



Thermal analysis of the $MCMgAl9Zn1$ magnesium alloy

L.A. Dobrzański*, M. Król, T. Tański, R. Maniara

Division of Materials Processing Technology, Management and Computer Techniques in Materials Science, Institute of Engineering Materials and Biomaterials, Silesian University of Technology, ul. Konarskiego 18a, 44-100 Gliwice, Poland

* Corresponding author: E-mail address: leszek.dobrzanski@polsl.pl

Received 22.09.2008; published in revised form 01.12.2008

ABSTRACT

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

Design/methodology/approach: The experiments were performed using the novel Universal Metallurgical Simulator and Analyzer Platform. Material used in this experiment is experimental magnesium alloy made as-cast.

Findings: The research shows that the thermal analysis carried out on UMSA Technology Platform is an efficient tool for collecting and calculating thermal parameters. The formation temperatures of various thermal parameters are shifting with an increasing cooling rate.

Research limitations/implications: This paper presents results for one alloy – $MCMgAl9Zn1$ only cooled with two different solidification rates i.e. 0.6 and 2°C/s, for assessment of the liquidus, solidus temperatures and describing a beginning of nucleation of $\alpha(Mg)-\beta(Mg-Mg_{17}Al_{12})$ eutectic. Further investigations should be concentrating on assessing an influence of different solidification rate on microstructure and mechanical properties.

Practical implications: The parameters described can be applied in metal casting industry for selecting magnesium ingot preheating temperature for semi solid processing.

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

Keywords: Casting; $MCMgAl9Zn1$; Thermal analysis; UMSA

MATERIALS MANUFACTURING AND PROCESSING

1. Introduction

Most magnesium alloys show very good machinability and processability and even the most complicated die-cast parts can be easily produced. Cast, moulded, and forged parts made of magnesium alloys are also inert gas weldable and machinable. Another aspect is the good damping behavior, which makes the use of these alloys even more attractive for increasing the life cycle of machines and equipment or for the reduction of sonic emission. Pure magnesium shows even higher damping properties than cast-iron, although these properties are highly dependent on the prior heat treatment [1-3].

In order to effectively control microstructure development during the melting, solidification as well as further materials processing is necessary to understand all metallurgical phenomena taking place. Knowledge of the solidification process as well as the influence of liquid and/or semi solid metal treatment on micro and macro structure characteristics is of primary importance.

The simple and very effective method, which makes it possible to determine a curve of the crystallization process – the cool curve $T=F(t)$, is thermal analysis.

Advanced Thermal Analysis (TA) techniques monitor the temperature changes in sample as it cools through a phase transformation interval. The temperature changes in the materials are recorded as a function of the heating or cooling time in such a manner

that allows for the detection phase transformation. In order to increase accuracy, characteristic points on the cooling curve have been identified using the first derivative curve plotted versus time [4-5].

In the metal casting industry, an improvement of component quality depends mainly on better control over the production parameters. Thus, computer aided cooling curve thermal analysis of alloys is used extensively for the evaluation of several processing and material parameters. Thermal analysis of alloys can provide information about composition of the alloy, the latent heat of solidification, the evolution of fraction solid, the types of phases that solidify and even dendrite coherency. There are also many other uses for TA such as determining dendrite arm spacing, degree of modification and grain refining in aluminum alloys, the liquidus and solidus temperatures, characteristic temperatures related to the eutectic regions and intermetallic phase formation [6-9].

The goal of this paper is to present the new methodology to determine the thermal characteristics of magnesium alloy based on customized UMSA computer controlled rapid solidification experiments.

2. Experimental procedure

2.1. Material

The investigations have been carried out on MCMgAl9Zn1 experimental magnesium alloys in as-cast made in cooperation with the Faculty of Metallurgy and Materials Engineering of the Technical University of Ostrava and the CKD Motory plant, Hradec Kralove in the Czech Republic. The chemical composition of the investigated materials is given in Table 1. A casting cycle of alloys has been carried out in an induction crucible furnace using a protective salt bath Flux 12 equipped with two ceramic filters at the melting temperature of $750\pm 10^\circ\text{C}$, suitable for the manufactured material. In order to maintain a metallurgical purity of the melting metal, a refining with a neutral gas with the industrial name of Engesalem Flux 12 has been carried out. To improve the quality of a metal surface a protective layer Alkon M62 has been applied. The material has been cast in dies with betonite binder because of its excellent sorption properties and shaped into plates of $250\times 150\times 25$.

Table 1.
Average chemical composition (wt%) of the MCMgAl9Zn1 alloy

Al	Zn	Mn	Cu	Si	Fe
9.399	0.84	0.24	0.0018	0.035	0.007

2.2. Test sample

The experiments were performed using a pre-machined cylindrical test sample with a diameter of $\phi=18\text{mm}$ and length of $l=20\text{mm}$ taken from the ingot. In order to assure high repeatability and reproducibility of the thermal data, the test sample mass was 9.3g within a very closely controlled range of $\pm 0,1\text{g}$. Each sample had a predrilled hole to accommodate a supersensitive K type thermocouple (with extra low thermal time constants) positioned at the center of the test sample to collect the thermal data and control the processing temperatures.

2.3. Thermal analysis

The thermal analysis during melting and solidification cycles was carried out using the Universal Metallurgical Simulator and Analyzer (UMSA) [10]. The melting and solidification experiments for the AZ91 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.

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

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

where T_{liq} and T_{sol} are the liquidus and solidus temperatures ($^\circ\text{C}$), respectively, and t_{liq} and t_{sol} the times from the cooling curve that correspond to liquidus and solidus temperatures, respectively [11].

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°C/s , that was equivalent to the solidification process under natural cooling conditions, and a 2°C/s average solidification rate. The Argon gas at 8 bars pressure and at a flow rate of up to 125 LPM (Litres Per Minute) was used to cool the outer surface of the test sample to accelerate the solidification process.

Fraction solid (FS) was determined by calculating the cumulative surface area between the first derivative of the cooling curve and the so-called base line (BL). The BL represents the hypothetical first derivative of the cooling curve that does not exhibit phase transformation/metallurgical reactions during the solidification process. The area between the two derivative curves (calculated between the liquidus and solidus temperatures) is proportional to the latent heat of solidification of the given alloy. Therefore, the latent heat directly delivered to the test sample affected the fraction liquid evolution. Similar calculations were performed for the fraction solid except that fraction solid was proportional to the latent heat released during the solidification [12-15].

3. Results and discussions

Thermal analysis of the MCMgAl9Zn1 magnesium alloy revealed that the solidify process of material cooled at 0.6°C/s started at $599.2\pm 3.9^\circ\text{C}$ and was completed at $412.2\pm 1.6^\circ\text{C}$, as well for material cooled at 2°C/s solidify process started at $603\pm 3.6^\circ\text{C}$ and finished at $399.7\pm 2.7^\circ\text{C}$. Below those temperatures the alloy was at solid state.

Temperature vs. Time cooling curves recorded for the test samples heated to 700°C and solidified at 0.6°C/s and 2°C/s are presented in Figure 1. Two visible temperatures were observed on the cooling curves. More information about the liquidus and solidus

temperatures and nucleation of eutectic were characterized based on the first derivative curves. The representative first derivative vs. temperature curves are presented in Figure 2. The temperatures of metallurgical reactions are pointed out by numbers and the corresponding numerical values are presented in Table 2.

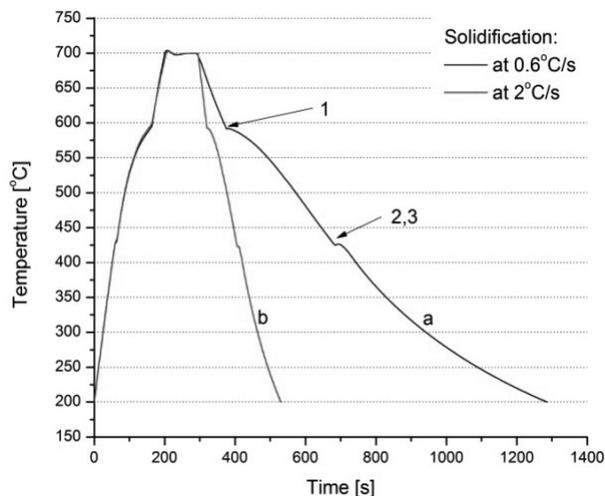


Fig. 1. Temperature vs. time curves of the MCMgAl9Zn1 alloy test samples recorded during melting and solidification at 0.6°C/s (line a) and 2°C/s (line b). The numbers correspond to the various metallurgical reactions as presented in Table 2

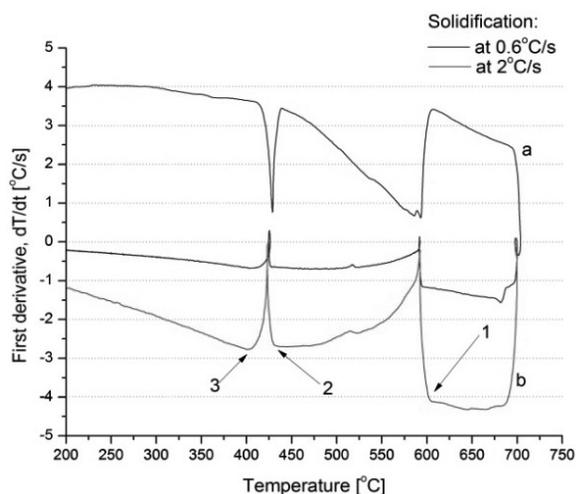


Fig. 2. First derivative (heating/cooling rate) vs. temperature curves of the test samples recorded during melting and solidification at 0.6°C/s (blue line) and 2°C/s (red line). The numbers correspond to the various metallurgical reactions as presented in Table 2

The liquidus temperature of the alloy solidified at 0.6°C/s was found at 599.2°C where the first crystallites nucleated from the melt (Figure 2, line a). That point was characterized based on sudden change in the first derivative curve (point 1 in Figure 2). Next

change on the first derivative curve was characterized at 428.2°C and corresponded to the nucleation of the $\alpha(\text{Mg})$ - $\beta(\text{Mg-Mg}_{17}\text{Al}_{12})$ eutectic (point 2 in Figure 2). The end of the solidification alloy, what is corresponded to the non-equilibrium solidus temperature was observed at 412.2°C (point 3, Figure 2).

The cooling curves for the MCMgAl9Zn1 alloy that solidified under a 2°C/s solidification rate are presented in Figure 1 and 2 (line b). The liquidus temperature was shifted up by approximately 4°C, i.e., to 603°C as compared with the material solidified at 0.6°C/s. The same observations were made for the nucleation of the $\alpha(\text{Mg})$ - $\beta(\text{Mg-Mg}_{17}\text{Al}_{12})$ eutectic that was shifted up by approximately 5°C, i.e., to 433.3°C as compared with the alloy solidified at 0.6°C/s. The end of the solidification alloy was observed at 399.7°C.

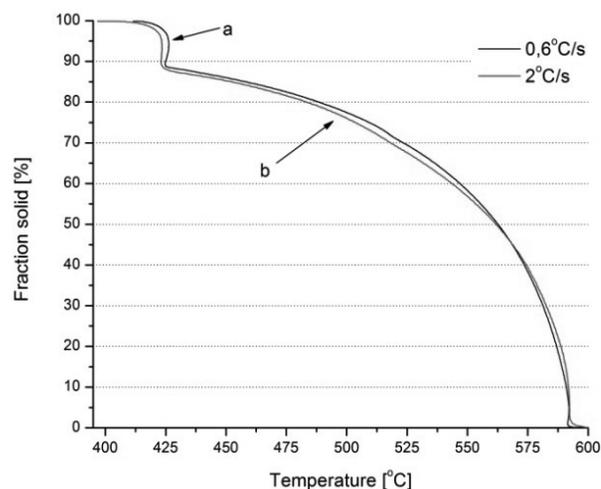


Fig. 3. Fraction solid vs. temperature curves of the test sample that solidified at 0.6°C/s (line a) and 2°C/s (line b)

Figure 3 presents a fraction solid vs. temperature curves of the test sample that solidified at 0.6°C/s and 2°C/s. It was observed for material solidified at 0.6°C/s cooling rate, that the beginning of nucleation temperature of the $\alpha(\text{Mg})$ - $\beta(\text{Mg-Mg}_{17}\text{Al}_{12})$ eutectic, started where material was at 88.5% solid state. Similar observations were made for material solidified at 2°C/s cooling rate, that the nucleation temperatures of the $\alpha(\text{Mg})$ - $\beta(\text{Mg-Mg}_{17}\text{Al}_{12})$ eutectic where alloy was at 85.2% solid state.

4. Conclusions

The subject of the research is conducted with the evaluation of the influence of the crystallization cooling rate on the phase crystallization temperature in MCMgAl9Zn1 alloy. The research show that the thermal analysis carried out on UMSA Technology Platform is an efficient tool for collect and calculates data about temperature and time of phase transformations, liquidus and solidus temperatures as well.

Solidification parameters are affected by the cooling rate. The formation temperatures of various thermal parameters are shifting with an increasing cooling rate.

Table 2.

Non-equilibrium thermal characteristics of the MCMgAl9Zn1 alloy test samples obtained during the solidification process at 0.6°C/s and 2°C/s solidification rates

Points	Thermal characteristics	Solidification rates [°C/s]			
		0.6		2	
		Temp. [°C]	Fraction solid [%]	Temp. [°C]	Fraction solid [%]
1	Nucleation of the $\alpha(\text{Mg})$ (Liquidus temperature)	599.2±3.9	0	603±3.6	0
2	Beginning of nucleation of $\alpha(\text{Mg})$ - $\beta(\text{Mg-Mg}_{17}\text{Al}_{12})$ eutectic	428.2±0.7	88.5	433.3±1.3	85.2
3	End of solidification process (Solidus temperature)	412.2±1.6	100	399.7±2.7	100

Acknowledgements

The authors would like to thank Dr. M. Kasprzak from the Silesian University of Technology in Poland for his valuable contributions to the UMSA Technology Platform's configuration. This scientific work is fragmentary financed within the framework of scientific financial resources in the period 2007-2008 as a research and development project R15 0702 headed by Prof. L.A. Dobrzański.

References

- [1] K.U. Kainer, *Magnesium – alloys and technologies*, Wiley-VCH Verlag GmbH & Co. KG aA, Weinheim, 2003.
- [2] L.A. Dobrzański, T. Tański, L. Cizek, J. Domagała, Mechanical properties and wear resistance of magnesium casting alloy, *Journal of Achievements in Materials and Manufacturing Engineering* 31/1 (2008) 83-90.
- [3] L.A. Dobrzański, T. Tański, J. Domagała, L. Cizek, Mechanical properties of magnesium alloys, *Journal of Achievements in Materials and Manufacturing Engineering* 24/2 (2007) 99-102.
- [4] S.G. Shabestari, M. Malekan, Thermal analysis study of the effect of the cooling rate on the microstructure and solidification parameters of 319 aluminum alloy, *Canadian Metallurgical Quarterly* 44/3 (2005) 305-312.
- [5] L. Backerud, G. Chai, J. Tamminen, *Solidification characteristics of aluminum alloys*, Vol. 2: Foundry Alloys, AFS Skanuminium, Stockholm, 1990.
- [6] L.A. Dobrzański, W. Kasprzak, M. Kasprzak, J.H. Sokolowski, A novel approach to the design and optimization of aluminum cast component heat treatment processes using advanced UMSA physical simulations, *Journal of Achievements in Materials and Manufacturing Engineering* 24/2 (2007) 139-142.
- [7] D. Emadi, L.V. Whiting, S. Nafisi, R. Ghomashchi, Applications of thermal analysis in quality control of solidification processes, *Journal of Thermal Analysis and Calorimetry* 81 (2005) 235-242.
- [8] L.A. Dobrzański, W. Kasprzak, J. Sokolowski, R. Maniara, M. Krupiński, Applications of the derivation analysis for assessment of the ACAISi7Cu alloy crystallization process cooled with different cooling rate, *Proceedings of the 13th Scientific International Conference "Achievements in Mechanical and Materials Engineering" AMME'2005, Gliwice–Wisła, 2005*, 147-150.
- [9] Method and Apparatus for Universal Metallurgical Simulation and Analysis – United States Patent, Patent No.: US 7,354,491 B2, Date of Patent: Apr. 8.
- [10] J.H. Sokolowski, M.B. Djurdjevic, Ch.A. Kierkus, D.O. Northwood, Improvement of 319 aluminum alloy casting durability by high temperature solution treatment, *Journal of Materials Processing Technology* 109 (2001) 174-180.
- [11] H. Yamagata, H. Kurita, M. Aniolek, W. Kasprzak, J.H. Sokolowski, Thermal and metallographic characteristics of the Al-20% Si high-pressure die casting alloy for monolithic cylinder blocks, *Journal of Materials Processing Technology* 199 (2008) 84-90.
- [12] W.T. Kierkus, J.H. Sokolowski, Recent Advances in CCA: A new method of determining baseline equation, *AFS Transactions* 66 (1999) 161-167.
- [13] M.B. Djurdjevic, W.T. Kierkus, G.E. Byczynski, T.J. Stockwell, J.H. Sokolowski, Modeling of fraction solid for 319 aluminum alloy, *AFS Transactions* 14 (1999) 173-179.
- [14] W. Kasprzak, J.H. Sokolowski, W. Sahoo, L.A. Dobrzański, Thermal and structural characteristics of the AZ50 magnesium alloy, *Journal of Achievements in Materials and Manufacturing Engineering* 29/2 (2008) 179-182.
- [15] H. Yamagata, W. Kasprzak, M. Aniolek, H. Kurita, J.H. Sokolowski, The effect of average cooling rates on the microstructure of the Al-20%Si high pressure die casting alloy used for monolithic cylinder blocks, *Journal of Materials Processing Technology* 203 (2008) 333-341.