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

Reports supporting requests for additional beam time

Reports can 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:** *Reactivity of Ti_3SiC_2 and liquid metallic alloys: from brazing applications to graphene-like material synthesis*	**Experiment number**: MA-1598
Beamline: ID15A & ID31	**Date of experiment**: from: 25/01/2013 to: 28/01/2013	**Date of report**: 04/02/2013
Shifts: 9 (ID15A) 6 (ID31)	**Local contact(s)**: Simon Kimber (ID15A) Christina Drathen (ID31)	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

- Dr. Andrieux Jerome*, Lab. des Multimatériaux et Interfaces, Université Lyon 1
- Dr. Dezellus Olivier*, Lab. des Multimatériaux et Interfaces, Université Lyon 1
- Samer Nassim*, Lab. des Multimatériaux et Interfaces, Université Lyon 1

Report summary:

The objectives addressed by the present proposal were to highlight the evolution of Ti_3SiC_2 in contact with a liquid metallic alloy (LMA) in terms of composition and structure. *In-situ* XRD experiments carried out on ID15A were chosen to follow Ti_3SiC_2 evolution during the reactivity with the LMA whereas *ex-situ* high resolution XRD (ID31) was selected to determine the structural modifications of Ti_3SiC_2 and identify the secondary phases formed.

No scientific result was obtained after this beamtime, mainly due to technical issues both from the beamline and the setup, even if 3 additional beamtime shifts were given (end of the beamtime on Tuesday 29th instead of Monday 28th). First of all, we gratefully acknowledge ID15 staff for the additional beamtime given. Secondly, we are strongly motivated in solving the setup issues in order to authorize unique in-situ liquid/solid reactivity studies. Once the setup will be validated, we will resubmit new proposals. Our only request might be to have a beamtime starting on Wednesday after a MDT day.

Technical aspects:

Technical aspects of the allocated beamtime are splitted in two paragraphs.

1. Beamline issues

Allocated beamtime was 9 shifts on ID15, starting on January, Friday 24th. Beamline staff support was excellent on Friday with, in addition of the local contact who did the alignement of the slits, beamstop and detector, the help of T. Buslaps for the detector physical installation, M. DiMichiel for Perkin Elmer detector tests and advices of use, A. Mauro for connection of eurotherm controllers to SPEC and M. Scheel for beam tuning. In addition, problem of front end closing due to local beamline interlock malfunction was observed and solved by the beamline staff. The induction furnace was ready to be installed only on Friday end of the afternoon and we did the furnace installation and connection on Friday night. On Saturday, we did the furnace alignment and then we prepared the first experiment. However, we found out many beamline problems. First problem was beam instability observed on ID15B. Tests were realized by M. DiMichiel and S. Kimber to understand the origin of this instability and to know the influence of this instability on ID15A beam. Second problem was SPEC communications with motors and counters. We asked the intervention of BLISS support to solve this problem. Third problem was the remote control of eurotherm controllers by SPEC due to wrong configuration of the eurotherm as well as bad temperature regulation on the sample for the same reason. Finally, we were able to run the first experiment on Sunday afternoon, after using 7 shits for beamline tuning and solving issues. MA-1598 beamtime was extended by 3 additional shifts until Tuesday 29th 8 a.m., and this additional beamtime was used to conduct 3 experiments. None of them was successful. Lack of scientific results is also due to setup issues as described in the following part.

2. Solid/liquid reactivity set-up

The furnace used for MA-1598 beamtime is available in the Sample Environment Pool at ESRF (Figure 1) and allows working under vacuum (10^{-5} mbar). However, the setup for liquid/solid reactivity studies was not existing before this beamtime. As soon as beamtime was allocated to this proposal, we got in contact with B. Gorges and H. Vitoux in order to develop the reactivity set-up (Figure 2). In addition, we came at ESRF to help in the development and the preliminary tests of the set-up in September (1 day), October (2 days) and December (3 days). More in details, the sample solid powder is placed in a graphite crucible (A, Figure 2) heated independantly by a resistive heater (B, Figure 2). The alloy (compacted powder) is placed in a graphite tube and melted by induction (C, Figure 2). In this way, the sample powder can be heated first to be desoxidized and the beginning of the reaction is controlled by the melting of the alloy. We wish to mention that a lot of work was done by H. Vitoux in order to prepare the furnace for MA-1598 and we gratefully acknowledge H. Vitoux and B.Gorges.

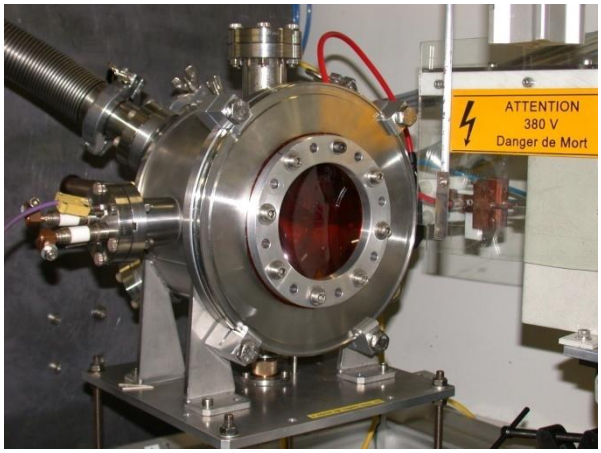

Figure 1. Induction furnace available at SEP, ESRF

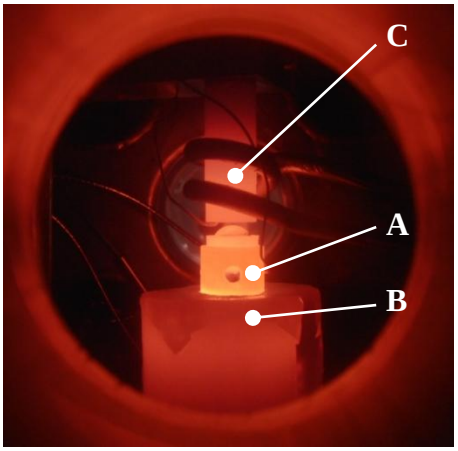

Figure 2. Liquid/solid reactivity set-up

Following MA-1598 beamtime, three technical issues were identified:

- Vacuum quality. Starting with a residual pressure of 10^{-5} mbar at 900°C, no wetting of the powder by the liquid alloy was observed. Different attempts were made during the beamtime to improve the vacuum quality and to decrease the oxygen partial pressure in the chamber, but still we were not able to observe the wetting of the powder by the liquid alloy. This explains the lack of scientific results after the beamtime.
- Temperature limitation on the sample to 900 °C. It is due to the heater capacity and the offset observed between the heater and the sample due to heat loss.
- No precise control of the volume of liquid in contact with the powder.

Despite the failure of MA-1598 beamtime, we are strongly motivated to develop a set-up allowing in-situ liquid/solid reactivity studies at ESRF, as this kind of studies is the heart of our research activity. We have already submitted a request for budget at the University Lyon 1 in order to make different modifications on the actual setup. For example, we are planning to heat both the sample and the alloy by induction, designing a new induction coil, in order to avoid the temperature limitation from the resistive heater. Then, we will design and build an automated system allowing the control of the liquid alloy slide on the powder. In addition, the use of getter inside the furnace combined with vitreous carbon as a material for the crucible are two approaches amongst others we will explore to improve the vacuum quality.

Finally, the 6 shifts of beamtime on ID31 were mainly based on samples obtained during the in-situ reactivity study carried out on ID15. Following the absence of successful experiment on ID15, we ask to postpone ID31 beamtime. It is to note that we will synthetize the samples ex-situ and be able to do the ID31 part of the proposal in the coming months.