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: 3D characterization of morphology, crystallography and internal stress effects of sub-grain defects intentionally introduced in superelastic NiTi wires**	**Experiment number**: MA-4494
Beamline: ID11	**Date of experiment**: from: 11/11/2020 to: 16/11/2020	**Date of report**: 16/02/2021
Shifts: 18	**Local contact(s)**: Dr LAWRENCE BRIGHT Eleanor, Dr WRIGHT Jonathan	*Received at ESRF:*

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Report:

Experimental setup

The ultimate aim of the experiment is to characterize the microstructure of a superelastic NiTi as it evolves due to martensitic transformation and coupled plastic deformation processes induced by tensile loading. For this purpose an in-situ 3DXRD experiment using a scanning pencil beam of ~200 nm was tailored and realized in configuration shown in Figure 1a. A 30 μm superelastic wire heat treated so to obtain rather large grains of ~5 μm, as checked by EBSD measurement (Fig 1b), was used in order to limit the number of grains within the scanned gauge volume thus facilitating the 3DXRD post-processing. Furthermore, a ~5 μm wide Pt layer was deposited over the wire's circumference serving as a marker (Fig.1b) in order to track the same gauge volume of the sample throughout all deformation stages probed during the experiment. In total 7 deformation stages, including the referential stress-free virgin stage, were probed as indicated by filled red circles I-VII in Tensile stress-El. Resistivity-Tensile strain graph (Fig. 1c) recorded during the experiment. All the expected deformation processes are well indirectly detected by the evolution of the electrical resistivity that was also measured during the experiment (blue curve in Fig. 1c) The deformation stages were selected to characterize expected deformation mechanisms consisting of the elastically strained austenite structure at the edge of martensitic transformation (II), stress-induced martensitic structure (III), martensitic structure deformed at two different extends via deformation twinning and dislocation slip (IV, V), plastically deformed subgraines of martensite structure retained after unloading (VI), plastically deformed subgrains of austenite structure reversed by stress-free heating (VII). At each stage, the same region of interest (ROI) was scanned in y direction along the wire cross-section using the y-step of 0.3 μm and in omega range of 180 deg. using the step of 0.1 deg. The ROI was centred around the wire axis as shown in Fig. 1b thus representing an inner cylindrical core of the wire with diameter of 20 μm and height of ~15 μm .

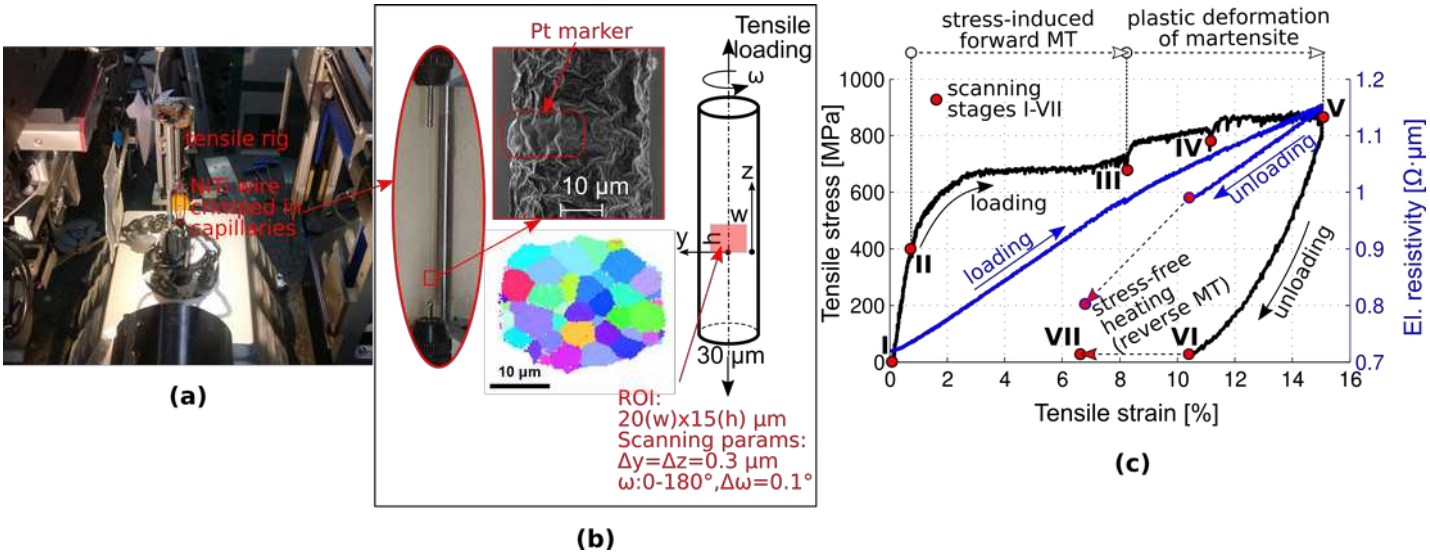

Figure 1: Experimental setup of 3DXRD experiment (a) using scanning 200 nm pencil beam to characterize deformation processes in in-situ loaded 30 µm superelastic NiTi wire (b) at significant deformation stages (c).

Data post-processing

Using the selected pencil beam scanning diffraction setup (68x48x1000 scans per scanning stage) a large data set was recorded (18.2 TB in total). The data set was largely reduced down to 242 GB by data segmentation as individual diffraction frames were sparse due to low number of grains illuminated. A script for parallelized peak search and storage into column files was implemented using ImageD11 package. In addition, sklearn package was used to implement peaks clustering in order to reduce duplicated peaks in omega scans.

A script for parallelized reconstruction of grain cross-sections and strain fitting was implemented based on grain indexing by ImageD11 applied to peaks collected from all y-omega scans at a single z position, inverse radon transformation of sinograms constructed for individual indexed grains, and strain fitting by refining unit cell parameters at each point on a sinogram.

In Figure 2 an example of a reconstructed cross-section is shown in terms of grains distribution (Fig. 2a), axial strain component (Fig. 2b), and axial stress component (Fig. 2c) related to loading stage II, i.e. elastically strained austenite prior the stress-induced martensitic transformation.

Further post-processing is being performed in order to reconstruct crystallography, strain, and stress in loading stages III-VII.

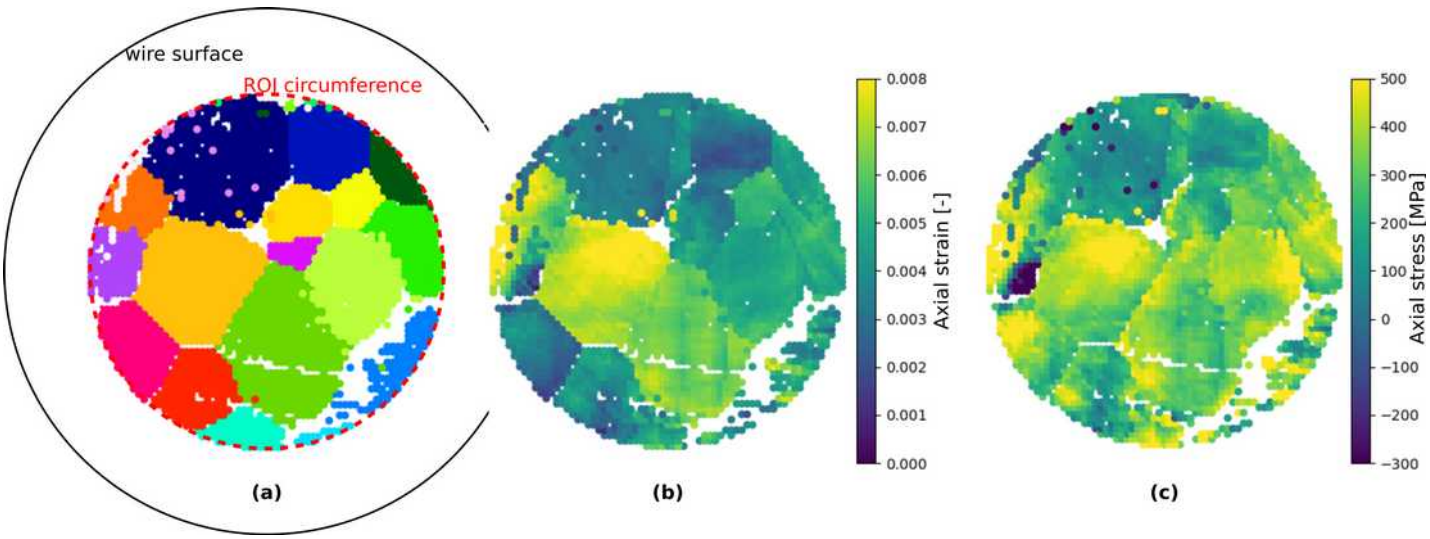

Figure 2: One of the reconstructed NiTi wires cross-sections in terms of grain morphology (c), and axial strain (b) and stress (c) related to loading stage II (see Fig. 1c).