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ENGINEERING
SUPPLEMENTARY-RESULT
NEGATIVE-RESULT

The effect of Y on the microstructure and mechanical performance of an Mg-Al-Y casting alloy

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Abstract

Environmental gains of electric cars can be optimized with the use of lightweight and recyclable magnesium in the vehicle's structural components. Ductility improvement of low-density Mg-Al alloys will extend their use in automotive body applications. The authors achieved 63% ductility improvement in Mg-6wt%Al with trace Y (1.5 ppm) due to the β-phase refinement and predicted that higher levels would not perform as well. As predicted, 0.3wt% of Y addition investigated in this study led to lower mechanical performance and β-phase refinement than those obtained with trace additions. The tensile ductility and yield strength increased by ~13% and 16%, respectively, and the compression strain to fracture by ~22%. Scanning electron and optical microscopy, X-Rays diffraction, mechanical testing and thermodynamic calculations were used to investigate the effect of 0.3wt% Y on the microstructure of Mg-6wt%Al. The matrix dissolution revealed the close association of the Al_2Y and the β-$Mg_{17}Al_{12}$ phases.

Keywords: Magnesium; Mg-Al alloys; Matrix extraction; Tensile ductility; Compressive strain

1. Introduction

Future autonomous cars will rely on computer systems, sensors, and satellite navigation which require extensive electronics that increase the vehicle's weight (Gao et al., 2014). Due to their high strength-to-weight-ratio, magnesium (Mg) structural alloys are a viable solution for vehicle-weight reduction in future cars. Mg-6wt%Al (Mg-6Al) casting alloys demonstrate the optimum level of strength and ductility for most automotive applications but further increase in their ductility will lead to their extended use in crashworthy car-body components (Friedrich & Mordike, 2006). The $Mg_{17}Al_{12}$ precipitate, the main second phase, improves strength but reduces ductility (Friedrich & Mordike, 2006; Nave et al., 2000).

Studies have shown that yttrium (Y) additions can improve the ductility of Mg-Al alloys (Su et al., 2010; Tahreen et al., 2016). When Y is in the solid solution of α-Mg then it can improve its mechanical properties via: (1) decrease of the Stacking Fault Energy (SFE) activating the pyramidal <c+a> dislocations (Sandlöbes et al., 2012) or (2) forming long period stacking ordered (LPSO) phases when a transition metal is present (Kawamura & Yamasaki, 2007). However, with Al present, the solubility of rare earths in α-Mg is nil, instead brittle precipitates (Pourbahari et al., 2017; C. Wang et al., 2015; L. Wang et al., 2019) form. Researchers have observed that the modification of β-$Mg_{17}Al_{12}$ in Mg-Al-Y alloys is responsible for improved mechanical properties (Boby et al., 2013; Cai et al., 2018; Kashefi & Mahmudi, 2012; S.-R. Wang et al., 2009). Previous work (Korgiopoulos & Pekguleryuz, 2020) by the authors has shown that the refinement of β-$Mg_{17}Al_{12}$ improves the ductility of Mg-6Al cast alloys at trace Y additions. This has been attributed to co-precipitation (at close temperatures) of the Al_4MgY phase with the $Mg_{17}A_{12}$ phase. Thermodynamics also predicted that the formation temperatures of the two phases deviate at higher Y levels leading to a loss in nucleant effectiveness.

2. Objective

The current work investigates the effect of Y additions (0.3wt%) to see if β-phase refinement and ductility improvement seen in trace levels of Y are also observed at higher Y levels and to elucidate the role of Y on the mechanical properties of Mg-6Al based alloys.

3. Methods

Mg-6Al-0.3Y (in wt%) alloy was synthesized using commercial purity (99.98%) Mg, 99.9% pure Al granules and 99.9% pure Y rods. The total mass of the alloying additions was 617 gr using 95% recovery factor for Al and 60% for Y. The alloying additions were made at 720 °C in a graphite crucible under CO_2/SF_6 protective atmosphere. A graphite crucible was used because it is affordable, and it does not react with magnesium. The cleaning after casting is efficient and leaves no residuals that could possibly contaminate the subsequent castings. The molten alloy was then poured under protective atmosphere into a preheated (400°C) steel mold to produce a flat plate. The actual composition (in wt%) of the alloy according to inductively coupled plasma atomic-emission spectroscopy is: 6.26%Al, 0.29%Y, 0.02% impurities (Fe, Mn) with Mg as balance. A scanning electron microscope (SEM-Hitachi SU3500) with an energy dispersive X-ray spectroscopy (EDS) detector was used for microstructural investigation. The grain size of α-Mg was measured with the intercept method using a Nikon-Epiphot 200 optical microscope after etching the samples with 4.2gr picric acid, 10 ml acetic acid, 10 ml distilled water and 70 ml ethanol. The ImageJ software (Rasband, 2011) was used to measure the precipitates and the grain size. The crystallographic information was obtained by XRD (Bruker D8 Discovery X-Ray Diffractometer-Cu source) in the bulk sample and after matrix extraction by using 5% acetic acid. The mechanical properties were evaluated by tensile and compression testing (MTS 810) at room temperature with a strain rate of $0.001s^{-1}$. Thermodynamic calculations (FactSage with FTlite database) (Bale et al., 2002) based on the CALPHAD method have been performed in equilibrium and non-equilibrium conditions (Scheil cooling).

4. Results and discussion

Mg-6Al-0.3Y alloy consists of partially divorced β-$Mg_{17}Al_{12}$, interdendritic Y-enriched precipitates and the Al enriched *a*-Mg phase as determined by EDS (Fig. 1). Y is detected in Mg-Al-Y and Mg-Al-Mn-Fe-Y precipitates. Both precipitates are closely associated with $Mg_{17}Al_{12}$ suggesting that they act as nucleation sites. The Y-enriched precipitates range from fine to coarse and present in three different morphologies, namely, spherical, square and plate-like, with the expectation that only the finer precipitates can act as refiners for the β phase. Table 1 shows the composition of the β-phase and Al_2Y as per the EDS analysis. No Y was detected in the *a*-Mg solid solution. There is close association (Figs 1 and 2) of the Al_2Y precipitates with the $Mg_{17}Al_{12}$. Similar association has been observed before by the authors (Korgiopoulos & Pekguleryuz, 2020) in Mg-6%Al based alloys for lower Y additions. XRD detects (Fig. 3a) Mg-Al solid solution and β-$Mg_{17}Al_{12}$ peaks in the bulk alloy. After the matrix extraction (Fig. 3b), the matrix disappears and the peaks for Al_2Y and $Mg_{17}Al_{12}$ become stronger. The peak intensity of spectra b is lower due to the low amount and small size (average size ~10-50 microns) of the extracted precipitates.

The $Mg_{17}Al_{12}$ (Table 2) in Mg-6Al-0.3Y is 25% finer than that in the binary Mg-6Al, but 41% coarser than the alloy with trace Y amount. Additionally, high Y addition forms even coarser (~87μm^2) Mg-Al-Mn-Fe-(Y) and Mg-Al-Y precipitates. The grain size of the cast Mg-6Al-0.3Y is also higher than the alloy

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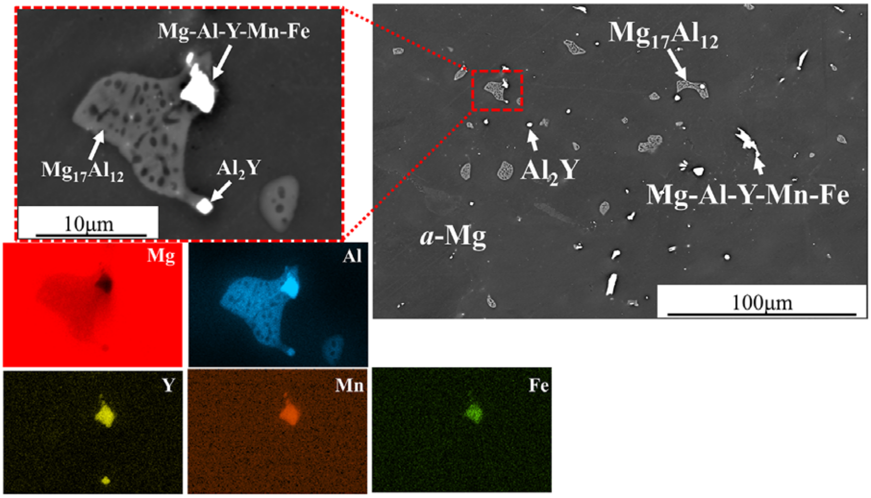

Figure 1. SEM/BSE as-cast microstructure of Mg-6Al-0.3Y and EDS maps. The red square shows the close associations of Y enriched precipitates with $Mg_{17}Al_{12}$.

Table 1. EDS on the precipitates after matrix extraction

Precipitate	Mg (at%)	Al (at%)	Y (at%)
$Mg_{17}Al_{12}$	57.2 ± 0.4	42.8 ± 0.4	–
Al_2Y	2.3 ± 1.2	61.5 ± 1.9	36.2 ± 0.7

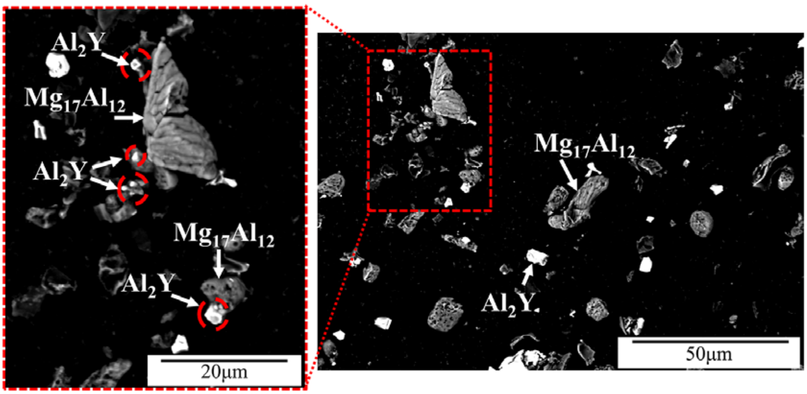

Figure 2. Mg-Al-0.3Y alloy after matrix extraction. The red circles show the Al_2Y precipitates associated with the $Mg_{17}Al_{12}$ phase.

with trace Y level and it is attributed to the coarsening of the Al_2Y phase (Chang et al., 2013; Pan et al., 2008; Zou et al., 2005).

Mg-6Al-0.3Y has the highest tensile yield strength (YS) of the three alloys (Table 3). Compared to the Mg-Al with trace Y, the tensile ductility (% El), ultimate tensile strength (UTS), and the compressive strength are lower (Tables 3 and 4). According to thermodynamic simulations conducted by the authors (Table 5), the solubility of Y in Mg-Al-Y is practically nil. Instead, Y forms precipitates (ordered intermetallics) with Al such as Al_4MgY, Al_3Y and Al_2Y. In Mg-Al-0.3Y, Al_2Y forms earlier (at higher temperature) than the β-phase (Table 5) and has time to coarsen losing its effectiveness as a nucleant of the β-phase and embrittling the alloy.

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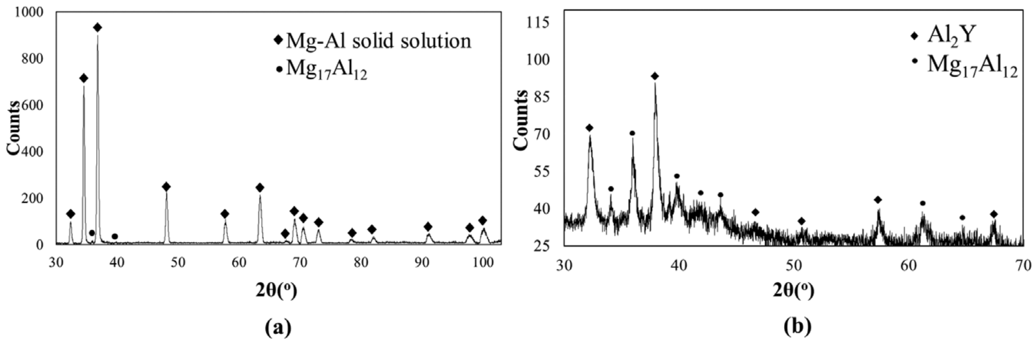

Figure 3. XRD results (a) Bulk alloy, (b) after matrix extraction.

Table 2. Precipitates size and α-Mg grain size as measured with Image J

Alloys	$Mg_{17}Al_{12}$ Size (μm^2)	Mg-Al-Mn-Fe-(Y) Size (μm^2)*	Mg-Al-Y Size (μm^2)	α-Mg Grain Size (μm)	Reference
Mg-6Al	32 ± 15	1.56 ± 0.06	–	96 ± 13	(Korgiopoulos & Pekguleryuz, 2020)
Mg-6Al with trace Y	17 ± 5	1.41 ± 0.20	–	85 ± 15	(Korgiopoulos & Pekguleryuz, 2020)
Mg-6Al-0.3Y	24 ± 6	7 ± 3	14 ± 10	105 ± 17	Current work

*Only precipitates in Mg-6Al-0.3Y alloy contain Y.

Table 3. Tensile properties of as cast samples at room temperature

Alloy	UTS(MPa)	YS(MPa)	El (%)	Reference
Mg-6Al	206 ± 5	74 ± 6	8 ± 2	(Korgiopoulos & Pekguleryuz, 2020)
Mg-6Al with trace Y	235 ± 13	72 ± 11	13 ± 3	(Korgiopoulos & Pekguleryuz, 2020)
Mg-6Al-0.3Y	209 ± 17	86 ± 10	9 ± 2	Current work

Table 4. Compression properties of as cast samples at room temperature

Alloy	CS(MPa)	CYS(MPa)	Strain(%)	Reference
Mg-6Al	276 ± 8	105 ± 8	23 ± 1	(Korgiopoulos & Pekguleryuz, 2020)
Mg-6Al with trace Y	271 ± 3	97 ± 5	28 ± 2	(Korgiopoulos & Pekguleryuz, 2020)
Mg-6Al-0.3Y	258±6	69±6	28±2	Current work

5. Conclusions

The addition of 0.3wt% Y improves the tensile properties and the strain to fracture in compression of the binary cast alloys Mg-6wt%Al. The improvement in ductility and UTS is not as significant as lower (trace level) Y additions. Al_2Y precipitates (determined via XRD and SEM/EDS) are in close association with the $Mg_{17}Al_{12}$ phase indicating their role as nucleants but the early formation of the Al_2Y in the liquid in Mg-Al-0.3Y leads to its coarsening, resulting in some loss in mechanical properties compared to the alloy with the trace level of Y.

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Table 5. Equilibrium and non-equilibrium (Scheil) thermodynamic calculations

Alloy(wt%)	Equilibrium – Y solubility in α-Mg (wt%)	Scheil - Y solubility in α-Mg (wt%)	Y enriched phases	Scheil-Formation temperature of $Mg_{17}Al_{12}$	Scheil-Formation temperature of Y enriched precipitates
Mg-0.01 Y	0.01	0.01	$Y_{10}Mg_{24}Mg_{24}$	–	–
Mg-0.3Y	0.03	0.29	$Y_{10}Mg_{24}Mg_{24}$	–	–
Mg-6Al-0.01Y	5.51E-21	0.00007	Al_4MgY	439°C	460°C
Mg-6Al-0.3Y	5.51E-21	0.0027	Al_2Y/Al_3Y (Scheil), Al_4MgY	439°C	555°C (Al_3Y), 610°C (Al_2Y), 510°C (Al_4MgY)

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Author contributions. K. Korgiopoulos and M. Pekguleryuz conceived and designed the study. K. Korgiopoulos conducted data gathering, their analysis and interpretation. Both authors wrote the article.

Data availability statement. The raw/processed data cannot be shared at this time as the data also forms part of an ongoing study.

Conflict of interest. The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this article.

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Peer Reviews

Reviewing editor: Dr. Eva Unger
Lund University, Lund, Sweden, 221 00

This article has been accepted because it is deemed to be scientifically sound, has the correct controls, has appropriate methodology and is statistically valid, and has been sent for additional statistical evaluation and met required revisions.

doi:10.1017/exp.2020.63.pr1

Review 1: The effect of Y on the microstructure and mechanical performance of an Mg-Al-Y casting alloy

Reviewer: Ali Arslan Kaya

Date of review: 13 November 2020

Conflict of interest statement. Reviewer declares none.

Comments to the Author: This is a useful and detailed work. The only comment which seems unjustified scientifically is the following: "These changes in mechanical properties are not due the well127 known effect of Y on SFE and LPSO since Y has negligible solubility in the a-Mg according to the thermodynamic calculations and EDS results"

I strongly advise that this comment should be changed. Although the solubility of Y in Mg is low, it is not zero. And even ppm levels of a foreign atom in any host metal can alter the SFE of some crystal planes and directions. Therefore such an effect of "low Y" levels in the system studied cannot be ruled out. And frankly speaking, this study presents no finding to justify the claim made in "Conclusions". There exist many studies showing the effect of Y on SFEs in Mg in literature. In short, this claim is highly misleading and therefore, need be changed.

Thank you.

Score Card

Presentation

3.7 /5

Is the article written in clear and proper English? (30%)	4/5
Is the data presented in the most useful manner? (40%)	4/5
Does the paper cite relevant and related articles appropriately? (30%)	3/5

Context

4.5 /5

Does the title suitably represent the article? (25%)	5/5
Does the abstract correctly embody the content of the article? (25%)	4/5

Does the introduction give appropriate context? (25%)	4/5
Is the objective of the experiment clearly defined? (25%)	5/5

Analysis

4.0 /5

Does the discussion adequately interpret the results presented? (40%)	4/5
Is the conclusion consistent with the results and discussion? (40%)	4/5
Are the limitations of the experiment as well as the contributions of the experiment clearly outlined? (20%)	4/5

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doi:10.1017/exp.2020.63.pr2

Review 2: The effect of Y on the microstructure and mechanical performance of an Mg-Al-Y casting alloy

Reviewer: Dr. V Pavlyuk

Date of review: 15 October 2020

Conflict of interest statement. Reviewer declares none

Comments to the Author: The information on the mass of prepared alloys and the mass losses during the preparation should be added to the description of the experimental procedure.

The obtained alloys were controlled by means of X-ray diffraction (XRD). What type of radiation was used (FeK, CuK, or other)? Indicate this in the manuscript.

The authors must add the results of XRD Rietveld refinements, please provide more details and explanations about the structure refinements for all observed phases.

Usually, magnesium alloys melt in tantalum crucibles. Explain why graphite crucibles have been chosen?

The EDS data should be reported with the standard deviations for all elements (Table 1).

Score Card

Presentation

3.3 /5

Is the article written in clear and proper English? (30%)	4/5
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Context

3.5 /5

Does the title suitably represent the article? (25%)	4/5
Does the abstract correctly embody the content of the article? (25%)	4/5
Does the introduction give appropriate context? (25%)	3/5
Is the objective of the experiment clearly defined? (25%)	3/5

Analysis

3.2 /5

Does the discussion adequately interpret the results presented? (40%)	3/5
Is the conclusion consistent with the results and discussion? (40%)	3/5
Are the limitations of the experiment as well as the contributions of the experiment clearly outlined? (20%)	4/5