

Introduction :

A new method enabling the preparation of nanoporous structures in metals such as pure *Ti*, *Nb*, *Fe*, and *Cr* was developed [Wada, 2011]. Dealloying in the metallic melt is a selective dissolution phenomenon of a mono-phase bi-alloy solid precursor: one component (referred as soluble component) being soluble in the metallic melt while the other (referred as targeted component) is not. When the solid precursor contacts the metallic melt, only atoms of the soluble component dissolve into the melt inducing a spontaneously organized bi-continuous structure (targeted+sacrificial phases), at a microstructure level. This sacrificial phase is finally removed by chemical etching to obtain the final nanoporous materials.

The aim of this beamtime was to follow the full process in situ.

Objectives :

This projects aims at characterizing the full dealloying process in a metallic melt. The experiment was composed into 2 parts i) to follow the dealloying front in metallic melt bath and ii) to follow the dissolution of sacrificial phase.

FeCrNi, FeNi and TiCu precursors with Mg melt bath was studied to obtain porous FeCr, Fe and Ti.

Experimental setup :

X-Ray tomography, X-Ray diffraction and Diffraction Contrast Tomography set ups were installed and we could easily switch from one setup to an other but they couldn't be used simultaneously.

The beamline staff was very helpful to help us adapting our setup to the line.

Results :

Dealloying was followed by *in situ* X-Ray Diffraction and by *ex situ* X-Ray tomography.

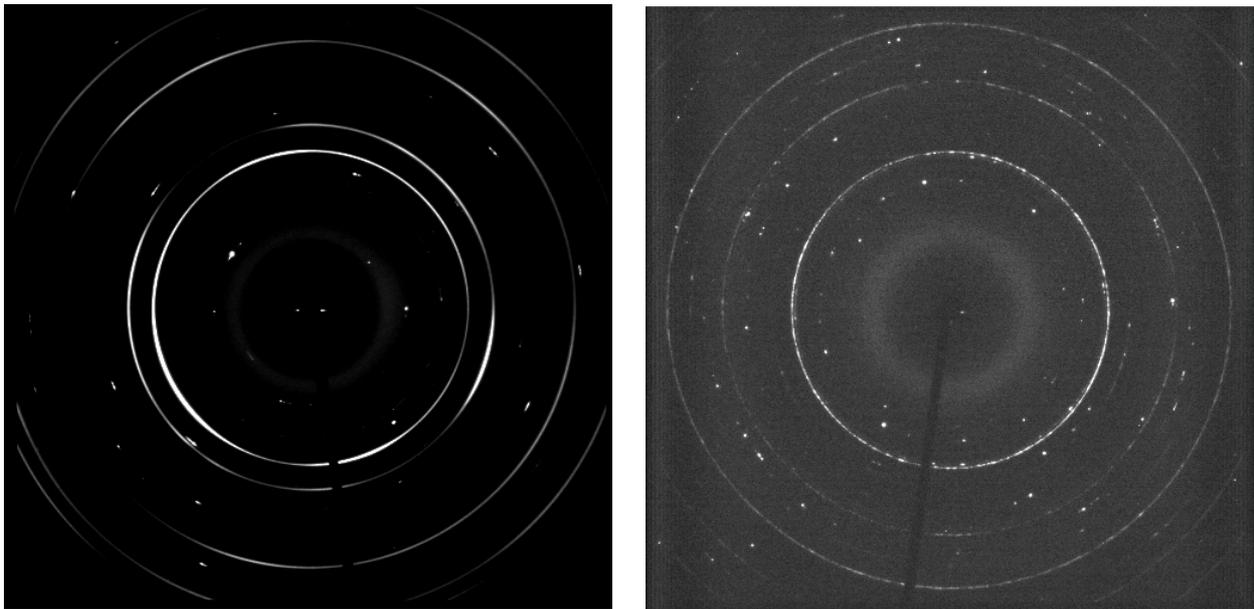


Fig. 1 (left) the initial step (right) after dealloying

During dealloying an allotropic transformation from FCC to BCC is expecting. Before and after dealloying X-Ray Diffraction patterns are different (as show in Fig. 1) so our first goal is achieved.

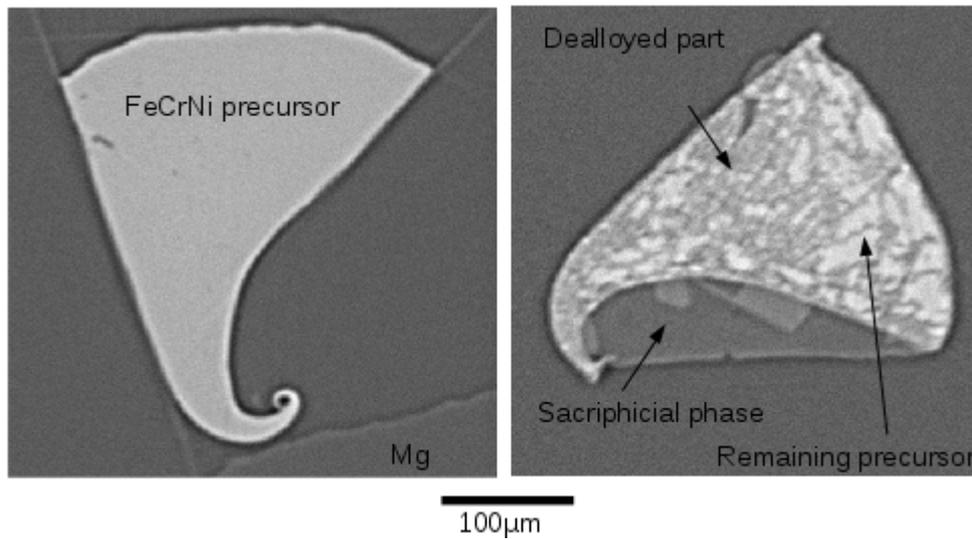


Fig. 2 : (left) precursor and Mg (right) after 1min dealloying at 770°C

Dissolution of sacrificial phase was followed *in situ* by X-Ray Diffraction (cf Fig. 3) and a X-Ray tomography scans were performed before and after dissolution (cf Fig. 4)

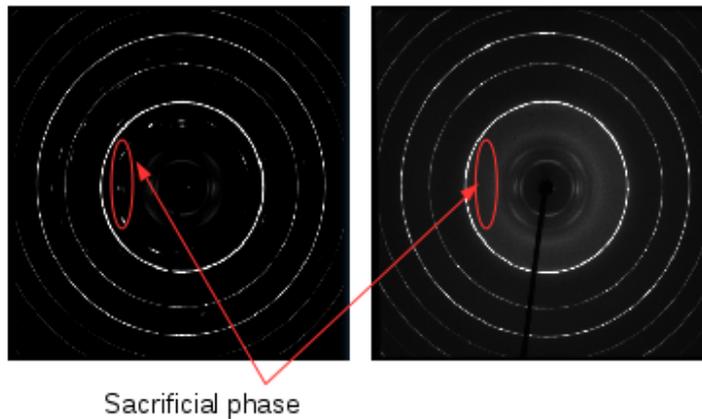


Fig. 3 : (left) dealloyed sample before etching (right) dealloyed sample after etching

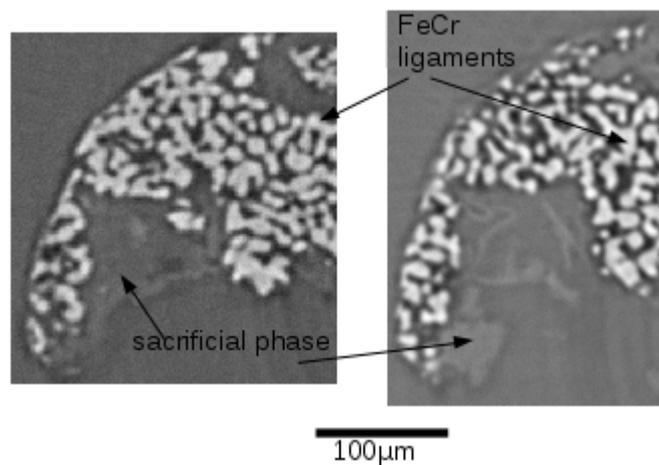


Fig. 4 : (left) dealloyed sample before etching (right) dealloyed sample after etching

Some sacrificial phase is remaining. The X-Ray Diffraction windows is smaller than X-Ray tomography windows so dissolution looks complete in X-Ray Diffraction pattern therefor it looks in progress in the X-Ray Tomography image.

We performed Diffraction Contrast Tomography scans on some restored precursors and dealloyed samples. Scans are not yet reconstructed but from diffraction pattern we are expecting goods results.

Conclusion :

During this beamtime we succeed by following the elaboration process of our foam by X-Ray tomography, X-Ray diffraction and shows the feasibility of using Diffraction Contrast Tomography of our samples. The analysis of the results is part of Morgane Mokhtari PhD works.

[Wada, 2011 a] T. Wada, K. Yubuta, A. Inoue, H. Kato. Mater. Lett., 65(2011), p. 1076