

# Effect of cooling rate on the solidification behaviour of MC MgAl6Zn1 alloy

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## Manufacturing and processing

### ABSTRACT

**Purpose:** The goal of this paper is to present the thermal characteristics of magnesium alloy using the novel Universal Metallurgical Simulator and Analyzer Platform.

**Design/methodology/approach:** The objective of this work is determine the liquidus, solidus temperature and beginning nucleation temperature to understanding crystallization of magnesium alloys.

**Findings:** The research show that the thermal analysis carried out on UMSA Technology Platform is an efficient tool for collect and calculate thermal parameters. It was determined that the higher solidification rate decreases the solidus temperature. In addition, it was observed that the beginning of nucleation of  $\alpha(\text{Mg})$ - $\beta(\text{Mg-Mg}_{17}\text{Al}_{12})$  eutectic temperature constituent increases when the solidification rate increases.

**Research limitations/implications:** This paper presents results for one alloy – MC MgAl6Zn1 only, cooled with three different solidifications rate i.e. 0.6, 1.2 and 2.4°C/s, for assessment for the liquidus, solidus temperatures and describe a beginning of nucleation of  $\alpha(\text{Mg})$ - $\beta(\text{Mg-Mg}_{17}\text{Al}_{12})$  eutectic.

**Practical implications:** The parameters described can be applied in metal casting industry for selecting magnesium ingot preheating temperature for semi solid processing to achieve requirements properties or to predict how to prepare parameters to heat treatment.

**Originality/value:** The paper contributes to better understanding and recognition an influence of different solidification condition on non-equilibrium thermal parameters of magnesium alloys.

**Keywords:** Casting; MC MgAl6Zn1; Thermal analysis; UMSA

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## 1. Introduction

Magnesium is the lightest structural metal of all. Although magnesium does not occur in nature in a metallic form, magnesium compounds occur worldwide. In its pure form,

magnesium is soft, mechanically weak, and hence, not generally used for structural applications. By careful selection of alloying elements, alloys can be produced - both for general-purpose and for special applications. As the other metallic alloy systems, a combination of well-known hardening mechanisms (solid solution hardening, particle dispersion hardening, work hardening, and

grain boundary hardening) determines the mechanical properties of magnesium alloys. Alloying additions influence other properties including reactivity of the melt, castability, and corrosion performance.

Magnesium alloys were used for a wide variety of applications, including pyrotechnics and metallurgical, chemical, electrochemical, and structural applications. Magnesium is the 8th most abundant element on the earth making up approximately 1.93% by mass of the earth's crust and 0.13% by mass of the oceans [1-4].

The most popular magnesium alloys are alloys with aluminium. Aluminium is the most important alloying element for magnesium. Its maximum solid solubility in the Mg-Al system is 12.7 wt% at the eutectic temperature 437°C. The eutectic contains 32 wt% aluminium, and its composition is Mg<sub>17</sub>Al<sub>12</sub>. Commercial alloys contain less than 10 wt% aluminium and, according to the equilibrium diagram, should solidify into a homogeneous matrix of magnesium with aluminium in solid solution. Commercial Mg-Al alloys usually contain a small amount of zinc (0-2 wt%), which strengthens them somewhat. Higher zinc levels increase the quantity of eutectic constituents, lower the solidification temperature, and may cause hot shortness (cracking). In the absence of solution heat treatment, maximum ductility is obtained by using alloys of low aluminium and zinc content (e.g., AM60 alloy). Further improvement of ductility is possible with even smaller amounts of aluminium, but castability suffers somewhat. High-temperature applications require improved creep properties, which are achieved by decreasing the aluminium content. Further improvements are possible by introducing elements that form finely dispersed particles in the matrix. In high-pressure die-casting parts that solidify rapidly, silicon addition causes formation of finely dispersed Mg<sub>2</sub>Si particles; this is the basis of Mg-Al-Si alloys [5-9].

This article presents a new methodology to determine the thermal characteristics of magnesium alloy based on customized UMMA computer controlled rapid solidification experiments.

The paper should begin with the introduction in which the state-of-the-art of the issue concerning the paper will be presented generally and concisely. It is necessary to quote references taking into consideration the remarks included in the section "References". It is necessary to present the aim of works included in the paper and clearly emphasise the originality of solutions and content-related approach to the issue worked out and described by authors. Exemplary section headings and range of the subsequent sections of the paper are given roughly which we wish you to adopt during the preparation of your paper.

## 2. Experimental conditions

The investigations were carried out on test pieces of MCMgAl6Zn1 magnesium alloys in as-cast state. The chemical composition of the investigated materials is given in Table 1. Casting cycle of alloys was carried out in an induction crucible furnace using a protective salt bath Flux 12 equipped with two ceramic filters at the melting temperature of 750±10°C, suitable for the manufactured material. In order to maintain a metallurgical purity of the melting metal, refining with a neutral gas of the industrial name - Emgesalem Flux 12 was carried out. The

material was cast in dies with bentonite binder because of its excellent sorption properties.

Table 1.

Chemical composition of investigation alloy

The mass concentration of main elements, %								
Al	Zn	Mn	Si	Fe	Pb	Ce	Mg	Rest
5.62	0.46	0.16	0.034	0.07	0.034	0.01	93.6	0.008

The experiments were performed using a pre-machined cylindrical test sample with a diameter of  $\varnothing=18$  mm and length of  $l=20$  mm (Figure 1). Each sample had a predrilled hole to accommodate a supersensitive K type thermocouple positioned at the centre of the test sample to collect the thermal data and control the processing temperatures.

The thermal analysis during melting and solidification cycles was carried out using the Universal Metallurgical Simulator and Analyzer (UMMA) [10-12]. The melting and solidification experiments for the MC MgAl6Zn1 alloy were carried out using Argon as cover gas. The data for Thermal Analysis (TA) was collected using a high-speed National Instruments data acquisition system linked to a personal computer. Each TA trial was repeated three times.

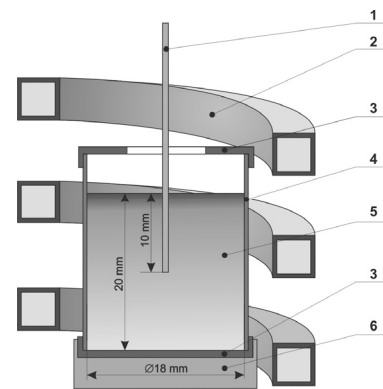


Fig. 1. Scheme of the UMMA Thermal Analysis Platform experimental set-up: 1 – low thermal mass thermocouple, 2 – heating and cooling coil, 3 – thermal insulation, 4 – steel foil, 5 – test sample, 6 – ceramic base

The TA signal in the form of heating and cooling curves was recorded during the melting and solidification cycles. The temperature vs. time and first derivative vs. temperature as well as fraction solid vs. temperature were calculated and plotted. Representative cooling and first derivative (cooling rate) vs. temperature curves is presented on Figs. 2 and 3.

The cooling rates for these experiments were determined using the following formula:

$$CR = \frac{T_{liq} - T_{sol}}{t_{sol} - t_{liq}} \left[ \frac{^{\circ}C}{s} \right] \quad (1)$$

where:  $T_{liq}$  and  $T_{sol}$  are the liquidus and solidus temperatures ( $^{\circ}C$ ), respectively, and  $t_{liq}$  and  $t_{sol}$  the times from the cooling curve that correspond to liquidus and solidus temperatures, respectively [13, 14].

Table 2.  
Characteristic points obtained from thermal-derivative analysis

Point	Temperature	Time	Description
I	$T_N$	$t_N$	Nucleation of $\alpha$ -phase (liquidus temperature)
II	$T_{Dmin}$	$t_{Dmin}$	The $\alpha$ -Mg dendrite minimum (undercooling) temperature
III	$T_{DKP}$	$t_{DKP}$	Coherency point
IV	$T_G$	$t_G$	The $\alpha$ -Mg dendrite growth temperature
V	$T_{(Mg+Si+Al+Mn)}$	$t_{(Mg+Si+Al+Mn)}$	Crystallization of $\alpha$ -Mg, $Mg_2Si$ and phases contains Al and Mn
VI	$T_{(Mg+Si+Al+Mn)f}$	$t_{(Mg+Si+Al+Mn)f}$	End of crystallization of $Mg_2Si$ and phases contains Al and Mn
VII	$T_{E(Mg+Al)N}$	$t_{E(Mg+Al)N}$	Beginning of nucleation of $\alpha(Mg)-\beta(Mg-Mg_{17}Al_{12})$ eutectic
VIII	$T_{E(Mg+Al)min}$	$t_{E(Mg+Mg_{17}Al_{12})min}$	The $\alpha(Mg)-\beta(Mg-Mg_{17}Al_{12})$ minimum (undercooling) temperature
IX	$T_{E(Mg+Al)G}$	$t_{E(Mg+Al)G}$	The $\alpha(Mg)-\beta(Mg-Mg_{17}Al_{12})$ eutectic growth temperature
X	$T_{sol}$	$t_{sol}$	End of solidification (solidus temperature)

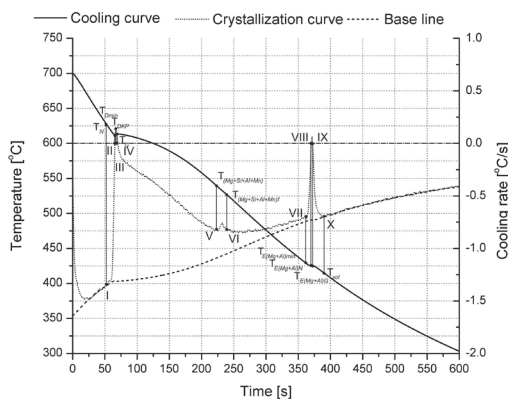


Fig. 2. Representative cooling and first derivative (cooling rate) vs. temperature curves recorded during the solidification cycles of MC MgAl6Zn1 alloy at a 0.6 °C/s solidification rate

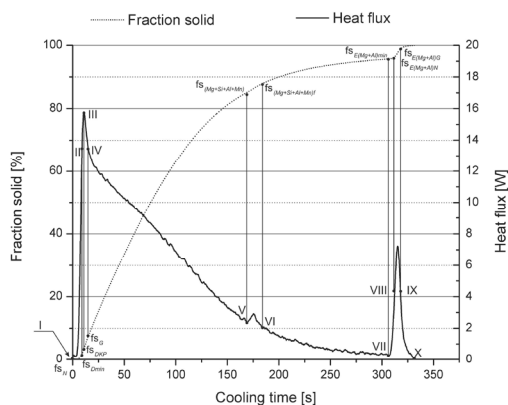


Fig. 3. Representative curves illustrate changes of heat flux and fraction solid of MC MgAl6Zn1 at a 0.6 °C/s solidification rate

The procedure comprised of the following steps. First, the test sample was heated to  $700\pm 2^\circ\text{C}$  and isothermally kept at this temperature for a period of 90 s in order to stabilize the melt conditions. Next, the test sample was solidified at cooling rate of approximately  $0.6^\circ\text{C/s}$  that was equivalent to the solidification process under natural cooling conditions, and  $2^\circ\text{C/s}$  average solidification rate. The Argon gas at 8bars pressure and at a flow rate up to 125LPM (Litres Per Minute) was used to cool the outer surface of the test sample to accelerate the solidification process.

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### 3. Results and discussions

Representative thermal analysis of the magnesium alloys were presented in Figure 2. Three visible temperature arrests were noted on the cooling curves. More detailed information pertaining to the alloy's thermal characteristics such as non-equilibrium liquidus, nucleation of the  $\alpha(Mg)-\beta(Mg_{17}Al_{12})$  eutectic, etc. were determined using the first derivative curves. The temperatures of the metallurgical reactions are presented in Table 3. Based on the cooling curve analysis, the non-equilibrium liquidus temperature of MC MgAl6Zn1 alloy that solidified under  $0.6^\circ\text{C/s}$  was found approximately  $618.9^\circ\text{C}$ . At this temperature the first magnesium dendrites, most likely, nucleated from the melt. Latent heat evolved and caused the temperature of the surrounding melt to rise. This point was clearly visible as a sudden change in the first

derivative curve. With further cooling, the magnesium dendrites continued to grow. The precipitate of phases contained aluminium and manganese and  $Mg_2Si$  was observed approximately at  $537.1^\circ C$  were participation of fraction solid at this point was approximately 85%. Process was finished at  $522.8^\circ C$ , where participation of fraction solid obtained 89.8%. At  $428.3^\circ C$  the next change in the first derivative curve was observed and corresponded to the nucleation of the  $\alpha(Mg)-\beta(Mg_{17}Al_{12})$  eutectic. It was found that non-equilibrium solidus temperature was approximately  $417.1^\circ C$ , where fraction solid obtained a 100%.

The non-equilibrium liquidus temperature of MC MgAl6Zn1 alloy that solidified under a  $1.2^\circ C/s$  was found approximately  $612.3^\circ C$ . The precipitate of the phases contained aluminium and manganese and  $Mg_2Si$  was observed at  $537.4^\circ C$  and finished at  $513.6$ . The nucleation of the  $\alpha(Mg)-\beta(Mg_{17}Al_{12})$  eutectic was observed at  $429^\circ C$ . The solidification sequence of the MC MgAl6Zn1 alloy finished when the solidus reaction was completed at  $412.7^\circ C$ .

Based on the first derivative of the cooling curve analysis, the liquidus temperature of the alloy solidified under  $2.4^\circ C/s$  was found at  $617^\circ C$ . At this temperature the first crystals of Mg nucleate from the liquid metal. A further decrease in the temperature resulted in nucleation of the intermetallic phases contained aluminium and manganese and  $Mg_2Si$  at  $537.1^\circ C$ . The nucleation of the  $\alpha(Mg)-\beta(Mg_{17}Al_{12})$  eutectic was observed at  $435.4^\circ C$ . The solidification process finished approximately  $405.8^\circ C$  when fraction solid obtained a 100%.

Figures 4-6 show the variation of the  $\alpha$ -magnesium ( $Mg_{Den}$ ) nucleation temperatures as a function of cooling rate and  $\alpha+\beta$  eutectic nucleation ( $T_{E(Mg+Al)N}$ ) temperature and solidus temperature ( $T_{Sol}$ ). Standard errors calculated for each measured

data point also were included in the graphs. It is seen that formation temperatures of the various phases are changed when the cooling rate is increased.

Changes of liquidus temperature are not observed during the examination, this temperature is constant for all cooling rates. The  $\alpha+\beta$  eutectic nucleation temperature increase with increase cooling rate from 0.6 to  $2.4^\circ C/s$ , the  $\alpha+\beta$  eutectic nucleation temperature increases from approximately  $424$  to  $435^\circ C$ . Due to increase of the cooling rate the solidus temperatures decrease. When the cooling rate is increased, the solidification range is increased from  $201^\circ C$  for the  $0.6^\circ C/s$  cooling rate to  $212^\circ C$  for  $2.4^\circ C/s$  cooling rate.

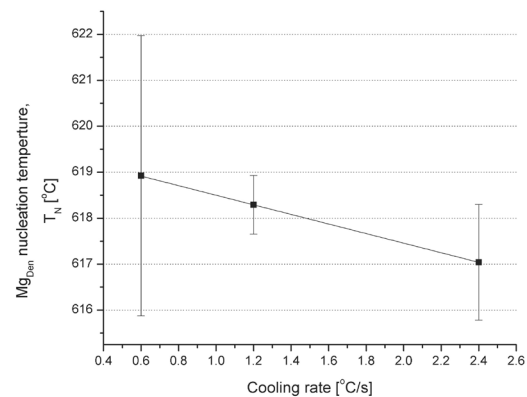


Fig. 4. Variation of the  $Mg_{Den}$  nucleation temperature as a function of cooling rate

Table 3.

Non-equilibrium thermal characteristics of the MCMgAl6Zn1 alloy test samples obtained during the solidification process at a 0.6, 1.2 and  $2.4^\circ C/s$  solidification rates

Points	Solidification rate, $^\circ C/s$					
	0.6		1.2		2.4	
	Temp. $^\circ C \pm$ STDV	FS, %	Temp. $^\circ C \pm$ STDV	FS, %	Temp. $^\circ C \pm$ STDV	FS, %
I	618.9±3.04	0	618.2±0.6	0	617±1.2	0
II	612.7±0.08	1.9	612.4±0.3	4.5		
III	613±0.08	2.8	612.3±0.1	6.4	605.2±0.6	8.9
IV	613.2±0.2	4.8	612.5±0.2	9		
V	537.1±0.65	85.1	537.4±1.6	85	537.1±0.5	83.4
VI	522.8±2.5	87.6	513.6±4.4	89.8	512.5±0.5	88.8
VII	428.3±0.47	95.4	429±1.2	95.7	435.4±1.9	93.1
VIII	424.7±0.67	96.6				
IX	424.9±0.63	97.9			Not observed	
X	417.1±1.3	100	412.7±3.3	100	405.8±2.4	100

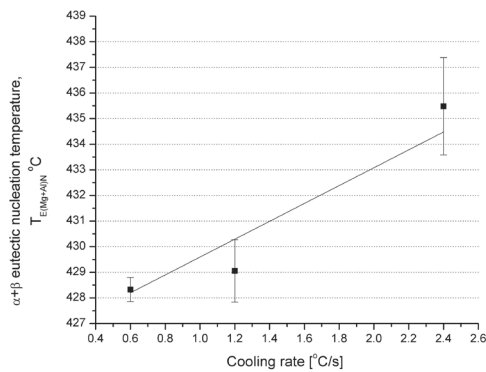


Fig. 5. Variation of the  $\alpha+\beta$  eutectic nucleation temperature as a function of cooling rate

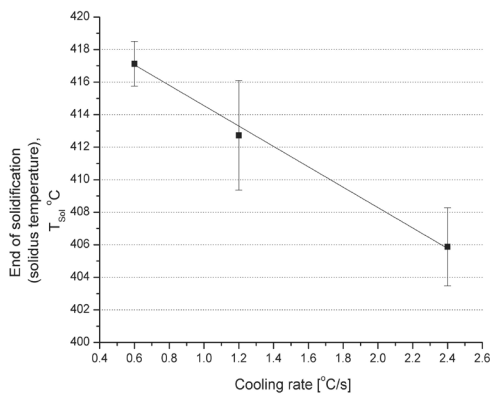


Fig. 6. Variation of the solidus temperature as a function of cooling rate

## 4. Conclusions

The research shows that the thermal analysis carried out on UMMA Technology Platform is an efficient tool to collect and calculate data about temperature and time of phase transformations, liquidus and solidus temperatures as well.

The results of the analyses can be summarized in the following conclusions:

- Solidification parameters are affected by the cooling rate. The formation temperatures of various thermal parameters are shifting when increasing a cooling rate.
- Increase of the cooling rate has not an influence on the beginning of  $\text{Mg}_{\text{Den}}$  nucleation temperature. Increasing the cooling rate increases significantly the  $\alpha+\beta$  eutectic nucleation temperature, solidification range and decreases the solidus temperature.

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## Additional information

The paper was published also in the Archives of Materials Science.

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