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Study of the microstructural evolution of precipitation in AZ91 alloy

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Abstract

Magnesium-based alloys have been the subject of several scientific research projects. Their excellent mechanical properties have allowed their use in various fields such as aeronautics and automotive. Phase transformations play a crucial role in the mechanical properties improvement; therefore the understanding of structural hardening due to the precipitation reaction is important. The main objective of this work is to study the influence of the aging temperature on the precipitation mechanism in AZ91 () alloy. It has been found that at high aging temperatures continuous precipitation is appeared and characterized by fine precipitates, however, discontinuous precipitation is observed under cellular form at low temperatures. With increasing ageing time continuous precipitates can be also observed and they block the growth of discontinuous precipitates. This phenomena influence directly the mechanical properties of such alloys, for that microhardness measurements are done to evaluate the mechanical properties variations as function of temperature, ageing time and so on.

Keywords: AZ91; continuous and discontinuous precipitation; aging; hardening.

1. Introduction

The good properties of magnesium such as low density (1.74 g.cm-3) and its high specific strength make its alloys potential candidates for replacing steel and other heavier materials [1]. Currently, the most widely used Mg-based alloy in the industry is AZ91 [2] industrial castings foundry casting alloy, it is based on the binary alloy Mg-9Al (wt. %.) with Zinc additions of 0.5-1.0 % by weight and about 0.3 % by weight of manganese. This system is the seat of a hardening due to the precipitation reaction but in reality this hardening is not important compared to the Al-Zn system. The main reason for the poor response of the structural hardening is due to the appearance of the continuous and discontinuous precipitation reaction [3,4]. While the first corresponds to germination and growth of particles of the $Mg_{17}Al_{12}$ phase but in the form of rods, platelets and needles in a massive way at the grain and is homogeneous, and significantly improves the properties (hardness, mechanical strength) of the material, the discontinuous precipitation produced, locally from the grain boundaries, a duplex cell structure formed of alternating lamellae of the same precipitate and solute solute-solute solid solution, it is heterogeneous, and it deteriorates the mechanical properties. In the present work, we followed the effect of aging temperature on the precipitation mechanism in the AZ91 alloy.

2. Experimental method

An AZ91 magnesium alloy was used in this study having a chemical composition of 9 wt. % Al, 1 % Zn, 0.5% Mn. Slices were thermally treated at T6, which included homogenization for 24 hours at 425 °C, cold water quenching, and artificial aging at 150 or 300 °C for different times.

For microstructural characterization samples were prepared, polished and etched. The microstructure of the samples was analyzed by Jeol 7100 scanning electron microscopy equipped with an Energy Dispersive X-ray Spectroscopy (EDX) and another of the TESCAN VEGA TS 5130 MM type. Our study was completed by X-ray diffraction with a PANalytical Empyrean powder diffractometer. Microhardness measurements are done by using ZWICK apparatus ($Hv_{0.1}$) using 100 g load.

3. Results

Chemical analysis of the studied alloy has been done by energy dispersive X-ray spectroscopy (EDS). Figure 1 and the inserted table (table 1) show the energy dispersion diagram and both concentrations of aluminum (Al) and Zinc (Zn) present in this alloy.

Table1: AZ91 alloy composition.

Element	Wt. %	At. %
MgK	89.74	91.02
AlK	9.52	8.70
ZnK	0.73	0.28
Total	100	100

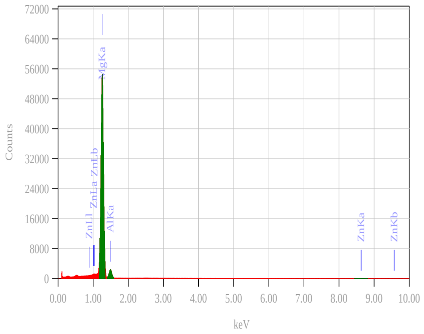

Figure 1. EDS spectrum of AZ91 alloy.

3.1. Metallographic study

Figure 2 (a-b) shows the microstructure of the AZ91 quenched state. The microstructure shows the presence of α-Mg and eutectic phases partially separated; the intermetallic compound $Mg_{17}Al_{12}$ is distributed in the form of islands within the α-Mg phase. The difference in the image contrast between the inner and outer regions of the dendrites shows that the segregation of aluminum formed during the solidification of the alloy. There is therefore a gradient of aluminum concentration in the α phase, as one approaches the β phase [5]. After annealing at 425 ° C (Fig. 2c) causes the complete dissolution of the eutectic and homogenized aluminum is appeared throughout the matrix.

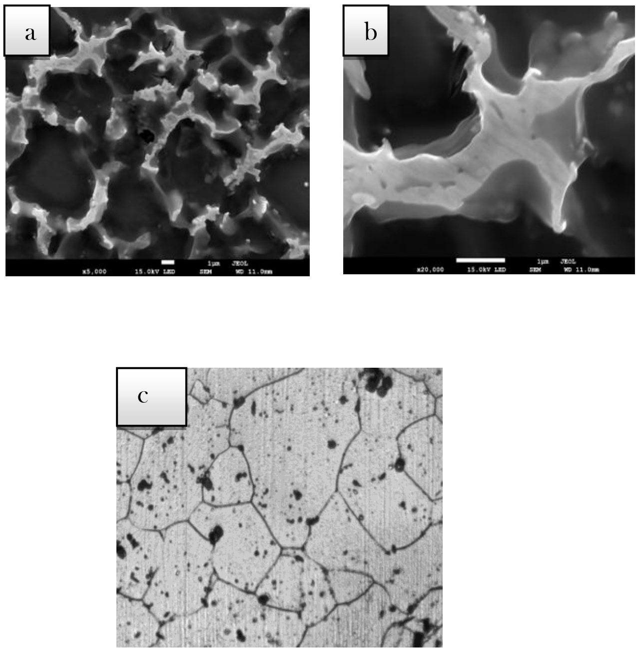

Figure 2. SEM (a-b) and optical microscope micrographs of the AZ91 sample after water quenching.

Figure 3 (a-b) shows the microstructure of a sample aged at 150 ° C for 10h. The first stages of lamellar precipitation consist in the formation of small β-germs on a grain boundary with strong disorientation. It is controlled by the diffusion of solute atoms along the reaction front (RF) which was initially a grain boundary (GB). The micrograph observed in Figure 3c gives a general view of which types of precipitates are appeared in this alloy: intergranular precipitates (interfacial type) and precipitates that form on dislocations (inside the grain).

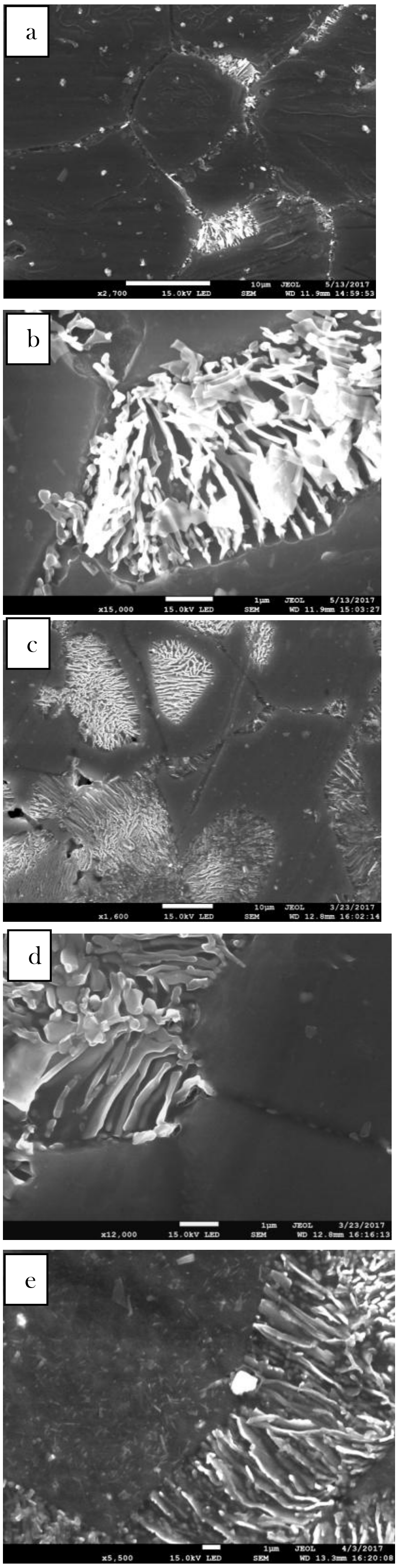

Figure 3. Microstructure of a homogenized AZ91 sample, tempered and aged 10h (a-b), 35h (c-d) and 60h (e)

At the first aging time after quenching (2h at 200 ° C) we observed in different regions of the matrix the formation of precipitate cells on the grain boundaries (Fig. 4a) with a cell morphology of $Mg_{17}Al_{12}$ precipitate similar to that observed by Azzeddine et al [6]. One of the phenomena observed after prolonging the aging time (24h and 35 h at 200 ° C.) is the formation of the β phase ($Mg_{17}Al_{12}$), in the form of needles almost perpendicular and oriented in two clearly defined directions, forming a structure identical to that of Widmanstätten (Fig. 4 b-c) [7].

After a relatively short holding time (10 min at 300 ° C), one can observe precipitate cells formation on the grain boundaries (Fig. 5a) and particles in the form of needles inside grains, confirming the start of the continuous precipitation (Fig. 5b). The increase of the aging time to 300 °C allowed a greater development of the needles of the β phase inside the grains (Fig 5c). At this temperature the discontinuous precipitation has almost disappeared because on the one hand the diffusion in volume prevents its growth and on the other hand its germination becomes difficult.

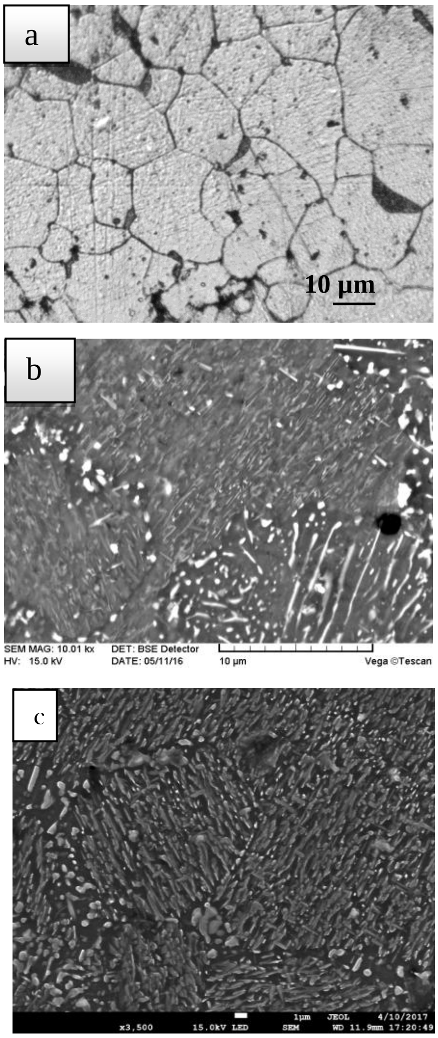

Figure 4. Microstructure of a homogenized AZ91 sample, quenched and aged 2 h (a), 24h (b) and 35 h (c) at 200 °C obtained by optical microscope (a) and (b-c) by SEM.

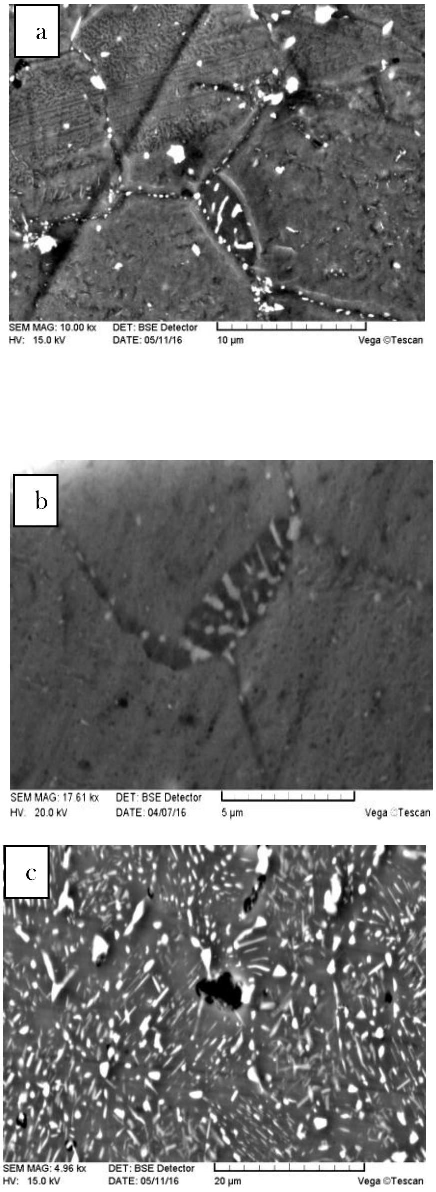

Figure 5. Microstructure of a homogenized AZ91 sample, quenched and aged for 10 minutes (a-b) and 10h (c) at 300 °C, obtained by scanning electron microscope.

3.2. Results of X-ray diffraction

X-ray diffraction spectra of the AZ91 material, aged at 150 ° C for 10h, reveal the existence of β phase (all peaks are indexed in figure 6).

After aging for 50h (Fig. 6) the quantity of the β phase is relatively increased, this effect is confirmed by the increase of the XRD peaks intensity of this phase.

X-ray spectra of AZ91 alloy aged at 200 ° C up to 40 hours (Fig. 7) show the presence of four lines associated with Mg17Al12 precipitated β phase. The extension of the aging time results in the coexistence of α_0 supersaturated phase and (α + β) precipitates. Therefore, the spectrum of this intermediate state (1h30 at 200 ° C) brings together at the same time the peaks of these three phases (α_0, α and β). The splitting peaks of intermediate state explain the coexistence of α and α_0. With the extension of aging time (10h at 200 ° C) we observe the peaks of α and β only with the disappearance of α_0 phase. On the other hand, the analysis of the X-ray diffraction spectrum corresponds to the prolongation of the aging time from 10 hours to 40 hours, shows a continuous displacement of the peaks in the direction of the decrease of the angle θ with respect to the aged state 1.5 hours, which confirms that this reaction is continuous.

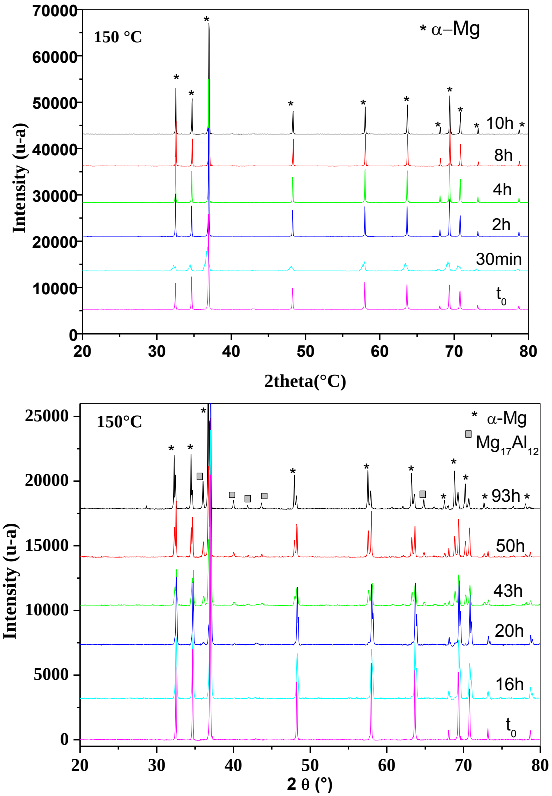

Figure 6. RX diffraction spectra of the AZ91 alloy homogenized, quenched and aged at 150 ° C.

X-ray diffraction spectra of the aged samples at 300 ° C for different times are shown in Figure 7b and it shows that the peaks of the second solid solution are relatively shifted to the of pure magnesium lines, due to the substitution in a larger amount of magnesium by aluminum that has smaller atomic radius (r_{Al} = 1.27 A° and r_{Mg} = 1.60A°) (this solid solution having lattice parameters a and c lower than the supersaturated solid solution). The intensity of the most intense peak related to the formation of the precipitated phase β which increases with aging time increasing; this is explained by the increase in the volume fraction of the precipitated phase during the isothermal aging (result in good agreement with the metallographic results). The extension of the aging time up to 2h shows the appearance of the other peaks which are still associated with the precipitated phase β. A continuous displacement of the peaks was observed after an extension of the aging time; these results being confirmed by microstructural observations, continuous precipitation is the most dominant reaction at this temperature.

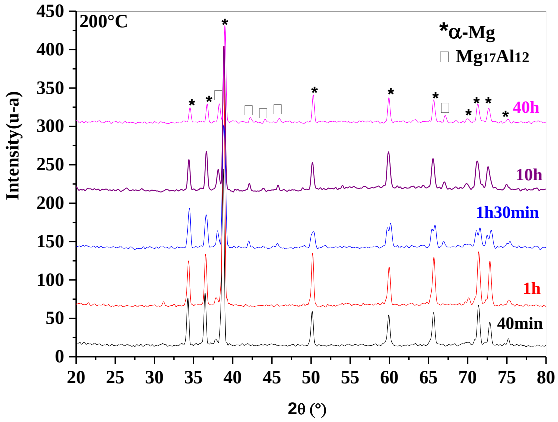

Figure 7. X- ray diffraction spectra of the AZ91 alloy homogenized, quenched and aged at 200 ° C.

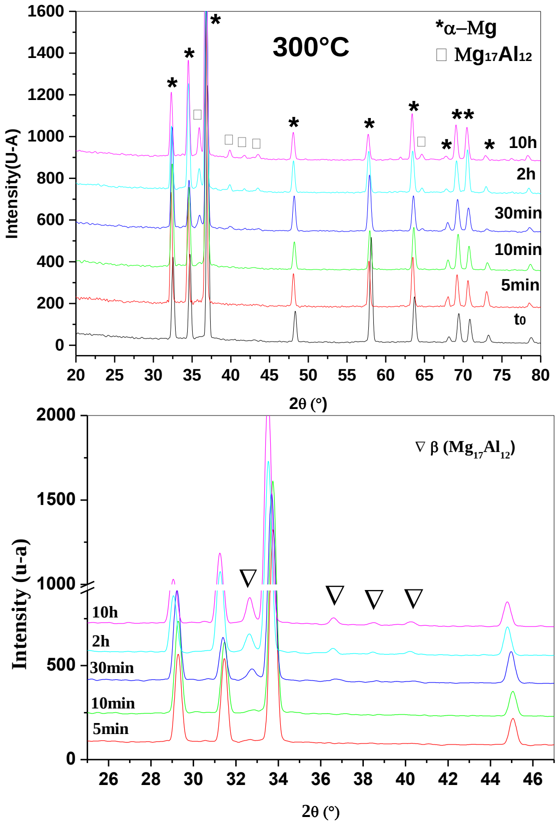

Figure 8. X- ray diffraction spectra of the AZ91 alloy homogenized, quenched and aged at 300 ° C.

3.3. Microhardness results

The age-hardening curves for AZ91 aged at different temperatures are shown in Fig. 9. The hardening curves consist of a typical response during the first aging times characterized by an increase in micro hardness values. No hardness peak was observed for AZ91 aged at 150 °C in 60 hours, although the hardness increases steadily with the aging time. This suggests that the age hardening at this temperature was relatively slow. A notable feature of these curing reactions as a function of aging time can be explained as follows:

1. High microhardness values are measured at the first ageing time and can be explained by the appearance and growth of continuous and discontinuous precipitated phases.
2. A relatively decrease in these values is observed after long ageing times from 200 to 300 °C. Since it is known that long ageing times lead to the coalescence of the formed phases so it causes a decrease in the measured microhardness values.
3. A plateau of microhardness values is observed during aging at 200 °C which can be associated with the braking of discontinuous precipitation by the appearance of continuous precipitation.

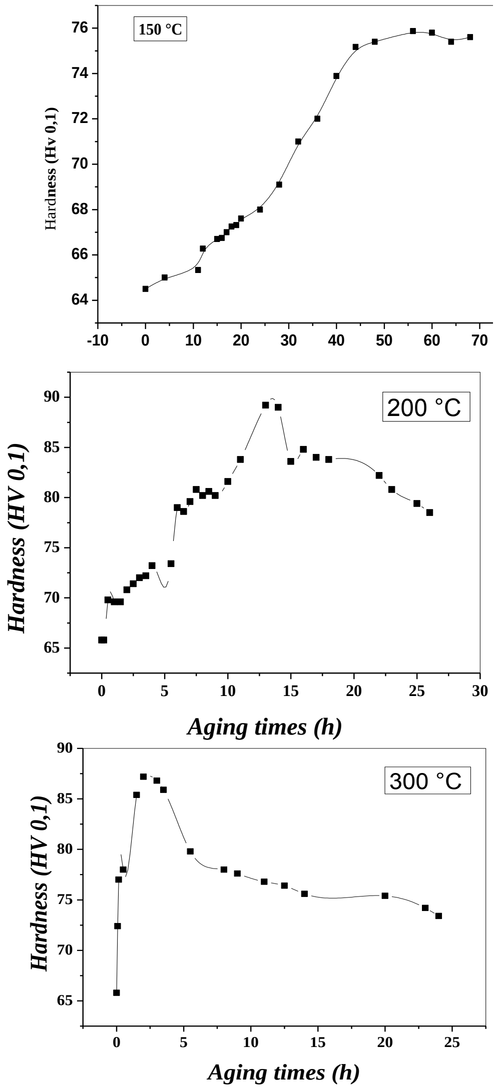

Figure 9. Variation of the microhardness as a function of the annealing time at 150, 200 and 300 ° C the AZ91 alloy.

4. Conclusion

The effect of temperature on precipitation mode in AZ91 alloy revealed that continuous precipitation develops at high temperatures (at 300 ° C), while discontinuous precipitation dominates at low temperatures (at 150 and 200 ° C).

The characterization by X-ray diffraction samples allowed to highlight the mode of reaction of the precipitation after the heat treatments applied.

The intensity of the most intense peak related to the formation of the precipitated phase β, increases with the aging time. This is explained by the increase in the volume fraction of the precipitated phase during isothermal aging. This result is in good agreement with the metallographic results.

Discontinuous precipitates are favored when the grain boundary diffusion process is dominant while continuous precipitates are formed from a quenched solution when bulk diffusion becomes faster

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