EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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:** Stress-assisted diffusion (SAD) of hydrogen atoms in anisotropic polycrystals	**Experiment number**: MA-4484
Beamline: ID-11	**Date of experiment**: from: Feb 3rd, 2021 to: Feb 9th, 2021	**Date of report**: March 2021
Shifts: 18	**Local contact(s)**: Jonathan Wright	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Hamidreza Abdolvand, Assistant professor at the University of Western Ontario, London, Canada.

Report:

Summary

Zirconium alloys are widely used in the core of various types of nuclear reactors. Following the diffusion of hydrogen within the microstructure of zirconium, a brittle phase called zirconium hydride might form. Formation of hydrides is significantly detrimental to the integrity and in-service performance of reactors matrials. Thus, understanding the underlying mechanism and consequences of hydride formation is crucial in increasing the lifespan of nuclear reactor core components. This research aims to employ a novel experimental technique, scanning three-dimensional synchrotron X-ray diffraction, on two hydrided zirconium samples to have a more comprehensive understanding of hydride formation. The experiment includes measuring 3D stresses at different temperatures to determine how hydride precipitation and dissolution affect the stress fields close to hydrided grain boundaries and triple points.

Scanning 3D-XRD experiment setup and samples

The Scanning 3D-XRD experiment was conducted at ID-11, at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France. The experiment benefited from the new generation of Exremely Brilliant synchrotron source (EBS) at ESRF, where 200 nm monochromatic X-ray beam was be used to measure 3D stress fields. The experimental setup is shown in Fig.1. The samples can be heated *in-situ* using a vaccum furnace.

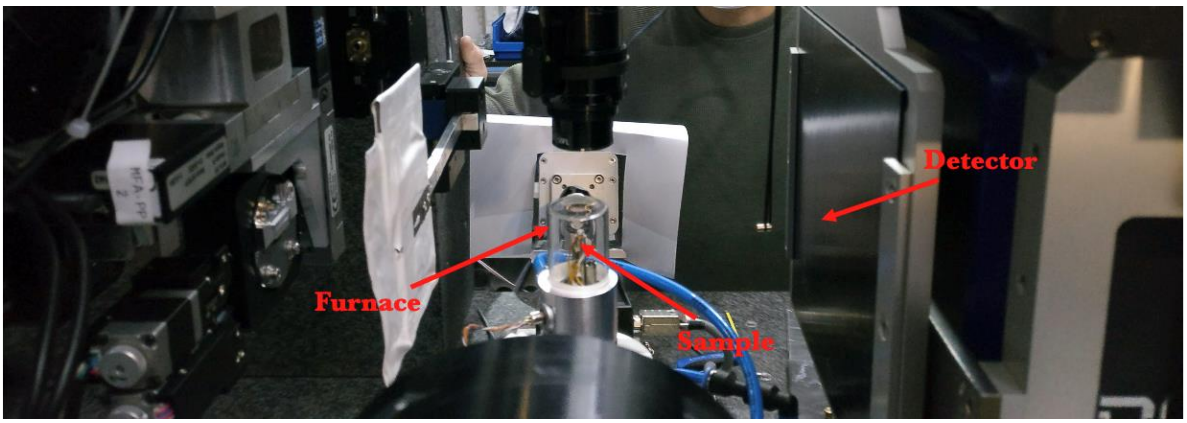


Fig. 1- The experimental setup at ID-11, ESRF

Two catholically charged zirconium samples were heat treated and electron backscatter diffraction measurement were conducted on the samples to detect the hydrides forming at the grain boundaries and triple points. Using focus ion beam (FIB), two tri-crystal samples were cut in the form of micropillars with different orientation relationship, e.g. hard-soft, between the zirconium crystals. Microscopic images of Sample#1 and #2 and their corresponding EBSD maps are shown in Fig.2a and 2b, respectively.

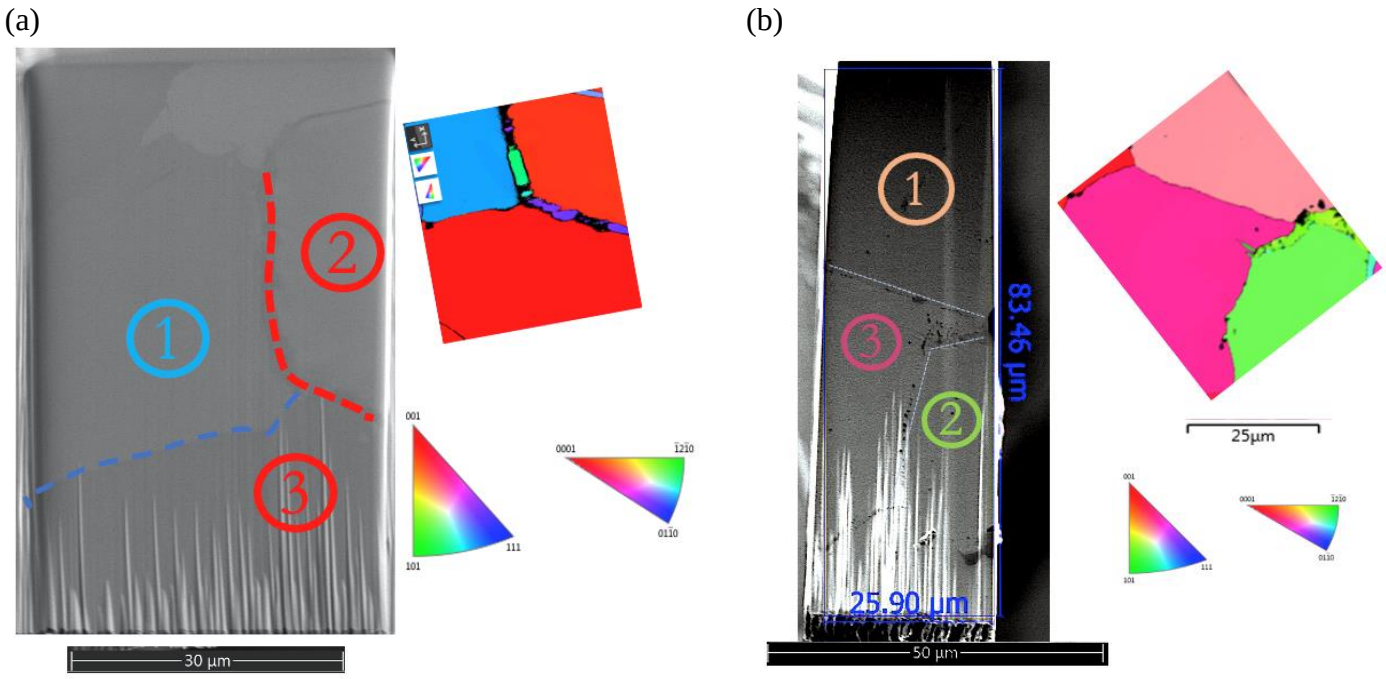


Fig. 2 – Microscopic images of (a) Sample#1 and (b) Sample#2. The grain numbers are based on the color in their corresponding EBSD map. EBSD maps are rotated in order to align the grains with the microscopic image.

During each temperature stage, the diffraction patterns were collected layer-by-layer to cover the height of the specimen. The height of each layer is approximately 1.5µm. A full scan was performed on Sample#1 at room temperature. For Sample#2, diffraction patterns were collected at 5 different temperatures during the experiment. The final temperature for each heating/cooling stage is listed below:

- T_0 = 24.5 °C
- T_1 = 280 °C
- T_2 = 380 °C
- T_3 = 430 °C
- T_{final}= 24.7 °C

The purpose of the mentioned heating stages was to heat the sample beyond the Terminal Solid Solubility limit (TSS) to dissolve all the hydrides. The final stage is designed in order to reprecipitate the dissolved hydride.
The post-processing of the collected dataset is done by coding in Python. The preliminary results of the experiment are presented below.

Results

1. Diffraction peaks

After measuring all the layers of the two samples at each temperature stage, the diffraction peaks and the shape of the grains in each layer are captured. Fig.3 illustrates the diffraction peaks for a test layer of Sample#2. The peaks marked with indicators below them are the known phases within that layer, e.g. α-Zr, δ- and γ-hydride. As shown in Fig.3, some unmarked phases are also detected within the sample. Detection of these phases is a part of the future work in post-processing.

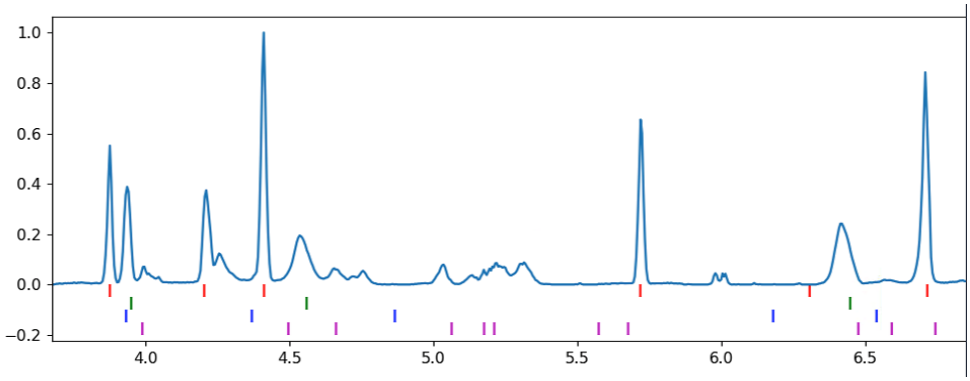
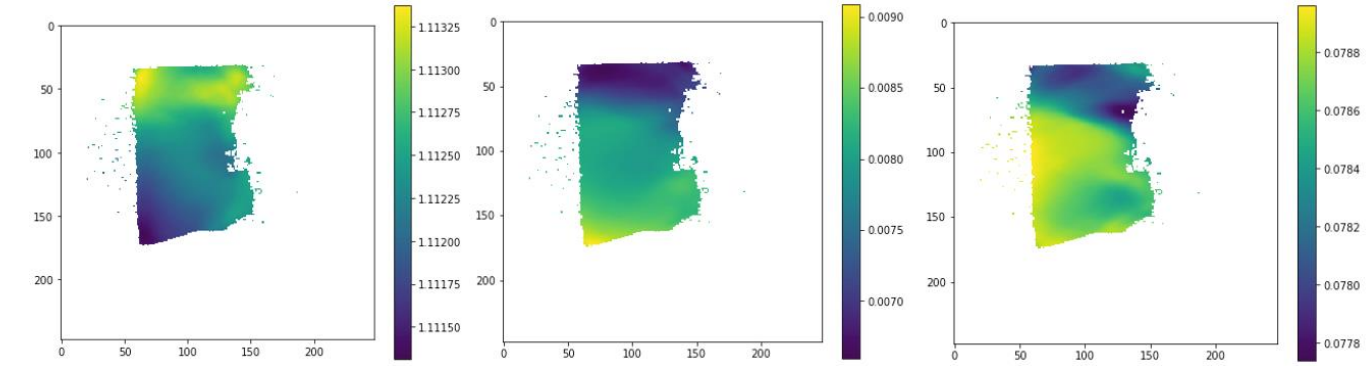


Fig. 3- Diffraction peaks measured for the test layer. Red, green, blue and pink marks respectively represent zirconium (HCP), δ-hydride (FCC), ε-hydride (FCT) and γ-hydride (ordered tetragonal)

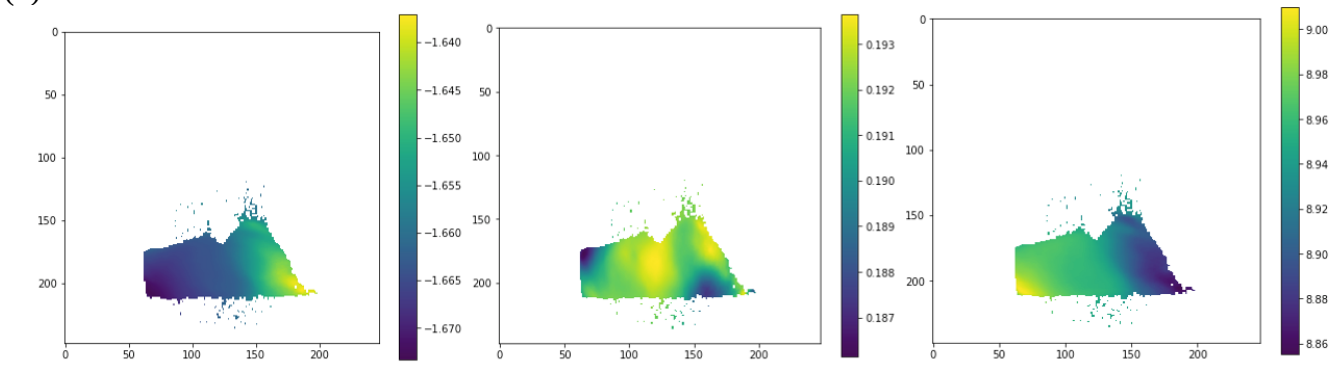
2. Grain shapes

The test layer consists of 3 adjacent grains. The Rodriguez Vector components for all the three grains are presented in Fig.4.

(a) Grain#1



(b) Grain#2



(c) Grain#3

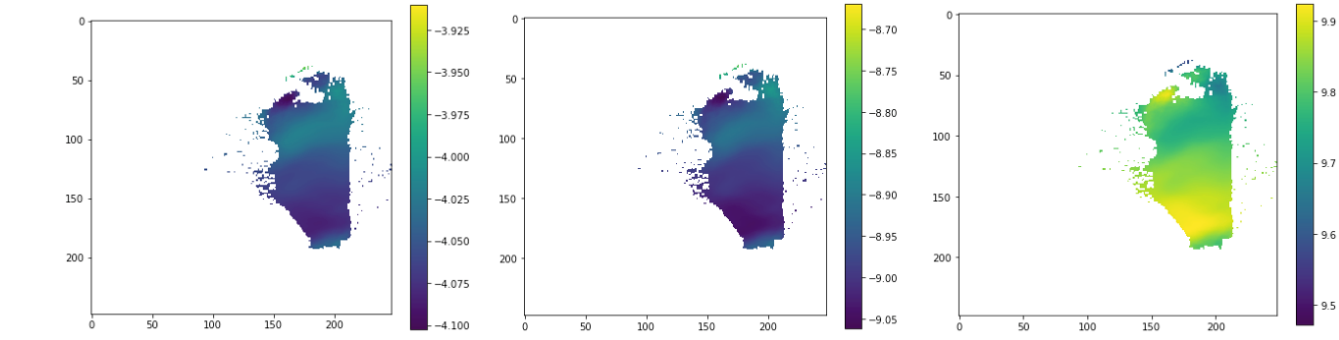


Fig. 4- Rodriguez Vector components captured for (a) Grain#1 (b) Grian#2 and (c) Grain#3

3. Phase map

Using the diffraction peaks and unit cell parameters for possible phases in the test layer, the phases at different regions of the layer can be identified. Once the phase of all the data points in this layer is identified, the phase map of the whole layer can be captured.

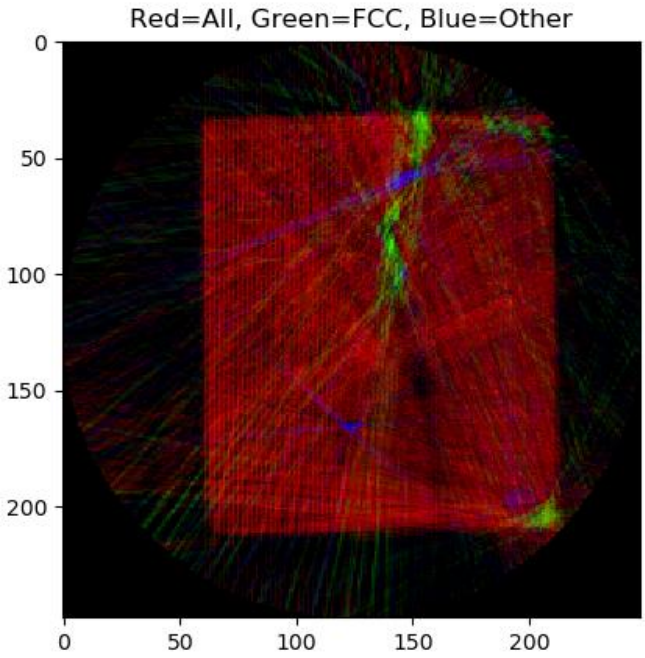


Fig. 5- The phase map for the entire test layer. Red regions are zirconium, green regions are delta hydrides and blue regions are other unknow phases at this layer (possibly zirconium oxide or other hydride phases)