MICROSTRUCTURE OF AlN PARTICLES REINFORCED AZ91D Mg-BASED METAL-MATRIX COMPOSITES

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Abstract
This paper investigated the microstructure of AlN particles reinforced AZ91D Mg-based metal-matrix composites (MMCs). The AZ91D is initially placed inside a graphite crucible and heated to 680-700°C in a resistance-heated furnace. The molten alloy is stirred with a vane operated at 350 rev./min for 3 minutes. Preheated AlN particles are simultaneously added to the stirred alloy with addition of 1, 2 and 5 wt. % of 1 μm AlN particles. The microstructure of the AZ91D and AlN_p reinforced AZ91D Mg MMCs specimens were observed by optical microscope. The mean grain size was determined using the linear intercept method. The clear microstructure of the eutectic structure is observed by polarization microscope. It was found that the β-phase Mg was almost eliminated through homogenization by SEM observation.

Keywords: metal-matrix composites, homogenization, eutectic solidification.

1 Introduction

Magnesium (Mg) alloys are gaining more recognition as a lightest structural material for light-weight applications, due to their low density and high stiffness-to-weight ratio. Even so, Mg alloys have not been used for critical performance applications because of their inferior mechanical properties compared to other engineering materials. Hence, many researchers attempt to fabricate Mg-based metal-matrix composites (Mg MMCs) by varied methods to obtain light-weight materials with excellent mechanical properties [1-5].

Regarding to fabrication of Mg MMCs, Ugandhar et al. [6] successfully synthesized Mg MMCs with sub-micron size SiC particulate reinforcements using an innovative disintegrated melt deposition (DMD) technique followed by hot extrusion. G. Cao et al. [5] studied the tensile properties and microstructure of cast AZ91D/AlN nanocomposites. Generally, after adding ceramic particles in the AZ91 matrix, the microhardness or yield strength and tensile strength are improved, but the ductility is decreased. And also some ceramic particles or whiskers such as SiC, $A_{l8}B_4O_{33}$ and B_4C react with AZ91D alloy if solidification processing is utilized. In recent years, ceramic nanopowders were used to reinforce the metallic materials [5]. According to the Orowan strengthening mechanism, finer particles are more efficient to improve the mechanical properties. Semenov et al. [7] presented the tribological contact characteristics of R18 tool steel in interface with AZ91D magnesium alloy hardened with SiC dispersed powder filler and by severe plastic deformation (SPD) - specifically, equal-channel angular pressing (ECAP). SPD of the original material leads to reduction of the molecular component of the friction coefficient. A lot of research was conducted in studying the AZ91 alloy reinforced by different ceramic particles.

AlN has been identified as a potential grain refiner for magnesium alloys using the edge-to-edge matching calculations. C. Zhang et al. [8] in situ synthesized ($AlN+Mg_2Si$)/Mg composites with uniform distribution of reinforcing particulates, and investigate the scale effect of reinforcing particulates on damping capacity. H.M. Fu et al. [9] studied the grain refinement by AlN particles in Mg-Al based alloys. Their experimental results indicate that the maximum grain refining efficiency of AlN in Mg–Al alloys occurs in samples cast from a melt temperature of 765 ◦C. Under these conditions, an addition of 0.5 wt% AlN reduces the grain size of Mg–3wt% Al alloy from 450 to 120μm. No further reduction is observed when more AlN is added to the melt. In the investigation of Maung Aye Thein et al. [10], Mg chips are recycled to produce nanostructured Mg–5wt%Al reinforced with 1, 2 and 5 wt% nanosized AlN particulates by mechanical milling. It was found that grain size played an important role in controlling ductility of the composites. Wu et al. [11] studied the influence of heat treatment on the properties of the consolidated AZ91D Mg alloy chips. Their experimental results show that heat treatments revealed that the age hardening effect was related to the transformation of the microstructure. Over aging during age heat treatment was believed to be caused by the formation of a lamellar structure composed of alternating layers of $Mg_{17}Al_{12}$ phase and magnesium matrix. S. Guldberg and N. Ryum [12] studied the microstructure and crystallographic orientation relationship of solidified Mg- $Mg_{17}Al_{12}$-eutectic. The effects of the addition of small amounts of Sr on the microstructure were also studied. The contribution of grain refinement to the strength level was discussed by the classical Hall-Petch equation [13, 14].

Previous studies mainly focused on the increase of the mechanical properties of the matrix alloy by incorporation of SiC particles for Mg MMCs [1-7]. The research focused on microstructures observation of AlN_p-reinforced AZ91 Mg MMCs was inadequate. The purpose of the present work is to investigate the microstructure of AlN particles reinforced AZ91D Mg-based metal-matrix composites to tell their constituents.

2 Experimental details

2.1 Materials preparation

The matrix used in this work is magnesium alloy AZ91D with 9.0 % aluminium. Its chemical composition is shown in **Table 1**. AlN particles with weight fraction of 1, 2, 5% within MMCs is used as the reinforcement phase. The commercially-available AlN powder with a particle diameter about 1μm, purity of ≧99.0% was added into AZ91D to form Mg-based metal-matrix composites.

Table 1 Chemical composition of AZ91D

Elements	Al	Zn	Mn	Si	Fe	Cu	Ni	Be	Mg
Wt%	9.0	0.69	0.20	0.05	0.001	0.00	0.001	0.001	Balance

The melt-stirring technique is used to fabricate the present Mg MMCs. Experimental setup is shown in **Fig. 1**. The AZ91D is initially placed inside a graphite crucible and heated to 680-700°C in a resistance-heated furnace. The molten alloy is stirred with a vane operated at 350 rev/min for 3 minutes. Preheated AlN particles are simultaneously added to the stirred alloy. Then the composite melt is finally poured into a metallic mold. The Mg MMCs containing AlN_p with different weight fraction of 1, 2, and 5 wt% are prepared for further mechanical and thermal testing. According to ASM standard the recommended solution treating for AZ91 casting is 16–24 hr at 413 °C and ageing time is 16 hr at 168 °C [15]. In this work homogenization heat treatment (T4) was performed at 400 °C for only 6 hr in argon atmosphere followed by water quench at 25 °C.

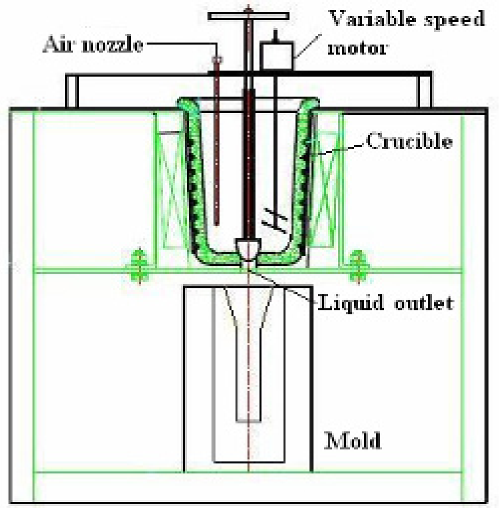

Fig.1 Setup configuration

2.2 Metallographic observations

The microstructure of the AZ91D and AlN_p reinforced AZ91D Mg MMCs specimens were observed by optical microscope. The surfaces of present MMCs were examined by scanning electron microscope (SEM, Hitachi-S3500). The grain structures of etched samples were observed using polarized light optical microscopy. Because no columnar zone was observed in any of the sample ingots, the mean grain size at the centre of each examined cross section was used to represent the grain size of that ingot. The mean grain size was determined using the linear intercept method.

3 Results and discussion

Fig. 2 through 5 show polarized light optical microstructure of the surface of AZ91D MMCs containing 0, 1, 2, 5 wt % of 1μm AlN particles, respectively. Their grain boundary can be clearly observed. There are lamellar structures and mesh structures within grain boundaries of all kinds of MMCs tested.

Fig. 6 indicates the field emission scanning electron microscopy (FESEM) image of 5wt%AlN_p/AZ91D MMCs, which is accompanied with the energy dispersive spectrometry (EDS) microanalysis. The AlN particles andβ phase can be seen in the FESEM image as indicated.

Fig. 7 through 10 show the SEM micrograph and EDS analysis of the strip in the lamellar structure (eutectic structure) of AZ91D/1wt%AlN_p/T4 MMCs. Figs. 11 through 13 indicate those images of AZ91D/2wt%AlN_p/T4 MMCs. Figs. 14 through 16 show those images of AZ91D/5wt%AlN_p/T4 MMCs.

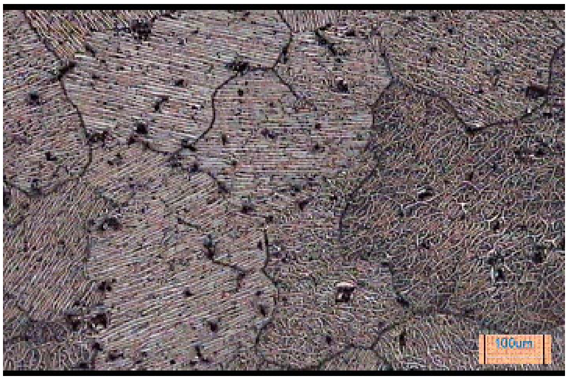

Fig.2 Polarized light optical microstructure of AZ91D/T4, 100 X

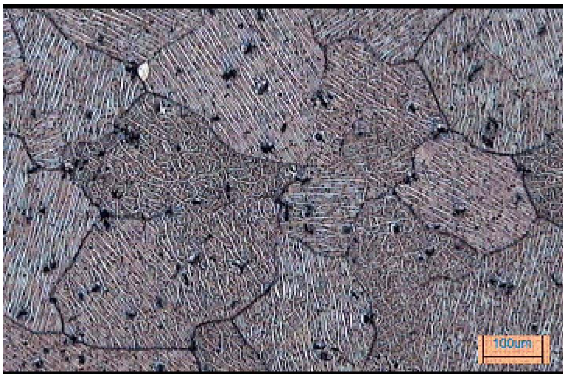

Fig.3 Polarized light optical microstructure of 1wt%AlN_p/AZ91D/T4, 100 X

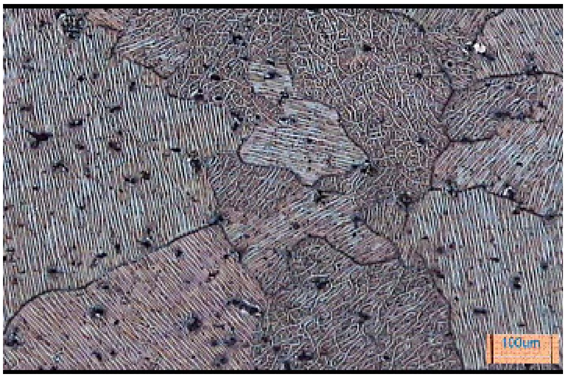

Fig.4 Polarized light optical microstructure of 2wt%AlN_p/AZ91D/T4, 100 X

The EDS analysis on the long stripes in the lamellar structure and meshes show that the Mg and Al elements are present, as given in **Figs. 8, 10, 12, 13, 15**, and **16**, suggesting that the long stripes areβ phase ($Mg_{17}Al_{12}$). **Fig. 9** shows that the major element in the matrix of the lamellar structure is Mg, indicating that the lamellar structure should be a mixture of Mg andβphase. The lamellar structure is similar to the discontinuous precipitation of the Mg–Al based alloys. Wu ea al. [11] indicated that discontinuous precipitation dominated at intermediate aging temperatures. The evolution of microstructure during aging indicates that the age hardening effect relates to the formation of the lamellar structure. By observing **Figs. 7, 11** and **14**, the β precipitates of the as-cast ingot of Mg MMCs could be almost dissolved into the grain through T4 heat treatment.

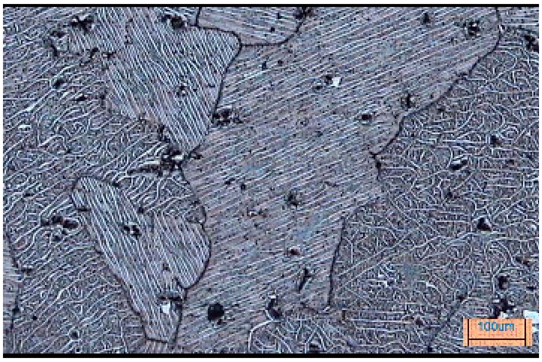

Fig.5 Polarized light optical microstructure of 5wt%AlN_p/AZ91D/T4, 100 X

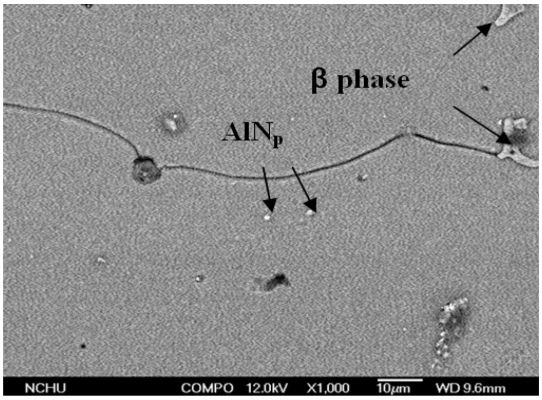

Fig.6 FESEM image of 5wt%AlNp/AZ91D/T4 MMCs

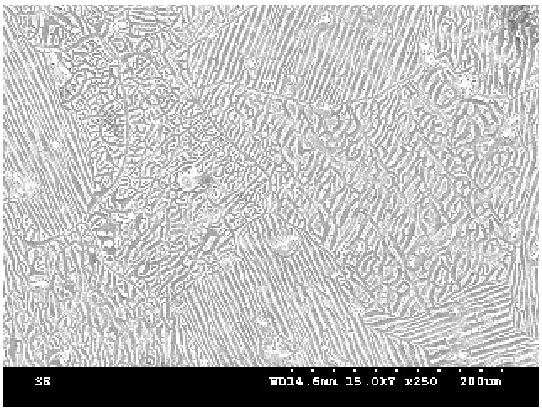

Fig.7 SEM micrograph of AZ91D/1wt%AlN_p/T4 MMCs

Element	Weight%	Atomic%
O K	12.92	18.75
Mg K	66.92	63.90
Al K	20.16	17.35
Totals	100.00	

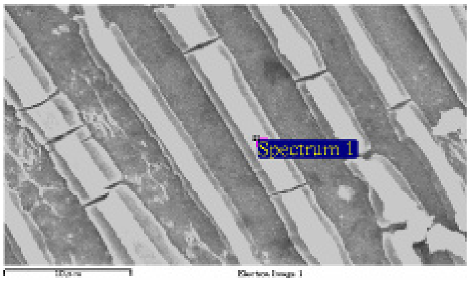

Fig.8 EDS analysis of the strip in the lamellar structure of AZ91D/1wt%AlN/T4 MMCs

Element	Weight%	Atomic%
Mg K	90.27	91.15
Al K	9.73	8.85
Totals	100.00	

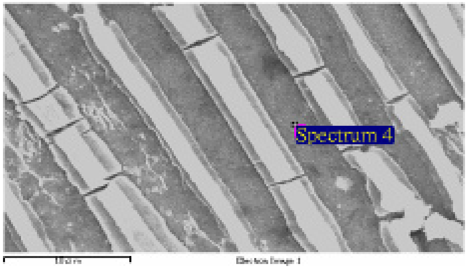

Fig.9 EDS analysis of the matrix in the lamellar structure of AZ91D/1wt%AlN/T4 MMCs

Element	Weight%	Atomic%
O K	22.19	30.99
Mg K	50.23	46.17
Al K	27.58	22.84
Totals	100.00	

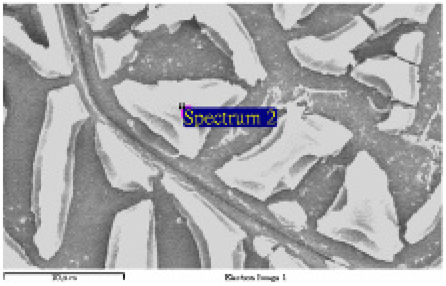

Fig.10 EDS analysis of the mesh structure of AZ91D/1wt%AlN$_p$/T4 MMCs

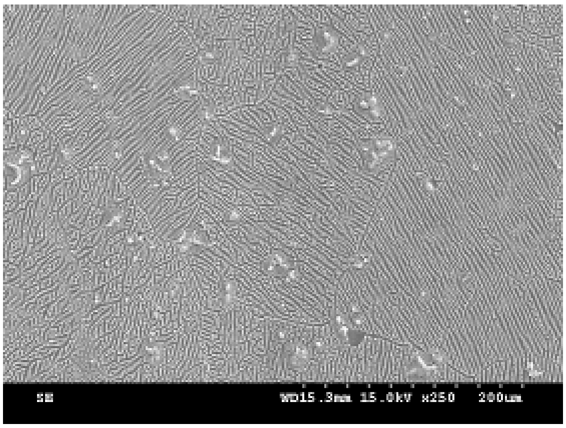

Fig.11 SEM micrograph of AZ91D/2wt%AlN$_p$/T4 MMCs

Element	Weight%	Atomic%
O K	17.15	24.50
Mg K	57.18	53.76
Al K	25.66	21.74
Totals	100.00	

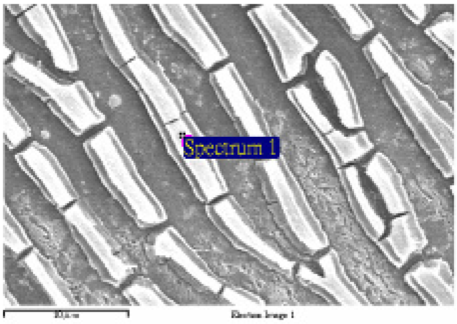

Fig.12 EDS analysis of the strip in the lamellar structure of AZ91D/2wt%AlN$_p$/T4 MMCs

Element	Weight%	Atomic%
O K	20.02	28.16
Mg K	56.15	51.97
Al K	23.83	19.87
Totals	100.00	

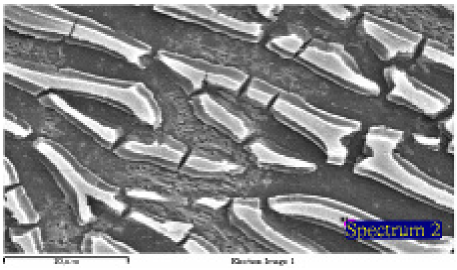

Fig.13 EDS analysis of the mesh structure of AZ91D/2wt%AlN$_p$/T4 MMCs

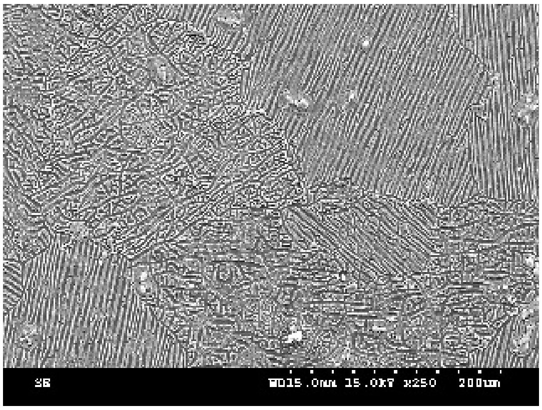

Fig.14 SEM micrograph of AZ91D/5wt%AlN$_p$/T4 MMCs

Element	Weight%	Atomic%
O K	18.51	26.16
Mg K	60.33	56.11
Al K	21.16	17.73
Totals	100.00	

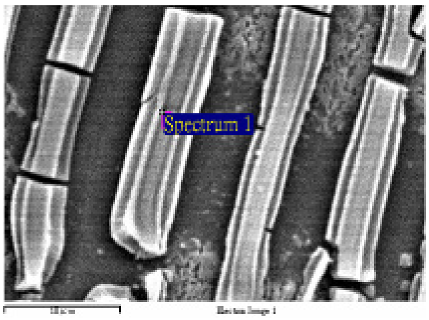

Fig.15 EDS analysis of the strip in the lamellar structure of AZ91D/5wt%AlN$_p$/T4 MMCs

Element	Weight%	Atomic%
O K	23.83	33.04
Mg K	47.90	43.71
Al K	28.27	23.25
Totals	100.00	

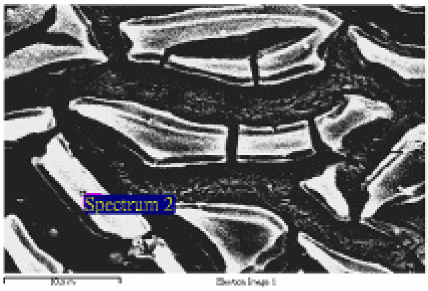

Fig.16 EDS analysis of the mesh structure of AZ91D/5wt%AlN_p/T4 MMCs

4 Conclusion

This study proposed and investigated the microstructure of AlN particles reinforced AZ91D Mg-based metal-matrix composites. The present Mg-based MMCs were fabricated by the melt-stirring technique. Based on the experimental results, the following conclusions and two important novelties could be drawn:

1. The clear eutectic structure of present AZ91D/AlN_p/T4 MMCs are observed by polarized light optical microstructure images, and its strip compositions ($Mg_{17}Al_{12}$) can be obtained by SEM micrograph and EDS analysis.
2. The homogenisation can dissolve the β precipitates of the as-cast ingot of Mg MMCs into the grain.

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