Elaboration of micro-porous powders by liquid metal dealloying

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Liquid metal dealloying (LMD) is a novel technique applicable on a wide range of metals to elaborate bicontinuous micro and nano composite and porous materials [1]. It consists in the selective dissolution of an element (Ni) from a precursor alloy (Fe-Ni) into a metallic liquid bath (Mg). Upon dissolution of Ni, Fe reorganizes into a porous connected structure.

Thanks to their large specific surface area, porous powders are good candidates as catalysts materials. Furthermore, powders being the key ingredient of metallic additive manufacturing and notably envisioned as promising feedstock for coating applications (cold-spray). This project aims at studying the dealloying reaction applied to powders in order to identify key process parameters enabling to control the final mophologies of the porous particules and their properties.

To elaborate dealloyed powders, Invar (Fe65Ni35) precursor powders were mixed with solvent Mg powders. This mixture was then heated and the dealloying reaction could happen upon the melting of Mg. Utilizing a powder mixture enabled precise control over the size of the Mg bath relative to the amount of precursor. Thus, the saturation of the Mg bath in Ni could be obtained, yielding partially dealloyed powders with a core-shell structure (Fig. 1). However, questions lingered about the role of intermetallic compounds in the dealloying reaction, the parameters influencing the ligaments composition and the kinetics of the reaction. To achieve a better understanding of the dealloying reaction, it was monitored using in situ X-ray diffraction at the ID11 beamline of the European Synchrotron Facility.

The various phases present throughout the dealloying reaction can be monitored and quantified via Rietveld analysis, as depicted in Fig. 2, revealing the kinetics of the process. These observations demonstrated how adjusting the thermodynamic conditions of the reaction (FeNi/Mg ratio, temperature) allows tailoring the microstructure of dealloyed powders, leading to the formation of core-shell structures with ligaments of predictable composition [2].

To assess the usability of these porous particles in additive manufacturing applications, compression tests were conducted on the porous particles. These tests highlighted the controllable mechanical weakening of the powders resulting from the dealloying reactions.

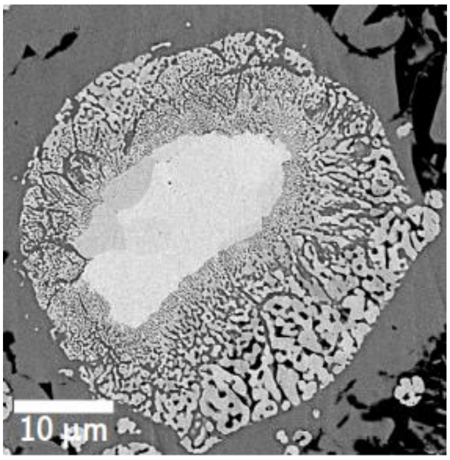

Figure 1: Partially dealloyed Invar powder presenting a core-shell morphology

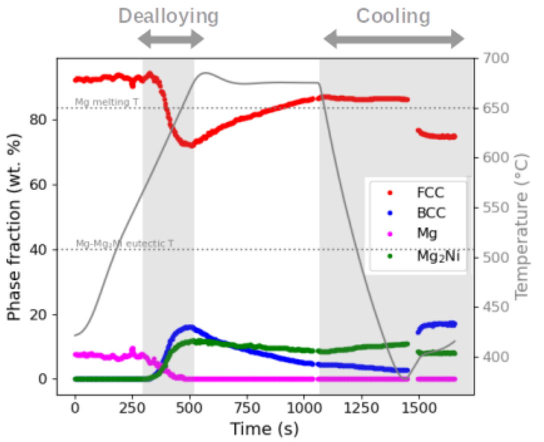

Figure 2: Evolution of phase fraction during the dealloying process monitored with in-situ XRD

References :

[1] Wada T, Yubuta K, Inoue A, Kato H. Dealloying by metallic melt. Materials Letters 15;65(7):1076–8, 2011.
[2] Lesage, L., Le Bourlot, C., Maire, E., Wada, T., Kato, H., Ludwig, W., Mary, N. and Geslin, P-A. Exploring equilibrium conditions in liquid metal dealloying of powders by in situ synchrotron X-ray diffraction. *Materialia*, *36*, p.102177, 2024.

Mots clés : porous metallic powders, dealloying, in-situ XRD