Materials Chemistry and Physics 194 (2017) 65–76

Contents lists available at ScienceDirect

Materials Chemistry and Physics

journal homepage: www.elsevier.com/locate/matchemphys

Microstructural evolution and strengthening behavior in in-situ magnesium matrix composites fabricated by solidification processing

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HIGHLIGHTS

- In-situ magnesium composites were fabricated using liquid stir-casting method.
- In-situ pyrolysis of cross-linked polymer has been utilized to obtain ceramic phases.
- Mg_2Si crystals were formed in magnesium and AE44 matrix composites but not in AZ91 matrix composites.
- The variation in size and morphology of Mg_2Si crystals with matrix materials are discussed.
- Strengthening mechanisms in in-situ composites are analyzed and discussed.

ARTICLE INFO

Article history:
Received 11 November 2016
Received in revised form
20 January 2017
Accepted 15 March 2017

Keywords:
Metal matrix composites (MMCs)
In-situ processing
Polymer derived ceramics
Microstructural evolution
Strengthening mechanisms

ABSTRACT

In-situ magnesium matrix composites with three different matrix materials (including Mg, AZ91 and AE44 Mg-alloys) were fabricated by injecting cross-linked polymer directly into the molten Mg/Mg-alloys, and having it convert to the 2.5 vol% SiCNO ceramic phase using liquid stir-casting method. In-situ chemical reaction took place within the molten slurry tending to produce 42 and 18 vol% Mg_2Si crystals in Mg and AE44 matrix composites, respectively but not in AZ91 matrix composite. Microstructural evolution of Mg_2Si crystals was discussed on the basis of availability of heterogeneous nucleation sites and amount of Al-atoms in the molten slurry. The observed micro-hardness and yield strengths are enhanced by factor of four to three as compared to their unreinforced counterparts, and Taylor strengthening was found to be the predominant strengthening mechanism in magnesium and AE44 matrix composites. Summation model predicted the yield strengths of the fabricated composites more preciously when compared to Zhang and Chen, and modified Clyne models.

1. Introduction

Mg-alloys and Mg-based metal matrix composites (MMCs) have significant potential to design the next generation automotive and aerospace vehicles owing to high specific strength and reduced fuel consumption [1–3]. Solidification processing is the most economical, viable and versatile process to fabricate MMCs for large scale manufacturing sectors. Pradep et al. [4] reviewed the important solidification principles underlying the microstructure evolution which governs the composite properties. Surappa [5] investigated particle-solidification front interactions in different MMCs, and concluded that intrinsic material properties as well as processing variables play a major role in controlling the microstructural evolution in the final MMCs. However, solidification processing is not well-suited for the dispersion of fine-sized particles due to chance of producing severe agglomeration by Vanderwals force of attraction, non-uniform particle distribution and poor wettability between ceramic-metal interfaces. Such large and hard agglomerates could be disintegrated by electromagnetic stirring (EMS) and ultrasonic agitation to some extent [6,7].

Ahmed et al. [6] justified that EMS results in an elevated shearing force which tends to break away the clustered nano-sized SiC particles and produces an intimate physical contact between nano-sized particles and the melt. Li et al. [7] proposed ultrasonic assisted melt processing technique that produces transient cavitations which tend to break up the clustered fine-sized particles. However, these techniques are more complex and too expensive for

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http://dx.doi.org/10.1016/j.matchemphys.2017.03.025
0254-0584/

commercial exploitation. Most of the technical challenges in solidification processing can be greatly minimized by adopting in-situ composite approach by which ceramic particles are generated within the molten state via chemical reaction between the added precursor and the host metal. In-situ composites offer superior microstructural/mechanical characteristics as compared to their conventional counterparts. For instance, in-situ MMCs comprised uniform dispersion of fine-sized thermodynamically stable ceramic particles, clean and unoxidized ceramic-metal interfaces along with high interfacial strength, improved hardness/yield strength and elastic modulus. Tjong et al. [8] discussed various possibilities of producing in-situ MMCs with different matrix materials and their potential applications. Shamekh et al. [9] investigated reaction mechanisms for in-situ AZ91 matrix composites and concluded that intensity of formation of TiC_x-TiB_2 phases increases monotonically with increasing process temperature and time.

Sachin et al. [10] applied an ultrasonic vibration to improve the dispersion of in-situ formed Mg_2Si particles in the molten AZ91 composite slurry. Sudarshan et al. [11,12] developed a novel processing approach to fabricate in-situ MMCs by utilizing pyrolysis of polymer precursor (polysilazanes) within the molten Mg. Noteworthy feature of this technique is that no chemical reaction between polymer precursor and the host Mg-alloy is required to produce ceramic particles as polymer contains all the constituents of ceramic phases within organic molecules itself.

Recently, N.M. Chelliah et al [13] fabricated magnesium matrix composites containing nano-sized SiCNO ceramic particles using two-stage (casting as well as friction stir processing) method. Further, they also analyzed the strengthening mechanisms and numerical models in two-stage processed composites. However, no attempt has been made to reinforce SiCNO phase or polymer derived ceramic (PDC) particles into commercial Mg-alloys such as AZ91 and AE44 to the best of our knowledge. Sudarshan et al. [11] observed that certain degree of chemical reaction between polymer precursor and the host Mg melt results in the formation of Mg_2Si particles at 800 °C. Mg_2Si is considered to be an attractive reinforcing phase owing to its superior mechanical and thermal characteristics to fabricate in-situ Al-Mg_2Si and Mg-Mg_2Si MMCs. However, the presence of coarsened Mg_2Si phase impairs the ductility of the composites.

Zhang et al. [14] refined the size of in-situ formed primary Mg_2Si phase by the addition of extra Si particles into molten Al-Mg-Si alloy. Li et al. [15] investigated the possibilities of evolving various morphologies in Mg_2Si crystals such as faceted octahedron, hopper octahedron, truncated octahedron and enormous dendrite in Al-Mg-Si alloy. Main objective of the present paper is two-fold: (i) to understand the microstructural evolution in in-situ MMCs dispersed with PDC particles at a temperature of 900 °C and (ii) to investigate their strengthening behavior and validate the numerical models for determining the yield strength of the composites. If Mg_2Si crystal is formed in the final MMCs, it is worthwhile to investigate how the morphology and size of Mg_2Si crystal differ with the matrix materials as it greatly influences the mechanical properties of the final MMCs.

2. Experimental methods

2.1. Matrix materials and polymer precursor

Commercial purity Mg (99.9%), AZ91 (99.7%) and AE44 (99.7%) Mg-alloys are chosen as the matrix materials. The chemical compositions of reinforced Mg-alloys are provided in Table 1. Poly (ureamethylvinyl)silazanes (PUVMS) (Ceraset, Kion Corporation), was utilized as a precursor for reinforcement.

The precursor in its native liquid form was thermally cross-linked at 400 °C for 5 h in Argon atmosphere to form a hard epoxy like material. Then organic epoxy was ball milled, yielding solid polymer powder (angular morphology) ranging in size from 1 to 100 μm with a density of 1.0 gcm^{-3}.

The chemical composition of as-received and cross-linked PUVMS polymer powder is provided in Table 2. The weight fraction of polymer precursor to be added into molten Mg/Mg-alloys can be estimated by the following formula [13]:

$$W_f^{precursor} = \frac{\rho_{PDC}}{0.85\rho_{matrix}} V_f^{PDC} \quad (1)$$

The factor of 0.85 indicates the ceramic yield from the cross-linked polymer which was determined experimentally.

2.2. Solidification processing of in-situ MMCs

In-situ MMCs were processed by using a commercially built bottom-poured melting furnace as illustrated in Fig. 1. One kilogram of the machined blocks (matrix material) was melted in a steel crucible using an electrical resistance furnace at a temperature of 700 °C (973 K). The steel crucible was continuously purged with Ar-5%SF_6 gas mixture to eliminate the risk of flammability. In all the casting experiments, the molten Mg were degassed by Argon (99.999% purity) gas for the period of 10 min before the reinforcement. The melt was then mechanically stirred by a 3-axial stirrer blade at a 600 rpm to create an appropriate vortex field. Subsequently, 3.2 wt% of pre-heated (at a temperature of 473 K) polymer powder was injected into the melt via particle feeder, and mixing was done for 5 min. The melt temperature was then increased to 900 °C (1173 K) and stirring process was continued for another 15 min to complete the in-situ pyrolysis. Finally, the composite slurries were bottom-poured into a preheated (at a temperature of 473 K) rectangular split-molds (200 mm length x 150 mm width x 20 mm thickness) made of plain carbon steel. These solidified composite specimens were designated as PP900-M, PP900-AZ, PP900-AE which were fabricated using pure magnesium, AZ91 and AE44 Mg-alloys as matrix materials, respectively. Here PP refers to polymer powder, and the three digit number 900 refers to pyrolysis temperature of the casting process. In addition, the castings from unreinforced Mg-alloys and pure magnesium were also fabricated.

3. Material characterization

Optical microscope (Leica DM2700, Germany), scanning electron microscope (JEOL JSM-6610LV, Japan) coupled with energy dispersive spectroscopy (EDS) and transmission electron microscope (Tecnai T20, FEI Company) were utilized for microstructural studies. Diffraction spectra of the as-cast composites were collected using an X-Ray Diffractometer (PANalytical X'pert-Pro (MPD), Netherlands). Micro-hardness measurements of as-cast composites were performed using Vicker's micro-hardness testing instrument (Wilson instrument, Model 401/402 MVD UK) at a load of 100 gf for dwell time of 15 s. All the micro-hardness measurements were performed taking due care to avoid indentation on or near the micro-porous area of the as-cast composites. Uni-axial compression tests were performed using Tinus Olsen mechanical testing machine to record the room temperature mechanical properties at a constant strain rate of 10^{-3} s^{-1}. Cylindrical specimens were machined from the center of as-cast composites by electrical discharge machining (EDM). Compression specimen had a diameter of 4 mm, and 6.5 mm height. Values of reported mechanical properties are average of ten micro-hardness measurements and three compression tests.