Effect of Flux on In-Situ Melting Shell Investment Casting of AZ91D Magnesium Alloy


Abstract — In this research, the effect of flux on investment casting of AZ91D magnesium alloy using in-situ melting technique was investigated. AZ91D granules together with different fluxes were charged into shell investment moulds and heated at 750 °C in argon protected environment in order to produce AZ91D investment casting. Surface of castings and the occurred mould-metal reaction was analysed. It was found that flux usage in conjunction with the granite is essential to produce a solid investment cast alloy. The results showed that usage of flux mixed with the granules as well as covering the granite on top with a layer of it produced the higher surface quality casting. Microscopic examinations revealed that flux developed a barrier layer which reduced mould-metal reaction and contributed to an improved approach in magnesium alloys investment casting process.

Keywords — AZ91D; Granules; Flux; Investment casting; In-situ melting; Mould-metal reaction

I. INTRODUCTION

Magnesium cast alloys, predominantly AZ91D which is a high-purity alloy with attractive mechanical and physical properties and excellent castability, has found wide applications in automotive and aerospace industries [1]. The current economic production demands as well as the needs for high precision components, encourage researchers to investigate on new ideas to find out suitable alternative processes. Investment casting process is usually used for production of high quality intricate shapes as well as thin-wall components requiring good surface finish and high degree of dimensional tolerance [2]. In other words, this method can be considered as one of the practical solutions for near net shaping of magnesium alloys [3]. However, due to high affinity of magnesium for oxygen, magnesium investment casters are faced with the challenge to eliminate mould-metal reactions during casting of the alloy. It is expected that resolving the problem will change the perspective of the foundry industry and encourage the aluminum investment foundries to incline to magnesium investment casting due to increasing demand for magnesium components. Many approaches have been of research concern to suppress or control mould-metal reaction such as using vacuum assisted [2], [4], different shell materials [5], [6] or controlling the process parameters [7], using protective gases and inhibitors [7], [8] during investment casting. However, commercially feasible techniques for investment casting of magnesium alloys and producing good surface finish are limited. As a result, the investment casting industry still needs reliable methods and techniques for producing higher quality products. Therefore, many researchers are still studying on this area because of inadequate established requirements. Shell investment casting process using in-situ melting technique is a new approach in order to produce magnesium alloy investment castings with good surface finish. One of the effective parameters affecting the cast alloy is flux usage during the process. Although melting of magnesium using flux is an aged method; but yet it is one of the common techniques amongst magnesium foundries [9]. Chloride compounds mainly MgCl2 and KCl together with a small amount of fluorides such as MgF2 and NaF are the chemical components of the flux formulations for the melting and casting of magnesium alloys [10], [11]. It is believed that employing flux decreases the surface tension [12], [13], as the preventive factor for gaining high degree of surface integrity in casting. It is reported that an increase in the amount of MgCl2 in MgCl2-KCl based fluxes together with CaF2 cause a decrease in magnesium melt interfacial tension which is an advantages for the melting process [14], [15]. This study aimed at investigating on the effect of different flux percentages and applications used with AZ91D granules to melt the granules and overcome mould-metal reaction during investment casting process using in-situ melting technique.

II. EXPERIMENTAL PROCEDURES

Ceramic moulds, shown in Fig. 1, with a total of five stuccoed layers having thickness of 7-8 mm were prepared...
according to the standard procedure for preparation of shell investment casting mould. The corresponding wax patterns made of low expansion wax (Maymar, Ltd, UK) were dipped into a slurry composed of zircon flour (ZrO2,SiO2) and colloidal silica (SiO2) for 30 s to develop a face coat on them. Viscosity of the slurry was set at 20-22 s using Zohn cup No. 5. Fine alumino-silicate sand (Al2O3,SiO2) was used for stuccoing. The prepared moulds were dewaxed at 200 °C for 30 min and fired at 800 °C for 60 min. Commercial granules of AZ91D magnesium alloy sized 4-5 in length, 1.5-2 mm in width and 0.8-1.2 mm in thickness with the main chemical composition of Mg-9Al-0.7Zn-0.2Mn (wt.%) were used as the melting material. The granules were charged into the prepared moulds and heated at 750 °C for 30 and 60 min in an electrical resistance heating furnace (Linn, VMK 135 S) under argon protected atmosphere. A flux composed of high purity chemical materials of MgCl2, KCl and CaF2 was used as the melting material. The granules were melted successfully during heating at 750 °C in argon protected atmosphere. Figs. 3(a-c) clearly show that no oxide layer formed on the granules due to utilizing argon atmosphere does not solely fulfill melting of the granules and other contrivance is required to contribute melting of the granules completely and producing appropriate casting.

Moreover, it is necessary to reduce surface tension and improve wettability to approach increased direct contact area of molten drops and create a unit pool of molten metal. It is worth noting that decreasing surface tension makes the alloy more able to replicate the mould shape due to effective contact to mould surface [21]. Figs. 2(a) and (b) also represent a large area of the product covered by adhesion of white patches of the investment showing severe mould-metal reaction taken place. Fig. 2(c) shows backscattered SEM (BSC) image of the granules at the surface of the casting after heating for 30 min. It is clear that the granules are completely separated without joining together and a space emerged between them. In fact, owing to the high surface tension originating from the poor wettability between the granules on the surface and the contraction of the metal during cooling, the granules separated from each other. Moreover, the highlighted area discloses that no oxide layer formed on the granules due to utilizing argon atmosphere during heating. As a result, although high temperature heating and free-oxygen atmosphere were used for in-situ melting of the granules, high surface/volume ratio together with the high surface tension of the granules prevented producing a complete solid product.

Four different cooling conditions including argon protected atmosphere (furnace cooling), closed environment and still air using normal and low permeable shell investment mould were experimented to investigate their effect on surface quality of the casting. The surface of the in-situ melted AZ91D investment castings was examined in order to explore the effect of flux on the process. Field emission scanning electron microscope (FESEM) equipped with energy dispersive X-ray spectroscopy (EDS) and X-ray diffraction (XRD) analyses were utilized for the research.

### III. RESULTS AND DISCUSSION

The authors proved and reported in their previous paper [16], [17] that the granules were melted successfully during heating in air. However, formation of magnesium oxide on the surface of the granules and its thickening during heating process encapsulated the molten metal inside an oxide casing and inhibited them from joining together to form a pool of molten metal. Consequently, a protective heating atmosphere is essential in order to inhibit oxide formation on the melted granules and let them join and produce a casting.

Figs. 2(a) and (b) display typical products of heating the granules in argon protected atmosphere at 750 °C for 30 min, it is clearly seen that the melting process is partially happened and even extended heating did not bring about any accepted product. In other words, a significant percentage of the granules, especially at the top part of the product, left unmelted. This problem can be attributed to high surface tension of the molten metal at mould-metal interface [18]. Therefore, due to low wettability [19], the small molten drops did not wet each other well. This indicates that surface or interfacial tension as a thermophysical property of molten metal plays a key crucial role on wetting small liquids over each [20]. As a result, it can be inferred that using controlled atmosphere does not solely fulfill melting of the granules and other contrivance is required to contribute melting of the granules completely and producing appropriate casting.

Fig. 3 demonstrates typical products of using 0.5, 1 and 1.5% flux on the top of the granules during heating at 750 °C in argon protected atmosphere. Figs. 3(a-c) clearly show that using insufficient flux did not prevent the granules on the top
of the moulds from suffering severe oxidation.

Fig. 2 Typical products of in-situ melted AZ91D granules after heating under argon protected atmosphere at 750 °C for (a) 30 min and (b) 60 min; (c) BSC image of the granules after heating

In contrast with the adequate amount of covering flux (1.5 wt%), no considerable oxidation and ignition were observed on the top of the melted granules. In other words, the molten metal was completely calm; thus no precautions are needed when the melted alloy cools in the air due to the formation of a barrier flux-magnesium product on the surface of molten metal. It is reported that this barrier layer can be MgCl₂·MgO which forms on the surface and protects the magnesium from further oxidation [22]. Figs. 3(d-f) indicate that the granules melted and produced solid products. However, a remarkable area of the castings surface still shows adherence of investment, and no significant progress was achieved in this issue. In other words, just the upper part of the casting is free of investment. It can be inferred that packing the granules together during heating and prevented the molten flux to go down through granules and provide any barrier to suppress the reaction between metal and mould.

Fig. 4 represents the cross-section BSE image of the interface of the alloy-adhered investment. It can be observed that a considerable amount of the alloy penetrated into the shell mould and formed a metal layer inside the shell. This can be occurred through the cracks formed in the investment. The diffusion of oxygen and magnesium vapour towards each other through the pores of the ceramic shell mould and their reaction inside the mould can be the reason to nucleate the cracks. Afterwards, due to low cooling rate, the molten alloy found the opportunity to flow into the created space and formed a metal layer inside the shell.

Fig. 3 Typical products of the granules heated at 750 °C under argon atmosphere and covered with (a, d) 0.5%, (b, e) 1% and (c, f) 1.5% flux.

Fig. 4 BSE cross-sectional image of the investment-casting interface

Figs. 5(a) and (b) represent two typical castings produced with application of 0.5% and 1% flux, respectively, at the bottom of the moulds together with 1.5% on the top of the granules. Although employing the flux at the bottom of the mould caused moderate improvements with disappearance of the investment adherence on the lower part of the castings, it still remained on the middle parts of castings surface. It is also seen that increasing the flux quantity from 0.5 to 1% at the bottom of the mould resulted in a slight progress in the process. Fig. 5(c) shows the product of granules charged into the flux-rubbed mould after heating. The investment adhesion was no longer observed on the surface; however, significant amount of the reaction products were emerged on the surface.

Fig. 6(a) demonstrates the top view of the black spot-shaped residue. It is seen that a granular morphology formed on the surface of casting. This may be inferred that the lack of good wettability between drops of molten magnesium alloy in the interface between molten metal and shell investment mould together with reaction between molten metal and mould or oxygen during cooling can be the reasons of such morphology.
The result of EDS analysis given in Fig. 6(c) confirms presence of MgO and carbon on the surface. It is worth noting that presence of carbon as a thin film on the surface of the magnesium oxide which is reported by other researchers as well [23], [24] is the reason for the black colour of the residues. The peaks Al, Zn and Si are ascribed to the existence of Mg_{17}(Al,Zn)_{12} and Mg_{2}Si, respectively, in the black residue. The former formed during melting of the granules and the latter produced due to reaction of molten metal with SiO_{2} of the mould material. Fig. 6(b) represents BSC cross-section of the black residue. The microstructure of the residue reveals that it is AZ91D.

Further attempts to suppress the mould-metal reaction and approach acceptable surface quality of castings were accompanied with just mixing the granules with 0.5, 1, 1.5 and 2% flux during charging the moulds. Fig. 7 demonstrates the picture of the castings produced after heating at 750 °C for 30 min. It can be observed that mixing 0.5 wt% flux with the granules is insufficient to produce enough wettability to join the melted granules together and produce a solid casting, while higher percentages showed inverse behaviour. It is interestingly clear that no more investment adhesion and black spots which are the products of mould-metal reaction are observed. In other words, using flux suppressed the mould-metal reaction and contributed to approach a possible remedy for overcoming magnesium alloys investment casting problem. However, it is seen that other product which are white in appearance emerged on the surface of the castings. They were intensified when the flux percentage was increased. Moreover, no cracking was observed in the mould during cooling outside the furnace. Indeed, presence of the flux can be considered as the only reason for the result. Fig. 8 shows typical casting produced by in-situ melting shell investment casting process. It is seen that a complete casting with less unmelted granules on the top was produced.
Fig. 8 Typical casting produced using 1% and 1.5% flux mixed with and on the top of the granules, respectively.

Figs. 9(b) and (c) show the EDS analysis results corresponding to two points of the layer denoted as point A, near the layer surface, and point B, close to the substrate, respectively. The spectra illustrate that the layer dominates in MgO at near the surface of casting, whereas it contains a considerable amount of chloride as well as MgO in the outer part. Presence of chlorine element in the spectrum indicates that the flux combined with MgO [25], [26] and caused magnesium to be prevented from severe reaction with oxygen. Existence of Al suggests that MgAl$_2$O$_4$ as the product of severe oxidation [24], [27], [28] in the inner part and its absence in the outer part of the layer confirm this claim.

Fig. 9 (a) BSC cross-sectional image of the white residue, (b) and (c) EDS spectra of the points indicated as A and B on the residue.

Fig. 10 displays XRD analysis pattern corresponds to the white residue. Detection of MgF$_2$ originated from the reaction of MgO with the fluoride compound of the flux [29] indicates suppression of mould-metal reaction during the process due to higher stability compared to MgO [30]. Potassium bearing compound can contribute to inhibit oxidation process due to consumption of oxygen and reacting with magnesium to form KClO$_2$ and K$_6$MgO$_4$. The absence of Mg$_2$Si in the XRD pattern reveals that the mould-metal reaction was confined to some extent by application of the flux.

Comparing the products of in-situ melting shell investment casting of AZ91D and the obtained results can be concluded that although mould-metal reaction lessened by utilizing of flux, however, high temperature of the process allow magnesium to evaporate and react with oxygen to form MgO.

IV. CONCLUSION

In this research, the effects of different amount and application conditions of fluxes on in-situ melted investment casting of AZ91D alloy were investigated. The following conclusions can be drawn:

1) In addition to application of protective atmosphere, melting flux is also required to charge with the granules in order to overcome the high surface tension between the granules and produce a pool of molten metal.

2) Mixing 1% flux with the granules suppressed mould-metal reaction and approached better surface finish for in-situ melted AZ91D investment cast alloy. In addition, usage of 1.5% flux on the top of the granules provided sufficient prevention from oxidation during cooling.

3) No detection of Mg$_2$Si and presence of MgF$_2$ on the surface of cast alloy be can be considered as the effective performance of flux in inhibition of magnesium-shell reaction during in-situ melting shell investment casting process.

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