



# Investigation on Reactions at Corners of Cast Part during Investment Casting of Reactive AZ91 Magnesium Alloy

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## Abstract

The magnesium alloy investment castings have greater potential for automobile and air-craft applications due to the higher strength to weight ratio of magnesium alloys and capability of the investment casting process to produce near net shape complex castings. The interfacial-mould metal reactions during investment casting of magnesium alloy inhibit successful production of quality castings. This paper presents the investigation done on the reactions at corners of AZ91 magnesium alloy cast part produced through investment casting. The stepped shape geometry of casting was selected to study the reactions at convex and concave corners of the cast part. The reacted surfaces were characterised using the SEM-EDX and XRD. The formation of oxides was observed on cast surface from characterisation. The temperature profile recorded at corners were helpful to understand the heat dissipation during the solidification of metal at corners. It was observed that the reactions occurred at the concave corner were more as compared to the convex corner of the cast part.

**Keywords:** Magnesium, Investment Casting, Corner, Reactions, Mg AZ91

## 1. Introduction

The research and development are growing rapidly in investment casting of the magnesium alloys for its present demand in automobile, aircraft, biomedical and defence sectors owing to excellent light-weight property [1-3]. The difficulties are reported by many researchers for the successful production of magnesium alloy casting with investment casting route. The interfacial reactions between ceramic mould and reactive magnesium melt are observed as the main barrier [4]. Alternative face-coat mould materials were assessed to prevent these reactions at the interface. The application of various face-coat oxides  $Al_2O_3$ ,  $Y_2O_3$ ,  $MgO$  etc. were observed as significant for suppressing the reactions to some extent [5-9]. The effectiveness of various protective gases to inhibit reactions and oxidation of

Mg AZ91 melt were studied by many researchers. The application of  $SF_6 + CO_2$  gas mixture was reported promising for inhibiting reactions [10-14]. In-situ melting and solidification technique was also observed to suppress the reactions to some extent, where ceramic moulds charged with Mg alloy granules and flux, heated in resistance heating furnace for 30 minutes under the presence of inert argon gas and allowed to solidify [15].

In the literature, limited work is reported on the effect of cast part geometry on reactions. The reactions were observed to be increased with increase in the cast part thickness due to slower cooling as a result of increased volume to heat transfer area [16]. The present research is focussed to study the mould metal reactions at corners of the cast part. The effect of type of corner (convex or concave) on interfacial mould-metal reactions is investigated.

## 2. Research Methodology

### 2.1. Selection of Geometry and Mould Making

The stepped shape geometry was selected as shown in Figure 1(a) to evaluate reactions at the corner. It can be seen that at a particular step, two types of corners are present one with a convex shape and another with a concave shape. The wax patterns are prepared by injecting molten wax into two-piece aluminium die. The detailed dimensions of wax pattern is presented in Figure 2. The thermocouple junctions were placed at corners on wax patterns as shown in Figure 1(b), therefore after mould development and dewaxing, junctions remain at the corner inside the cavity to record the temperature at corner during solidification of metal inside the mould.

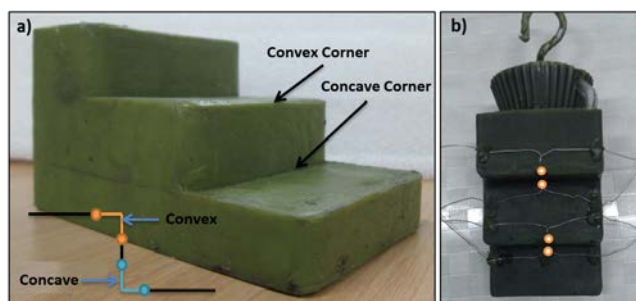


Fig. 1. (a) Step shaped wax pattern indicating the convex and concave corner (b) Thermocouple placement at corners

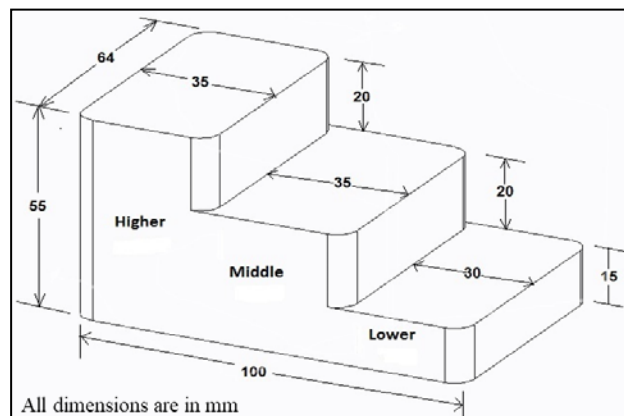


Fig. 2. Detailed dimension of stepped shape cast geometry

The moulds were developed on wax patterns using zircon flour ( $ZrSiO_4$ ) as a primary slurry material. Subsequent coatings were done using secondary slurry made up of aluminosilicate (molochnite). The compositions of primary and secondary slurry are mentioned in Table 1. Total 7 numbers of coatings were done to achieve sufficient mould thickness. The coarser stuccos were used during successive coatings and six hours of drying time provided prior to each coating. The moulds were dewaxed in resistance heating furnace at a temperature of  $200^{\circ}C$ - $250^{\circ}C$  and fired for 30 minutes at  $800^{\circ}C$  for complete removal of wax and any residues as shown in Figure 3.

Table 1.

Composition of slurry

| Slurry    | Ceramic Powder (%wt.)                                 | Binder (%wt.)          | Viscosity Zahn B4 Cup (sec) | Wetting Agent (%wt.)  |
|-----------|---|------------------------|-----------------------------|---|
| Primary   | Zircon Flour-325 mesh [ $ZrSiO_4$ ] (75%)             | Colloidal Silica (25%) | 35-40                       | n-Octanol +Diocetyl Sodium Sulfosuccinate (3 to 5% of binder) |
| Secondary | Molochnite Powder-200 mesh [ $Al_2O_3, SiO_2$ ] (45%) | Colloidal Silica (55%) | 12-15                       | -   |



Fig. 3. Firing of moulds in resistance heating furnace

### 2.2. AZ91 Magnesium Alloy Melting and Casting

The castings were produced by pouring commercial AZ91 magnesium alloy (having weightage composition of 9.05% Al, 0.92% Zn, 0.18% Mn and balance Mg). A graphite crucible was used for the melting of AZ91 magnesium alloy. The melting was carried out in a resistance heating furnace under the protective atmosphere of a gas mixture (3%  $SF_6$  + 97%  $CO_2$ ) as shown in Figure 4.

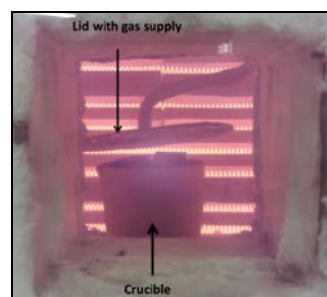


Fig. 4. Melting of AZ91 magnesium alloy under the protective gas supply

The pouring was carried out at  $680^{\circ}C$ , after purging of protective gas inside moulds. The reactions were observed on AZ91 magnesium alloy casting after knockout of mould as shown in Figure 5.



Fig. 5. AZ91 magnesium alloy casting

### 2.3. Characterisation of Reactions

Square sample of size 15mm\*15mm\*5mm was cut from the middle step of stepped shape casting as shown in Figure 6 to examine the reactions at corners. SEM-EDX was performed to quantify the interfacial mould metal reactions at the corner. Total five spots were considered in sequence moving from top edge of convex corner to the bottom edge of concave. At each place, one reacted spot (blackish appearance) and one nearby unreacted spot (silvery appearance) was considered for EDX measurement. The spot numbers 1 to 5 represents reacted regions whereas 1' to 5' represents un-reacted regions. XRD was also done to confirm the presence of reaction compounds on the casting surface.

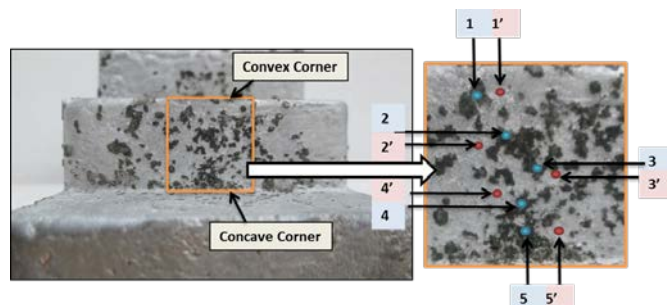


Fig. 6. Sample cut for SEM-EDX

### 2.4. Temperature Measurement

Thermocouples were connected to the Data Acquisition system (National instruments DAQ 9178) using temperature measurement module 9219 interfaced with a computer as shown in Figure 7 to measure the temperature at corners during solidification. The continuous recording of temperature at 1Hz frequency was carried out during solidification using LABVIEW software.

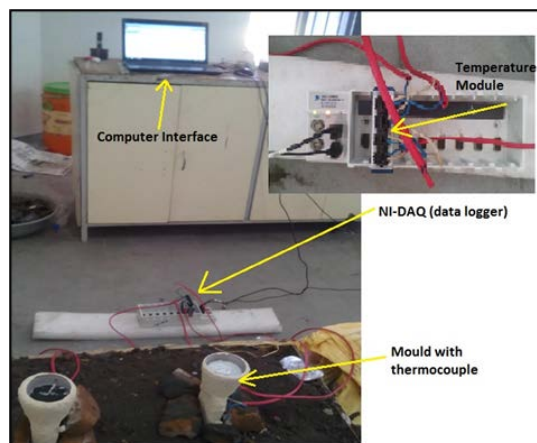


Fig. 7. Temperature measurement set up

## 3. Results and Discussion

The EDX spectra at reacted regions (spots 1 to 5) are presented in Figure 8 (a) to (e) whereas at un-reacted regions are presented in Figure 9 (a) to (e).

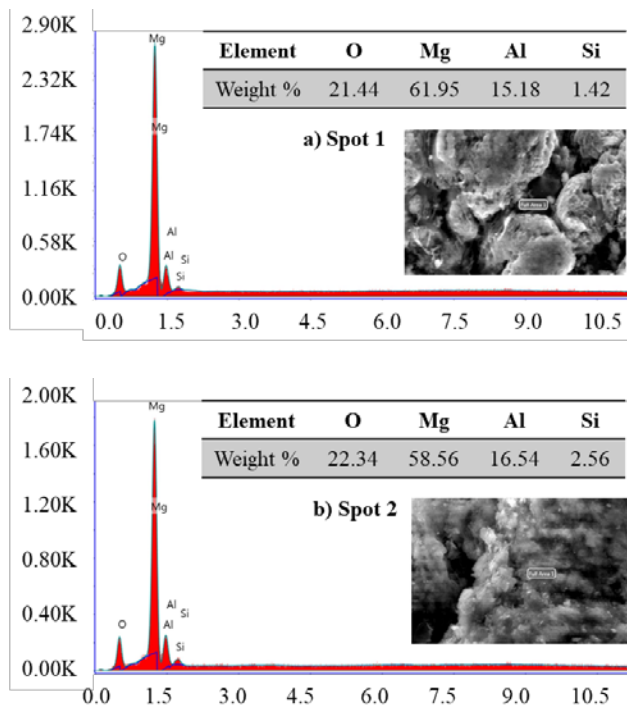




Fig. 8. (a) to (e) EDX spectra at reacted spots 1 to 5

Fig. 9. (a) to (e) EDX spectra at un-reacted spots 1' to 5'

The presence of reaction compounds  $MgO$ ,  $Mg_2Si$ ,  $MgAl_2O_4$ ,  $Mg_{17}Al_{12}$  etc. were detected in the XRD spectra as shown in Figure 10. The formation of magnesium oxide ( $MgO$ ) is the result of reaction of melt with the decomposed and diffused oxygen from mould (Eq.1). The formation of  $Mg_2Si$  is owing to the

reactions of melt with colloidal silica binder (Eq.2). The presence of nitride compounds  $Mg_3N_2$  and  $AlN$  is due to the possible reactions of alloying elements with atmospheric nitrogen. In addition, magnesium-aluminum compound  $MgAl_2O_4$  and  $\beta$ -phase  $Mg_{17}Al_{12}$  are also observed.

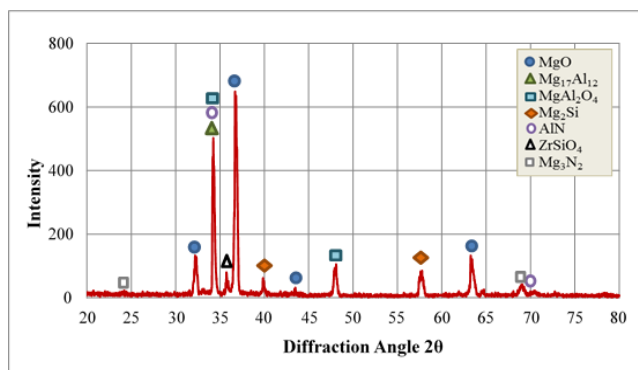
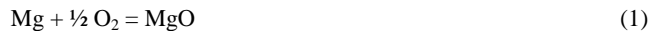


Fig. 10. XRD spectra of AZ91 magnesium alloy casting

However, owing to the higher affinity of magnesium with oxygen probability of oxides ( $MgO$ ) formation is maximum. This is also confirmed from EDX results, the amount of % oxygen detected was greater as compared to other elements. The summary of results representing oxygen content at each spot is presented in Table 2. It can be seen from the table that; oxygen content was increased from the spots of top edge near at convex corner moving towards the bottom edge at concave corner. The % oxygen content increased from 21.44% to 26.02% and 8.02% to 12.75% for reacted blackish regions and un-reacted silvery regions respectively. These results show that the oxides ( $MgO$ ) formed were more at regions near to concave corner as compared to the convex corner of cast surface. This indicates that at concave corner occurrence of reactions is more as compared to convex corner.

Table 2.  
Summary of SEM-EDX results

| Spots at reacted regions | % oxygen weightage | Spots at un-reacted regions | % oxygen weightage |
|--------------------------|--------------------|-----------------------------|--------------------|
| 1                        | 21.44              | 1'                          | 8.02               |
| 2                        | 22.34              | 2'                          | 8.95               |
| 3                        | 23.05              | 3'                          | 9.08               |
| 4                        | 24.17              | 4'                          | 10.75              |
| 5                        | 26.02              | 5'                          | 12.75              |

The mould cross section as shown in Figure 11(a) indicates that the thickness of mould at the convex corner is less (6.5 mm) as compared to concave corner (14 mm). The change in the mould thickness affects the mould permeability and heat dissipation. As the thickness of mould increases, permeability of mould also increases [17]. If the permeability is high than the chances for

reactions of melt with oxygen may increase, as melt having greater access to atmospheric oxygen. The heat dissipation rate at both the corners during casting solidification will be different owing to convex and concave shape profile. At the convex corner, the heat flow from metal to mould will be divergent type whereas at the concave corner it will be convergent type as shown in Figure 11(b). In addition, the heat transfer would be slow at concave corner due to higher mould thickness as compared to convex corner. This slower cooling at concave corner provides more time to melt to react with mould. This may be the reason for observation of greater reactions and oxide formations at concave corner as compared to convex corner.

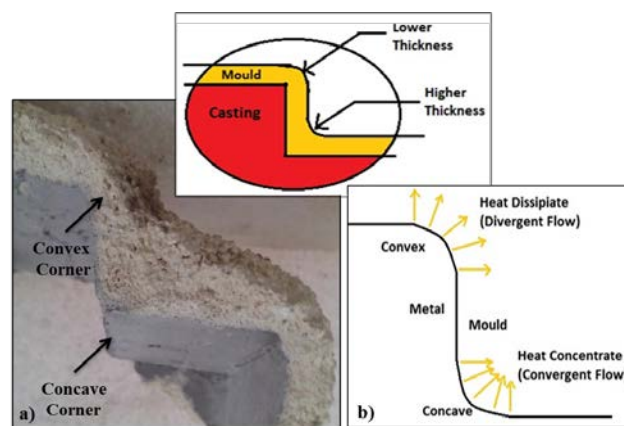


Fig. 11. a) The cross-sectional view of the ceramic mould, b) Heat dissipation at corners

The thermal profile recorded at corners during solidification is presented in Figure 12. It can be observed that there was a delay of around 35 seconds to reach the temperature of  $450^\circ C$  at interface of concave corner. This lag in cooling time provides more time to metal to remain in a liquid state. This resulted in larger reactions at the concave corner as compared to the convex corner of the cast part.

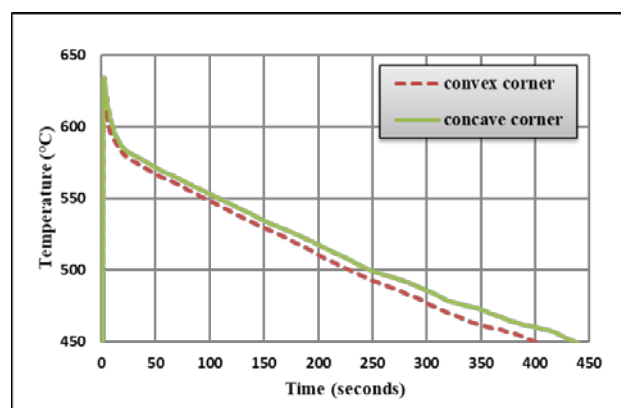


Fig. 12. Thermal profiles at corners of cast part during cooling

## 4. Conclusions

In this research, the investigation on mould-metal reactions at corners of cast part was carried out. The key conclusions are presented below.

- The oxygen detected on the cast surface at the concave corner has been around 5% higher than the convex corner. This indicates the larger formation of oxides at the concave corner as compared to convex corner from mould-metal reactions.
- The slower heat dissipation at the concave corner of the cast part due to convergent heat flow as well as larger mould thickness provided more time to a melt to react with mould at concave corner.

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