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The influence of Nd and Ho addition on the microstructure of Mg-7Al alloy

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ABSTRACT

Purpose: of this paper was to examine effect of Nd and Ho on the microstructure of as-cast and solid-treated Mg-7Al alloy, and to study the response to ageing by hardness changes measurements of investigated alloys during aging at 200ºC.

Design/methodology/approach: The microstructure of Mg-7Al-3Nd and Mg-Al-10Ho alloys after casting and heat treatment was studied by means of metallographic and scanning electron microscopy. The phase characterisation was carried out by X-ray diffraction and by energy dispersive X-ray microanalyser. The hardness changes during aging of both alloys were determined by Vickers hardness.

Findings: It was found that as-cast microstructure of Mg-7Al alloy doped with 3% Nd, and 10% Ho, is characterized by α-Mg solid solution with eutectic mixture of α-Mg and $Mg_{17}Al_{12}$ phases, intermetallic compounds Al_2Nd and $Al_{11}Nd_3$, and Al_2Ho respectively. During solution heat treatment eutectic phase was dissolved and intermetallics were transformed into Al_3Nd and Al_3Ho, whilst $Al_{11}Nd_3$ phase was remained in microstructure. Aging of investigated alloys plays slight role in mechanical properties at ambient temperature.

Research limitations/implications: The microstructure of supersaturated Mg-7Al-10Ho alloy is composed of α-Mg and thermally stable, fine and randomly distributed Al_3Ho phase, what may considerably improve the creep behaviour this alloy at elevated temperature. The creep tests in connection with microstructural analysis will be done in the future.

Practical implications: The development of creep resistant Mg-Al alloys with individual rare earth elements allows to apply these materials as responsible structural members working at elevated temperature. The present results extend the knowledge about effect of Nd and Ho addition on the microstructure of Mg-7Al alloys.

Originality/value: Microstructural studies of two new Mg-7Al alloys doped with 3% Nd and 10% Ho were presented.

Keywords: Mg-Al-RE alloys; Casting; Heat treatment; Microstructure

PROPERTIES

1. Introduction

Magnesium alloys are being extensively used in lightweight structural members, especially in automotive, aerospace and electronic industry, due to their features like low density, high specific strength, high damping capacity, excellent screening ability against electromagnetic radiation. However, range of application for Mg alloys at elevated temperatures (above 120°C) is limited attributable to poor creep resistance. Improvement of creep strength is important to apply these alloys as responsible structural members working at elevated temperature. Cast alloys based on Mg-Al system, according to ASTM alloy designation AZ, AM, AS and AE, have been developed for applications in automotive industry. AZ (Mg-Al-Zn) and AM (Mg-Al-Mn) alloys are characterized by a good castability, corrosion resistance, strength and ductility at ambient temperature, as well as by poor creep resistance at elevated temperature [1,2]. AS (Mg-Al-Si) alloys exhibit slightly better creep strength, but reduced ductility and difficulties in die casting due to the presence of Mg_2Si [3].

Microstructure of Mg-Al alloys is typical for the eutectic mixture of the α phase and $Mg_{17}Al_{12}$ particularly at the grain boundaries. This intermetallic compound has a low melting point (458°C) and is brittle, furthermore the interface α-Mg/$Mg_{17}Al_{12}$ demonstrates brittle nature as a result of lattice misfit [4]. Moreover in most commercial alloys the eutectic is a fully or partially divorced [5]. The instability of eutectic phase contribute to weak creep resistance [6], therefore to improve mechanical properties of Mg alloys at elevated temperature, the rare earth elements are added. A number of Mg-Al alloys with rare earth elements (e.g AE41, AE42) in the form of mischmetal, that contains Ce, La and Nd mainly, were developed [3,7,8]. These alloys have improved creep properties due to formation thermally stable intermetallic phases, however there is currently insufficient knowledge of the effect of individual rare earth elements on microstructure and in consequence on creep properties.

The positive individual effect of Nd and Ho on mechanical properties in Mg-RE alloys were reported [9]. In the present work, the effect of 3 wt. % of Nd and 10 wt. % of Ho on the microstructure of Mg-7Al alloy in as-cast and supersaturated condition, and on the age hardening, have been studied.

2. Experimental procedure

The microstructure of two Mg-7%Al alloys, with 3 wt. % of Nd (Mg-7Al-3Nd) and 10 wt. % of Ho (Mg-7Al-10Ho) addition, as-cast and after heat treatment were examined. Both alloys were melted in steel crucibles in an electrical resistance furnace under a protecting flux containing 38-40% KCl, 3-5% CaF_2, 5-8% $BaCl_2$, 1.5% MgO and <8% (NaCl + $CaCl_2$) according with Russian standards. The alloys were prepared from pure magnesium (99.96%), aluminum (99.99%), neodymium (99,85%) and holmium (99.83%). During smelting neodymium and holmium were introduced into melts in form of the master alloys Mg-28.8% Nd and Mg-44.8% Ho. The melts of the alloys were cast into steel mold to produce cylindrical ingots with a diameter of 15 mm. The mould was heated preliminary up to about 200°C.

Light (LM) and scanning electron microscopy (SEM) studies were carried out on both casts in the "as received" as well as after supersaturated state. The supersaturation was provided at 560°C for 3 hours in argon protective atmosphere and quenched in water, and aging at 200°C in silicon oil. Specimens for microstructural investigation were prepared by mechanical grinding and polishing and etched in solution containing 1ml nitric acid, 20 ml acetic acid, 24 ml distilled water and 75 ml glycol. The phase characterisation was carried out by X-ray diffraction using Cu K_α radiation and by energy dispersive X-ray microanalyser (EDS). The hardness changes during aging of both investigated alloys were determined by Vickers hardness at the load 5 kG.

3. Results and discussion

3.1. Microstructure of Mg-7Al-3Nd

The metallographic examination of Mg-7Al-3Nd alloy as-cast has shown that microstructure is characterized by of α-Mg matrix and eutectic mixture α-Mg and $Mg_{17}Al_{12}$ phases with characteristic dendritic morphology (Fig. 1). The growth morphologies of eutectic in Al-Mg system depends on Al content and cooling rate [5]. After supersaturation at 560°C for 3 hours and quenching in water eutectic phase was not observed (Fig. 2).

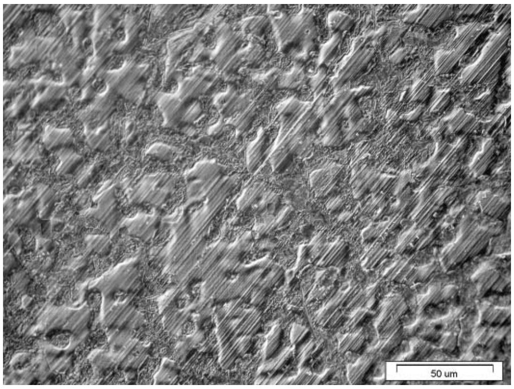

Fig. 1. Microstructure of as-cast Mg-7Al-3Nd alloy (LM)

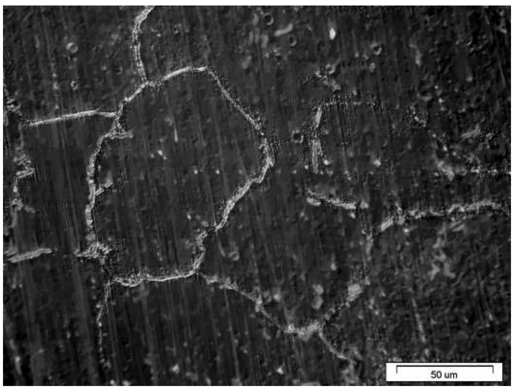

Fig. 2. Microstructure of supersaturated Mg-7Al-3Nd alloy (LM)

The XRD analysis indicates the presence of intermetallic phases Al_2Nd and $Al_{11}Nd_3$ in α-Mg matrix microstructure of as-cast Mg-7Al-3Nd alloy (Fig. 3). After supersaturation eutectic was dissolved, however $Al_{11}Nd_3$ phase was still evident in the solid solution α-Mg and the Al_3Nd phase was formed.

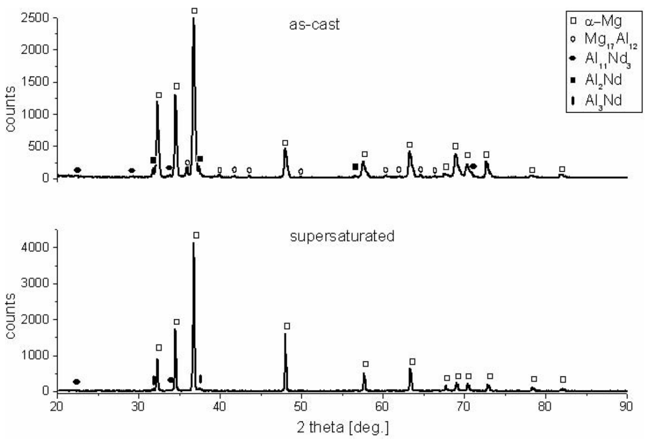

Fig. 3. X-ray diffraction pattern of as-cast and supersaturated Mg-7Al-3Nd alloy

SEM studies allow to identify phases and their distribution in the alloy (Figs. 4,5). The eutectic component $Mg_{17}Al_{12}$ phase is visible on BSE image as grey phase. Al_2Nd precipitates, of an irregular shape and size about 2-10 µm, are distributed mainly inside grains.

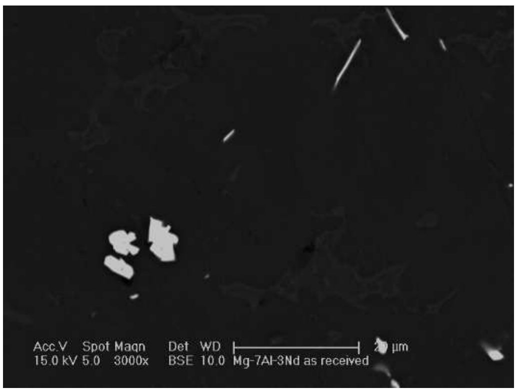

Fig. 4. Microstructure of as-cast Mg-7Al-3Nd alloy (BSE)

The clusters of a few Al_2Nd precipitates are observed in microstructure. The $Al_{11}N_3$ needle like shaped phase was observed mainly at grain boundaries. The size of this precipitates is 5-20 µm in length. In supersaturated state the eutectic is dissolved in the matrix. Intermetallic compound $Al_{11}Nd_3$ remains and also Al_3Nd was observed in microstructure (Fig. 5).
The composition of observed phases measured using EDS analyzer is given in the table 1. The content of aluminium dissolved in α-Mg matrix is higher than its maximal solubility at room temperature [10]. It suggests that Al content from dissolved $Mg_{17}Al_{12}$ phase caused transformation of Al_2Nd during super-saturation into Al_3Nd.

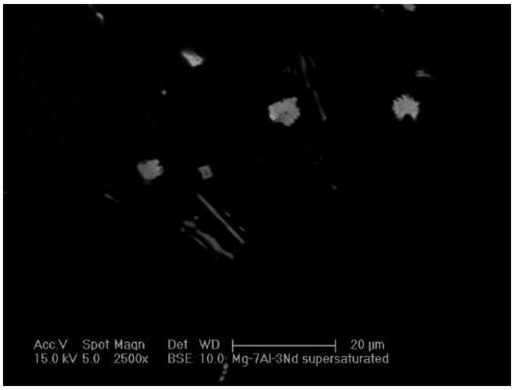

Fig. 5. Microstructure of supersaturated Mg-7Al-3Nd alloy (BSE)

Table 1.
Chemical composition of observed phases in Mg-7Al-3Nd alloy

	Phase		Element in atomic %		
			Mg	Al	Nd
as-cast	α-Mg	hexagonal	94,48	5,22	-
	$Mg_{17}Al_{12}$	cubic	66,73	33,27	-
	Al_2Nd	hexagonal	-	68,73	31,27
	$Al_{11}Nd_3$	orthorhombic	-	77,57	22,43
super-saturated	Al_3Nd	hexagonal	-	74,39	25,61
	$Al_{11}Nd_3$	orthorhombic	-	79,04	20,96

3.2. Microstructure of Mg-7Al-10Ho

Analogously to Mg-7Al-3Nd alloy as-cast, the microstructure of Mg-7Al-10Ho alloy consists of α-Mg matrix and eutectic compound $Mg_{17}Al_{12}$, whilst after supersaturation at 560°C within 3 hours and quenching, eutectic is dissolved (Fig. 6).

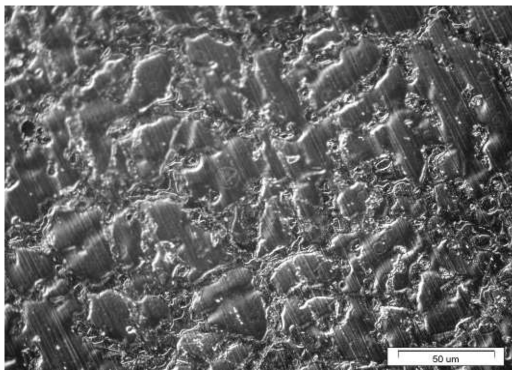

Fig. 6. Microstructure of as-cast Mg-7Al-10Ho alloy (LM)

The XRD analysis of as-cast Mg-7Al-10Ho alloy indicates the occurrence of intermetallic phase Al_2Ho in microstructure (Fig. 7). After supersaturation the eutectic was dissolved, however intermetallic phases were difficult to identify from X-ray diffraction pattern due to insufficient intensity of diffracted peaks (Fig. 8).

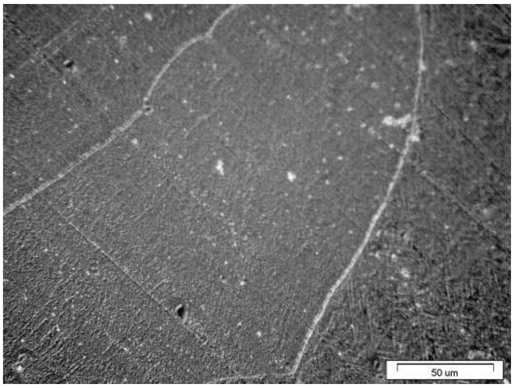

Fig. 7. Microstructure of supersaturated Mg-7Al-10Ho alloy (LM)

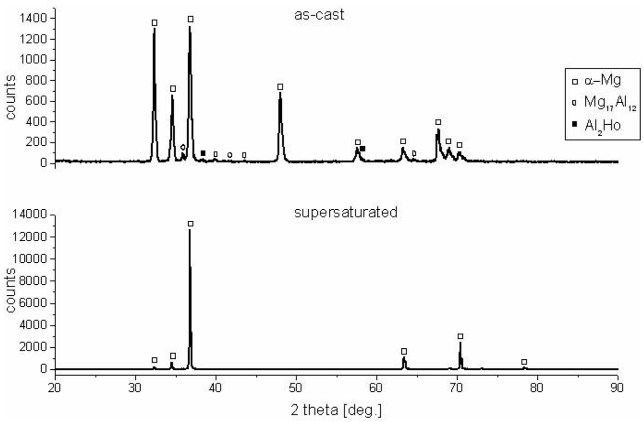

Fig. 8. X-ray diffraction pattern of as-cast and supersaturated Mg-7Al-10Ho alloy

The eutectic, visible on BSE micrograph as grey phase are situated in chains (Fig. 9). Al_2Ho precipitates of an irregular shape, are randomly distributed in microstructure, from fine (less than 1 μm) to large at about 5 μm. The clusters of few Al_2Ho precipitates are formed in microstructure. In the supersaturated Mg-7Al-10Ho alloy the eutectic is not present (Fig. 10).

Two times smaller Al content in the solid solution than in the as-cast Mg-7Al-3Nd alloy were detected (Table 2). It can be associated with higher rare earth element content, that strongly reacts with Al forming Al_2Ho. Similarly to supersaturated Mg-7Al-3Nd alloy, Al content caused transformation of Al_2Nd intermetallic phase into Al_3Nd.

Table 2.
Chemical composition of observed phases in Mg-7Al-10Ho alloy

Phase			Element in atomic %		
			Mg	Al	Nd
as-cast	α-Mg	hexagonal	97,59	2,41	-
	$Mg_{17}Al_{12}$	cubic	69,72	30,04	-
	Al_2Ho	cubic	-	64,88	35,12
super-saturated	Al_3Ho	hexagonal	-	75,88	24,12

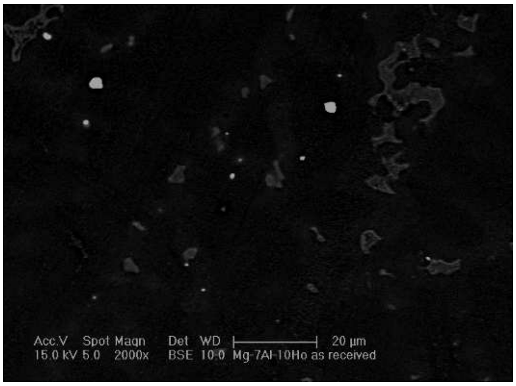

Fig. 9. Microstructure of as-cast Mg-7Al-10Ho alloy (BSE)

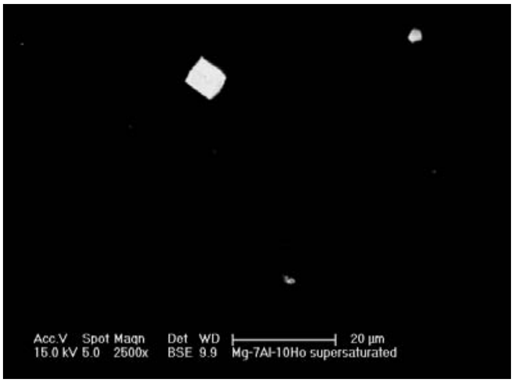

Fig. 10. Microstructure of supersaturated Mg-7Al-10Nd alloy (BSE)

3.3. Hardness results determined by Vickers indentation

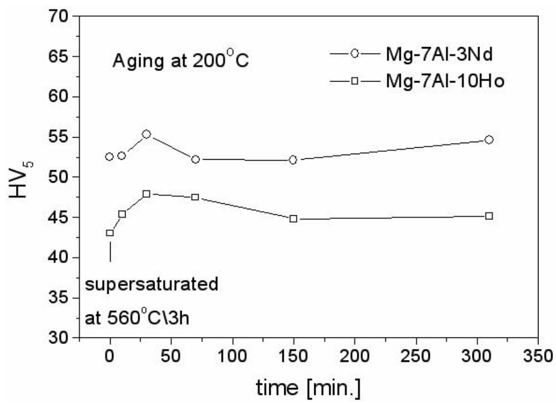

Fig. 11. The Vickers hardness changes during aging at 200°C for Mg-7Al alloys with 3% Nd and 10% Ho addition

A small increase in the Vickers hardness for both alloys during aging at 200°C was measured (Fig. 11). Mg-7Al alloy with 10% Ho exhibits better response to ageing treatment.

Higher HV_5 values for alloy with Nd addition are connected with precipitation of Al_3Nd and $Al_{11}Nd_3$.

Sufficient mechanical properties of Mg-Al-RE alloys at room temperature and good mechanical properties at elevated temperature were reported [11,12]. The creep tests of investigated alloys are required to estimate their creep behaviour.

3.4. The influence of Nd and Ho addition on microstructure of Mg-7Al alloy

Nd and Ho additions in Mg-7Al alloys influence the formation of intermetallics in α-Mg matrix with $M_{17}Al_{12}$ phase. In solid solution of Mg-7Al alloy with Nd, more Al is dissolved as compared to the alloy with Ho. It can be connected to the content of rare earth elements in both alloys.

After the solution heat treatment eutectic is dissolved in both alloys. It was reported that rare earth metals added to Mg-Al alloys suppress formation of undesirable $M_{17}Al_{12}$ phase [14]. Thermally stable intermetallics remains in the microstructure of investigated alloys, however during solution treatment Al_2Nd and Al_2Ho were enriched in Al, forming Al_3Nd and Al_3Ho phases respectively. Solubility of rare earth metals in Mg solid solution depends on atomic size [9], and decrease with increasing content of aluminium [13].

In heat-treated Mg-7Al-3Nd alloy, orthorhombic $Al_{11}Nd_3$ needle shaped compound precipitates at grain boundaries. The decomposition of $Al_{11}Nd_3$ phase above 150°C was reported [15].

It seems that Mg-7Al alloy with 10% Ho should have better creep properties at elevated temperature than alloy with 3% Nd, due to a fine, stable and randomly distributed Al_3Ho phase in microstructure. To confirm this inference, creep tests and microstructure analysis should be carried out.

4. Conclusions

The microstructure of as-cast Mg-7Al alloy with 3% Nd or 10% Ho consists of α-Mg solution, eutectic α-Mg + $Mg_{17}Al_{12}$ and intermetallic compounds Al_2Nd, $Al_{11}Nd_3$, and Al_2Ho respectively.

There was no evidence of eutectic α-Mg + $Mg_{17}Al_{12}$ presence in both solution heat treated alloys, but the particles of Al_3Nd, $Al_{11}Nd_3$, and Al_3Ho accordingly were detected.

Al content of dissolved in α-Mg solid solution was two times higher in Mg-7Al-10Ho than in Mg-7Al-3Nd alloy.

Aging both of alloys plays a small role in mechanical properties at ambient temperature.

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