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IN-SITU SYNTHESIS OF Mg_2Si-REINFORCED AMC

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Abstract

Aluminium Matrix Composites (AMC) are advanced materials composed of an aluminium alloy reinforced with ceramic or metallic particles, fibres, or whiskers to enhance properties such as strength, stiffness, wear resistance, and thermal stability. These composites offer a high strength-to-weight ratio, improved mechanical performance, and corrosion resistance, making them ideal for aerospace, automotive, and defence applications.

The purpose of the research was to synthesise, homogenise and characterise Al-Mg_2Si composite samples. We plotted the equilibrium phase diagram and Scheil curves using the Thermo-Calc software. Based on the results of the thermodynamic simulations of equilibrium and non-equilibrium solidification, we prepared the appropriate raw materials and melted them in a BN-coated steel crucible in an induction furnace. The most appropriate ratio was calculated to be Mg/Si = 1.74. Due to the high casting temperature, shrinkage and gas porosity occur in the samples. The cooling rate during solidification should be as high as possible to achieve the appropriate microstructure. We also performed simple thermal analysis (ETA) and plotted cooling curves in the Origin software and differential scanning calorimetry (DSC). Microstructural characterisation was also carried out to define the type, proportion and morphology of phases in the as-cast and homogenised state.

According the thermodynamic calculation under all conditions the primary phase Mg_2Si solidifies first, then the $Al_{13}Fe_4$ phase and finally the eutectic (α_{Al} + Mg_2Si). It is also possible that the eutectic (α_{Al} + Mg_2Si + β_{Si}) solidifies last. The results of the DSC and metallographic analysis show the primary Mg_2Si phase, the eutectics (α_{Al} + $Al_{13}Fe_4$), (α_{Al} + Mg_2Si) and (α_{Al} + Mg_2Si + β_{Si}) in the as-cast samples. The solidification of the investigated composite takes place in the range between 750 ºC and 540 ºC. Upon the homogenisation process, the rounding of the edges of the primary solidified Mg_2Si particles, and the rounding and growth of the eutectic Mg_2Si phase was observed in the microstructure.

Keywords*: Aluminium matrix composite (AMC), primary Mg_2Si, Thermodynamics, DSC*

1. INTRODUCTION

Composites are mixtures of two or more solid materials that are, in principle, mechanically separable and exhibit complementary properties [1]. They consist of a matrix and a reinforcement phase [2]. In recent years, aluminium matrix composites (AMCs) have gained increasing attention in advanced structural and functional applications. Due to their low density and high performance, aluminium and its alloys are widely employed in the automotive and aerospace industries [3, 4]**.** AMCs offer exceptional properties such as high specific strength, excellent wear and corrosion resistance, low density, and high specific stiffness. Aluminium composites can be reinforced with particles, whiskers or short fibres, continuous fibres, and monofilaments. Among the particle-reinforced AMCs, composites strengthened by the intermetallic Mg_2Si phase (Al-Mg_2Si) have recently emerged as particularly attractive to researchers and engineers. These composites can be produced using relatively simple casting techniques, whereby the Mg_2Si phase is formed "*in situ*" [5] through a chemical reaction between silicon and magnesium. Al-Mg_2Si composites represent a new class of ultralightweight materials with significant potential for aerospace, automotive, and other high-performance applications [6]. The Mg_2Si compound is characterized by low density, a high melting point, a high elastic modulus, and excellent hardness [7].

Nevertheless, during solidification, Mg_2Si particles tend to crystallize as coarse, irregularly shaped particles that are often inhomogeneously distributed within the matrix. This can adversely affect the mechanical properties, particularly tensile strength. These issues can be mitigated through homogenisation treatment, which reduces the effects of microsegregation [8]. During homogenisation, the particles of both, the primary and eutectic Mg_2Si phases, become more spheroidal [6].

The objective of research was to synthesize, homogenise, and characterise Al-Mg_2Si composite samples subjected to various annealing durations, employing light and electron microscopy as well as differential scanning calorimetry (DSC).

2. EXPERIMENTAL

To produce the Al-Mg_2Si composite, designed to contain 18–20 wt.% Mg and 10–11 wt.% Si with a Mg/Si mass ratio of 1.74, 1629 g of AlSi10Mg alloy and 139 g of high-purity magnesium (99.9%) were weighed. Due to the high reactivity and rapid oxidation of magnesium, an additional 10 wt.% excess was added. The composite was melted in an induction furnace using a steel crucible coated with BN. The melt was intermittently stirred with a steel rod, ensuring the temperature did not exceed 770 °C. After melting, the alloy was cast into two moulds: a steel bar-shaped mould preheated to 450 °C, and a Croning measurement cell for simple thermal analysis (ETA). Homogenisation of the samples was carried out in an electric resistance furnace—initially at 430 °C for 4 hours, followed by further annealing at 520 °C for durations of 12, 24, 48, and 96 hours. The equilibrium phase diagram and Scheil solidification curves were calculated using the Thermo-Calc software. After the thermal analysis (ETA) was performed, and cooling curves were plotted using Origin software. Additionally, differential scanning calorimetry (DSC) was conducted. Microstructural characterization was carried out to identify the type, volume fraction, and morphology of phases in both, the as-cast and homogenised samples.

3. RESULTS AND DISCUSION

Based on thermodynamic calculations using the Thermo-Calc software, equilibrium solidification proceeds in the following sequence: primary Mg_2Si and $Al_{13}Fe_4$ phase, primary α_{Al} dendrites, eutectic (α_{Al} + Mg_2Si + $Al_{13}Fe_4$), and finally eutectic (α_{Al} + Mg_2Si + $Al_{13}Fe_4$ + β_{Si}). Non-equilibrium solidification, as predicted by the Scheil model, occurs in the following order: solidification of the primary Mg_2Si phase, followed by primary $Al_{13}Fe_4$ phase, and then the eutectic (α_{Al} + Mg_2Si) (Fig. 1). In cases where only magnesium is a fast-diffusing element—or when neither magnesium nor silicon are fast diffusing—the eutectic (α_{Al} + Mg_2Si + β_{Si}) also forms at lower temperatures. Experimental thermal analysis (ETA) revealed that solidification of the primary Mg_2Si phase begins at 705.9 °C. The eutectic (α_{Al} + Mg_2Si) solidifies at 580.0 °C, followed by the eutectic (α_{Al} + β-Al_3Mg_2) at 447.8 °C. Final solidification is completed at 433.9 °C. From the heating DSC curve of the as-cast sample, the melting temperatures of the following phases were determined: at 450.6 °C the eutectic (α_{Al} + β-Al_3Mg_2); at 525.5 °C the eutectic (α_{Al} + Mg_2Si + β_{Si}); at 589.5 °C the eutectic (α_{Al} + Mg_2Si); and at 598.7 °C the eutectic (α_{Al} + $Al_{13}Fe_4$). The primary Mg_2Si phase melts over a temperature range from 620.2 °C to 783.4 °C. From the cooling DSC curve of the same sample, the solidification sequence was determined as follows: solidification of the primary Mg_2Si phase begins at 751.5 °C, followed by the eutectic (α_{Al} + $Al_{13}Fe_4$) at 589.6 °C, and the eutectic (α_{Al} + Mg_2Si) at 582.9 °C. Significant differences were observed in the melting and solidification enthalpies of the primary Mg_2Si phase, particularly between samples K-0 and K-96 (Fig. 2), indicating that the sample homogenised for 96 hours contains the lowest amount of primary Mg_2Si. It was further observed that the eutectic (α_{Al} + β-

Al_3Mg_2) is present only in the as-cast sample and disappears after homogenisation. Light and scanning electron microscopy revealed that the polygonal morphology of the primary Mg_2Si phase begins to round after 24 hours of homogenisation and becomes significantly more rounded after 96 hours (Fig. 3). With increasing homogenisation time, the eutectic (α_{Al} + Mg_2Si) also becomes finer and more spheroidized (Fig. 3).

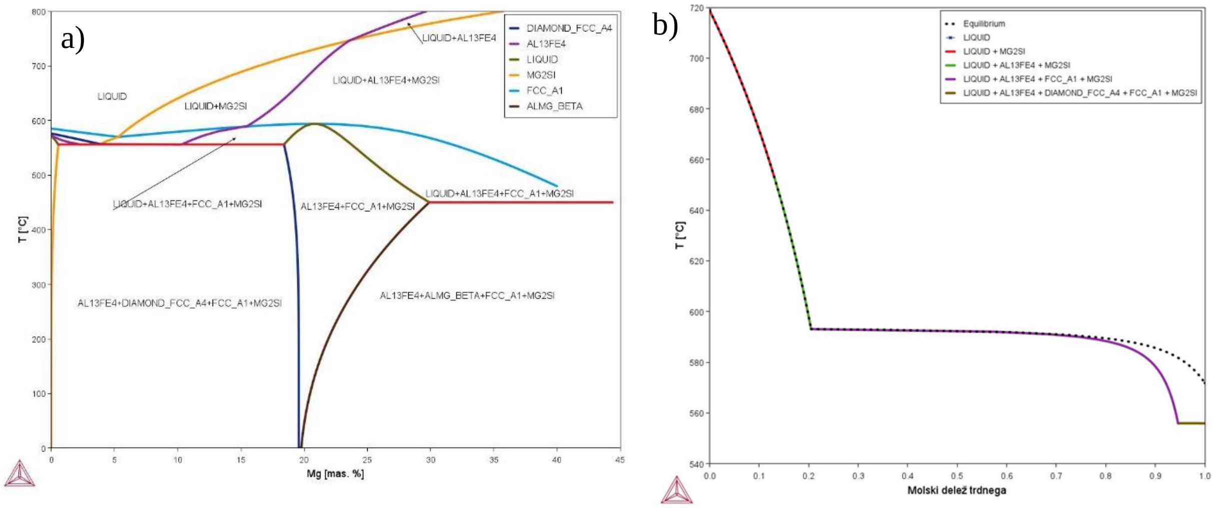

Figure 1. Thermodynamic calculation of Al-Mg-Si-Fe system, a) equilibrium isopleth phase diagram and b) Scheil model of solidification

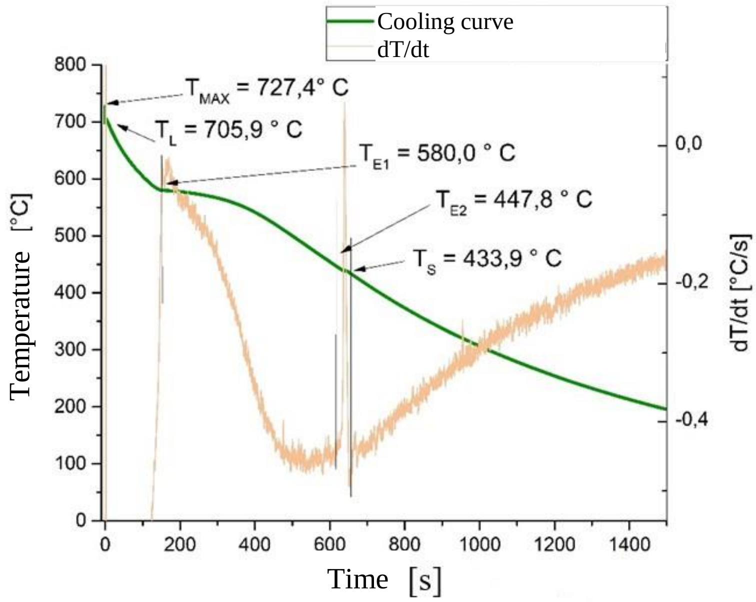

Figure 2. Cooling curve of synthetised Al-Mg_2Si composite

Point and area EDS analyses (Fig. 4) confirmed the presence of the following phases in the microstructure of both, the as-cast and homogenised samples: the primary Mg_2Si phase, the eutectic $Al_{13}Fe_4$ phase, mixed α_{Al} crystals enriched with magnesium, and the eutectic (α_{Al} + Mg_2Si).

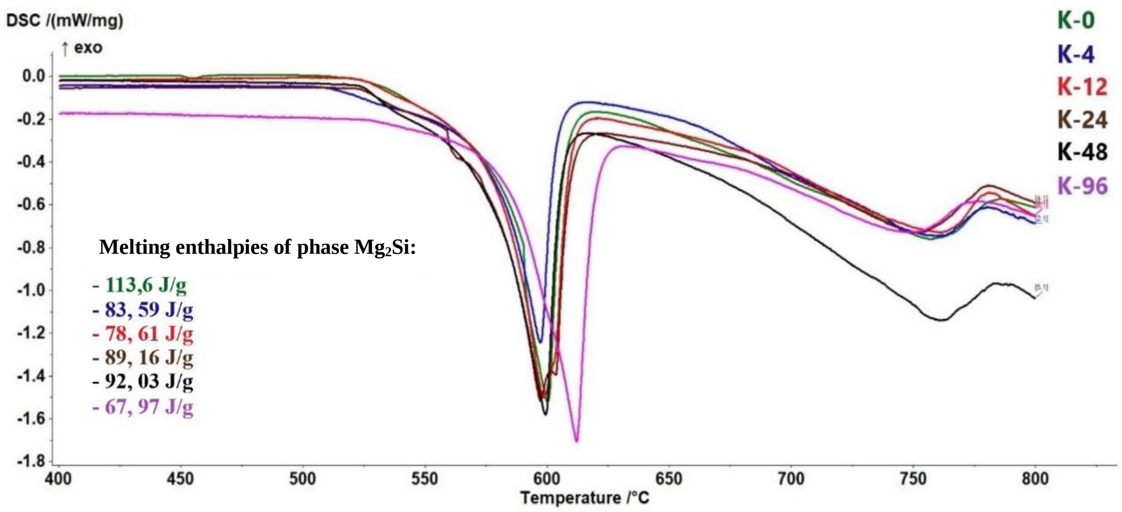

Figure 3. Comparison of heating DSC curves of samples K-0, K-4 and K-96

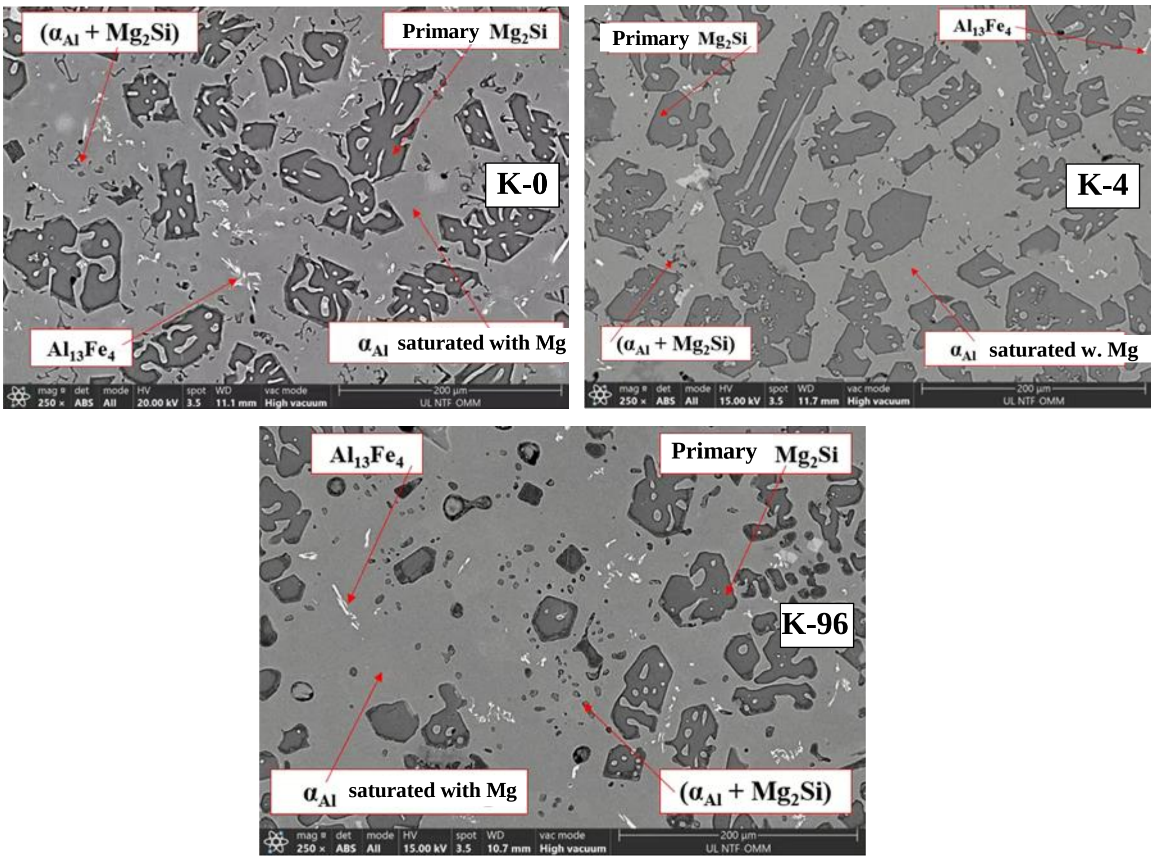

Figure 4. Microstructures of samples K-0, K-4 and K-96

4. CONCLUSIONS

The investigation of the synthesized and homogenised in-situ aluminium matrix composite (AMC) revealed that, based on the DSC heating curve, solidification occurs in the following sequence: at 751.5 °C solidification of the primary Mg_2Si phase begins; at 589.6 °C solidification of $Al_{13}Fe_4$ phase follows; and at 582.9 °C the eutectic (α_{Al} + Mg_2Si) solidifies. In the case of sample K-96, the eutectic (α_{Al} + Mg_2Si + β_{Si}) solidifies last, at 540.0 °C. The microstructure of both, the as-cast and homogenised samples, contain the following constituents: the primary Mg_2Si phase and the eutectics (α_{Al} + $Al_{13}Fe_4$), (α_{Al} + Mg_2Si), and (α_{Al} + Mg_2Si + β_{Si}). The eutectic (α_{Al} + β-Al_3Mg_2) is present only in the as-cast sample and disappears after homogenisation. Morphological changes were observed in the primary Mg_2Si phase: polygonal particles began to round after 24 hours of homogenisation and became significantly more rounded after 96 hours. With increasing homogenisation time, the (α_{Al} + Mg_2Si) eutectic also became finer and more spheroidized. Based on the measured melting enthalpies, it was concluded that the initial as-cast sample (K-0) contains the highest amount of primary Mg_2Si phase, while the sample homogenised for 96 hours (K-96) contains the lowest.

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