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-4482**
Beamline: ID06	**Date of experiment:** from: 24.2.2021 to: 2.3.2021	**Date of report:** 1.3.2021
Shifts: 12	**Local contact(s):** Can Yildirim, Carsten Detlefs	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

J. Keckes[a], M. Reisinger[b], J. Todt[a], M. Meindlhumer[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:

Due to the travel restrictions, the in-situ experiment could not be performed as originally described in the poposal. The users also could not mail the in-situ heating system, which control/operation is very complicated. For this reason, an ex-situ experiment was performed by the beamline scientists Can Yildirim and Carsten Detlefs. The users prepared chips of Cu and Cu-Al alloy thin films, which were cycled up to 50.000 times to a temperature of up to 600°C in home laboratory (Fig. 1). In this way, various degress of thermal farigue were simulated.

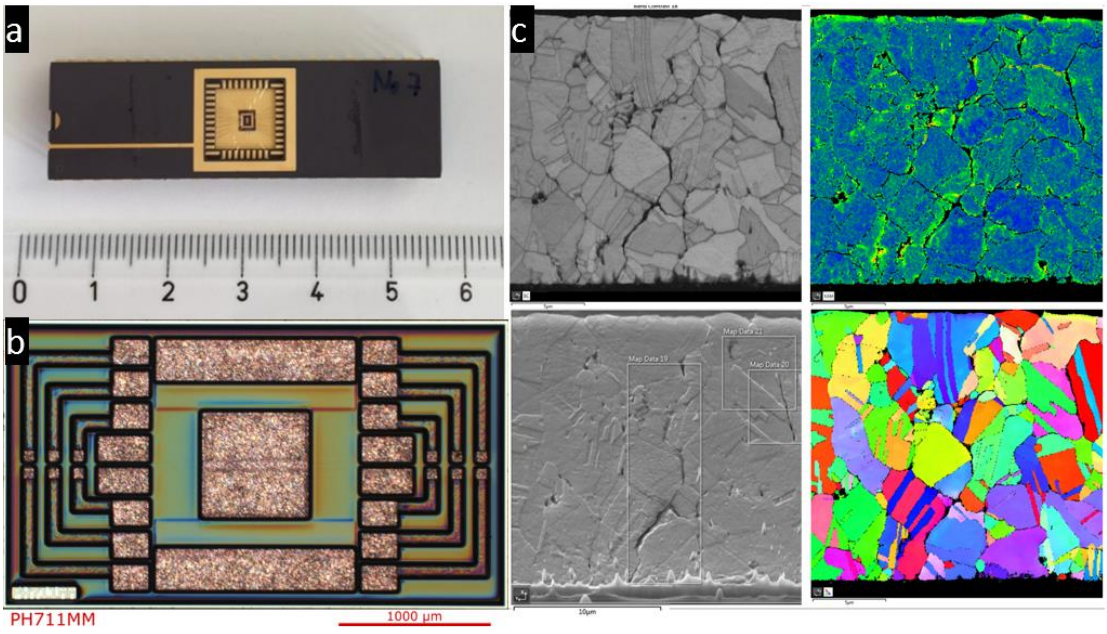

Figure 1: A view of the experimental package (a) used to cycle the Cu and Cu-Al films in the centre of the chip (b) and SEM-EBSD images of the Cu microstructure (c).

The chips were mounted on SEM holders and measured in transmission geometry using DFXM at 17keV. By a sequence of omega-eta and theta scans, it was possible to map mosaicity and strain gradients within virgin and thermally cycled Cu and Cu-Al grains.
Since the experiment was done on 24.2.-2.3.2021, only preliminary data are shown.
The ex-situ data revealed nicely strain and orientation gradients across the grains, which however depend on the number of the applied thermal cycles. The virgin grains indicated relatively homogenoues orientation and strain magnitudes, whereby the cycled samples showed steep gradients or even abrupt changes (cf. Fig. 2).
Importantly, it was possible to observe a formation of new grain boundaries in charactreized grains (indicated by a dashed line in Fig. 2). This observation is very unique and was not reported before.
Consequently, the new resubmitted proposal was modified and the in-situ characterization of grain boundaries evolution will be a new topic of the future experiment.

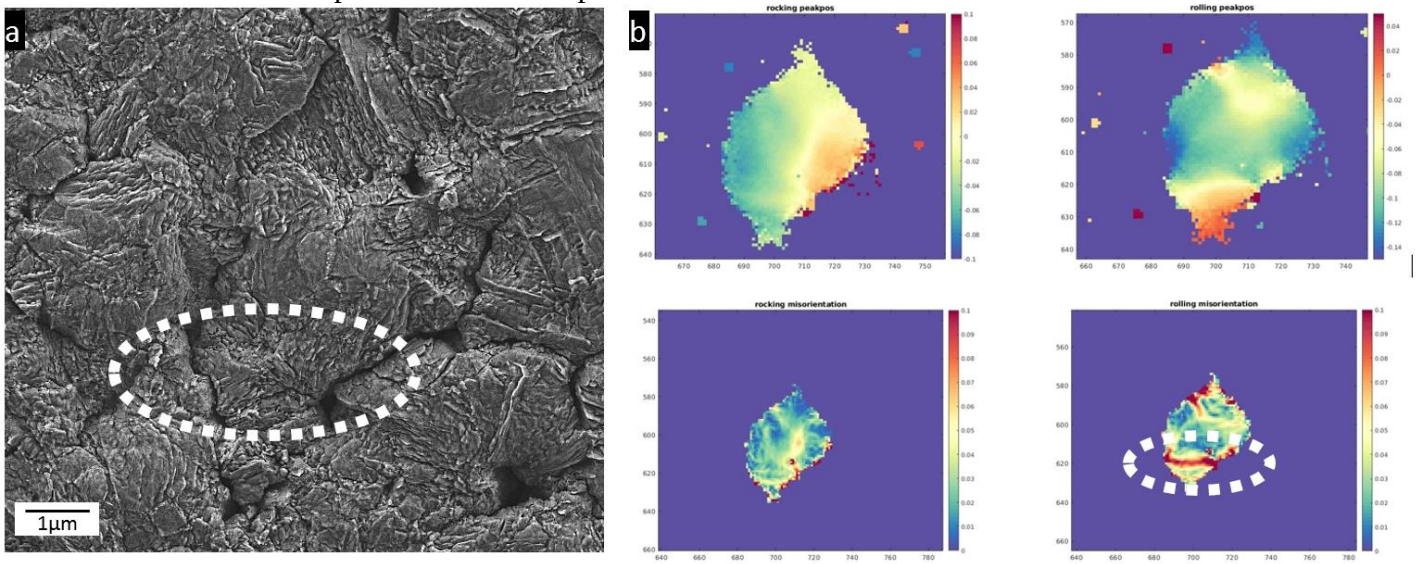

Figure 2: SEM miccrograph of a surface of a representative Cu grain after 50.000 thermal cycles (a). In (b) results from the DFXM indicate an evolution of misorientaion and strain gradients across the grain volume. The data reveal a formation of a new grain boundary, which is indicated by a dotted line.