EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:
https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report")*,
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Revealing crystal structure of novel high-pressure tungsten borides	**Experiment number**: **HC-5074**
Beamline: ID-27	**Date of experiment**: from: 15 November 2022 to: 18 November 2022	**Date of report**: 28.02.2023
Shifts: 9	**Local contact(s)**: Mohamed Mezouar, Tomasz Poreba	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Inexpensive superhard materials find numerous industrial applications. Several theoretical and experimental studies suggest that mechanical characteristics of some tungsten borides could exceed those of widely used tungsten carbide, WC. Nevertheless, there are still many inconsistencies on possible crystal structures, chemical compositions, stability fields, and mechanical properties of tungsten borides. The aim of the proposed experiment was to apply methods of single-crystal X-ray diffraction (XRD) in laser-heated DACs in order to determine crystal structures and chemical compositions of possible high-pressure phase(s) in W-B system.

A mixture of boron and tungsten were loaded into a DAC equipped with 120 um Boehler-Almax diamonds; neon was used as a pressure medium and as a pressure standard. The sample was laser-heated at 95 GPa to a maximum 3000(100) K. We observed formation of at least two novel tungsten boride phases: a high-pressure polymorph of tungsten tetraboride, HP-WB_4, that crystallizes in sp.gr. $C2/m$ (Z = 2, a = 2.8651(18), b = 4.8677(9), c = 4.6863(17) Å, β = 97.42(5)°, R_1 = 6.3%) and tungsten hexaboride, WB_6, with sp.gr. $I2/m$ (Z = 2, a = 7.3256(12), b = 2.7508(2), c = 8.8094(7) Å, β = 103.034(14)° , R_1 = 3.3%). Crystal structure of HP-WB_4, is similar to one of LP-WB_4. In the crystal structure of LP-WB_4 (Figure 1, left), the metal atoms form the flat hexagonal close packed layers perpendicular to *c*-axis that follow ABABA… sequence. Covalently-bonded boron networks are located between layers of tungsten atoms. The networks consist of two puckered layers connected through short B-B bonds. In the crystal structure of high-pressure polymorph the boron layers flatten, while tungsten layers have no translational shift i.e. follow simple AAA… sequence (Figure 1, center). Due to higher metal:boron ratio, the crystal structure of WB_6 appears to be more complex and consists from WB14 polyhedra connected through common edges and faces (Figure 1, right). It resembles motifs found in other high-pressure transitional metal borides composed of MBx polyhedra such as FeB_4, Fe_2B_7 and Co_5B_{16}.

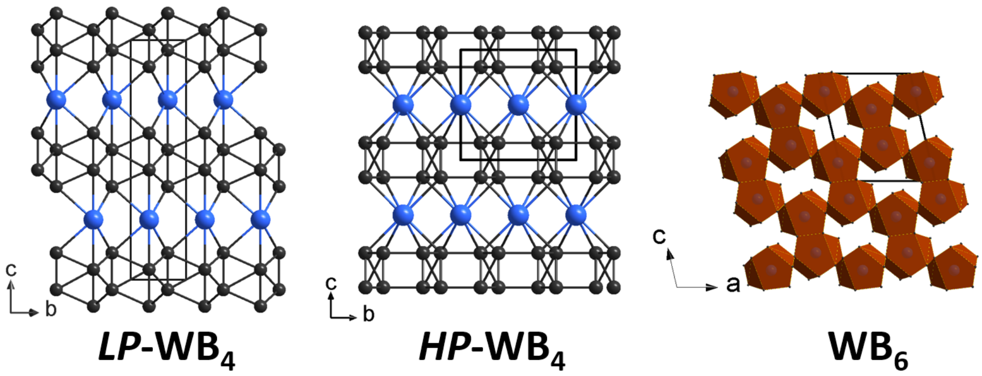

Figure 1. Crystal structure of two novel high-pressure phases of boride of tungsten: left – LP-WB_4 (obtained earlier), center - HP-WB_4 and right - WB_6. Blue spheres – tungsten, black spheres – boron; orange - WB14 polyhedrons.