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: Examining the influence of temperature on γ and γ' lattice misfit strain in Ni base superalloys designed for additive manufacture**	**Experiment number**: ME-1610
Beamline: ID22	**Date of experiment**: from: 31/01/2023 to: 04/02/2023	**Date of report**:
Shifts: 12	**Local contact(s)**: Andrew Fitch	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr Alexander Lund*, University of Bath, UK

Jiraphant Srisuriyachot*, University of Bath, UK

Dr Stephane Forsik*, Carpenter Technology Corporation, United States

Report:

Batches of experimental nickel and cobalt-nickel powder superalloys with a high-volume fraction of γ' strengthening precipitate were atomized, heat treated under vacuum and sealed under protective atmosphere. The microstructure of each powder batch consists of a matrix with an FCC structure and between 50 vol.% and 70 vol.% of γ' precipitate with an ordered FCC_L12 structure. The goal of the experiment conducted at the ESRF was to measure the lattice parameters of the γ and the γ' phases as a function of the temperature during a heat/cool cycle between room temperature and 871 °C that is supposed to mimic a typical aging cycle in manufacturing conditions. We are planning to use the data to calculate the crystallographic misfit between the two phases and correlate it with 3D printing trials that we are currently performing.

Before the experiment, 0.5 mm quartz capillaries were filled with powder, sealed under nitrogen atmosphere, and prepared for analysis. A hot blower was used to heat up the samples between room temperature and 871 °C and back to room temperature. XRD signal was continuously collected during the heat/cool cycle but the temperature was stabilized at predetermined steps (260 °C, 538 °C and 871 °C) to collect more signal. The entire heat/cool cycle was repeated three times with different heating/cooling rates: slow heat/rapid cool, slow heat/slow cool and finally rapid heat/slow cool. All seven batches of powder were exposed to the same heat/cool cycles.

The major output of the experiment is a series of diffraction patterns. Figure 1 is an example of the intensity of X-rays scattered as a function of the 2θ angle. Major peaks correspond to the γ and γ' phases and their position was used to calculate the lattice parameters. Other minor peaks correspond to second phases such as carbides or Laves phase but are yet to be fully indexed. In the example in Figure 1, the γ/γ' misfit is derived from the lattice parameters a_γ = 3.6396 Å and a_γ = 3.6422 Å giving a misfit of δ = 0.073 %, calculated as follows:

$$\delta = \frac{2 \times (a_{\gamma'} - a_{\gamma})}{(a_{\gamma'} + a_{\gamma})}$$

Part of the results will be published in a paper on cracking mitigation during the design of high γ' superalloys for additive manufacturing to be submitted to the journal *Metallurgical and Materials Transactions A*. The bulk of the results will be submitted to the TMS Superalloys 2024 conference and published in the proceedings in September 2024.

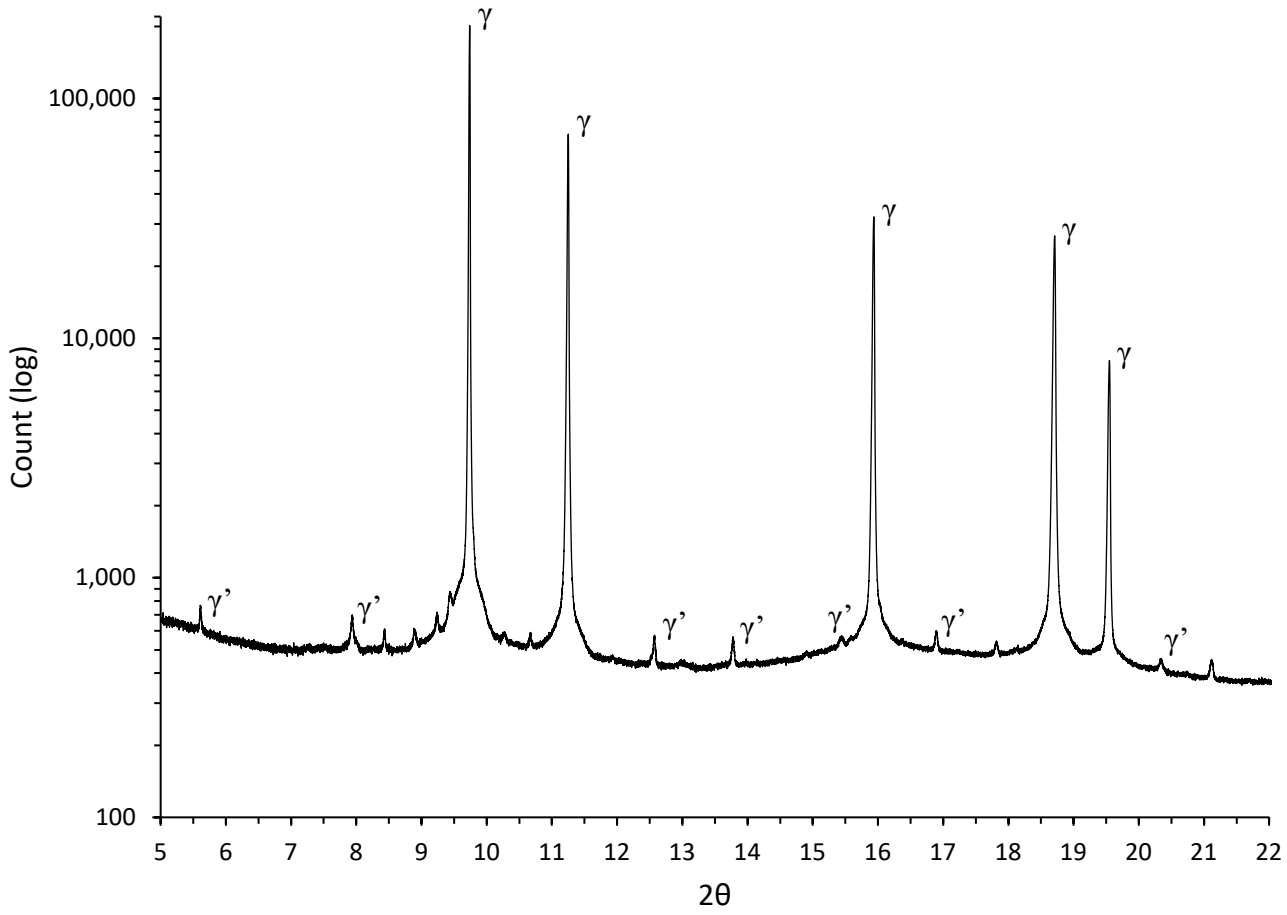

Figure 1: Typical XRD diffraction showing peaks corresponding to the γ and γ' phases. Other smaller peaks have yet to be indexed.