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: In-situ observation of sub-grain formation and strain gradient evolution during thermal fatigue of Cu grains within Cu interconnects**	**Experiment number**: **MA-4969**
Beamline: ID06	**Date of experiment**: from: 05.10.2021 to: 13.10.2021	**Date of report**: 1.3.2022
Shifts: 21	**Local contact(s)**: Can Yildirim, Carsten Detlefs	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

J. Keckes[a], M. Reisinger[b], T. Ziegelwanger[a], K. Hlushko[a], C. Yildirim[c], C. Detlefs[c]

[a]Montanuniversitaet Leoben Department of Materials Physics Jahnstrasse 12 AT - 8700 LEOBEN

[b]Laboratory KAI GmbH KAI GmbH Europastraße 8 AT - 9524 VILLACH

[c]European Synchrotron Radiation Facility, 71 avenue des Martyrs, CS40220, 38043 Grenoble Cedex 9, France

Report:

As specified in the proposal, the users performed in-situ heating experiments on custom chips designed for ID06 (Fig. 1a,b). The chip was used to heat the Cu and Cu-Al pads in the centre with dimensions of 700 x 700 x µm^2 and a thickness of 20 µm. The aim was to evaluate the effect of thermal cycling on the thermal fatigue of the metals.

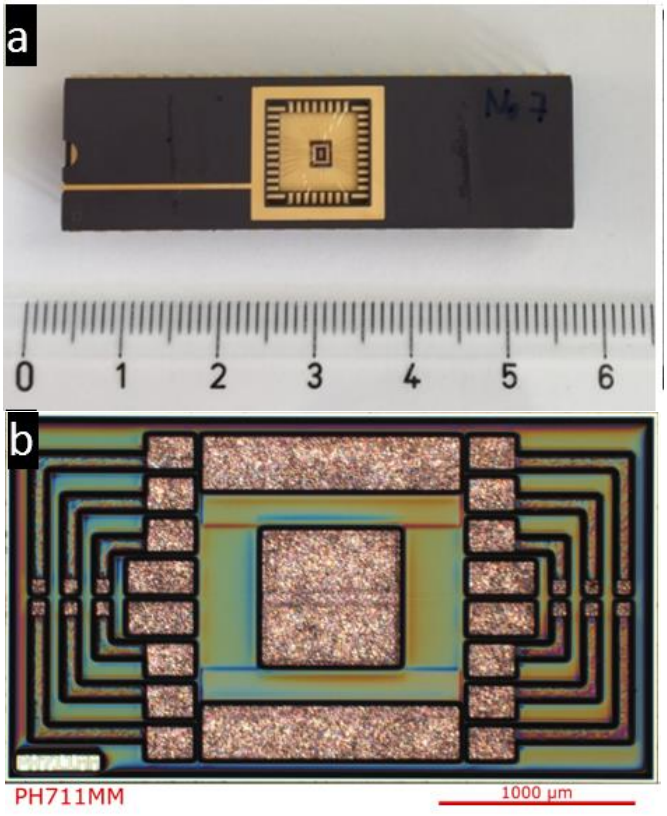

Figure 1: A view of the experimental package (a) used to cycle the Cu and Cu-Al pads in the centre of the chip (b).

Nine samples of Cu and Cu/Al were measured during the experiment. At first, the sample was mounted into to goniometer and then a suitable grain was found, which was mapped using DFXM at 17keV (Fig. 2).

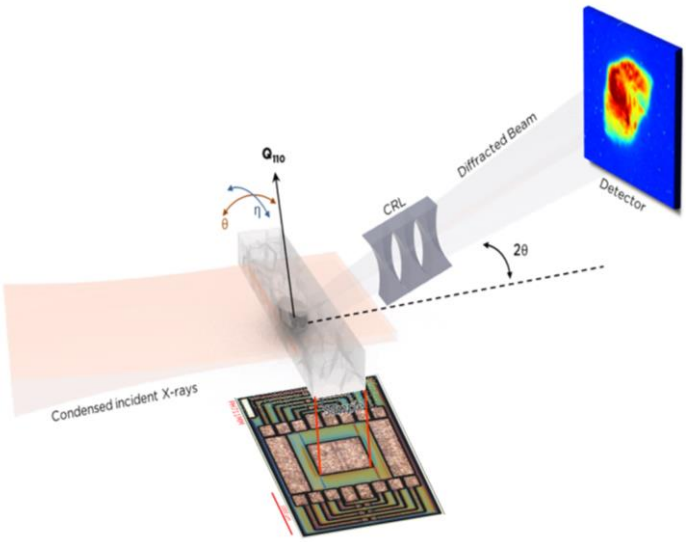

Figure 2: A schematic description of the experimental setup with the angles.

Then the chip was heated applying 10, 100, 300, 500, 1000, 3000 and 5000 cycles in the temperature range of 100-450°C using a custom heating electronics.
After every heating step, the grains were measured by a combination of θ–η and θ/2θ measurements (Fig. 2).
Additionally, some grains were in-situ heated in the goniometer and characterized at high temperature up to 450°C.
The measurements were used to follow the evolution of strain distributions and changes in the mosaicity.
The data reveal very complex changes in the intragranular strain distributions and grain orientations after every heating cycle.
Currently, the results are being evaluated and the users are optimistic to submit two manuscripts based on the data from this experiment.

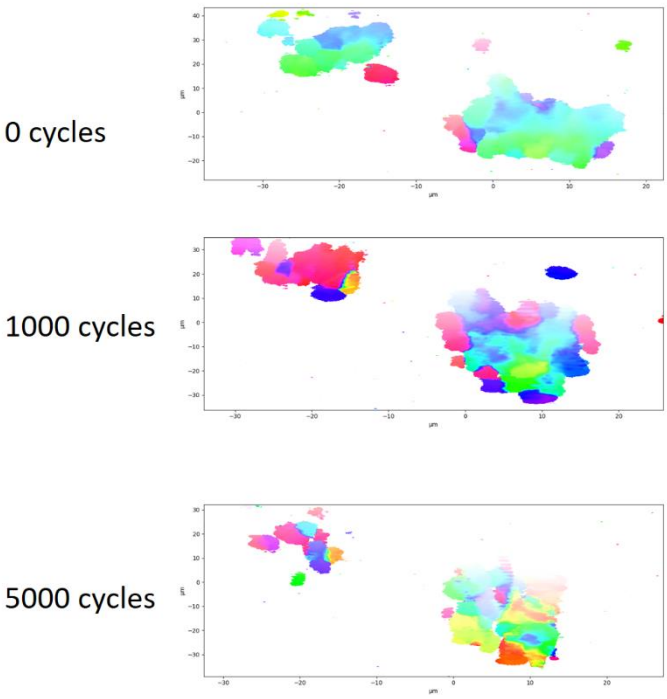

Figure 3: Results from a mosaicity characterization of a typical Cu grain in-as deposited state and after 1000 and 5000 temperature cycles.