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:** Undoped and P-doped W-Nb-O catalysts: Influence of heat-treatment conditions in crystallization processes	**Experiment number**: A25-2-1068
Beamline: BM25	**Date of experiment**: from: 12 MAY 2023 to: 16 MAY 2023	**Date of report**:
Shifts: 12	**Local contact(s)**: DR. JUAN RUBIO ZUAZO	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

During the time at BM 25-SpLine, the first 3 shifts were dedicated to the measurement of X-Ray difractograms of samples that present crystalline and pseudocrystalline structures (a set of 23 samples) in the SixC diffractometer. Powder samples were measured in capillars, without any inconvenience during that time.

Preliminary results seen in **Figure 1** show that, as an example, there are some differences when comparing the XRD patterns obtained at laboratory scale and at the ESRF. Then, it can be observed the presence of additional diffraction peaks that, undoubtedly, will help to understand the phase transition and identify the new structures that undergo upon calcination in solids that present caesium, tungsten and niobium (pseudocrystalline to distored orthorhombic M1 phase). Same results were obtained in catalysts that also present phosphorous in the structure.

Afterwards, the 9 shifts left were dedicated to HAXPES experiments in samples that present cesium, phosphorous, tungsten and niobium in different proportions and with different activation temperatures.

In this case, the first part of the experiment was dedicated to the identification of the regions present in the studied sample (in terms of kinetic energy), depending on its composition, and the elucidation of the analysis time, reaching to a compromise between resolution and time. Then, a total amount of 10 samples were measured (two sets of five catalysts).

During that time, the most relevant issue was due to the nature of the samples. Thus, this type of catalysts based on mixed metal oxides present a heavily marked semiconductor behaviour, so differential charge was observed. The solution for this problem was to increase notably the element region in where the measures took place, i.e., a larger kinetic energy range, which also led to a strong increase in the time per sample analysis. In addition to that, this differential charge observed in all the solids analysed made it difficult to stablish a general protocol in terms of number of acquisitions. In any case, this problem was overcome by manually displacing the recorded

spectra after the measurement in order to maximize the resolution of the spectra. Nevertheless, interesting results were obtained.

Preliminary results, as an example, are displayed in **Figure 2**. There it is shown the full range HAXPES spectra (survey) of a sample synthesized with caesium, tungsten, niobium and phosphorous in the structure and previously heat-treated at 1000 ºC (it is also the same sample shown in **Fig.1**, XRD results). Interestingly, all elements have a signal in the HAXPES spectra except for caesium, element that it is present in the conventional XPS spectra recorded in our lab. Then, as a conclusion, it is suggested that caesium is only incorporated in the surface of the structure, being that surface incorporation enough to produce a phase transition upon heat-treatment into the orthorhombic phase known as M1.

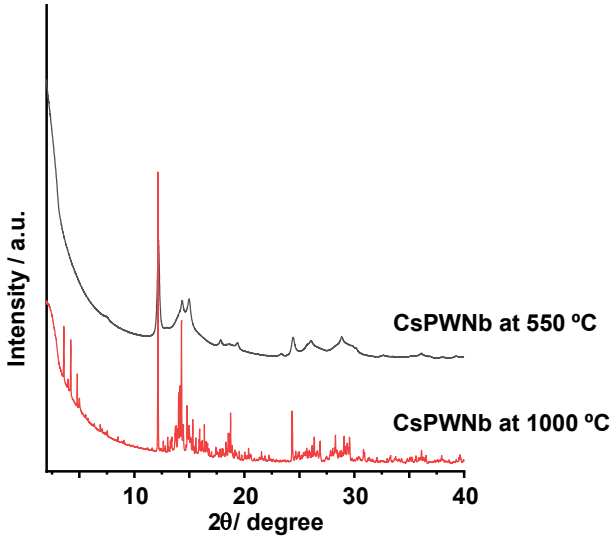

Figure 1. Comparison of the XRD pattern obtained for CsPWNb oxide heat treated at 550 (black) and 1000 ºC (red).

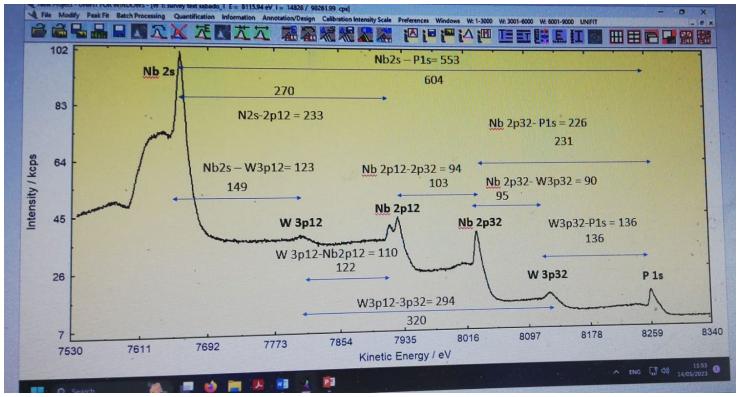

Figure 2. HAXPES survey obtained for a CsPWNb oxide treated at 1000 ºC.

Further analysis of the spectra, expected in the coming months, will allow to stablish a relationship between crystalline structure and chemical (bulk and surface) composition at different activation temperatures. We would also like to express our most sincere gratitute to the SpLine staff for their interest and hep with the experiment.