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:** Residual stress in tungsten fibre-reinforced composites	**Experiment number**: MA-3147
Beamline: ID11	**Date of experiment**: from: 03.02.2017 to: 06.02.2017	**Date of report**: 02.07.2020
Shifts: 9	**Local contact(s)**: Wolfgang Ludwig	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Interim Report:

To investigate the residual stress in tungsten fibre-reinforced tungsten composites (W_f/W) produced by chemical vapour deposition (CVD-W_f/W) or a powder metallurgical process (PM-W_f/W) transmission diffraction maps were collected for various samples in the MA3147 experiment. Samples of the pure constituents W fibre, CVD W, PM W and various W_f/W samples with different interlayer materials mainly in the as fabricated but for some samples also in a heat treaded state have been investigated. Beside the residual stress in general, the difference between the two manufacturing routines have been of interest.

1. Specimen preparation

Due to the strong absorption of W only thin samples can be used for the collection of transmission diffraction maps. For that reason, single fibre composite samples as shown in Figure 1 were manufactured. These systems consist of a single W fibre possibly coated by an interlayer and embedded in a W matrix produced either by CVD or by PM. The interlayer was deposited on the fibres by a physical vapour deposition process. For CVD-W_f/W the matrix was produced by the reaction of WF_6 and H_2 at a temperature of 600°C for 6h. PM-W_f/W was produced by hot isostatic pressing for 4h at 2000 MPa at a temperature of 1500°C. After the production, the samples were electrolytically thinned and then cut to the needed height using a wire saw.

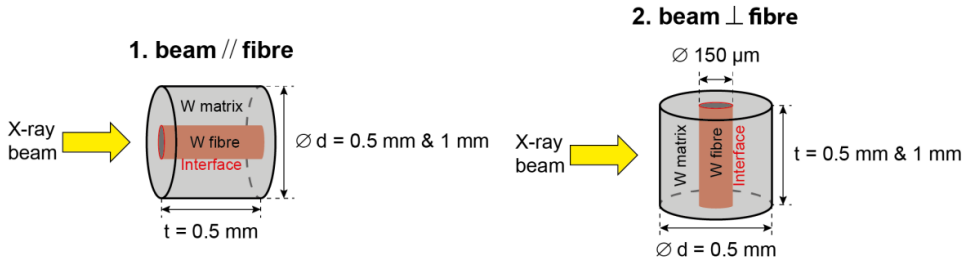

Figure 1 *Experimental setup and sample dimensions.*

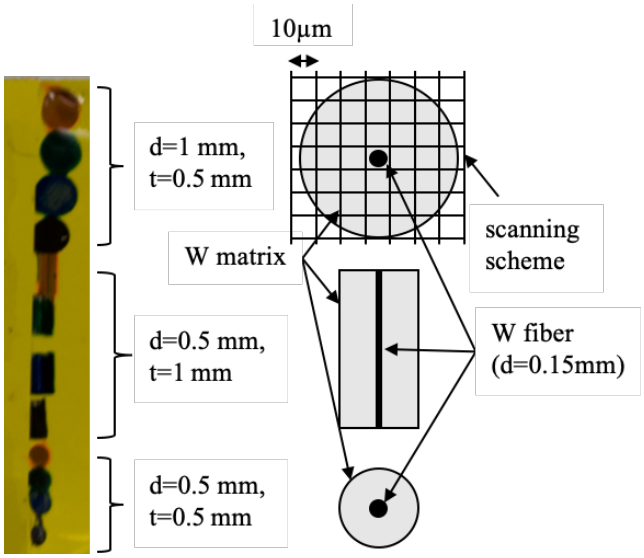

Figure 2 *Mounted* CVD-W_f/W *specimens (left) and corresponding schematically sketch (right) with scanning scheme.*

2. *Experimental*

We used a highly energetic beam of 68 keV, with a wavelength of 0.18 Å and a beam size of ~20x20 µm² in a transmission diffraction set up. A FReLoN 2k camera (2048x2048 px) with an exposure time of 133ms for each image was used.

An example for the mounting of the CVD-W_f/W samples is shown in Figure 2. Several specimens were attached to a Kapton tape using a Cyanolit glue ” to be able to perform multiple scans without changing the specimen holder.

For the PM-W_f/W, specimens with a 1µm, 2µm and 3µm thick the Er_2O_3 interlayer were measured. For the CVD-W_f/W, specimens with different interlayer materials (Er_2O_3, Y_2O_3, ZrO_2,YSZ, TN, without interlayer) and interlayer thickness ranging from 10 – 1000 nm were measured.

3. *Result*

We scanned 78 specimens and depending on the size of the specimen, up to 8000 single images per specimen were collected.

An example of a collected scan is shown in Figure 3 a). Due to a lack of manpower data analysis is still ongoing.

As the first step the aszimuthal integration has been performed with an automated script for several specimens as shown for one sample in Figure 3 b). The analyses of the data will be done with python scripts by using pyFAI in the near future.

Table 1 Specimens

Material	*Interlayer material*	*Scanned specimens*
Single W fiber	-	13
CVD-W	-	2
PM-W	-	5

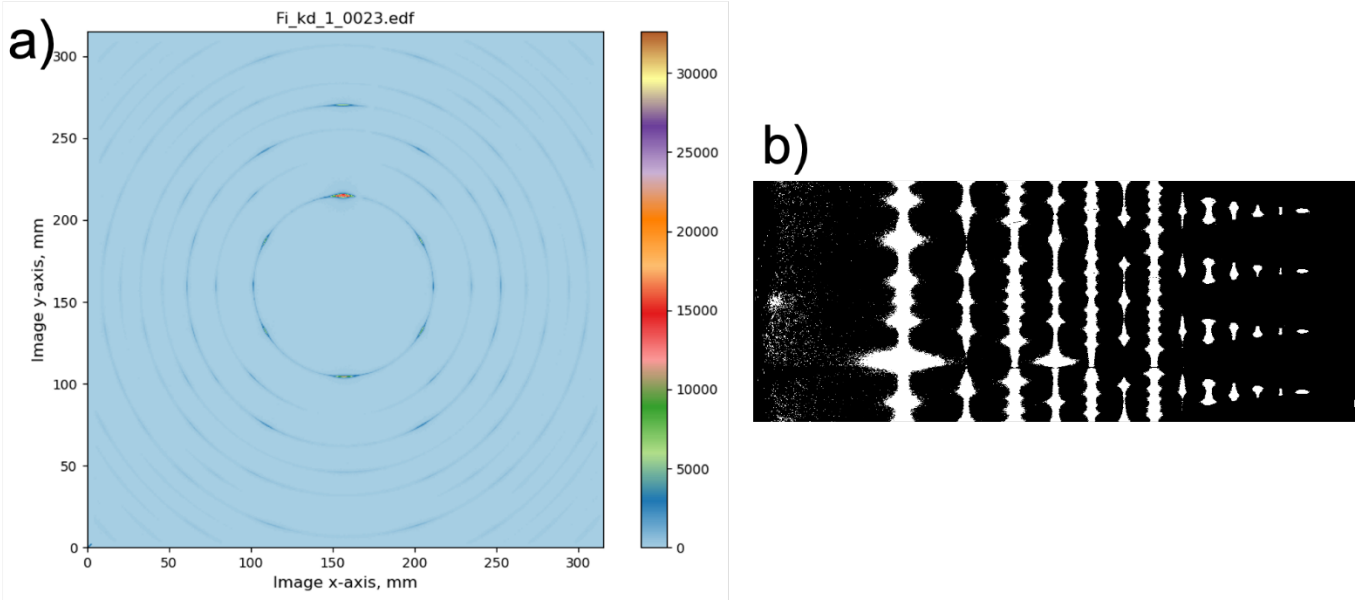

Figure 3 Diffraction patterns for a pure W fiber with a diameter of 150 µm. a) collected scan b) after aszimuthal integration.