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:** Analysis of deformation mechanisms in nanocrystalline NiTi wire under various thermomechanical loads via in-situ synchrotron x-ray diffraction texture evolution experiments	**Experiment number:** MA-5425
Beamline: ID15A	**Date of experiment:** from: 04 October 2022 at 08:00 to: 08 October 2022 at 08:00	**Date of report:** 29 December 2022
Shifts: 11	**Local contact(s):** CHECCHIA Stefano	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

We have performed in-situ synchrotron x-ray diffraction measurements in transmission on the ID15A beamline during the beamtime as indicated above. The diffraction measurements were synchronized with thermomechanical tests of nanocrystalline NiTi wires, with the aim to study the deformation mechanisms acting during such thermomechanical loading conditions.

A custom made tensile tester MITTER was installed on the beamline and the beam (gauge volume) was placed at the center of the wire sample. The tester enables performing thermomechanical loading tests while controlling test temperatures at desired values within a range of -40 °C to 450 °C. Recording of the 2D diffraction images and stress-strain-temperature-electric resistance of the wire was synchronized via TTL signal throughout the testing.

The experimental parameterization is summarized in Table 1. The acquired 2D diffraction images will be analyzed to extract lattice plane responses (i.e., integrated intensity, lattice strain, peak width) along sample direction(s) of interest and evolution of texture of involved phases during testing (Rietveld analysis).

Table 1. The synchrotron x-ray diffraction parameterization for our measurement.

monochromatic x-rays	energy: 85 keV (wavelength 0.14586 Å)
beam size	150 × 150 μm^2
detector	Pilatus3 X CdTe 2M detector (active area of 253.7 (W) × 288.8 (H) mm^2 and pixel size of 172 × 172 μm^2);
sample-to-detector distance	935.075 mm
exposure time for each measurement	0.01 s
calibrant	CeO_2 standard (NIST Standard Reference Material SRM-674a)

The NiTi wire under study is characterized by a reversible transformation between the parent cubic austenite (B2) and product monoclinic martensite (B19′) phases under temperature change (shape memory effect) and/or external stress (superelasticity). With further straining, the material accommodates plastic deformation mainly via deformation twinning, kinking and kwinking, assisted by dislocation slips. These deformation processes cause characteristic discontinuous lattice rotations in the material, which will be captured by the evolution of texture. The study thus attempts to analyze information on texture evolution (and also combining studys on texture modelling and TEM analysis of microstructures) in order to reveal deformation/transformation mechanisms activated during various thermomechanical loads frequently used in engineering applications of NiTi, as thermal actuator cycling, functional fatigue, low temperature shape setting and two way shape memory effect (TWSME). Fig. 1 illustrates the experimental setup. Fig. 2 instantiates the thermomechanical testing, during which the NiTi wire sample was subjected to loading-unloading-heating-cooling cycles.

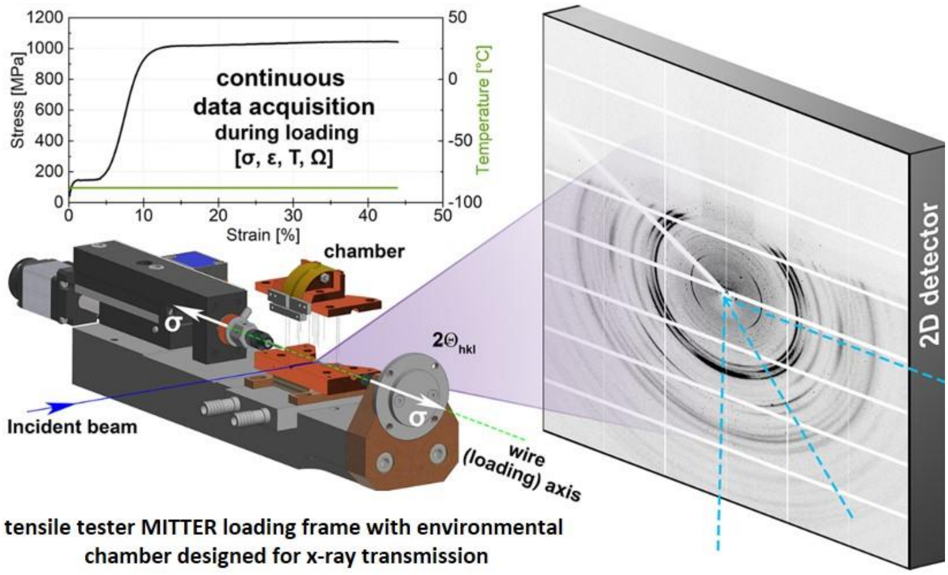

Figure 1. In-situ synchrotron x-ray texture experiment. 2D x-ray diffraction images are recorded continuously during the thermomechanical tensile test on NiTi shape memory alloy wires.

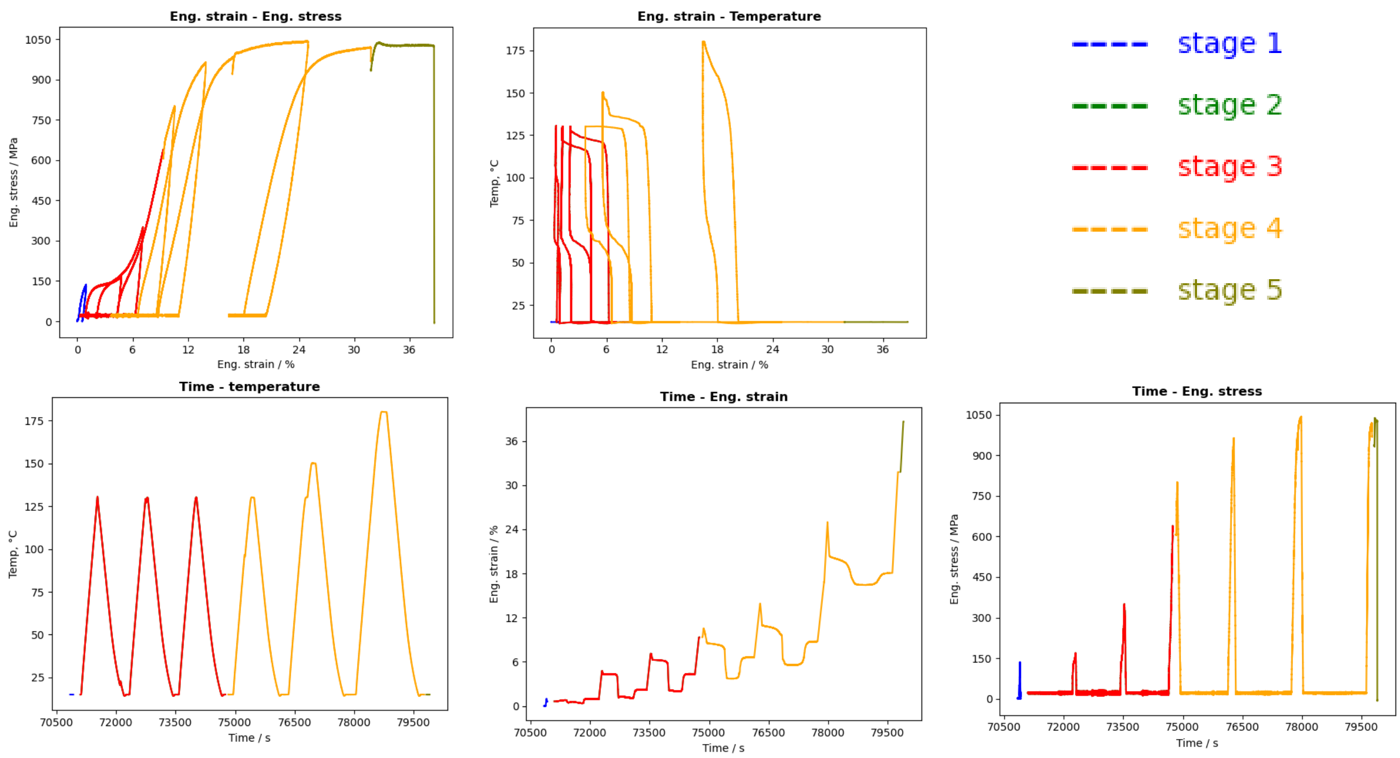

Figure 2. Example of the thermomechanical tests recording time versus. engineering stress-strain and temperature; this data was synchronized with 2D diffraction images throughout the test via TTL signal.