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Thermal analytical investigations of the magnesium alloy AZ91

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Summary

The results of thermal derivative analysis (TDA), differential scanning calorimetric (DSC) measurements and microstructure investigations of commercial AZ91 magnesium alloy are presented. The performed examinations allowed to determine the microstructure after solidification process and also precipitation process during continuous heating of supersaturated solid solution. The α-phase and α+γ semi-divorced eutectic were observed in as-cast material, whereas both discontinuous and continuous precipitates of γ phase were revealed after heating supersaturated AZ91 alloy.

Key words: Metallography, AZ91 alloy, TDA, DSC, Microstructure

1. Introduction

Magnesium alloys are light metallic structural materials with a unique combination of properties, which are very attractive in such applications as the automobile, aerospace and electronic industries. The use of magnesium alloys has become significant due to a one-third lower density of magnesium compared with aluminium, improved damping ability, a higher resistance to corrosion and better mechanical properties. Most commercial magnesium alloys are based on Mg-Al system comprising the AZ and AM alloy series. Phase equilibrium and thermodynamic quantities in this system provide crucible information for design and development of magnesium alloys; they also form the basis for the understanding of their behavior [1-5].

The most popular of these alloys is AZ91. The ternary magnesium alloy contains about 9wt% Al and 1wt% Zn, with an addition of about 0.4wt% Mn to improve corrosion resistance. The maximum solid solubility of aluminium in magnesium (α phase with hexagonal structure) is reasonably high at 12.9wt% Al at the eutectic temperature of 710 K. The equilibrium concentration at 473 K is about 2.9wt% Al so that a large amount of aluminium is available for γ precipitates. The γ phase has a stoichiometric composition of $Mg_{17}Al_{12}$ (at 41.4wt% Al) and an α-Mn cubic unit cell. Compared with binary Mg-Al alloys, in commercial ternary alloys type AZ91 no new phases occur, if the Al to Zn ratio is greater than 3:1. Zinc is presumably substituting for aluminium in the precipitate γ-phase [2-18].

Microstructure of the Mg-Al alloys can be formed during solidification process (like mould casting or high-pressure die casting) and hest treatment processes (homogenization, quenching and ageing). In Mg-Al alloys during solidification process fully or semi-divorced eutectic can occur [1, 4]. Sometimes in cast material the α+γ lamellar structure due to discontinuous precipitation process can be observed. During ageing process γ-phase out in two form, as discontinuous or continuous precipitates dependence on ageing temperature [2-23].

In this paper, the following thermal analyses were demonstrated:

- Thermal derivative analysis (TDA) – for the determination of solidification process, and
- Differential scanning calorimetric (DSC) measurements – for the determination of the precipitation process in solid state.

2. Experimental procedures

The commercial AZ91 magnesium alloy with a chemical composition of 8.5÷9.5wt% Al, 0.45÷0.9wt% Zn 0.17÷0.4wt% Mn was used in this study. The investigated AZ91 alloy was gravity cast into chill - about 40 mm diameter rods. TDA measurements were carried out with Crystaldigraph computer recorder (NiCr-NiAl thermocouple).

Samples of about 3 mm thickness and 4 mm diameter were cut off from the rods. They were subjected to the heat treatment process (Tx) at protective argon atmosphere including: solution annealing for 24 h at 693 K; water quenching (at approximately 278 K); and heating during DSC measurements up to 675 K at heating rate of 2 K/min. The measurements were carried out with NETZSCH 404 calorimeter, used ZrO_2 crucible and under a vacuum. (In preliminary investigations the influence of the samples on the crucible material was tested. The reaction between magnesium and ZrO_2 at the investigated temperature range were not observed).

Microstructure examination was carried out by means of a light microscope, (LM) Neophot-32 (Carl-Zeiss Jena) and scanning electron microscopes (SEM, Joel JSM-5400 type and Philips XL30ESEM FEG type) on polished and etched microsections.

3. Results

Figure 1 shows experimentally the TDA curves: solidification cure and their first derivative. According to equilibrium phases diagram, in AZ91 alloy after solidification process only α-phase should occur. Because of non-equilibrium solidification condition of described experiment in AZ91 alloy, the L⇔α+γ eutectic reaction was presented.

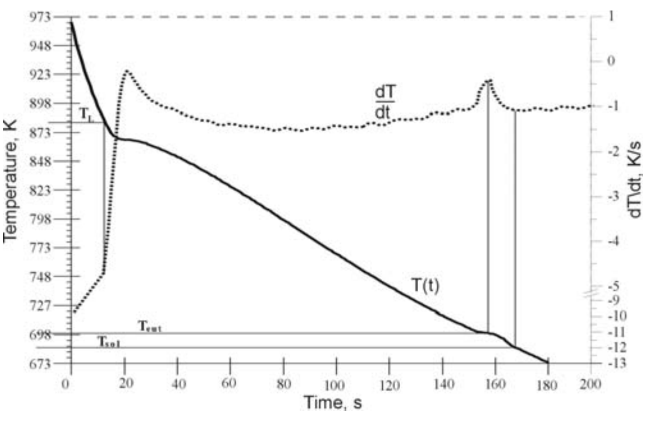

Fig. 1. TDA results: solidification curve T(t) and their first derivative dT/dt of the magnesium alloy AZ91

Non-equilibrium solidification conditions cause the formation of large crystals of the primary α-phase (depleted in alloying elements – especially in Al) and pushing the Al admixture away into interdendritical spaces. At the last stage of solidification the α+γ semi-divorced eutectic is formed. Figs. 2 and 3 present the microstructure obtained after described solidification process. The semi-divorced eutectic is clearly visible at Fig. 3.

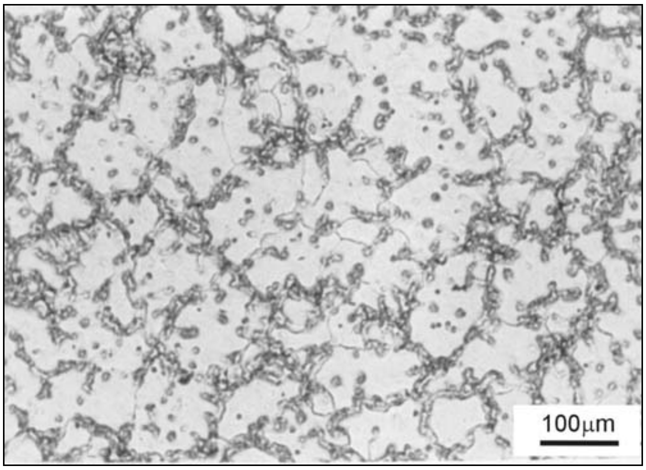

Fig. 2. Microstructure of as-cast magnesium alloy AZ91; light microscopy

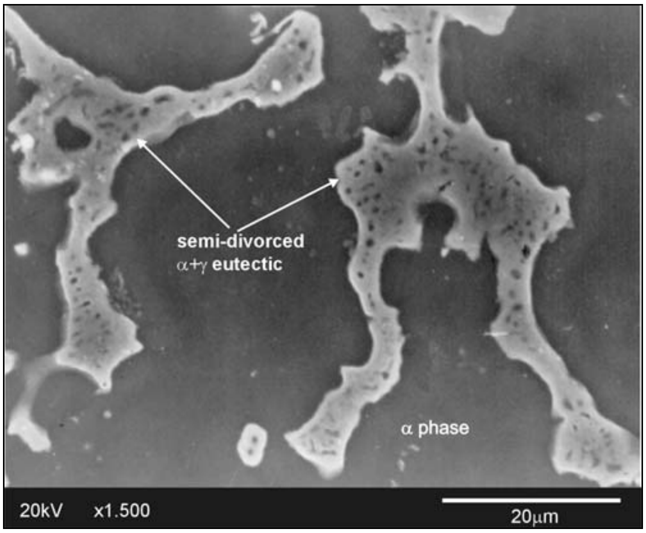

Fig. 3. Microstructure of as-cast magnesium alloy AZ91; SEM

Figure 4 shows experimentally the DSC signal curve obtained during heating supersaturated AZ91 alloy from room temperature up to 675 K at heating rate of 2 K/min. Large amount of peaks corresponding to phase changes are visible. Superposition of phase change peaks could testify that perpetual process of precipitation can occur. During heating process discontinuous or continuous precipitation leads to the formation of secondary γ phase from supersaturated solid solution.

Figs. 5-6 show images representing the variation in precipitation type after perpetual heating process of the supersaturated AZ91 alloy at heating rate 2 K/min. Both continuous and discontinuous precipitates are observed.

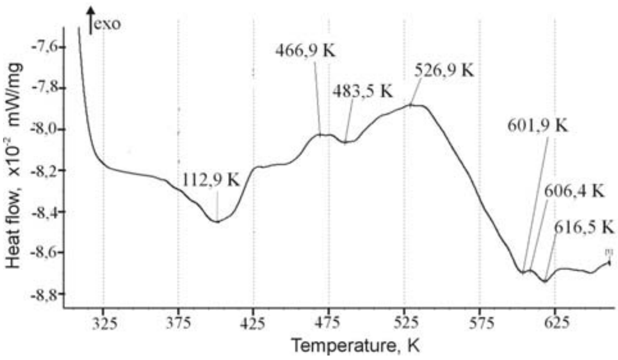

Fig. 4. DSC curve of the magnesium alloy AZ91 after heating supersaturated solid solution at heating rate 2 K/min.

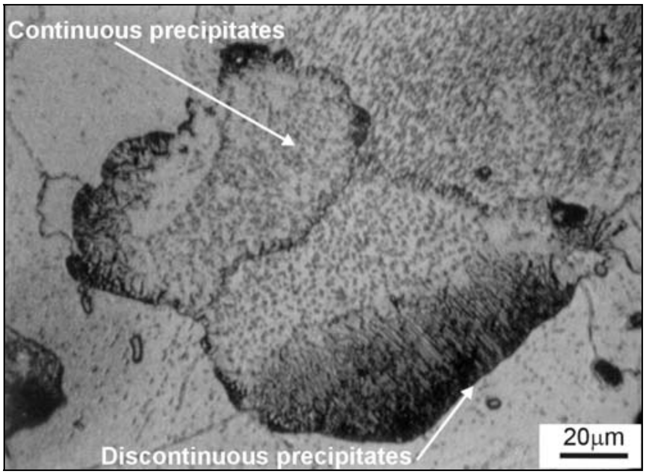

Fig. 5. Microstructure of the AZ91 magnesium alloy after heating supersaturated solid solution up to 675 K (heating rate 2 K/min.); light microscopy

The discontinuous precipitation is the cellular growth of alternating layers of γ phase and near-equilibrium matrix phase (α) at the high angle boundaries. This heterogeneous reaction leads to the formation of a lamellar structure behind a moving grain boundary. Continuous precipitation forms in all the remaining regions in the supersaturated matrix. In AZ91 alloy, however, these two types of precipitations can occur [2, 4-6].

It should be noted that in both cases of precipitates the γ-phase in AZ91 alloy has a plate-like morphology. Discontinuous precipitates usually have lamellar morphology because of process kind. In the investigated alloy continuous precipitates also have plate-like morphology with a visible crystallographic orientation compatibility to matrix grains, described in paper [2].

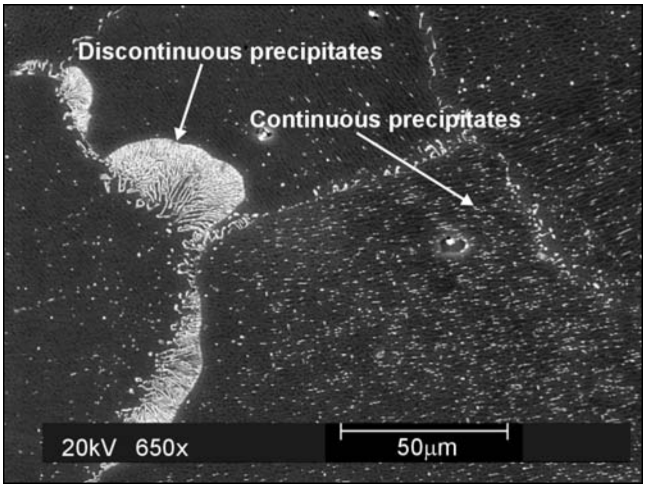

Fig. 6. Microstructure of the AZ91 magnesium alloy after heating supersaturated solid solution up to 675 K (heating rate 2 K/min.); SEM

4. Conclusions

(1) During non-equilibrium solidification process of the AZ91 magnesium alloy, the α-phase and α+γ semi-divorced eutectic are formed.
(2) Perpetual heating process of supersaturated solid solution cause the formation of secondary γ phase due to both continuous and discontinuous precipitation.
(3) Discontinuous precipitates have a characteristic lamella structure while continuous precipitates also have plate-like morphology.

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