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.

	Experiment title: Multiaxial stress-strain transfer across indenter-sample interface during in-situ indentation of soft high-entropy shape memory alloys and hard thin films	**Experiment number**: MA-5322
Beamline: ID13	**Date of experiment**: from: 15.11.2022 to: 21.11.2022	**Date of report**:
Shifts: 18	**Local contact(s)**: Aicha Asma Medjahed, Manfred Burghammer	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

As specified in the proposal, the users performed an *in situ* indentation experiment with the indentation device (provided to the beamline ID13 during the LTP MI-1355) using a diamond tip coated with nanocrystalline diamond using chemical vapour deposition. Following the deposition the indenter tips edges were cut by focused ion beam milling to ensure uniform contact during testing. A set of SEM images of the tip is presented in Fig. 1.

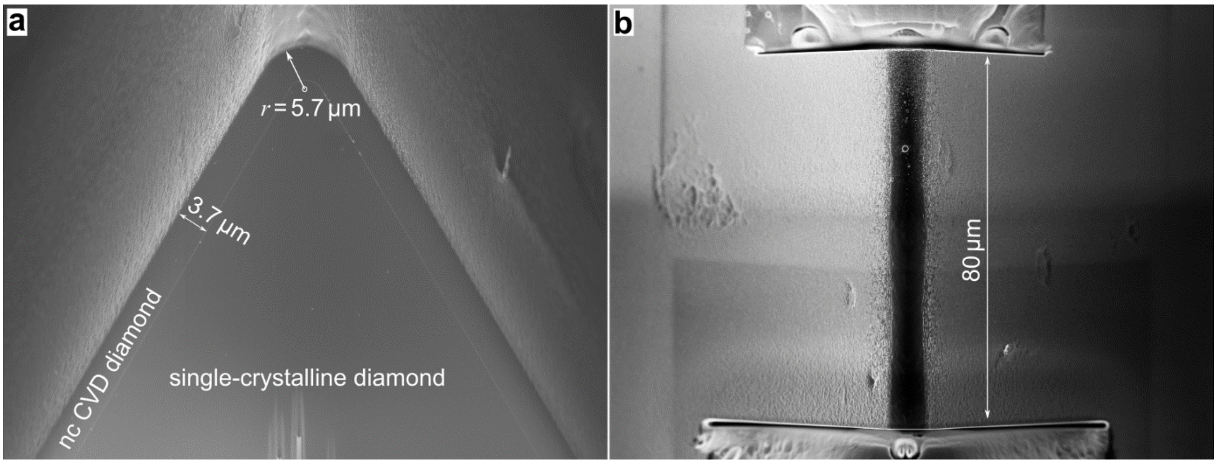

Figure 1: SEM images of the indenter tip from the side (a) and top (b). A uniform diamond coating of 3.7 µm thickness was deposited on the single-crystalline dimaond wedge, therefore the resulting radius of the tip is 5.7 µm (a). The side faces were polished by focused ion beam milling leading to 80 µm width (b) of the tip in beam direction.

The tip was mounted in the holder presented in Fig. 2a and pre-aligned using the optical microscope present at the ID13 beamline. Additionally a test imprint on Al was conducted to ensure alignment parallel to the indenter frame. Careful adjustment of the alignment was done by a set of absorption scans at different rotations around the *z*-axis to optimize the positioning of the tip. Afterwards the misalignment of the tip was corrected using the ball-head screws on the tip holder. In Fig. 2b, an absorption scan of the tip confirming the suitable alignment of the diamond tip is presented.

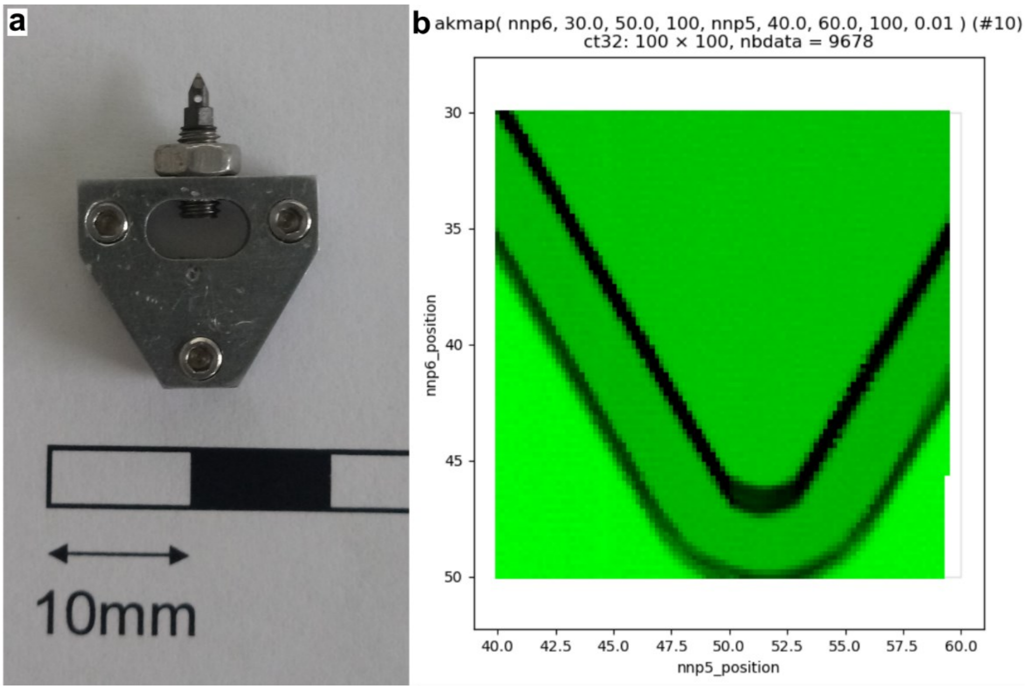

Figure 2: (a) The indenter tip mounted in the tip holder. The three ball head screws are used for fine tuning the alignment of the tip when installed in the tips cage. (b) Absorption scan of the tip after alignment using the rotation axes of the ID13 beamline and the ball-head screws.

In total 5 samples of different elastic-plastic behaviour and hardness were *in situ* tested during the experiment. Each indentation experiment was carried out in 6 steps, (i) before loading, (ii) at loads of 0.2, 0.5, 1.0 and 2.0 N loads and (iii) after the experiment. A typical load-displacement curve is shown in Fig. 3a. In Fig. 3b-f the results obtained from SAXS and WAXS data during indentation of a bulk nanostructured high-entropy alloy at a load of 0.5 N are presented.

The diffuse scattering around the beam stop is integrated for every acquisition and then used to compound a micrograph sensitive primarily to electron density variations (Fig. 3b). Here, the tip and the smaple can be easily identified, as well as the sample surface due to its higher roughness and the interface between the single-crystalline diamond and the nanocrystalline dimaond deposited by CVD. Additionally, averaged FWHM distributions are evaluated from the data (Fig. 3c) showing an highly localised FWHM increase attributed to the high gradients of stresses of 1st (Fig. 3d-f) order within the X-ray gauge volume.

Furthermore, for every 2D map σ_{yy}, σ_{yz} and σ_{zz} distributaions (Fig. 3d-f) are evaluated giving new and unprecedented insights into the stress transfer across the indenter-sample interface.

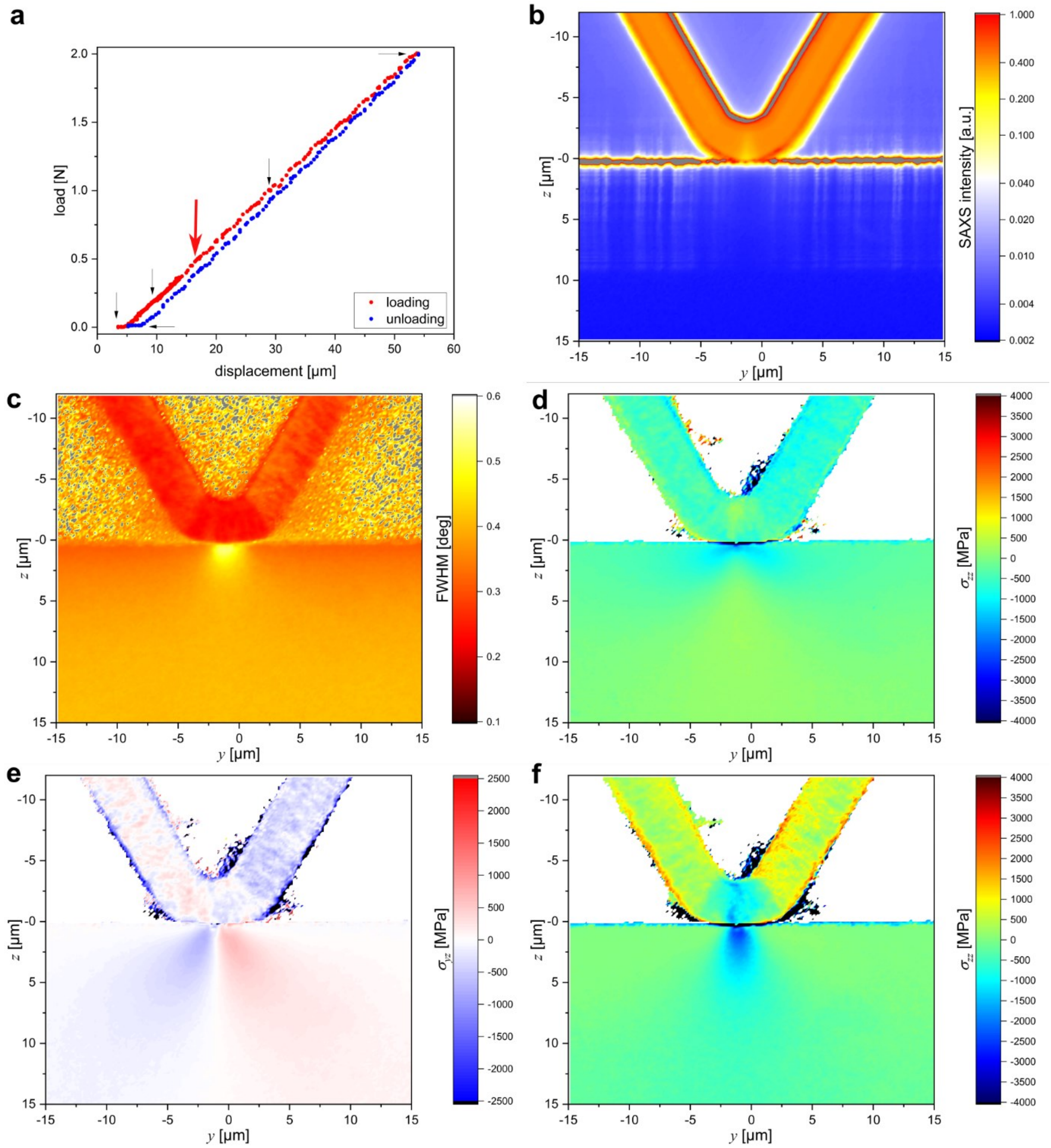

Figure 3: Results obtained from SAXS and WAXS data during indentation of a bulk nanostructured high-entropy alloy: the load-displacement curve (a) and the data obtained at a load of 0.5N (b-f) as indicated by the arrow. In (b) a SAXS micrograph compounded from the diffuse scattering around the beam stop for each acquisition is presented showing the indenter tip in contact with the sample, while in (c) average FWHM distributions from diamond and the HEA sample are shown. σ_{yy}, σ_{yz} and σ_{zz} distributaions at 0.5 N load for both the diamond tip and the HEA sample are presented in (d-f), respectively.