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MICROSTRUCTURAL AND MECHANICAL CHARACTERIZATION OF THE THIXOCASTED ZK60-T6 MAGNESIUM ALLOY WITH RARE EARTH ELEMENTS

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Abstract. *This paper aims to study the effects of the T6 thermic treatment composed by solubilization and artificial aging, in the microstructure and mechanical properties of the ZK60 Magnesium Alloy produced by thixocasting, with standard composition and with the addition 1.5% in weight of rare earth. The results demonstrate that this casting process engender ingots with homogeneous chemical composition, free of pipes, blowholes and internal oxidation. Moreover, the microstructure of the alloy consists in a matrix of globular grains with heterogeneous shape, with the a-Mg phase reinforced by the precipitates Mg-Zn, Mg-Zn-RE and Mg-RE at the grain boundaries. Furthermore, the mechanical properties were tested using the Vickers micro hardness, comparing the results for the standard composition alloy, as casted and after the T6 treatment, as well for the alloy with rare earth, which presented a hardness increase after the thermal treatment.*

Keywords: *Magnesium, Rare Earths, Casting, Semi-solids, T6*

1. INTRODUCTION

Magnesium alloys have the lowest density of all the metals, high specific mechanical resistance and good machinability, making it an interesting material for the automotive and aircraft industry where weight reductions result in increasing efficiency and performance.

The addition of mischmetal (MM) to the ZK60 alloy has already proved the formation of intermetallic phases with high melting point, thermochemical stability and has also, improved both mechanical and corrosion resistance in comparison to the unalloyed ZK60.

Mg-Zn-Zr aloys exhibit one of the highest mechanical properties among all magnesium-alloys. Moreover, recent studies have proved that the addition of rare earth elements improves the behavior of such alloys in corrosive environment. Furthermore, new studies with Mg-Zn-Zk alloys may show new alternatives to the use of Magnesium as light structural material.

Given all the highlights of this alloy, this paper presents the development of a laboratorial production scale of ZK60 alloy (Mg-6 wt.%, Zn-1 wt.%, Zr - up to 0.45 wt.%), which was further, modified by the addition of 1.5% in weight of mischmetal (a mixture of rare earths with reduced cost). The effects of precision casting process in the semi-solid state (thixocasting), and artificial aging heat treatment (T6), using solubilization temperatures of 250°C, 350°C and 400°C and aging temperatures 150°C, 200°C and 250°C were studied by Vickers hardness (HV), Optical Microscopy (OE), Scanning Electron Microscopy (SEM), and Energy Dispersive X-ray Spectroscopy (EDS). The promising results of all the tests conducted, confirms that such process contributes as a prominent stem for arising the industrial scale production of this alloy.

2. EXPERIMENTAL PROCEDURE

The ZK60 alloy was obtained from the following raw materials: Metallic magnesium, alloying metal Zirmax (Mg-33.3-Zr wt.%), electrolytic zinc and for the modified alloy, Mischmetal (55-Ce wt.%, 24-La wt.%, 15-Nd wt.%, 4-Pr wt.%). The solidus and liquidus temperatures were obtained based on the ASM standard composition for the ZK60 alloy in addition to the method presented by Mordike (2006), in regards to the following binary phase diagram.

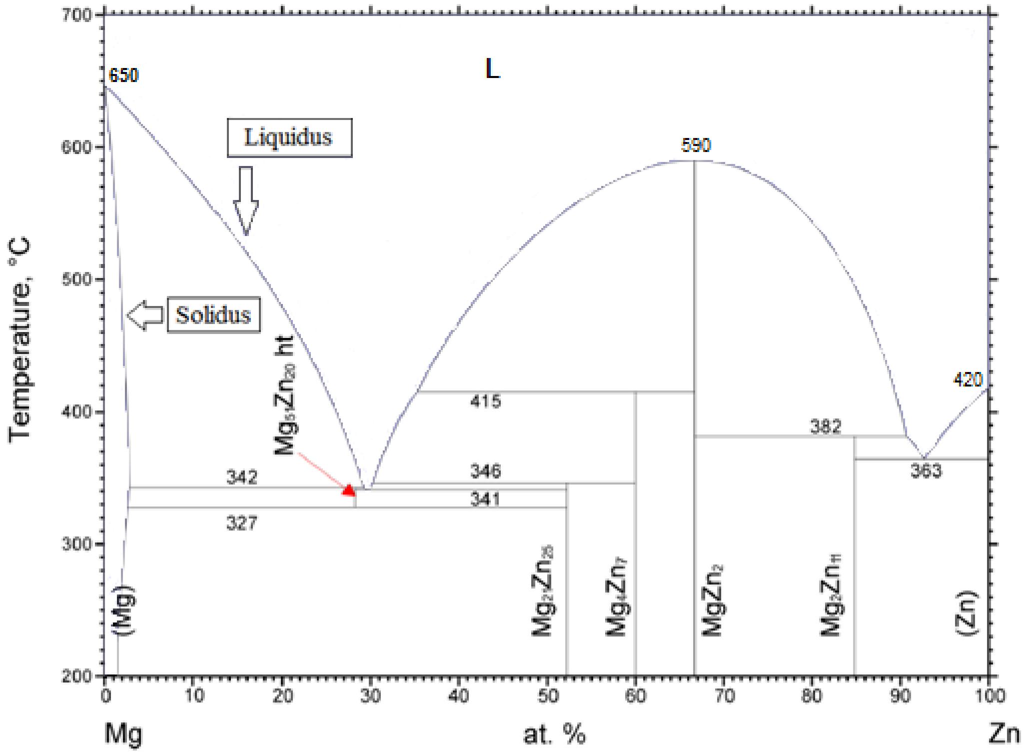

Figure 1. Adapted Mg-Zn phase diagram (Miettinen, 2008)

The alloys were obtained by thixocasting, that is, using mechanical stirring while the material contained in the ladle is in the semi-solid state. A furnace used is resistance heated, equipped with a stir blade and has its atmosphere protected by argon at a flow rate of 15 L/min, maintaining an absolute pressure of approximately 0.75 bar. The ladle and the blade are made of SAE 1020 steel. The melt volume was determined to fill 40% of the ladle, engendering an ingot with 124mm in diameter and 245mm in height.

The T6 heat treatment was performed on the furnace model Analógica NA-1100-TM, and the atmosphere was protected with argon with a flow rate of 10L/min and an absolute pressure of 1.25 bar.

The specimens were microstructurally characterized by means of optical microscopy and scanning electron microscopy, in order to identify the precipitation profile resulting from the aging process. In addition, EDS was used to identify by chemical microanalysis the elements present inside the grains and at their boundaries. Finally, the samples have been mechanically characterized by the Vickers microhardness concerning the standard ISO 6507-1:2005, which states the application of 1.96 N load for 20s, by a indenter made of diamond, with pyramidal shape tip, angled at 136° in regard to the edges, and a quadrangular base. The equipment used was the testing machine Mitutoyo MVK GI.

The experimental factors investigated were the type of the alloy, the solubilization temperatures T1, T2 and T3, and the artificial aging temperatures T4, T5, T6, all these factors concerning both the alloys with or without with or without rare earths added as casted. The statistical analysis was conducted using the tool ANOVA, and the software Minitab, using the data obtained on each sample and test, as inputs. As presented by the Tab. 1, a total of 20 different processes were studied, with 3 reproductions for each one, therefore resulting in the analysis of 60 experimental conditions.

Table 1. Statistical methodology applied in the analysis of the experimental results (Machado, 2015)

Processes	Conditions
1	As casted, with Mischmetal
2	As casted, without Mischmetal
3	Solubilized at 400°C/24h and aged at 150°C/12h, with Mischmetal
4	Solubilized at 400°C/24h and aged at 150°C/12h, without Mischmetal
5	Solubilized at 400°C/24h and aged at 200°C/12h, with Mischmetal
6	Solubilized at 400°C/24h and aged at 200°C/12h, without Mischmetal
7	Solubilized at 400°C/24h and aged at 250°C/12h, with Mischmetal
8	Solubilized at 400°C/24h and aged at 250°C/12h, without Mischmetal
9	Solubilized at 350°C/24h and aged at 150°C/12h, with Mischmetal
10	Solubilized at 350°C/24h and aged at 150°C/12h, without Mischmetal
11	Solubilized at 350°C/24h and aged at 200°C/12h, with Mischmetal
12	Solubilized at 350°C/24h and aged at 200°C/12h, without Mischmetal
13	Solubilized at 350°C/24h and aged at 250°C/12h, with Mischmetal
14	Solubilized at 350°C/24h and aged at 250°C/12h, without Mischmetal
15	Solubilized at 300°C/24h and aged at 150°C/12h, with Mischmetal
16	Solubilized at 300°C/24h and aged at 150°C/12h, without Mischmetal
17	Solubilized at 300°C/24h and aged at 200°C/12h, with Mischmetal
18	Solubilized at 300°C/24h and aged at 200°C/12h, without Mischmetal
19	Solubilized at 300°C/24h and aged at 250°C/12h, with Mischmetal
20	Solubilized at 300°C/24h and aged at 250°C/12h, without Mischmetal

The T6 heat treatment was performed on the furnace model Analógica NA-1100-TM, and the atmosphere was protected with argon with a flow rate of 10L/min and an absolute pressure of 1.25 bar.

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3. RESULTS

3.1 Chemical composition of the casted ingots

The chemical composition of each casted ingot was obtained by X-ray Fluorescence (XRF), using the instrument EDX-8000. The results are presented the Tab. 2 and Tab. 3.

Table 2. Chemical composition of the ingot casted with mischmetal

Mg	Zn	Ce	La	Nd	Mn	Pr	Zr	Others
92.717	5.757	0.517	0.299	0.167	0.14	0.07	0.312	0.021

Table 2. Chemical composition of the ingot casted with mischmetal

Mg	Zn	Ce	La	Nd	Mn	Pr	Zr	Others
93.135	6.28	0.085	0.094	0.041	0.014	-	0.351	-

3.2 Microstructures as casted

The mechanical stirring during the solidification produces a homogeneous microstructure consisting on a matrix of globular grains of the α-Mg phase, with average grain size of 100 μm and heterogeneous shape, reinforced by an extensive net of precipitates along the grain boundaries. One stems from the preferential segregation of rare earth elements (Fig. 3) and zinc (Fig. 3 and Fig. 4), on the liquid phase during the solidification interval, which can be addressed to the solute solubility gap that exists between the liquid and the solid states.

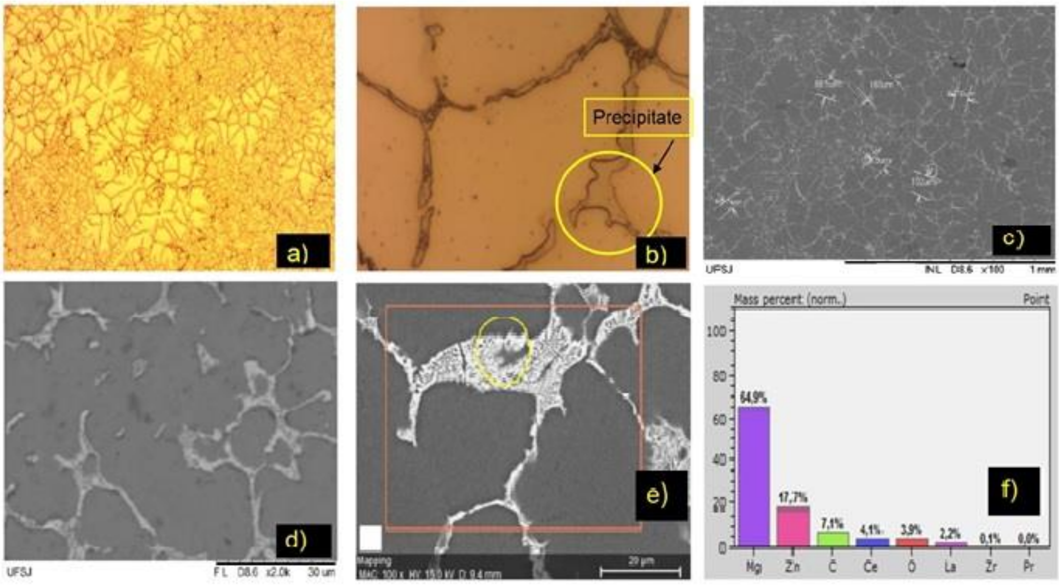

Figure 3. Images a and b, optical microscopy of the alloy ZK 60 with 1.5wt % of Mischmetal, as casted, showing heterogeneous grain size and precipitates at the grain boundaries. Images c and d, SEM presenting the morphology of the precipitates, the first smooth and the second lamellar. Images e and f, EDS mapping outlining the mass percentage of each element present on the alloy.

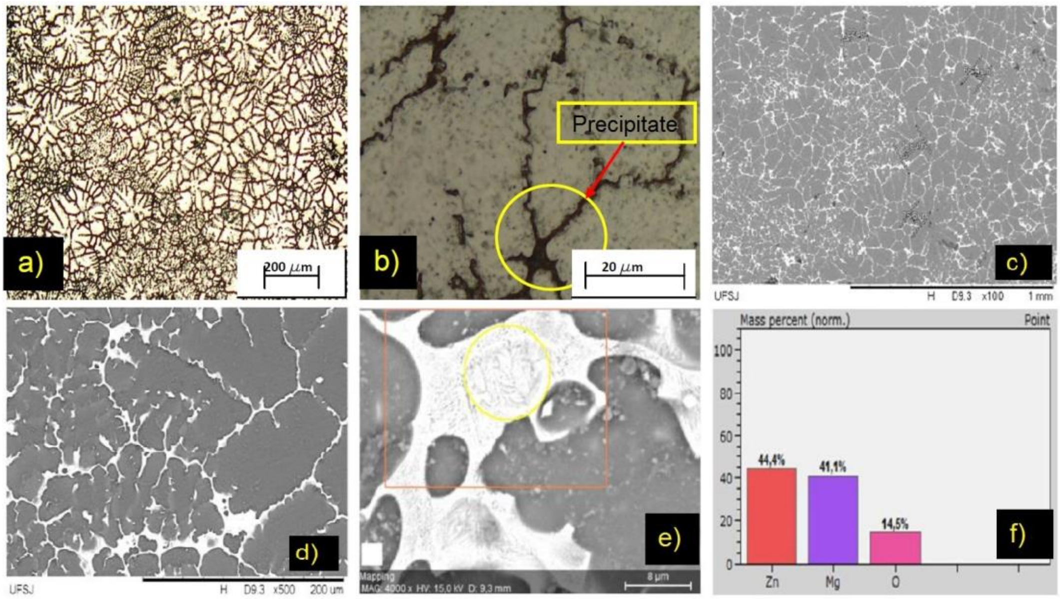

Figure 4. Images a and b, optical microscopy of the ZK60 alloys without Mischmetal, as casted, heterogeneous grain sizes and precipitates at the boundaries. Images c and d, scanning electron microscopy showing the morphology of the Mg and Zn precipitates with lamellar shape. Images e and f, EDS mapping presenting the mass percentage of each element of the alloy.

3.3 Effects of the heat treatment on the microstructures and mechanical properties

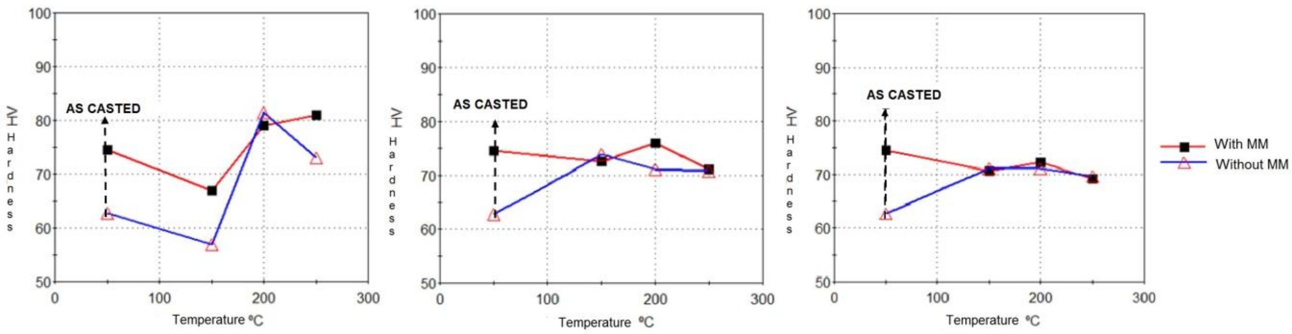

Figure 2. Comparison between the T6 heat treatment effects on the alloy with MM and without MM

The best mechanical properties were obtained through the T6 thermal treatment due to the redistribution of the precipitated phases, the comparison of the results with the sample as casted is presented on the Fig. 2, by comparing the hardness measurement results for each one in regards to the aging temperature used. The control and distribution of the phases comes up with temperature variations at the solubilization, quenching and aging, in the sequence.

As shown by the Fig. 2, the best result was obtained using the solubulization temepratue at 400C for 24h, followed by aging at 200°C and 250°C, which presented a hardness increase of 30% for the alloy without MM and of 8% for the alloy with MM, when compared to the alloys with and without mischmetal, as casted.

4. CONCLUSION

The ingots obtained have homogeneous chemical composition, are free from blowholes, defects and internal oxidation. The raw solidification microstructure is characterized by a matrix composed by globular grains of α-Mg reinforced by a net of precipitated of the MgZn, MgZnRE and MgRE types, segregated along the grain boundaries. After several heat treatments T6, followed by microhardness hardness tests, the best experimental condition was revealed the solubilization at 400°C for 24h, with aging at 200°C or 250°C for 12h, both for the alloy with and without mischmetal. When compared to the alloys as casted, the hardness increase of the aged alloys can be addresses to the homogenous precipitation of nanoparticles at the grains interior.

5. ACKNOWLEDGEMENTS

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