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# In situ melting and solidification assessment of AZ91D granules by computer-aided thermal analysis during investment casting process

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#### ABSTRACT

Computer-aided thermal analysis was carried out to precisely measure the thermal characteristics of AZ91D magnesium alloy granules during in situ melting and solidification in investment casting process. Ceramic shell moulds of two different thicknesses equipped with highly sensitive thermocouples at three different locations were prepared to provide a range of heating and cooling rates during in situ melting and solidification. The results revealed that dissimilar thermal regimes were experienced by the granules at different locations of the mould during heating, which led to asynchronous melting of the granules. It was found that both melting commencement and completion were increased with increasing heating rate; in contrast inverse behaviour was observed during cooling. The onset and the end of solidification temperatures and duration decreased with increasing cooling and solidification rates. The information from this approach is very crucial for suppressing mould-metal reaction especially during cooling in casting of magnesium alloys by investment casting process.

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#### 1. Introduction

Due to their attractive and unique properties, magnesium alloys, are considered as the advanced materials and the most capable eco-materials to widen their application scopes, especially in the transportation industry [1], where weight reduction to increase fuel efficiency and energy saving are crucial [2]. The promising physical and mechanical properties of these alloys coupled with their high affinity with oxygen [3] and the problems which are encountered during their processing such as oxidation and ignition [4] necessitate further investigation on thermal properties and solidification of the alloys. This allows the development of new and advanced technologies to address such problems as well as to control the microstructure to approach premium components [5].

In situ melting is a new technique proposed by the authors for the manufacture of magnesium components by investment casting process [6] using minute sized of the magnesium alloys such as chips, turnings, and swarf. Due to the unique characteristics of this technique, the problems associated with the conventional investment castings can be resolved. Recycling of the small sized magnesium waste into the product is of the outstanding advantage of this technique as well. Therefore, melting and solidification behaviour of the materials using this technique are the key process parameters that affect the microstructure of cast alloy.

Although heating and cooling curves provide some information on melting and solidification of materials, respectively, they are unable to represent the required parameters evidently. Therefore, the corresponding derivative curves are mandatory to disclose small peaks related to little transformation heats and provide accurate analysis. Computer-aided thermal analysis (CATA), as a non-destructive, quantitative, fast and important supplementary technique [7], has received wide attention in materials science and metal casting industry to evaluate melt quality and control several processing parameters [8]. In this research, the characteristic temperatures of AZ91D as a well-known magnesium alloy [9] having granular shape were determined at different heating and cooling rate using CATA.

#### 2. Experimental procedures

Ceramic shell investment casting moulds with two different thicknesses of 6–7 mm and 3–4 mm with the aim of providing different heat transfer rates were prepared according to the conventional procedure using zircon-colloidal silica slurry and alumina–silicate sand as stucco. Commercial AZ91D alloy granules





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Fig. 1. The AZ91D granules used throughout the experiments.

 Table 1

 Chemical composition (wt.%) of AZ91D magnesium alloy granule.

Al	Zn	Mn	Si	Fe	Cu	Ni	Be	Mg
9.07	0.67	0.22	0.037	0.0021	0.0043	0.0006	0.0010	Rem.

with the size of  $4-5 \times 1.5-2 \times 0.8-1.2$  mm (Fig. 1) and chemical composition given in Table 1 were used for the investigation. The granules together with 2.5% self-made flux comprised of MgCl<sub>2</sub>, KCl and CaF<sub>2</sub> were charged into the moulds fabricated with three calibrated high sensitivity K-type (nickel-chromium) thermocouples located at the centre of different positions, namely bottom, middle and top of the mould cavity, as schematically shown in Fig. 2.

The moulds were heated in argon protected atmosphere in an electrical resistance gastight muffle heating furnace at  $700 \pm 3$  °C for 30 min followed by cooling to room temperature in still air. During heating and cooling, temperature–time data were recorded and plotted using a high-speed data acquisition system (EPAD-TH8-K), at a dynamic rate of 100 Hz/ch, linked to a computer with DEWESoft7 software. FlexPro data analysis software was utilised to plot and smoothen the corresponding first derivative heating and cooling curves. The curves were used to evaluate melting and solidification behaviour and to establish any characteristic temperatures, as schematically shown in Fig. 3. The experiments were repeated at least twice to achieve reproducible and reliable results.



Fig. 2. Schematic illustration of the set-up system.



**Fig. 3.** Representative first derivative heating and cooling curves indicating the position of characteristic points.



Fig. 4. Schematic picture showing the locations of cut samples.

Metallographic samples were cut from the vicinity of thermocouples tip (Fig. 4) to evaluate microstructure of the cast alloy. The samples were mounted, ground using SiC paper up to final 2400 grit, polished with colloidal silica suspension and then etched using 5–6 gr picric acid + 5 gr acetic acid + 100 ml ethyl alcohol + 10 ml distilled water solution for 15 s in order to reveal the microstructure of the samples. Optical and field emission scanning electron microscopy (FESEM), energy dispersive X-ray spectroscopy (EDS) and X-ray diffraction (XRD) analyses were carried out to evaluate the microstructure of in situ melted cast alloy.

#### 3. Results and discussion

#### 3.1. Thermal analysis of the granules during heating

Fig. 5a–d represents the heating and the corresponding first derivative heating curves of the granules located at the different positions of the thick mould after melting, respectively. In general, any sudden peak in the first derivative heating curve is considered as the characteristic temperature. Thermal characteristics of the heated AZ91D granules extracted from the first derivative heating curve are summarised in Table 2. The non-equilibrium melting of the eutectic phase,  $\alpha$ -(Mg) +  $\beta$ -(Mg<sub>17</sub>Al<sub>12</sub>), started at  $T_{eh}$ . Elevating the heating temperature led to a gradual increment of liquid and

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