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Grain refinement of AlN_p/AZ91D magnesium metal-matrix composites

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Abstract

The grain refinement of AlN particles reinforced AZ91D Mg-based metal-matrix composites (MMCs) was investigated in this paper. The Mg MMCs were prepared by the melt-stirring technique with addition of 1, 2 and 5 wt.% of 1 μm AlN particles. Observing the microstructures of MMCs, the β-phase of Mg was almost eliminated through homogenisation. The ultimate tensile strength of Mg MMCs with only 1 wt.% addition of AlN_p has increased to 174 MPa, which is by 10.1 % higher than that of pure AZ91D. After homogenisation, the ultimate tensile strength of Mg MMCs with 1 wt.% addition of AlN_p has increased to 253 MPa, which is by 60.1 % higher than that of pure AZ91D.

K e y w o r d s: grain refinement, metal-matrix composites, homogenisation, tension test

1. Introduction

Magnesium (Mg) alloys are gaining more recognition as the lightest structural materials 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 various methods to obtain light-weight materials with excellent mechanical properties [1–6].

Regarding to fabrication of Mg MMCs, Ugandhar et al. [7] successfully synthesized Mg MMCs with sub-micron size SiC particulate reinforcements using an innovative disintegrated melt deposition (DMD) technique followed by hot extrusion. 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, $Al_{18}B_4O_{33}$ 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. [8] 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 has been conducted in studying the AZ91 alloy reinforced by different ceramic particles.

AlN was identified as a potential grain refiner for magnesium alloys using the edge-to-edge matching calculations. 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 ℃. Under these conditions, an addition of 0.5 wt.% AlN reduces the grain size of Mg-3wt.%Al alloy from 450 to 120 μm. No further reduction is observed when more AlN is added to the melt. In the investigation of Thein et

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T a b l e 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

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 ageing 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.

Previous studies mainly focused on the increase of the mechanical properties of the matrix alloy by incorporation of SiC particles for Mg MMCs [1-8]. The research focused on how the AlN particle (AlN_p) and the heat treatment affected the mechanical properties of AlN_p-reinforced AZ91 Mg MMCs was inadequate.

The purpose of the present work is to investigate the grain refinement by AlN particles of reinforced AZ91D Mg-based metal-matrix composites, as well as their mechanical performances.

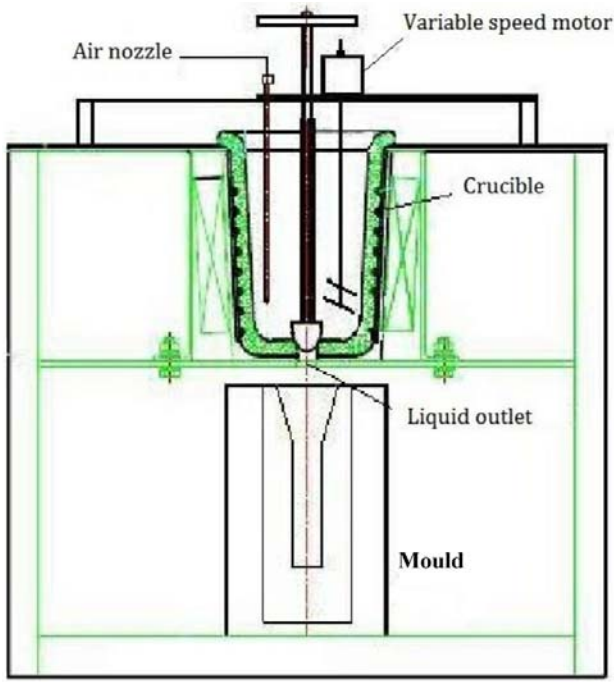

Fig. 1. Setup configuration.

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, and 5 % within MMCs are used as the reinforcement phase. The commercially available AlN powder with a particle diameter about 1 μm, purity of $\geq$ 99.0 %, and thermal conductivity of 140–180 $W\,m^{-1}\,K^{-1}$ was added into AZ91D to form Mg-based metal-matrix composites.

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 ℃ in a resistance--heated furnace. The molten alloy is stirred with a vane operated at 350 rev min^{-1} for 3 min. Preheated AlN particles are simultaneously added to the stirred alloy. Then the composite melt is finally poured into a metallic mould. The Mg MMCs containing AlN_p with different weight fraction of 1, 2, and 5 wt.% are prepared for further mechanical and thermal testing.

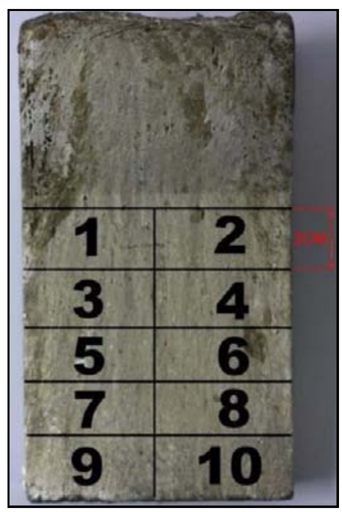

Fig. 2. Sampling position of hardness test.

According to ASM standard the recommended solution treating for AZ91 casting is 16–24 h at 413 ℃ and ageing time is 16 h at 168 ℃ [12]. In this work homogenisation heat treatment (T4) was performed at 400 ℃ for only 6 h in argon atmosphere followed by water quench at 25 ℃.

T a b l e 2. Hardness and tensile strength of samples with homogenisation and without homogenisation

	Hardness without homogenisation HV	Hardness with homogenisation HV	Tensile strength without homogenisation (MPa)	Tensile strength with homogenisation (MPa)
AZ91D	62.48	60.15	158	207
AZ91D/1wt.%AlN_p	68.65	67	174	253
AZ91D/2wt.%AlN_p	65	64.44	168	238
AZ91D/5wt.%AlN_p	67.02	64.95	165	222

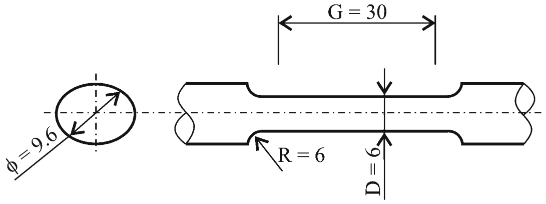

Fig. 3. Size of specimen prepared according to ASTM E8M-04, unit: mm.

2.2. Metallographic observations

The microstructure of the AZ91D and AlN_p reinforced AZ91D Mg MMCs specimens was 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.

2.3. Hardness tests and tension test

Vickers macrohardness measurements, using a load of 10 kgf, were carried out on the AZ91D and AlN_p reinforced AZ91D Mg MMCs with a Matsuzawa hardness tester (Model MV-1). Sampling position of hardness test is shown in Fig. 2, in which 10 positions were measured. Tension test of present MMCs was performed by Materials Test System of 5 tons with strain rate of 1 mm min^{-1}. Specimens for test were prepared according to ASTM E8M-04 (Fig. 3).

3. Results and discussion

3.1. Hardness and ultimate tensile strength

Table 2 indicates the hardness of samples with homogenisation and without homogenisation. It can be observed that hardness of MMCs with homogenisation

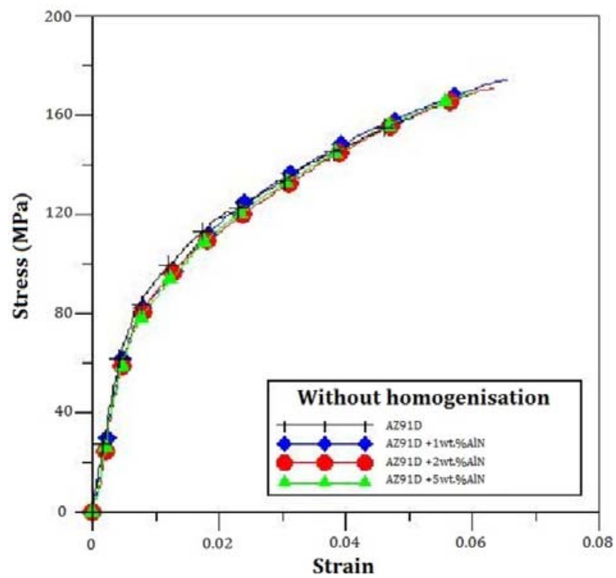

Fig. 4. Stress-strain curve of specimen without homogenisation.

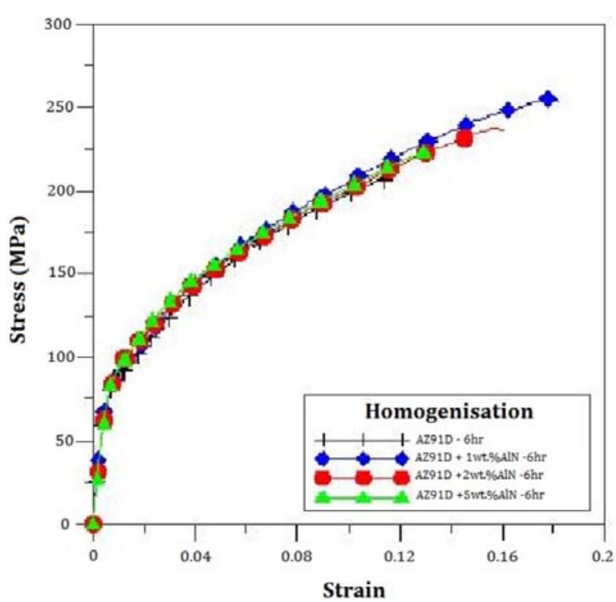

Fig. 5. Stress-strain curve of specimen with homogenisation.

Table 3. Elongation and yield strength of samples with homogenisation and without homogenisation

	Elongation without homogenisation (%)	Elongation with homogenisation (%)	Yield strength without homogenisation (MPa)	Yield strength with homogenisation (MPa)
AZ91D	3.2	6.5	78	69
AZ91D/1wt.%AlN_p	3.6	11.3	83	80
AZ91D/2wt.%AlN_p	3	9	80	77
AZ91D/5wt.%AlN_p	2	6	78.3	75

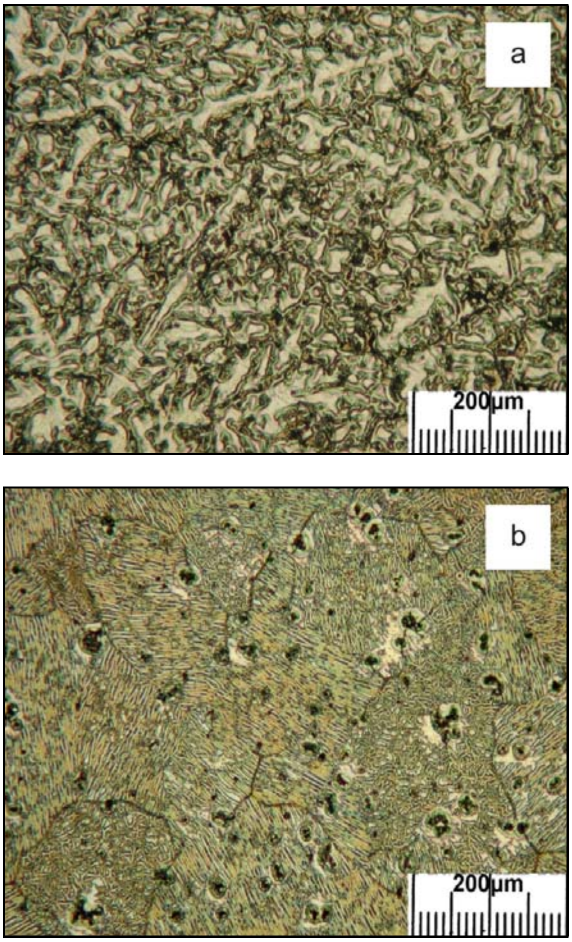

Fig. 6. Microstructure of AZ91D (a) before homogenisation, (b) after homogenisation, 100×.

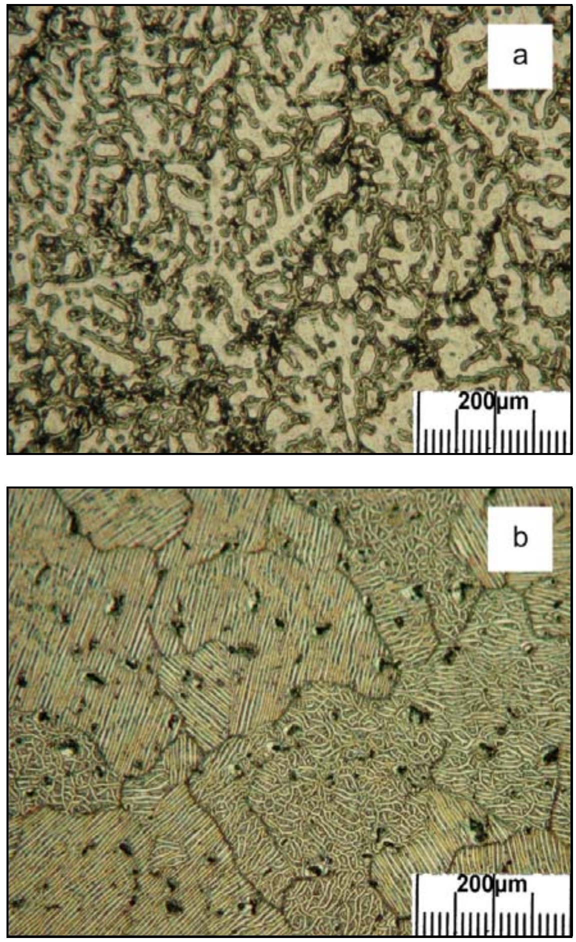

Fig. 7. Microstructure of 1wt.%AlN_p/AZ91D (a) before homogenisation, (b) after homogenisation, 100×.

is lower than that of MMCs without homogenisation. The homogenisation heat treatment at 400 ℃ for 6 h was found to be effective in dissolving the β precipitates in the as-cast ingot (see Figs. 6–9), which can decrease the fraction of the hard β phase ($Mg_{17}Al_{12}$) in MMCs to become softer than MMCs without homogenisation. The MMCs with 1 % AlN have the highest hardness among all percentages of MMCs both for MMCs with homogenisation and without homogenisation. The above result indicates that the hardness does not increase proportionally with increase of percentage of AlN_p added into AZ91D, since more AlN_p added more defects might occur in the MMCs during casting.

Figure 4 shows the stress-strain curve of tensile test of MMCs without homogenisation, and Fig. 5 shows that of MMCs with homogenisation. From the results of tensile test, Table 2 shows the tensile strength of MMCs with homogenisation and without homogenisation, Table 3 indicates the elongation of 2 groups (with homogenisation and without homogenisation) of MMCs, and Table 3 shows the yield strength of

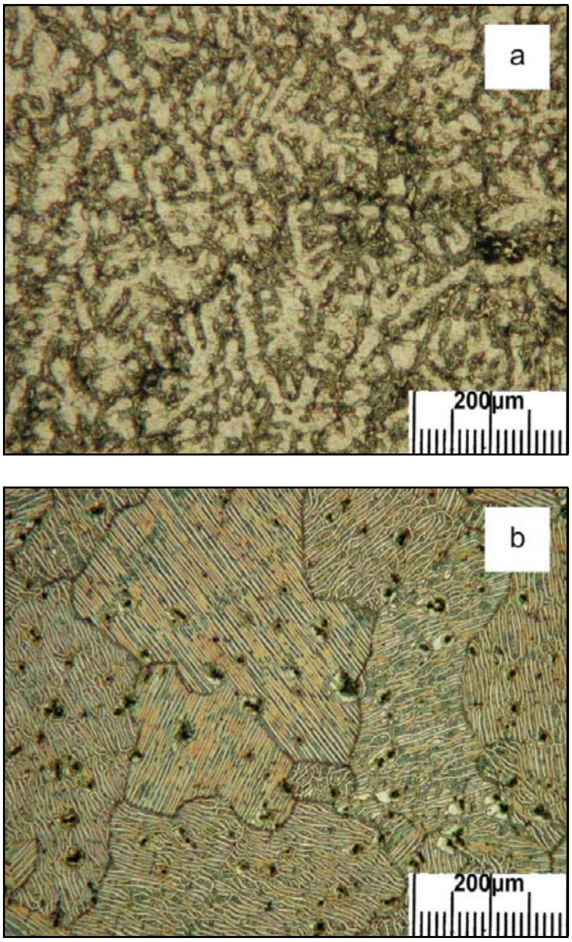

Fig. 8. Microstructure of 2wt.%AlN_p/AZ91D (a) before homogenisation, (b) after homogenisation, 100×.

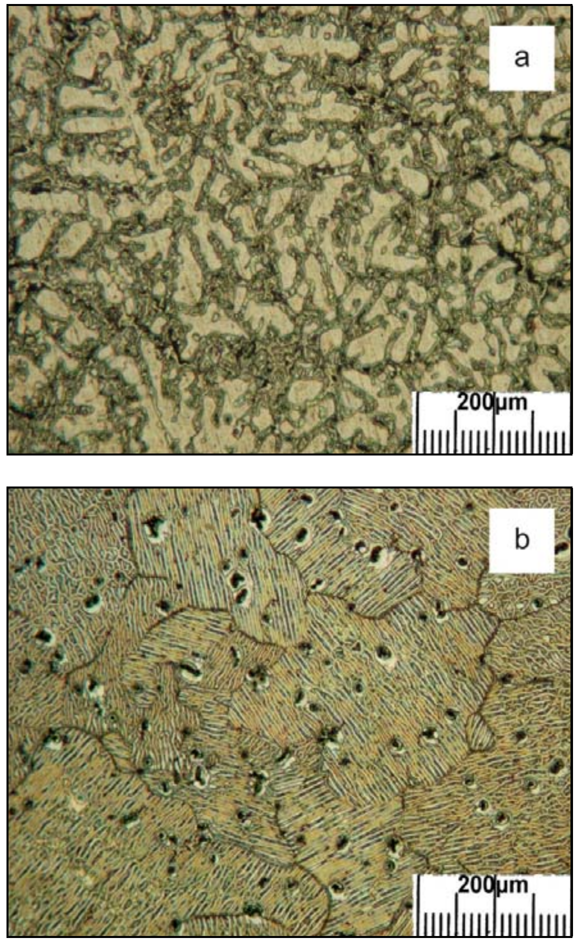

Fig. 9. Microstructure of 5wt.%AlN_p/AZ91D (a) before homogenisation, (b) after homogenisation, 100×.

2 groups of MMCs. The results show that additions of AlN_p increase the hardness, yield strength and tensile strength of AZ91D alloy, but reduce the elongation of such an alloy (except for 1wt.%AlN_p addition). It is found that AZ91D/1wt.%AlN_p performed excellent mechanical properties, such as elongation of 3.6 % (without homogenisation), elongation of 11.3 % (with homogenisation), yield strength of 83 MPa (without homogenisation), and yield strength of 80 MPa (with homogenisation). Before homogenisation, the ultimate tensile strength of Mg MMCs with only 1 wt.% addition of AlN_p has increased to 174 MPa, which is by 10.1 % higher than that of pure AZ91D. With homogenisation, the ultimate tensile strength of Mg MMCs with 1 wt.% addition of AlN_p has increased to 253 MPa, which is by 60.1 % higher than that of pure AZ91D.

In general, homogenisation can increase the tensile strength and elongation of present MMCs, and reduce their hardness and yield strength, because of its dissolving the hard β precipitates in the as-cast ingot.

3.2. Grain refinement

Figures 6–9 show typical optical microstructures at the centre of the examined cross sections of the sample ingots of AZ91D MMCs with different weight percentage of AlN_p, and Fig. 10 illustrates the variation of the mean grain sizes with the weight percentage of AlN_p additions. From Fig. 10 it can be seen that the addition of AlN_p reduced the grain size of the sample ingots from 300 μm to about 200 μm. The AZ91D/1wt.%AlN_p had the minimum average size compared to other percentage of AlN_p.

The contribution of grain refinement to the strength levels could be discussed on the basis of the classical Hall-Petch equation [13, 14]:

$$\Delta\sigma_{\text{Hall-Petch}} = K d_{\text{m}}^{-1/2}, \qquad (1)$$

where K is the Hall-Petch coefficient and d_{m} is the matrix grain diameter. Parameter K was calculated from the slope of $\Delta\sigma_{\text{Hall-Petch}}/d_{\text{m}}^{-1/2}$ plot using dif-

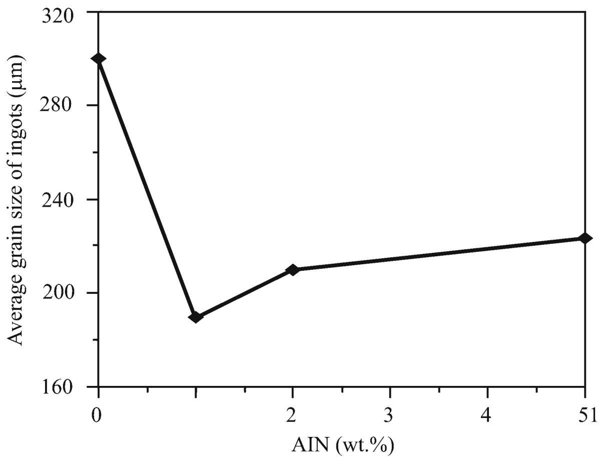

Fig. 10. Grain size variation of AZ91D/AlN$_p$ MMCs with wt.% of AlN.

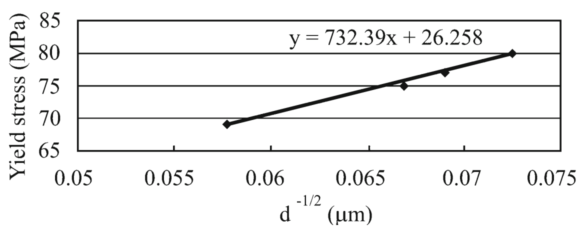

Fig. 11. Yield stress of AZ91D/AlN$_p$ MMCs with grain size to fit the classical Hall-Petch equation.

ferent grain sizes of the AZ91D MMCs material before and after annealing and their corresponding yield stress values.

The yield stress of AZ91D/AlN$_p$ MMCs as a linear function of grain size to fit the classical Hall-Petch equation is shown in Fig. 11, which indicates the contribution of grain refinement to the strength levels and verifies the accuracy of presented Hall-Petch equation.

The AZ91D/1wt.% AlN$_p$ had the minimum average size compared to other percentage of AlN$_p$ exhibiting highest yield strength.

4. Conclusion

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

1. The incorporation of AlN particles could improve the mechanical properties of AZ91D matrix alloy, such as hardness and yield strength.

2. The addition of AlN$_p$ reduced the grain size of the sample ingots, from 300 µm to about 200 µm, which increased the yield strength of the present MMCs.

3. The hardness and yield strength do not increase proportionally with increase of percentage of AlN$_p$ added into AZ91D, 1wt.%AlN$_p$/AZ91D MMCs exhibiting the highest hardness and yield strength.

4. Homogenisation can reduce present MMCs' hardness and yield strength, because the hard β precipitates could be dissolved within the as-cast ingot.

Acknowledgement

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