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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:** Effect of processing parameters on the reaction kinetics of Fe-Al intermetallics by combined radiography and diffraction	**Experiment number**: MA-3401
Beamline: ID15A	**Date of experiment**: from: 12.07.2017 to: 16.07.2017	**Date of report**: 05.09.2017
Shifts: 12	**Local contact(s)**: Marco Di michiel	*Received at ESRF:*

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Report:

Objective & expected results

The aim of this work was to study the effect of processing parameters on the reaction kinetics of Fe-Al intermetallics. The processing strategy consists in infiltrating molten Al or Al alloy into a porous Fe-based preform to produce Fe-Al intermetallics. X-ray radiography (XR) is combined with X-ray diffraction (XRD) to bring information about reaction velocity and reaction front between molten Al and solid Fe. Reaction velocity and front propagation will be evaluated by image analysis of XR, while XRD data will be used for the analysis of type and amount of the formed phases during post-infiltration heating. In addition, the use of laboratory X-ray computed tomography (XCT) will provide detailed information about the microstructure of the infiltrated samples.

Results & conclusions

Two porous preforms with different void sizes and content were selected for investiagtion, namely selective laser melting (SLM) and Kochanek (KE). In situ infiltration of Al melt into Fe preform were performed combining radiography and diffraction measurements where a dedicated furnace was used. The energy was set to 50 keV and beam size to 4x4 mm. Pilatus area detector was used for XRD measurements while PCO edge camera was used for XR imaging. The in situ infiltration experiments were performed under argon atmosphere. SLM and KE samples (10x10x1.5 mm^3) were used. The Fe preform is placed in the lower part of the crucible inside the furnace while the Al plate is mounted above the Fe preform. After heating, the Al melt is injected into the porous Fe preform be means of a pneumatic actuator. The experiments were carried out at ID15A beamline where the temporal and spatial resolutions were a crucial factor for choosing this beamline. The analysis of the obtained data is not completed yet. Preliminary observations of one selected successful infiltration are reported below.

Radiographies and XRD patterns were recorded during the infiltration experiments. An important result for this study was the measurement of a clear evolution of the Debye-scherrer rings from the beginning of the experiment until full infiltration (Fig. 1). This confirms the formation of new phases and that the infiltration is reactive. In addition, the obtained radiographies show changes in the microstructure although the Al was invisible. The difference in image contrast is an indication of the formation of new phases (fig. 2). A complete analysis of the radiography images will be performed by making the difference of 2 consecutive images to extract quantitaive information of the infiltration and reaction process.

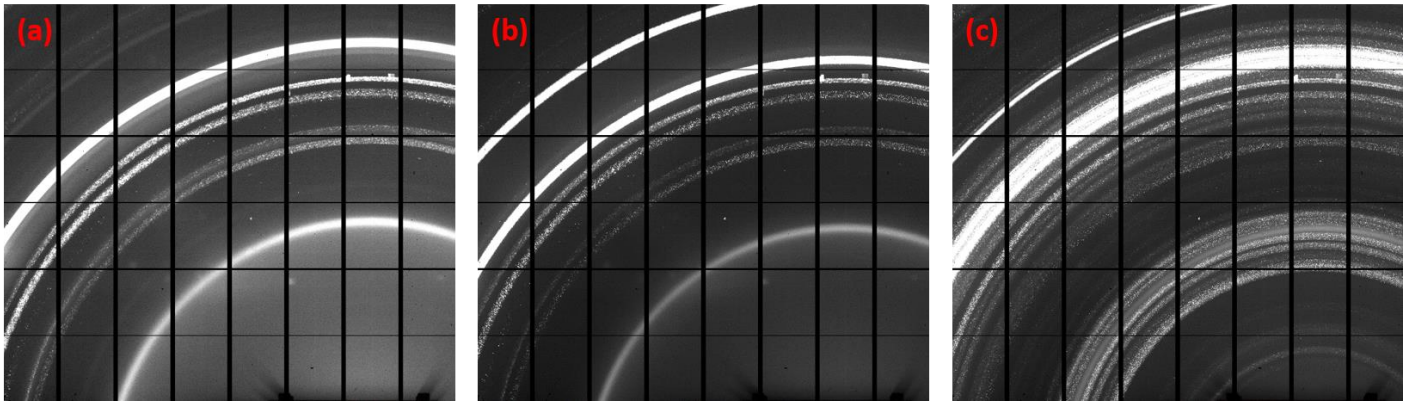

Fig. 1: The evolution of XRD patterns during infiltration of Al melt into KE sample; (a) before, (b) during and (c) after infiltration.

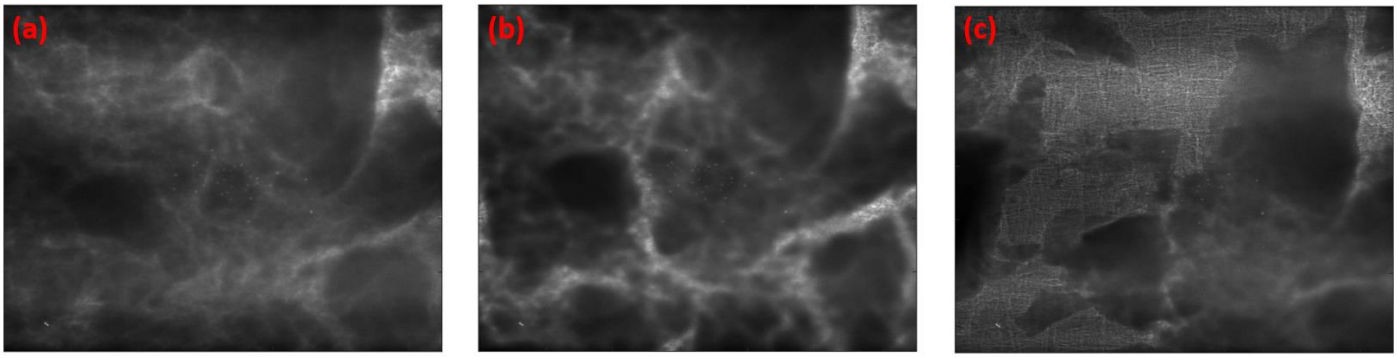

Fig. 2: The evolution of XR images during infiltration of Al melt into KE sample; (a) before, (b) during and (c) after infiltration.

Post-mortem analysis of the infiltrated sample by tomography gives more details about the reactive infiltration as shown in Fig. 3. The tomographic observations allowed to distinguish different phases where in particular, on top of sample in direct contact with the molten Al, a needle-like structure was observed. More details with different intensity contrasts are visible which confirmes the existence of new phases. Some parts with light contrast are also visible which are attributed to intermetalics with higher Fe contents or pure Fe grains (different contrast even in the “lighter” phases is observed) that didn’t react with Al or where Al didn’t reach. EBSD analysis is planned to identify precisely the formed phases to fully characterize the infiltrated material.

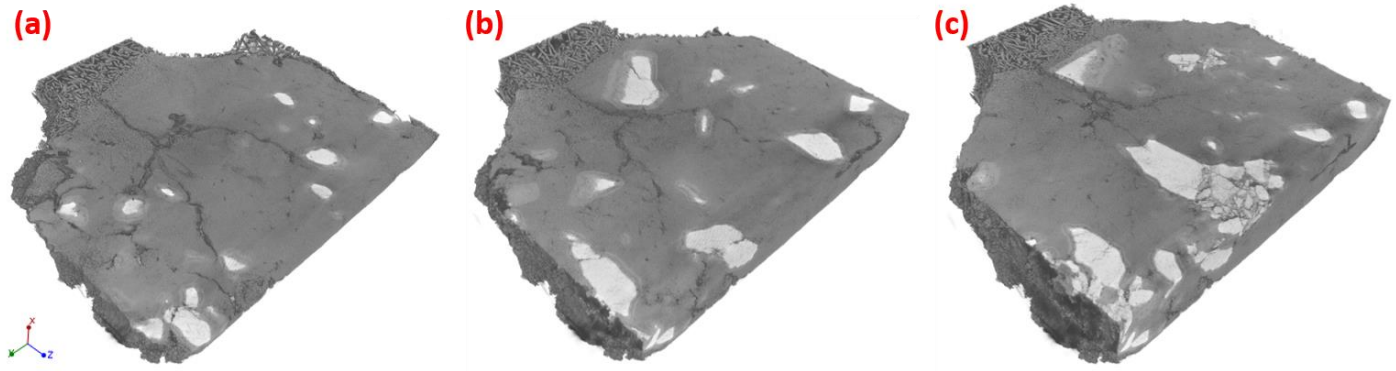

Fig. 3: (a), (b) and (c) Sections of tomographic data for infiltrated KE sample. The images show different structures and intensity contrasts which belong to different phases.

In summary, this experiment was very succeesful and demonstrates the importance of XR and XRD as powerful tools for observing infiltration and reaction process in situ. Full analysis of the data is in progress.