Magnesium Technology 2012
Edited by: Suveen N. Mathaudhu, Wim H. Sillekens, Neale R. Neelameggham, and Norbert Hort
TMS (The Minerals, Metals & Materials Society), 2012

SOLIDIFICATION STUDIES OF Mg-Al BINARY ALLOYS

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Keywords: Mg-Al binary alloys, Secondary dendrite arm spacing, Cellular-columnar transition

1. Introduction

The knowledge of as cast microstructure of Mg alloys is of prime importance to design the downstream thermo mechanical processes like rolling and annealing which then follows with commercial applications of Mg alloys in various industrial sectors [1]. Specifically, the emerging technologies like Twin roll casting that require minimal thermo mechanical processing; it is the solidified microstructure at the casting stage which determines the mechanical properties of the final product. Hence, the as cast microstructural information of Mg alloys becomes imperative but there has been little or no work on the same. In this regard, the present study focuses on the influence of the solidification parameters like thermal gradient (G), solidification velocity (V), cooling rate and solute content on the microstructural features of Mg-Al alloys. Directional solidification experiments and gravity experiments have been performed to explore the relationship between secondary dendrite arm spacing (SDAS) of Mg-Al alloys and the solidification parameters. The transition between cellular-columnar growth modes for Mg-Al alloys has also been investigated in context to the solidification parameters.

2. Experimental

Directional solidification experiments have been performed using a modified bridgemann furnace which consists of an induction furnace mounted on top and a middle section which houses the Mg sample resting on water cooled Cu mould. The sample length was fixed at 200 mm and the solidification experiments were carried out in alumina crucibles of 5.5 and 4.5 mm I.D and 300 mm in length. The furnace temperature was set to 710 °C. The experiments are started by placing the sample in the induction furnace column and then the sample is pulled down with the desired solidification velocity for 100 mm and then quenched with He gas. The directional solidification experiments enable to control the solidification parameters like thermal gradient (G) and solidification velocity (V) and the product of these two parameters (G.V) gives the cooling rate. The cooling rates that could be achieved through these experiments for Mg-Al binary alloys were in the range of 0.05 to 2.75 K/sec and the associated G and V are listed in table 1.

Table1: The parameters for directional solidification experiments.

Thermal gradient (K/mm)	Solidification velocity (mm/sec)	Cooling rate (K/sec)
8.5	0.00625	0.05
7.5	0.05	0.38
7.0	0.17	1.16
5.5	0.50	2.75

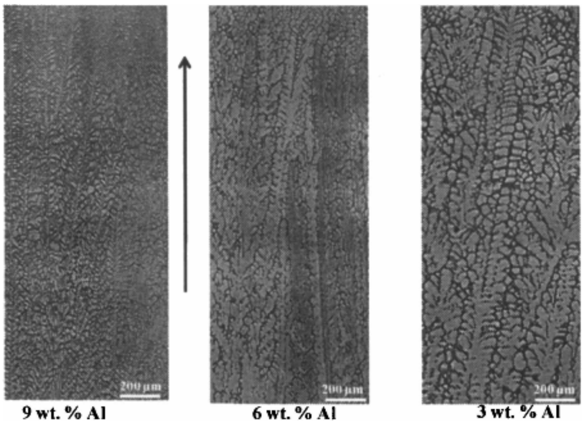
For intermediate and higher cooling rates, gravity casting experiments were performed. For example, casting in a wedge shaped Cu mould enables to attain different cooling rates along the varying cross section of the tapered mould. Three thermocouples were put at different positions from the tip of the wedge to attain cooling rates of 30, 75 and 110 K/sec. Casting experiments were also done in sand mould and graphite mould to obtain cooling rates of 1 and 20 K/sec respectively. Injection mould experiments were conducted in 4 and 6 mm Cu mould to obtain higher cooling rates but the exact measurement was not possible due to the limitations of data acquisition systems; however the cooling rates could be estimated from the Secondary dendrite arm spacing (SDAS) measurements which is discussed in next section.

3. Results and Discussion

3.1 Secondary dendrite arm spacing

Secondary dendrite arm spacing (SDAS) is an important length scale in microstructural solidification as it sets the diffusion distance in solid and liquid phase thus affecting the microsegregration and second phase formation in the solidified microstructure. SDAS investigation has been conducted for AZ91 alloy by many researchers [2-8] in the cooling rate range of 0.1 to 10^5 K/sec. A linear relationship (log-log scale) was proposed by Dube et al. [7] between SDAS and cooling rate for the cooling rate range mentioned above. However, the SDAS measurements from directional solidification experiments by Tensi and Rosch [3] and Pettersen et al. [2] for AZ91 alloy deviate from the proposed linear relationship in the cooling rate range of 0.1 to 1 K/sec. This deviation in SDAS data by directional solidification experiments cannot be ignored as the SDAS was very accurately determined. More over there is no study in literature which investigates the effect of solute content on the SDAS of Mg-Al alloys which could be of technical importance as the solute content is an important parameter for Mg as-cast alloy design.

The microstructural examination is done for directionally solidified Mg-Al alloys and for the gravity cast Mg-Al alloys. The optical micrographs for the directionally solidified Mg-Al (3, 6 and 9 wt. % Al) alloys at 0.375 K/sec is shown in Fig.1. The microstructure shows columnar growth for the alloys with the primary dendrite stem growing against the heat extraction direction. Substantial side branching is seen for all the alloys with microstructure becoming finer as the solute content increases from 3 and 6 to 9 wt. % Al. Secondary dendrite arm spacing (SDAS) is extracted from the optical microstructures using linear intercept method from all the solidified microstructure obtained through directional solidification and gravity casting experiments.



9 wt. % Al 6 wt. % Al 3 wt. % Al

Figure 1: The longitudinal sections of the directionally solidified Mg-Al alloys at the Thermal gradient (G):7.5 K/mm, Solidification velocity (V):0.015 mm/sec and Cooling rate: 0.38 K/sec. Arrow indicated the growth direction.

The variation of SDAS for Mg-Al binary alloys with cooling rate is shown in Fig 2. As seen from Fig. 2, the SDAS for all the Mg-Al binary alloys decreases with cooling rate but the relationship is not necessarily linear in the entire range of cooling rate. Particularly, the SDAS variation at lower cooling rates from 0.375 to 10 K/sec shows a different slope as compared to the SDAS in the higher cooling rate specifically greater than 20 K/sec. In this context, there cannot be a strict linear relationship between SDAS and cooling rate (log-log scale) for Mg-Al binary alloys as deduced for AZ91 alloy by Dube et al. [7].

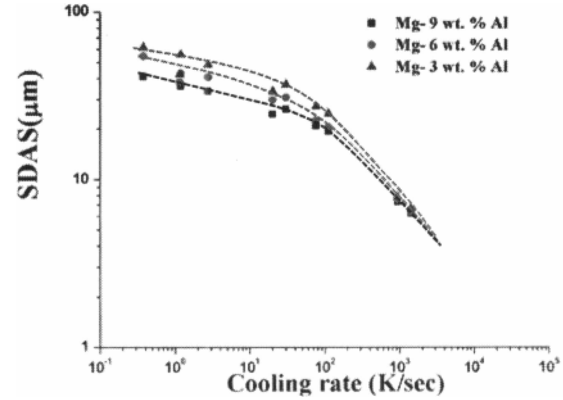


Figure 2: The variation of secondary dendrite arm spacing for Mg-Al alloy with cooling rate and Al concentration.

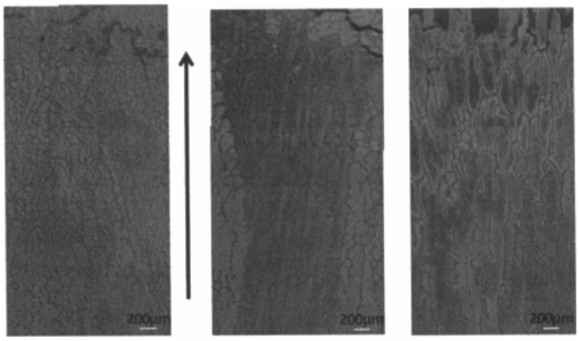
Interestingly, the SDAS becomes finer with increase in Al concentration and Mg-9 wt. % Al alloy has the lowest SDAS as compared to Mg-3 and 6 wt. % Al alloy at the same cooling rate. SDAS is primarily determined by the growth of the primary dendrite tip and the coarsening of the secondary branches which are already formed [9]. During columnar growth, higher solute content causes more undercooling at the growing dendrite tip which promotes dendrite tip splitting, as the solidification velocity or the cooling rate increases. Higher undercooling can also cause more perturbations to form along the sides of the main primary stem which then coarsen to from secondary branches. Since coarsening is diffusion driven process, higher solute content can also enhance the coarsening process. Thus, the combination of dendrite tip splitting and coarsening process can decrease the SDAS during columnar growth and both these processes are more effective for higher solute content. Even in equiaxed growth, SDAS is dictated by the coarsening of the secondary branches and the growth of secondary branches is similar to the growth of primary dendrite stem in columnar growth. Thus like the columnar growth, SDAS in the equiaxed growth is also expected to decrease as the solute content is increased. As stated earlier, the SDAS is dependent on the dendrite tip splitting of the primary dendrite stem and the coarsening of the secondary branches. One of these effects or the combination of both could be dominant in the lower cooling rate regime than the higher cooling rate which could explain the change in slope in Fig.2. In order to obtain the cooling rates for the injection mould casting experiments a linear relationship (log-log scale) was assumed between the SDAS and cooling rates measured for wedge casting experiments. This relationship gave the cooling rates of 933 and 1412 K/sec for the injection mould casting with 6 and 4mm diameter Cu mould respectively. More experiments are currently in progress for cooling rates greater than 100 K/sec for Mg-Al binary alloys to correctly ascertain the relationship between

SDAS and cooling rate, specifically in the higher cooling rate regime.

3.2 Cellular-Columnar transition

Columnar dendrite is the most common solidification form for many commercial alloys. Primary dendrite arm spacing (PDAS) is an important length scale that characterizes the columnar growth. PDAS formed in the solidified microstructure affects the mechanical properties and even the creep performance of cast Mg alloys is affected by PDAS [10]. Under specific conditions of solidification, alloys are expected to exhibit a transition from cellular to columnar growth which has been investigated for Al based alloys as functions of thermal gradient (G) and solidification velocity (V) [11]. However, the effect of solute content on the transition is not well studied. Specifically for Mg-Al binary alloys, there is no study undertaken in this regard.

As presented in table 1, directional solidification experiments were done at low cooling rate of 0.05 K/sec for Mg-Al (3, 6 and 9 wt. % Al) binary alloys and the optical microstructures are presented in Fig. 3. Mg-9 wt. % Al alloy shows columnar growth with secondary branches along the sides of the primary dendrite stem. Mg-6 wt. % Al alloy shows some side branching at the mushy zone near the quenched solid-liquid interface but the microstructure seems cellular far behind the quenched interface. Interestingly, Mg-3wt. % Al alloy looks entirely cellular at 0.05 K/sec with no side branching present at the growing primary dendrite stem.



9 wt. % Al 6 wt. % Al 3 wt. % Al

Figure 3: The longitudinal sections of the directionally solidified Mg-Al alloys at the Thermal gradient (G):8.5 K/mm, Solidification velocity (V):0.00625 mm/sec and Cooling rate: 0.05 K/sec. Arrow indicated the growth direction.

Primary dendrite arm or cell spacing could be measured from the microstructures for Mg-Al binary alloys for all the cases of cooling rate given in table 1 and a plot could be drawn showing the variation of PDAS with cooling rate for all the three alloys as shown in Fig. 4. It is noted that PDAS decreases for Mg-9 wt. % Al alloy with increase in cooling rate from 0.05 to 0.375 K/sec, however Mg-6 wt.% Al alloy shows a decrease in spacing at 0.05 K/sec when compared to 0.375 K/sec and the PDAS decreases again as the cooling rate is increased from 0.375 to 2.75 K/sec. Mg-3 wt. % Al alloy shows the similar trend as Mg-6 wt. % Al alloy but the spacing for Mg- 3 wt. % Al is lower when compared to Mg-6 wt.% Al alloys. This indicates that Mg-3wt. % Al will predominantly remain cellular when the cooling rate is less than 0.05 K/ sec; however at this cooling rate Mg-6 wt. % Al is still in the transition mode and will only adopt the cellular mode of growth at cooling rates lower than that required for Mg-3 wt. % Al. In this context, Mg-9 wt. % Al alloy will start showing cellular growth only at very low cooling rates when compared to Mg-3 and 6 wt. % Al alloys. This clearly emphasizes the influence of solute content on the cellular-dendrite transition in Mg-Al alloys.

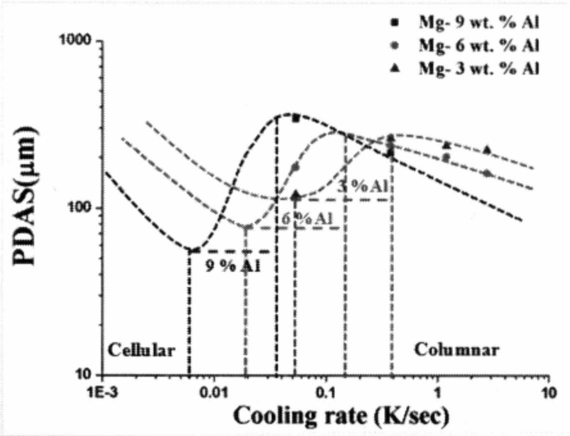


Figure 4: The variation of Primary dendrite arm spacing with cooling rate for Mg-Al binary alloys. The cellular-columnar transition is indicated for the alloys by the dashed lines.

The experimental trends discussed so far clearly emphasises the importance of undercooling at the dendrite tip on the various solidification growth modes. In the cellular mode, the increasing cooling rate decreases the undercooling at the tip of the growing cell which results in more cell flattening (increase in tip radius) and increasing the cooling rate further, splits the cell tip which marks the beginning of columnar growth with substantial side branching. Increasing the cooling rate beyond this point increases the undercooling due to more dendrite tip splitting (decrease in tip radius). Higher solute content will have more undercooling at the tip under both the cellular and columnar growth modes. Hence Mg-9 wt. % Al can achieve the transition to columnar mode at lower cooling rates than Mg-3 and 6 wt. % Al alloys.

4. Summary

Based on the directional solidification experiments and gravity casting experiments, following observation were made on the microstructural evolution of Mg-Al (3, 6 and 9 wt. % Al) alloys:

1. Secondary dendrite arm spacing (SDAS) decreases with cooling rate and increasing the Al content in Mg-Al binary alloys.
2. A linear relationship (log-log scale) cannot describe the SDAS variation in cooling rate as previously observed in case of AZ91 alloys and SDAS for the Mg-Al binary alloys showed that the deviation from linearity arises at lower cooling rates.
3. Cellular-columnar transition was studied for Mg-Al binary alloys with the aid of directional solidification experiments and it was observed that the transition is strongly dependent on Al concentration of the alloy.

All the as cast microstructural data of Mg-Al binary alloys are being used to test the solidification model in conjunction with the thermodynamic database.

5. Acknowledgement

Financial support from General Motors of Canada Ltd. and the CRD and MagNET research programs of the Natural Sciences and Engineering Research Council of Canada is gratefully acknowledged. We would also like to thank Dr. A. Javaid of CANMET for Mg alloy casting and Dr. D. H. Kang for thoughtful discussions.

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