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 using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

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.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal 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:** Morphological instability during reactive diffusion in Ni/Si couples : in-situ and real-time micro-tomographic characterization	**Experiment number**: ME-805
Beamline: ID19	**Date of experiment**: from: 16-Apr-2004 to: 18-Apr-2004	**Date of report**: 13-Aug-2004
Shifts: 9	**Local contact(s)**: Dr. Petra Pernot	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr Jerome PARET (*)
L2MP UMR6137, Faculté des Sciences de St Jérôme, case 142, 13397 Marseille cedex 20 (France).
Dr Nathalie BERGEON (*)
L2MP UMR6137, Faculté des Sciences de St Jérôme, case 142, 13397 Marseille cedex 20 (France).
Delphine BORIVENT (*)
L2MP UMR6137, Faculté des Sciences de St Jérôme, case 142, 13397 Marseille cedex 20 (France).
Dr Bernard BILLIA
L2MP UMR6137, Faculté des Sciences de St Jérôme, case 142, 13397 Marseille cedex 20 (France).
Gérard CLUGNET
L2MP UMR6137, Faculté des Sciences de St Jérôme, case 142, 13397 Marseille cedex 20 (France).

Report:

The origin of the higly irregular morphology of the phase Ni_3Si_2 formed during bulk reactive-diffusion in nickel/silicon binary couples is still not understood. This is particularly troublesome since nickel silicides grown by reactive-diffusion are used as ohmic contacts in integrated circuits. When dimensions are reduced, interfacial planarity becomes obviously a matter of concern. In the absence of a theoretical modelisation of this morphological instability, we aimed at experimentally characterizing the Ni_3Si_2 microstructure in order for us to select among the tentative mechanisms of the instability. Preliminary work on the project indicated that an **in-situ** and **real-time** microtomographic characterization should be feasible. On this basis, 9 shifts were allocated for tests to our experiment on the ID19 breamline.

As regard the original aim of the proposal, that is **in-situ** and **real-time** microtomography, the experiments were unsuccesful. This was due to a sample environment problem and not to the X-ray source or X-ray characteristics at the beamline. The practical difficulty of the experiments we performed lies in the fact that both half of the diffusion couple should be maintained in contact in some way in the furnace + tomograph before the reaction begins. It turned out that the sample holder, which we had tested in a furnace providing radiative heating, was not really suited to the particular furnace available at the ID19 beamline. The latter is based on conduction heating and requires that a lot of attention is put on the efficiency of thermal contacts. During the duration of the experiments, we never succeeded in achieving high enough temperatures at the sample to start the diffusive-reaction. This problem should however be solved by a different design of the sample holder.

Yet, some success was achieved when we performed **post-mortem** tomographic scans of samples we had prepared beforehand in our laboratory. 4 scans were obtained for 4 different durations of the interdiffusion processus : t1 = 6h, t2 = 13h, t3 = 24h and t4 = 48h. All the 4 experiments had been performed at the temperature θ = 750°C. It is clearly seen from the reconstructed images (Fig. 1) that the method efficiently provides a map of the microstructure. From such data volumes, one may easily extract statistical properties of the collection of Ni_3Si_2 needles such as radius, height, shape. We are still investigating the data in order to check if density variations in the pure silicon phase (into which needles are growing) are measurable.

As a conclusion, this preliminary round of experiments, although unsuccesfull at providing **in-situ** and **real-tim**e data, proved that microtomography is perfectly suited for characterizing such a complex 3D microstructure as the one we want to study. Given the characteristic sizes,velocities and resolution involved, the feasibility of real-time measurements is assessed.

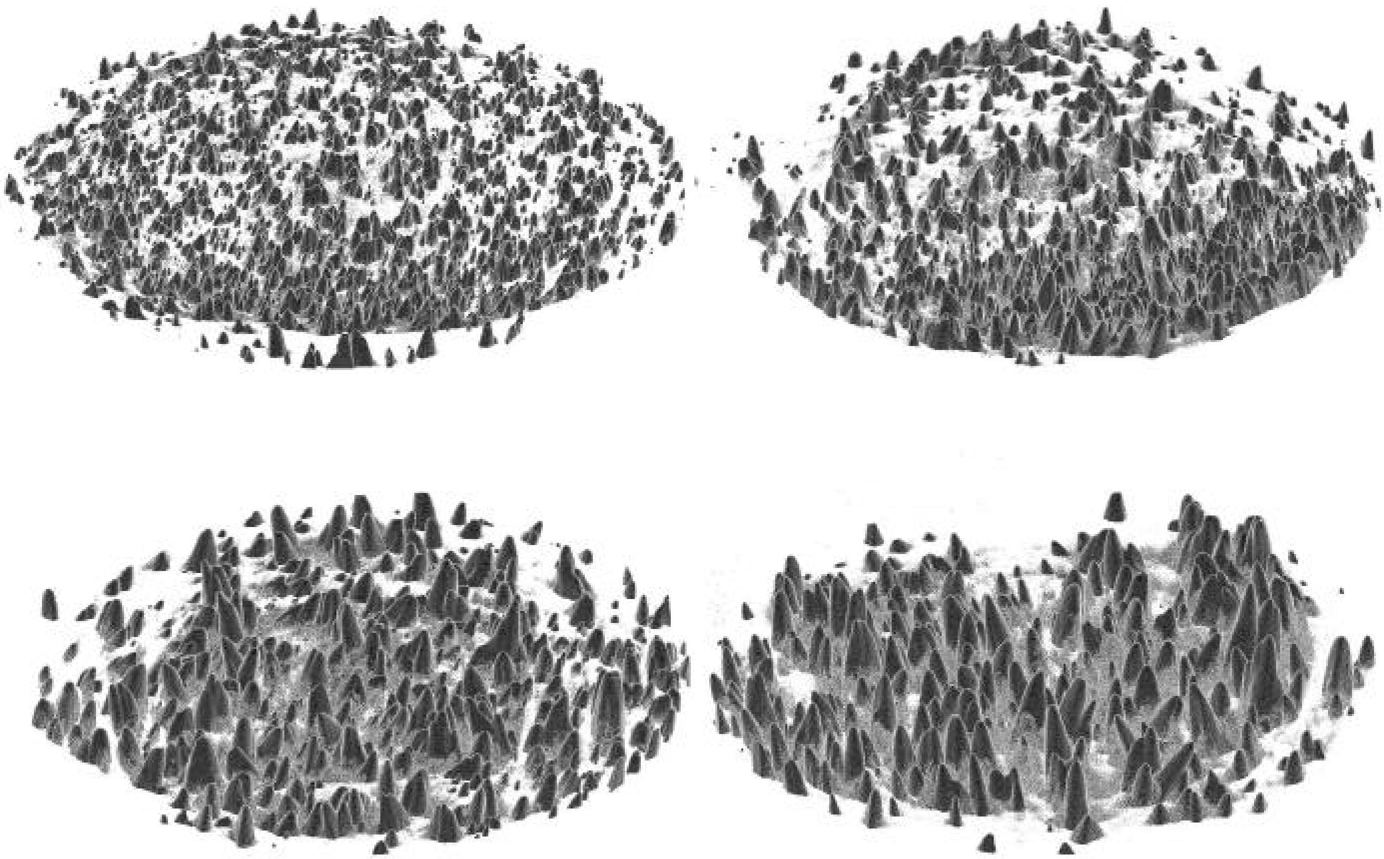

Figure 1 : Needle microstructure of the Ni_3Si_2 phase (grey/black). Experiments performed at θ = 750°C. From top left to bottom right : t = 6, 13, 24, 48h. Diameter of the samples : 3mm.

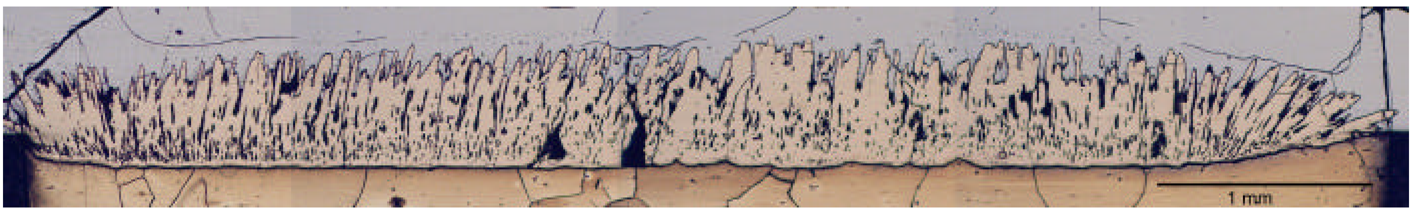

Figure 2 : Conventionnal observation by metallography and optical microscopy. θ = 750°C, t = 6h, diam. = 6mm.