Experimental report

23/01/2024

Proposal: 1-01-200 **Council:** 4/2023

Title: Phase evolution in Mg alloys powders for Additive Manufacturing during solidification and heat treatments

Research area: Materials

This proposal is a new proposal

Main proposer: Borja PILLADO RIOS

Experimental team:

Local contacts: Sandra CABEZA
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Samples: AZ31 (Mg-3%Al-1%Zn(wt.%))
Mg-Y-Zn: Y 6.9 Zn 2.5 CaO 0.3 Mg Bal (wt%)
Mg-Y-Gd-Zn: Y 6.4 Gd 5.7 Zn 2.4 CaO 0.3 Mg Bal (wt%)
Mg-Y-Nd-Zn: Y 6.4 Nd 5.3 Zn 2.4 CaO 1.3 Mg Bal (wt%)
WE54 (Mg-Y-Nd): Y 5 Nd(mm) 4 Mg Bal (wt%)
Mg-Ni-Y-Ce: Y 3.3 Ni 4.4 Ce(mm) 5.23 Mg Bal (wt%)

Instrument	Requested days	Allocated days	From	To
D1B	4	3	05/09/2023	08/09/2023

Abstract:

The aim of the present proposal is fully characterized the crystalline structure and phases of AZ31 Mg alloy powders. These powders are the starting raw material, which will be further used in Additive Manufacturing for printing objects. After a complete characterization with ex situ microstructural studies (X-rays, SEM, EDX, TEM, and DSC), we want to carry out neutron diffraction (ND) experiments during in-situ experiment in D1B of melting and solidification of feedstock powders at different cooling rates, and then followed by annealing towards simulating their evolution during AM. Room temperature and in-situ neutron diffraction experiments will be carried out during the heating and cooling treatments at different heating/cooling rates. This proposal is submitted for the allocation of a beamline for the fulfilment of the scientific objectives set out in the postdoctoral project of Borja Pillado Ríos, currently under contract with the Institut Laue Langevin (ILL) since February 1st, in the framework of the Scientific Partner agreement between the Ministry of Science and Innovation (MCIN) and the Institu

ILL-D1B 1-01-200 Phase evolution in Mg alloys powders for Additive Manufacturing during solidification and heat treatments

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The aim of this research was to employ neutron thermodiffraction to examine the crystallographic structures of three magnesium (Mg) alloys and track their transformations across a range of temperatures during different phases of the additive manufacturing (AM) process. MgYZn, MgYNdZn and WE54 were selected for this proposes. The interest of this alloys remains in the secondary phases that is formed intergranurally, which has a quasi-crystalline long-period stacking ordered (LPSO) structure. The LPSO structure offers potential enhancements in the strength and flexibility of magnesium alloys, thereby promising improved mechanical properties.Neutron powder diffraction (ND) experiments were executed at the D1B facility of the ILL, utilizing two distinct wavelengths: λGe(311) = 1.28 Å and λPG(002) = 2.52 Å. These experiments were performed using the vanadium induction furnace F2 of the D1B instrument.

Initially, 50 mg of each Mg alloy powder was individually examined in separate vanadium tubes to establish reference conditions at room temperature (RT). Subsequently, the furnace was configured for the thermal cycling experiments. For these experiments, specialized quartz tubes with dimensions of 330x10mm were employed to encase the samples while maintaining a precisely controlled 150 mbar Ar atmosphere, replicating the conditions used in the AM process. The temperature was raised in 50°C increments at a consistent rate of 10°C per minute until the respective melting points were attained. These experiments were carried out at temperatures of 200, 250, 300, 350, 400, 450 and 500°C (each for a duration of 60 minutes for 1.28 Å plus 30 minutes for 2.52Å). The samples were allowed to naturally cool to room temperature, where an additional measurement was taken. ND patterns were recorded during the cooling phase, with measurements conducted every 5 minutes.. The determination of structural parameters was achieved using the Rietveld method with the aid of the Mag2Pol software from ILL.

MgYZn Alloys Thermo-diffraction Study:

Figure 1 illustrates the diffraction of the MgYZn alloy across various temperature stages. The α-Mg peaks were indexed to the space group P63/mmc. Notably, lattice parameters increased as the temperature increased, as shown in Table 1. The estimated thermal expansion coefficients for the a and c parameters between 200 and 550°C were 3.4x10-5 and 5.8x10-5 ºC-1, respectively.

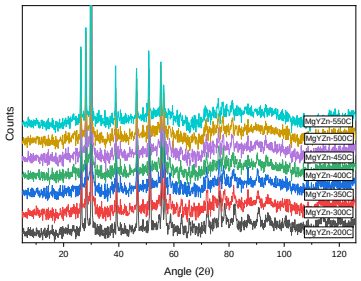

Figure 1. Diffraction patterns of MgYZn alloy in simulated AM fabricated condition

Furthermore, peaks associated with the secondary LPSO phase may be present in the diffraction patter. However, characterizing the precise nature of this phase has proven challenging. It is speculated that the liquefied Mg matrix may facilitate the formation of MgO due to oxygen present within the powder pores. This novel phase at 600°C is attributed to a cubic structure with a space group of Pm-3m and a lattice parameter of 3.5 Å. It predominantly forms under high-temperature, low-oxygen conditions and gradually diminishes as the metal solidifies.

MgYNdZn:

For the MgYNdZn alloy, the neutron thermodiffraction study aimed to examine its crystallographic structures as they evolve with temperature during different AM process phases. Neutron powder

diffraction (ND) experiments were conducted at the D1B facility of the ILL, employing wavelengths λGe(311) = 1.28 Å and λPG(002) = 2.52 Å, with the vanadium induction furnace F2 of the D1B instrument used for the experiments.

As seen in Figure 1, the diffraction patterns for MgYNdZn reveal that the alloy reaches its melting temperature before the cooling phase. The α-Mg peaks were indexed to the space group P63/mmc. The lattice parameters for the a and c axes increased with rising temperature (as shown in Table 1). The estimated thermal expansion coefficients for these parameters between 200 and 550°C are 3.4x10-5 and 5.8x10-5 ºC-1, respectively.

Additionally, at 344°C, new peaks were observed, indicative of a new phase formation. However, defining the nature of this new phase has posed a challenge. The presence of oxygen within the powder pores is thought to enhance the formation of MgO when the Mg matrix is liquefied. This newly formed phase at 344°C is attributed to a cubic structure with a space group of Pm-3m and a lattice parameter of 3.5 Å. It mainly emerges under high-temperature, low-oxygen conditions and gradually diminishes as the metal solidifies.

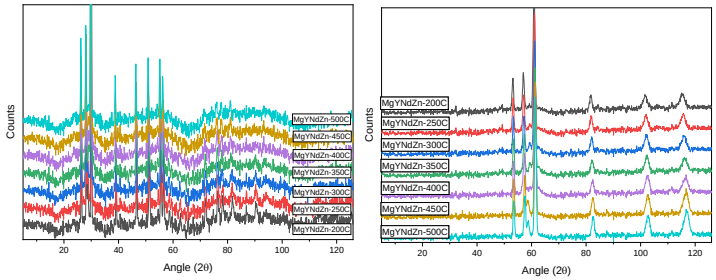

Figure 2. Diffraction patterns of MgYNdZn alloy in simulated AM fabricated condition

WE54 (MgYNd):

In the case of the WE54 (MgYNd) alloy Figure 3 showcases diffraction patterns at different temperatures, indicating that the alloy reaches its melting temperature before the cooling phase. The α-Mg peaks were indexed to the space group P63/mmc. Lattice parameters increase with rising temperature, as detailed in Table 1. The estimated thermal expansion coefficients for the a and c parameters between 200 and 550°C are 3.4x10-5 and 5.8x10-5 ºC-1, respectively.

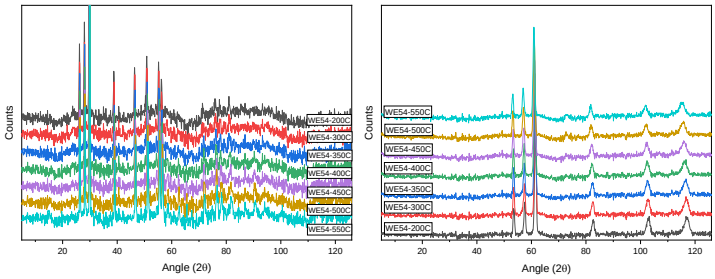

Figure 3. Diffraction patterns of WE54 alloy in simulated AM fabricated condition

Moreover, at 344°C, new peaks emerged, suggesting the formation of a novel phase. However, characterizing the exact nature of this phase has been challenging. It is speculated that when the Mg matrix liquefies, it may promote the formation of MgO due to the presence of oxygen molecules within the powder pores. This newly formed phase at 344°C is attributed to a cubic structure with a space group of Pm-3m and a lattice parameter of 3.5 Å. It predominantly forms under high-temperature, low-oxygen conditions and gradually diminishes as the metal solidifies.

CONCLUSIONS:

A comprehensive assessment of the structural transformations throughout the AM process of two Mg alloys AZ31 and BAS1 has been achieved in this beam time campaign. The preliminary analysis of the data has revealed the evolution of the alpha-Mg cell parameter against temperature until melting, serving as input for the additive manufacturing process. This results contributes to the knowledge of Mg powder metallurgy and their viability and optimization towards AM processing routes. A forthcoming paper is in preparation to be submitted to peer review scientific journalx.