the small sample cavity, the known equation of state of the rhenium gasket along with the first order vibrational mode of diamond were used as *in-situ* pressure gauges. The samples were precompressed to the targetted pressures (> 140 GPa) and laser-heated above 2000 K at our home laboratory in Bayreuth. Necessary arrangement had been made to have access to a laser-heating setup at the ESRF, although that setup has a larger beamsize than the one employed at our home institute. Further compressing and laser-heating of some of these samples at the ESRF resulted in diamond anvil failure. Despite the sanitary situation, two team members were able to go work onsite.

X-ray diffraction maps of all samples were collected and revealed diffraction lines belonging to pure La, diamond powder formed following the decomposition of heated paraffin, and of many other phases. On the best positions, single-crystal X-ray diffraction datasets were acquired in order to determine the crystal structure of these other phases. At 140 and 176 GPa, the unit cell of the LaH_{10} compound, was unambiguously identified. This single-crystal data confirms the reported powder X-ray diffraction data.[1–3]

However, of the utmost interest is that single-crystal data was also obtained on previously unobserved La-H phases. The full data analysis is currently ongoing and the unit cells as well as the position of the lanthanum atoms are extremely likely to be determined from these datasets. This data will be coupled with theoretical calculations to help find the position and amount of hydrogen atoms in these unit cells. Table 1 provides the lattice parameters and the number of reflections observed for a new La-H phase.

The next weeks will enable to more fully assess the quality of the collected single-crystal data. The single-crystal reflections are very intense, which is very impressive given the sample and beamsize. Nonetheless, having a crystal domain properly positioned on the center of rotation of the rotational stage is not trivial; despite a very careful centering procedure. As it stands, the best strategy appears to be to collect single-crystal datasets until a single domain, upon preliminary analysis appears to be perfectly centered. While a time-expensive approach, it is essential to obtain the best possible data. In any case, the beamtime was undoubtedly a great success and truly shows how much the ID11 has improved due to the EBS-ESRF.

Table 1: Single-crystal domains of LaH_x at a given pressure

Domains	*a (Å)*	*c (Å)*	*V (Å^3)*	*# reflections*
1	3.7633(17)	5.5869(16)	68.52(5)	37
2	3.7483(10)	5.5864(10)	67.97(3)	123
3	3.7575(14)	5.590(4)	68.35(6)	84
4	3.7523(7)	5.590(2)	68.16(3)	119

References

// [**1**] Drozdov, A. P. *et al. Nature* **569**, 528–531 (2019)
// [**2**] Somayazulu, M. *et al. Phys. Rev. Lett.* **122**, 27001 (2019)
// [**3**] Meng, H. *et al. Int. J. Hydrogen Energy* **42**, 29344–29349 (2017)