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: Measurement of temperature dependence of γ/ γ'-lattice misfit in single-crystal Ni-base alloys with different misfit signs**	**Experiment number**: ME-1557
Beamline: ID31	**Date of experiment**: from: 29.04.2021 to: 01.05.2021	**Date of report**: 20.02.2023
Shifts: 6	**Local contact(s)**: Veijo Honkimäki	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*** Bettina Camin, Hochschule Bremerhaven, An der Karlstadt 8, 27568 Bremerhaven, Germany**

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*** Gert Nolze, Federal Institute for Materials Research, Unter den Eichen, 12205 Berlin, Germany**

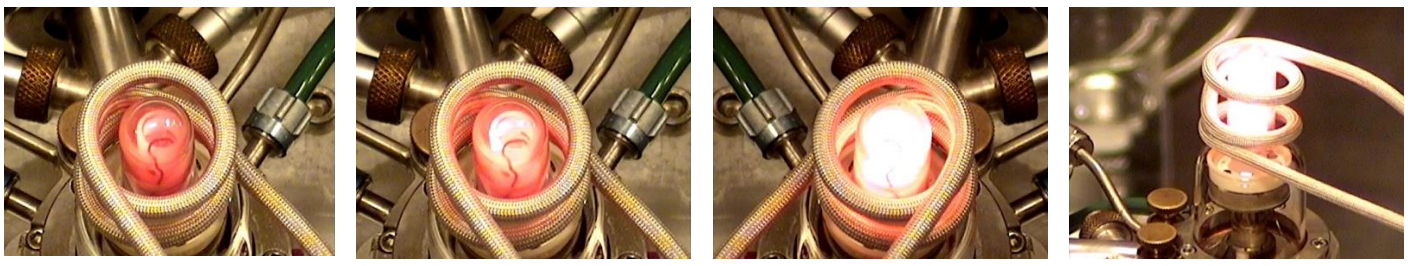
Report:

In general: The experiment was carried remotely. Our local contact, Veijo Honkimäki, did the experiments at ID31 personally due to covid-19 pandemic. Also due to the covid-19 pandemic, the changes of the both applicants each to new affiliations and also difficulties in downloading and opening the data (new format) this report is late. The beamtime was prolonged to the 2nd May 2021 due to technical problems at the beginning.

Experiments:

The samples were sent to the ESRF. The delivered samples had a square cross section of 2 × 2 mm², a length of 15 mm and were cut longitudinally from single-crystals with an axial orientation close to [001]. The prepared samples were glued on a sample holder and mounted in a furnace by the local contact. After alignment measurements, the samples were heated up to 1100 °C stepwise (Figure 1) and then also stepwise cooled down. To prevent oxidation, the diffraction measurements were carried under argon atmosphere at given temperatures during heating and cooling.

The objective of the investigation was to measure the temperature dependence of the lattice misfit of γ- and γ′-phases in 4 nickel-base superalloys: the commercial alloy - CMSX-4 and three model alloys C4 (19-90K+), A7 (19-89K) and A10 (19-90K) with positive, zero and negative misfit values at 1000 °C, respectively.



a b c d

Figure 1 Examples for stepwise heating up of the furnace (a-d)

The measurements were carried out in X-rays with energy E = 77.502 keV in transmission mode.

The diffraction pattern were recorded using a Pilatus3 2M camera, with a resolution of 1475 × 1679 pixel and pixel size of 172 μm. The measurement data were stored at the ESRF and then downloaded by the applicants for processing, see Table 1.

Table 1. Files with measurement results

Date	Sample	Dataset	Files	Size
14:10 29 Apr 2021	alignment	0001	23	576.1 MB
14:26 29 Apr 2021	CeO2	0001 (2021-04-29 14:26:14)	4	5.9 MB
16:02 29 Apr 2021	CeO2_2	0001 (2021-04-29 16:02:26)	4	9.4 MB
19:08 29 Apr 2021	A7	alignment	12	136.9 MB
10:37 30 Apr 2021	A7	tempscan	52	1.4 GB
11:14 30 Apr 2021	C4	alignment (2021-04-30 11:14:19)	16	275.1 MB
20:12 30 Apr 2021	C4	tempscan (2021-04-30 20:12:12)	46	3.2 GB
21:02 30 Apr 2021	A10	alignment (2021-04-30 21:02:19)	19	407.6 MB
08:21 1 May 2021	A10	tempscan (2021-05-01 08:21:53)	68	1.2 GB
17:27 1 May 2021	CMSX	alignment (2021-05-01 17:27:11)	103	2.2 GB
17:53 1 May 2021	C4	retry_alignment	17	172.2 MB

The data were opened using the silx view program, see Figure 2. After viewing in silx view, the diffractograms were saved in the usual TIF format needed for their quantitative processing.

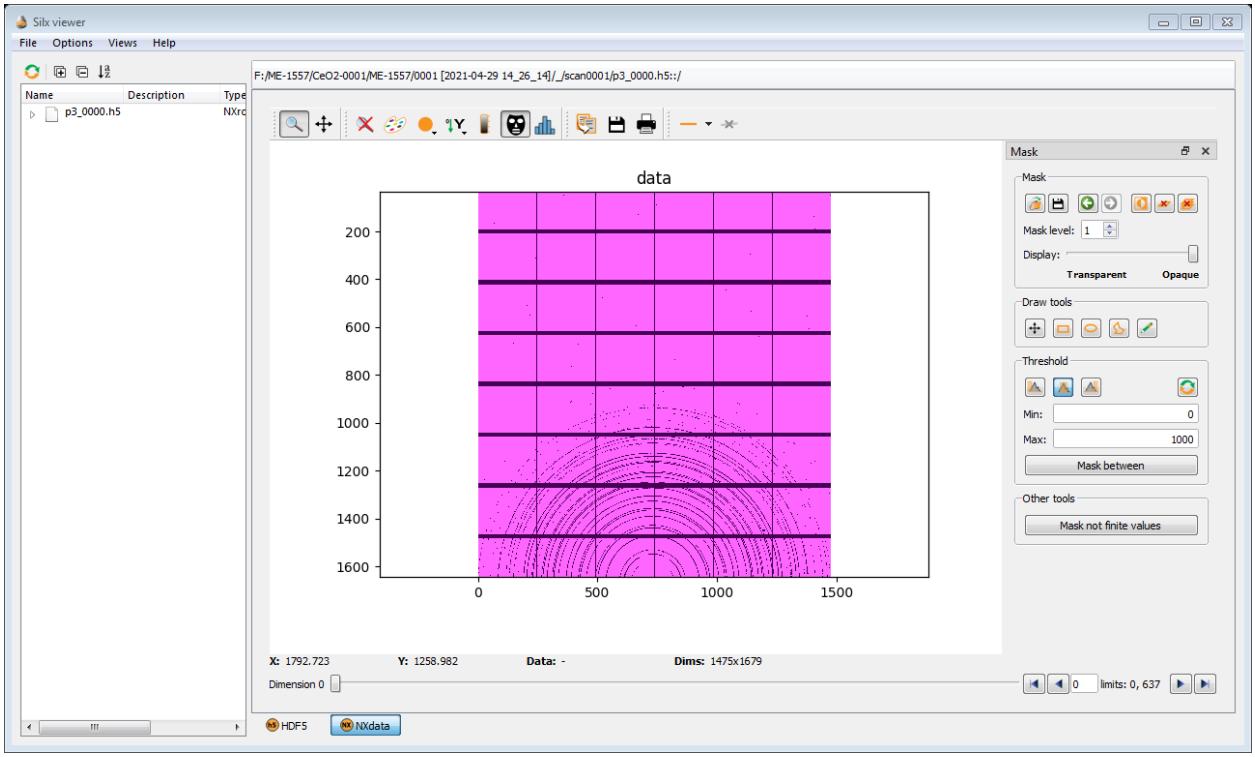


Figure 2 Viewing a diffractogram using the silx view program

In the first step, a calibration measurement using a powder of cerium (IV) oxide CeO_2 with a cubic (fluorite) lattice, having the parameter a = 5.41 Å was carried out (Figure 3).

Figure 4 shows a 1475 × 1679 pixel resolution diffractogram recorded from sample A7 at room temperature. As can be seen, the image of X-ray reflection on the diffractogram is very weak. However, by slecting a local 231 × 186 pixel area around the reflection, a distinguishable profile of its integral intensity can be obtained, see Fig. 4.

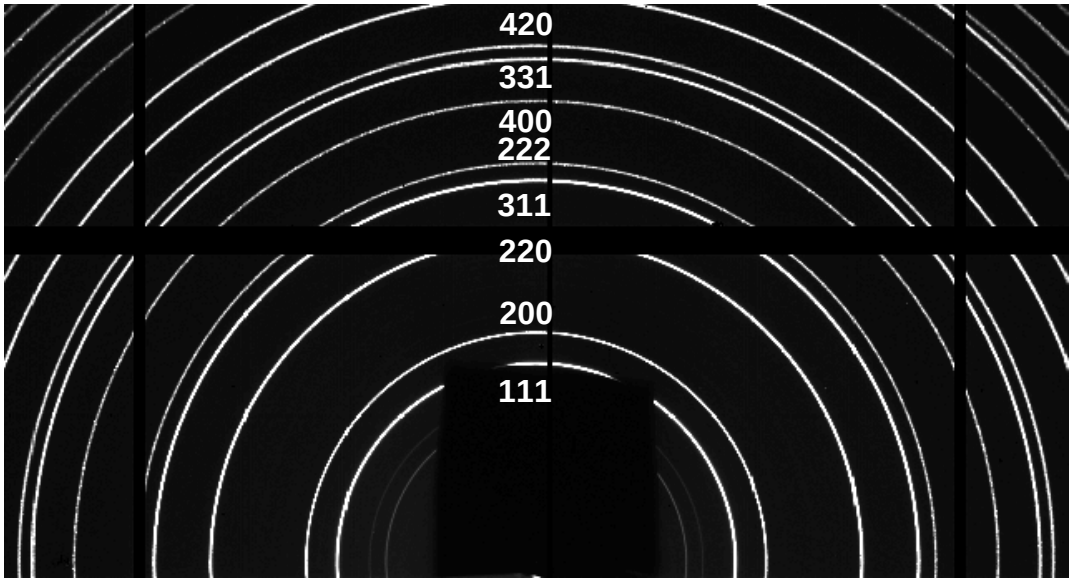
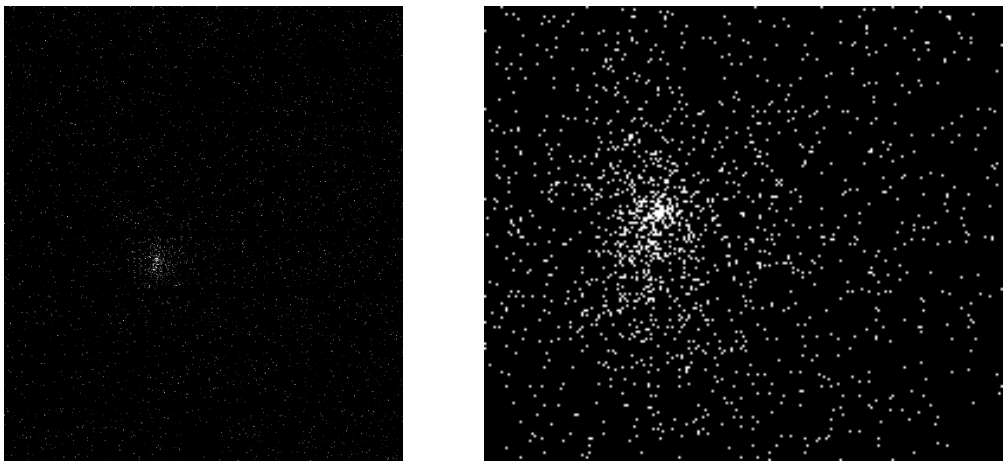


Figure 3 Calibration image of cerium (IV) oxide polycrystal CeO2. X-ray reflection indices are shown in the figure



(a) (b)

Figure 4 Image of a reflection on a diffractogram of resolution 1475 × 1679 pixels (a). Local area of 231 × 186 pixels selected around the X-ray reflection (b)

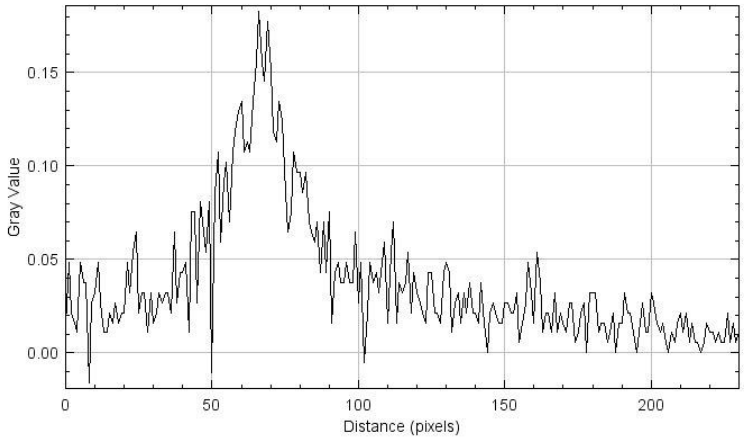


Figure 5 Integral intensity distribution over an area of 231 x 186 pixels around the X-ray reflection, see Fig. 4b

Summary:
The samples planned for this investigation have been prepared and delivered to the ESRF.
All planned experiments have been performed.
Due to some reasons X-ray reflections recorded from the samples have very low intensities.
Processing such noisy reflections needs the development of special software.
However, the development of such software takes a long time.
After obtaining the required results, it is planned to write and publish a paper which will be submitted to the ERSF.