EUROPEAN ACADEMIC RESEARCH
Vol. V, Issue 3/ June 2017

Impact Factor: 3.4546 (UIF)
DRJI Value: 5.9 (B+)

ISSN 2286-4822
www.euacademic.org

Quantitative analysis of phase precipitation of the magnesium alloy in dependence of various heat treatment processes

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Abstract:

Precipitation phases created in the microstructure of Mg-Al-Zn AZ61 Magnesium workable alloy, in dependence of heat treatments process regimes: solution heat treatment in temperatures 470°C for 5hours and precipitation heat treatment in temperatures 100°C, 150°C, 200°C, 250°C, 300°C in 1 and 15h., were exanimate in this study. Through this regimes in the AZ61 alloy microstructure by TEM-EDX analyzes are effectuated these precipitations phases: Al-Mn, Al4Mn, Al8Mn5, Mg-Si, Al-Mg-Mn. Light Microscopy after this regimes and controlling methods, distinguished and demonstrate precipitations particles phases of Al-Mn, Mg-Si, Al-Mg-Mn, Al-Mg-Si Al-Mg-Si-Mn, Al-Mg-Mn-Fe, Al-Mg-Si-Fe with size 0.3 µm – 50 µm, by having morphology of disc, needle, plate and rounded shape. Quantitative analyze of volume fraction by light microscopy, under this temperature, 100°C for 1h and 15h has effectuated the highest volume fraction of precipitation particles.

Key words: Magnesium alloy; Microstructure; heat treatment; phase precipitation; quantitative analyze

INTRODUCTION

AZ61 workable alloy consideration the designations of Mg and Mg alloys contains 6% Al and 1% Zn [1]. This alloy be used for instruments which have high mechanics operation and for the production of forget materials [2].

Given rise to the mechanical-thermal-physical properties of this alloy its demonstrating fine properties from corrosion [3].

The heat treatment of the metals and its alloys is most important phase of the finale tape of the production.

The initial structure may vary in wrought products from unrecrystallized to recrystallize and may exhibit only modest or additional strain from quenching after solution heat treatment. The rapidly solidification process offers significant improvements in the mechanical properties of this alloy [4].

Choice of time-temperature cycles for precipitation heat treatment should receive careful consideration.

One essential attribute of a precipitation-hardening alloy system is a temperature-dependent equilibrium solid solubility characterized by increasing solubility with increasing temperature.

Larger particles of precipitate result from longer times and higher temperatures; however, the larger particles must, of necessity, be fewer in number with greater distances between them [5] .

The objective is to select the cycle that produces optimum precipitate size and distribution pattern [6].

For the alloy AZ61 specimens chosen for this study, the thermal operations applied has one or more purposes depending upon the original state of the alloy and the production process involved.

One of the principal objectives is improved workability and optimized information about microstructure through the next steps of heat treatment. Consequently, the cycles used represent compromises that provide the best combinations of properties.

Heat treatment to homogenize and increase property of this Magnesium alloy is a three-step process:

- Solution heat treatment: dissolution of soluble phases
- Quenching: development of super saturation
- Precipitation heat treatments: to provide hardening by precipitation of constituents from solid solution.

MATHERIALS AND METHODES

Experimental work

The chemical composition from the AZ61 alloy, determinated by Wave Length Dispersive Electron Microbeam Analysis (WDX) is shown in Table 1.

Chemical Composition in Masse % (Reminder Mg)							
Alloys	**Al**	**Zn**	**Mn**	**Si**	**Fe**	**Ni**	**Cu**
AZ61 specimens	5.05	0.81	0.15	0.058	0.004	0.007	0.004

Table 1. ***Chemical composition in Masse % of AZ61alloy***

For the reason that Magnesium is a material that have a high affinity for the oxygen, so to protect it against oxidations and to arrive a quality of heat treatment must that the AZ61 magnesium specimens put inside an ampoule under Argon atmosphere with Titanium welding on of chip.

The ampoule is made from quarc glass with a length of 150mm and a diameter of 12mm.

The constituent thermal operation of the heat treatment process to show the structural changes of the alloy was the solution heat treatment of the specimens in elevated temperature 470 °C for time extends 5hours.

In most instances, to avoid those types of precipitation that are detrimental to mechanical properties the solid solution formed during solution heat treatment must be quenched rapidly enough (and without interruption) to produce supersaturated solid solution at room temperature – the optimum condition for precipitation hardening.

Immediately from the final hot working operation the ampoules with the specimens are depth in the water to be quenched and after they come broken in way to take the specimens. The last thermal operation of the heat treatment, after this process, to take more information about phases created in this alloy was the Precipitation heat treatments.

The effects of precipitation on mechanical properties are greatly accelerated and usually accentuated by reheating the quenched material in different temperatures, 100 °C, 150 °C, 200 °C, 250 °C and 300 °C for time extend 1 and 15 hours.

After this in the same way of the preview process they come quenching in water.

After this steps the specimens come under the metallographic preparation for Light Microscopy analyzes which investigate the microstructure (Table 2, 3, 4).

Stage N°	Support	Abrasive	Pressure (N)	DiscRotating Speed (rpm)	Time (sec)	Lubricant
1	paper	SiC 800	50-100	150	40	water
2	paper	SiC 1000	50-100	150	40	water
3	paper	SiC 1200	50-100	150	40	water
4	paper	SiC 2400	50-100	150	40	water
5	paper	SiC 4000	50-100	150	40	water

Table 2. Metallographic grinding preparation

Stage N°	Support	Abrasive	Pressure (N)	Disc Rotating Speed (rpm)	Time (min,sec)	Lubricant
1	MOL	Diamante spray 3µ	100-150	150	1'55"	Blue/liquid
2	NAP	Diamante spray 1µ	100-150	150	1'55"	Blue/liquid
3	CHEM		30-40	150	20-30"	Water/OP(S)[1](2/1)

Table 3. Metallographic polishing preparation

	Solution	Mas. cm^3		Solution	Mas. cm^3		Solution	Mas. cm^3
Acetic acid	Acetic acid	4,2	Nitric acid-Acetic acid	HNO_3	1	Oxalat acid	Oxal acid	1
	Water	10		Water	10			
	Acetic acid	10		Acetic acid	20		Water	100
	Ethylenglycol	70		Ethylenglycol	60			

Table 4. Metallographic etching preparation [7]

For the chemise analyze of the smaller parts (smaller than 3 µm), from AZ61 alloy specimes (table 5), is used the TEM-EDX (Transmission Electron Microscopy – Energy Dispersive Rötgen Analyze) analyze and for the topographic surveys, e.g. diffraction contrast microscopy.

Nr.	Conditions
1	Initial state
2	Homogenizing 470 °C 5 h
3	Homogenizing 470 °C 5 h + annealing 100 °C 1 h
4	Homogenizing 470 °C 5 h + annealing 100 °C 15 h
5	Homogenizing 470 °C 5 h + annealing 200 °C 1 h
6	Homogenizing 470 °C 5 h + annealing 200 °C 15 h

Table 5. Conditions of heat treatment process, for the specimens use for TEM investigations [7]

Electronic microscopy was a technique widely used in structural and chemical characterization of materials. It provides information about morphology, grain size, chemical composition, crystalline and identification of crystalline phases in materials.

This technique allows the quantitative analysis, structural studies by high resolution microscopy and spectroscopic analysis by electron energy loss.

The preparation of the discs (from the AZ61 alloy specimens) is made up of the following steps [8], [9]:

-selection of specimens
-removal of specimens
-pre-thinning
-final thinning

In AZ61 alloy specimens, the aim was to produce discs of about 100µm in thickness and 3mm in diameter, at the centre of which is the particular area of the specimens to be investigated.

RESULTS AND DISCUSSIONS

The purpose is to accelerate and accentuate the solidification of this alloy in way to achieve a nearly homogeneous solid solution giving optimize hardness results, through the precipitation-hard enable of the AZ61 wrought alloy.

The microstructure consisted of many particles of Mg α grains, separated with small particles of quasi-crystalline precipitation phases called "secondary phases", which with the increasing of temperature from 100°C - 470°C of homogenizing process diffusing in the Mg matrix.

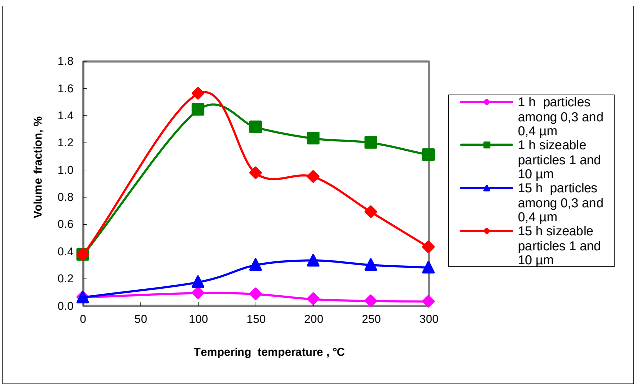
After, 5 hours solution heat treatments in temperature 470°C and 1h and 15 h in 100°C- 300°C, precipitation heat treatment the mirostructure was transformed in a conventional already homogenous structure, with polygonal grains [9] .

The Mg α grains was enriched in aluminum, manganese, siliceous, zinc, basically to the simultaneous dissolution of the phases and variability of the volume fractions.

After this long interval the continuous or discontinuous precipitation phases were crystallized in the boundary of α grain.

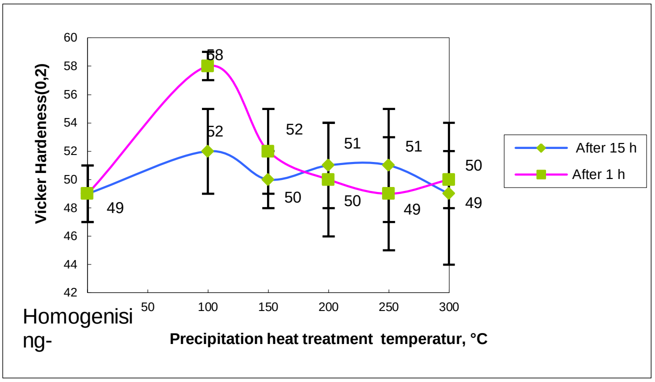
The maximum of homogenization is definite in the lower precipitation heat treatment temperature 100°C for 1h.

Quantitative analyze of volume fraction by light microscopy, under this temperature, 100°C for 1h and 15h has effectuated the highest volume fraction of precipitation particles (graphic 1).



Graphic 1. The variability of volume fraction (%) of the small particles with size 0.3-0.4µm and the sizeable precipitations particles with size from 1-10µm, from the precipitation heat treatment temperature. [9]

In connection with this, are shown the highest value of the macro hardness parameter of the AZ61 alloy microstructure property (graphic 2), by 58HV2 after 1h and 52HV2 after 15h tempering in temperature 100°C.



Graphic 2. The HV2 macro hardness curve for the specimens of the AZ61 alloy, after heat treated in different temperatures for 1h and 15 hours

Light Microscopy and TEM analysis revealed morphology of precipitation phases like, needle shape, rounded or disc shape, figures 1, 2, 3, 4, 5, 6 downstairs.

The Al-Mn (generally until 50 μm) and the Mg-Al-Mn (with a dimensions 3-10 μm) phase precipitations observed with establishing morphology in photos 1 and 2, after precipitation heat treatment 200°C for 1h, and solutiontion heat treatment, in 470°C, 5h.

The consequence of thermal treatment on the precipitation response of intermetallic precipitates particles created in the microstructure of AZ61 alloy is analyzed to give a mass percent and atomic percent information of them.

TEM-EDX analyze (table 6) demonstrate a 60-70 % Al Mass percent and 78-80% Al atomic percent and 20-30% Mn in mass percent, 18-22 % Mn atomic percent in type particles of Al:Mn with a relation 4:1through them.

Homogenizing regime	**Temperature °C**	**Time, h**	**Precipitation phases**
Solution heat treatment	**450°C 470°C 490°C**	**AZ**	**Al-Mn, Mg-Si, Al-Mg-Mn**
		1	
		3	
		5	
Precipitation heat treatment (after solution heat treatment 470°C, 5h)	**100°C**	**1**	**Al-Mn, Mg-Si, Al-Mg-Mn, Al-Mg-Mn-Fe,**
	150°C		
	200°C		
	250°C		
	300°C		
Precipitation heat treatment (after solution heat treatment 470°C, 5h)	**100°C**	**15**	**Al-Mn, Mg-Si, Al-Mg-Mn, Al-Mg-Si Al-Mg-Si-Mn, Al-Mg-Mn-Fe, Al-Mg-Si-Fe**
	150°C		
	200°C		
	250°C		
	300°C		

Table 6. Precipitation phases created in the AZ61 alloy under homogenizing regimes. [TEM-EDX, Sonde-WDX] [9]

The Al:Mg:Mn type particles have a mass and atomic percent of the elements of 16-20% Al, 67-73% Mg, 5-10 Ma.% Mn and 16-19% Al, 75-80% Mg, 4 -8% Mn atomic percent with a relation of 2.9 : 12.9 : 1. The figures 2, 3 and 4 shows the Mg-Al-Mn phase's precipitation of the AZ61 alloy take from Light microscopy and TEM investigations [9].

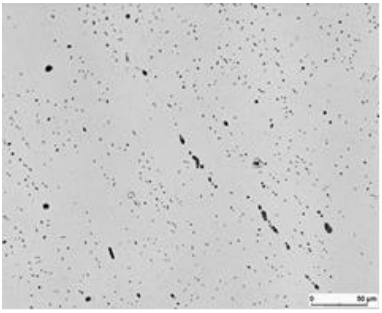


Figure 1. The Al-Mn phase precipitation in the microstructure of the specimens treated in temperature 200°C for 1 h (OP-S suspension)

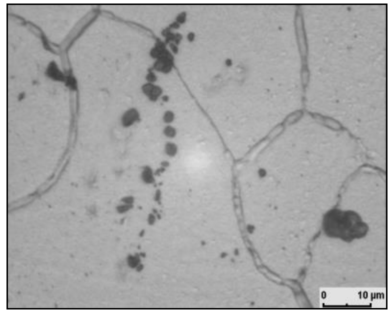


Figure 2. The Al-Mg-Mn phase precipitation after homogenizing in 470°C 5h (Pikrinsäure 30 sec.) take from Light Microscopy

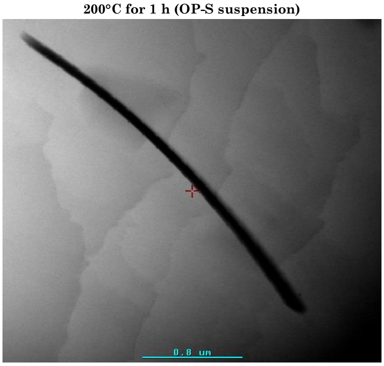


Figure 3. The Mg-Al-Mn phase precipitation, needle like of AZ61 alloy after specimens treated at 200°C for 1h. (photo from TEM investigation)

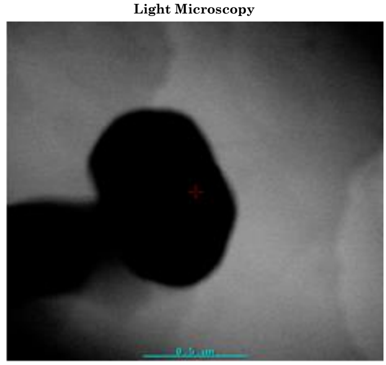


Figure 4. The Mg-Al-Mn phase precipitation, Polygonal shape like of AZ61 alloy after specimens treated at 200°C for 15h. (photo from TEM investigation)

The photos in figures 5 and 6, shown the Mg-Al-Si-Mn, Mg-Al-Si phase precipitation of AZ61alloy after heat treatment, in characteristics hexagonal and spherical form, taking from TEM investigations.

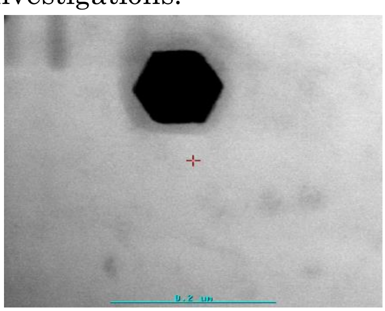


Figure 5. The Mg-Al-Si-Mn phases precipitation, hexagonally shape of AZ61 alloy after specimens treated at 100°C for 1h. (photo from TEM investigation)

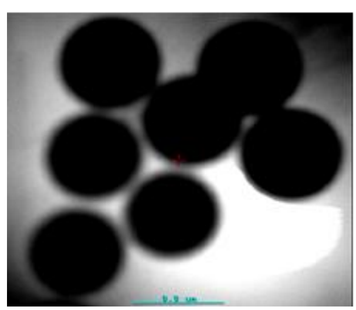


Figure 6. The Mg-Al-Si phase precipitation, spherical shape of AZ61 alloy after specimens treated at 200°C for 1h. (photo from TEM investigation) [9]

CONCLUSIONS

1. The effective temperature and time to give up the qualitative homogenizing microstructure was 470°C for 5h, for solution heat treatment of the alloy microstructure.
2. Through this regime in the AZ61 alloy microstructure by TEM-EDX analyzes are effectuated these precipitations phases: ***Al-Mn, Al_4Mn, Al_8Mn_5, Mg-Si, Al-Mg-Mn.***
3. The effective temperature and time to give the satisfying results, after microstructure investigations of the AZ61 alloy was the 100°C for 1h and 15h, precipitation heat treatment.
4. Light Microscopy after this regime and controlling methods distinguished and demonstrate precipitations particles phases of **Al*-Mn, Mg-Si, Al-Mg-Mn, Al-Mg-Si, Al-Mg-Si-Mn, Al-Mg-Mn-Fe, Al-Mg-Si-Fe*** with size 0.3 µm – 50 µm, by having morphology of disc, needle, plate and rounded shape.
5. Quantitative analyze of volume fraction by light microscopy and the highest value of the macro hardness parameter, was effectuated in temperature, 100°C for 1h and 15h.

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