An investigation on microstructural and mechanical properties of solid mould investment casting of AZ91D magnesium alloy

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ABSTRACT

In this work, AZ91D magnesium alloy was cast in solid plaster mould using vacuum assistance. The influence of process parameters and wall thickness on the microstructure and tensile properties of cast specimens was studied. Within the range of experimental parameters, it was found that casting and mould preheating temperatures have minor influence on mechanical properties. However, gating design proved to affect the microstructure and tensile properties of cast specimens. Compared to bottom filling, top filling led to casting defects that seriously impaired the mechanical properties. Generally, tensile properties increased with a decrease of section thickness, this improvement being explained by a reduction of dendrite arms spacing and grain size. Finally, the mechanical properties of solid mould investment cast specimens were compared with those obtained by foamed plaster casting, sand casting, permanent mould casting and die-casting.

1. Introduction

Investment casting methods are used for the production of short series of premium quality components having complex shapes and thin walls [1–3]. They are also useful for the fabrication of prototypes, particularly in high-pressure die-casting of magnesium alloys where smooth surface finish and near-net-shape are essential [4,5].

It is recognised that the mechanical properties of cast alloys depend on their microstructure: grain size and texture, secondary phases and porosity. However, there is little information about the influence of casting parameters on microstructure and mechanical properties of investment cast magnesium alloys [6–9].

Idris and Clegg [8,9] have studied the influence of investment casting process variables on mechanical properties of magnesium alloys cast into an alumino-silicate mould. However, no clear relationship between solidification conditions, microstructure and strength was established. Kim et al. [10] observed a decrease of the grain size and an increase in the hardness and the ultimate tensile strength with a decrease of the mould temperature. They also found that any casting temperature variation on the interval of temperature considered (650–710 °C) produced little variation on the hardness. In the case of conventional solid mould investment casting, Davenport and Orton [11] have studied the mechanical properties of investment cast AZ63 and AZ92 magnesium alloys, in the as-cast state. Their properties were similar to those obtained by sand casting, for specimens having comparable solidification time. A decrease in the casting temperature led to better tensile properties. Herrick [6] and Pellegrini [12] studied the mechanical properties of Mg–Al- and Mg–Zr-based alloys respectively. In particular, Herrick [6] reported that mould temperature (from 20 to 340 °C) and casting temperature (from 670 to 740 °C) as well as section thickness (from 1.6 to 12.7 mm) had minor influence on the average grain size. However, increasing the investment mould temperature from 20 to 340 °C or the pouring temperature from 670 to 740 °C proved to have a deteriorating influence on the average mechanical properties of all alloys and tempers investigated. The negative influence of mould and pouring temperatures was attributed to the presence in varying
quantity of undissolved phases after heat treatment. In the case of Mg-Al-based alloys, Herrick [6] found that the tensile properties were comparable to those obtained by sand casting methods. Among all the alloys studied, the combination of strength, ductility and castability was found to be best for AZ91C alloy [6,13].

Previous papers have presented the results of a study about the influence of process and mould parameters on the fluidity, surface finish and reactivity of magnesium alloys produced by using vacuum-assisted plaster mould investment casting [14,15]. In the present work, the influence of casting conditions on microstructure and mechanical properties of AZ91D magnesium alloy cast was examined. The following experimental parameters were studied: gating design, thickness of cast specimen, casting temperature and mould preheating temperature.

2. Experimental Procedures

2.1. Production of Cast Specimens

Tensile test specimens were produced using solid mould investment casting method with vacuum assistance, a process described in a previous paper [14]. AZ91D ingots were melted in a 430 stainless steel crucible under CO2–0.5% SF6 protective gas mixture. Moulds were also flushed with the CO2–0.5%SF6 gas mixture prior to and during pouring of the molten alloy in order to help reducing mould–metal reactions [9,16].

The pouring temperature and mould preheating temperature influence the surface finish of the cast specimens and mould–metal reactivity [9,15]. Unless specifically stated otherwise, pouring temperature and mould preheating temperature were set to 750 and 350 °C respectively.

2.2. Experimental Parameters

2.2.1. Gating Design

The design of the cluster and its gating is critical with thin components as filling must be completed before solidification significantly reduces melt flow. In investment casting, it is usual to pay relatively little attention to the design of the filling system of a casting [17]. The top-filled system is widely adopted, the gating system being usually short and wax patterns directly connected to the sprue [17–19]. In this way, mould cavities are filled rapidly at the lowest cost. However, previous works showed that bottom filling reduces melt turbulence, leading to better mechanical properties [17,19–26]. It must be noted however that the degree of improvement in mechanical properties depends on the nature of the alloy studied. Significant improvement was observed in Al-based alloys, while this effect was less clear for Fe- or Ni-based alloys [19,26].

In the case of magnesium alloys, such study has not yet been reported in the literature so far. The first objective of this study is thus to assess the influence of gating design on microstructure and tensile properties of AZ91D magnesium alloy. In this perspective, two types of gating system were used as illustrated in Fig. 1a. Some specimens were cast using bottom filling (Fig. 1b), for which the molten magnesium alloy enters the mould at its base, rises up and enters the specimen cavity more quietly. Other specimens were cast by direct filling (Fig. 1c), the molten alloy entering the specimen cavity from the top under the influence of gravity. The thickness of test specimens was set to 4.3 mm.

2.2.2. Pouring and Mould Preheating Temperatures

Both pouring temperature and mould preheating temperature influence mould–metal reactivity and cooling rate during solidification [15]. In order to study the influence of pouring temperature on microstructure and mechanical properties, 2.2 mm thick specimens were bottom filled at a mould preheating temperature of 350 °C and at casting temperatures of 700, 720, 740, 750 and 760 °C. This range of experimental pouring temperatures was selected in order to prevent excessive contamination of the melt and ensure complete filling. The effect of mould preheating temperature was studied by using 4.3 mm thick specimens, bottom filled at 750 °C. Mould preheating temperature was set to 250, 300 and 350 °C. It was found with experience that preheating at
temperature higher than 350 °C was detrimental to surface finish [15].

2.2.3. Thickness of Cast Specimens
During solidification, the cooling rate of cast specimens and their microstructure are influenced by wall thickness [27]. In this series of experiments, the influence of section thickness on microstructure and mechanical properties of cast specimens was investigated using both bottom and top filling. The thickness of specimens was set to 1.0, 1.3, 1.6, 2.2, 3.2 and 4.3 mm for top filling and to 1.6, 2.2, 3.2 and 4.3 mm for bottom filling. It was not possible to completely fill tensile specimens thinner than 1.3 mm using bottom filling, within the range of experimental casting parameters tested during this study. However, the surface filled was sufficiently large to be able to observe the microstructure and make porosity measurements on these specimens.

2.3. Microstructural Studies
Selected specimens were mechanically polished through successively finer grits of silicon carbide papers (down to grit size 1200) and diamond polishing suspension (particle size down to 0.1 μm). Polished specimens were etched with acetic glycol to reveal the general microstructure [28]. A different etchant was used to reveal the granular structure [29]. Specimens were examined by optical (OM) and scanning electron microscopy (SEM). Grain size was measured according to ASTM E112 [30].

Solidification time was estimated by measuring the secondary dendrite arm spacing (SDAS), using the following relation obtained for AZ91D magnesium alloy solidified under various conditions [31]:

\[
\lambda = 35.5 \tau^{-0.31}
\]

where \( \lambda \) (μm) is the secondary spacing and \( \tau \) (K s\(^{-1} \)) is the cooling rate during solidification. Measurements of SDAS were made at a minimum of ten different locations on dendrites revealing at least four secondary arms. Solidification time was calculated considering a liquidus of 595 °C and a non-equilibrium solidus of 425 °C, i.e., a liquidus–solidus interval of 170 °C [32].

The porosity of tensile specimens was evaluated by using density measurements in distilled water (Archimede’s method) and calculated using the following relation:

\[
\text{% porosity} = 100 \times \frac{D_{\text{a}} - D_{\text{t}}}{D_{\text{a}}}
\]

where \( D_{\text{a}} \) is the actual density of the specimen and \( D_{\text{t}} \) is the theoretical density of the alloy, taken as 1.81 g cm\(^{-3} \) for AZ91D magnesium alloys [32]. A minimum of three tensile specimens were used for these measurements.

2.4. Evaluation of Tensile Properties and Fractography
Tensile strength was evaluated with as-cast sub-size specimens of rectangular cross-sections having a width of 6.0 mm and a 25.0 mm gage length. Tensile tests were carried out at a deformation rate of 0.003 s\(^{-1} \), according to ASTM B557M [33]. The ultimate tensile strength (UTS) and the total elongation were evaluated from the stress–strain data obtained. The real elongation at break (\( \varepsilon \)) was calculated by subtracting the elastic strain (100 × fracture stress/elastic modulus) from the total elongation, according to ASTM B557M [33]. The tensile yield strength at 0.2% offset (YS) was calculated using a nominal elastic modulus (E) value of 45 GPa, since AZ91 magnesium alloy does not exhibit a definite elastic region [34–38].

The fracture surfaces of selected tensile test specimens were examined using a scanning electron microscope, equipped with an energy dispersive X-ray analyser (EDS). All specimens were coated with a thin layer of Au–Pd to prevent charging.

3. Results and Discussion
3.1. General Microstructure of Cast Specimens
Fig. 2 shows the microstructure of a 1 mm thick specimen cast at 750 °C in a mould preheated at 300 °C, revealing microconstituents typically found in specimens during this study. Primary equiaxed dendrites of \( \alpha \)-magnesium are visible (Fig. 2a). At higher magnification (Fig. 2b), a lamellar structure consisting of alternate \( \beta \)-phase (Mg\(_{17}\)Al\(_{12}\)) and \( \alpha \)-magnesium solid solution is detected along with large intermetallic \( \beta \)-phase.

Fig. 2 – (a) Optical micrograph of solid mould cast AZ91D alloy; (b) detailed view of the lamellar structure (1) and Mg\(_{17}\)Al\(_{12}\) phase (2).
phase. Cross-section of as-polished specimens also reveals the presence of intermetallic Al–Mn based paricles, most of them being located at the surface of cast specimens [39].

3.2. Influence of Gating Design

Fig. 3 shows typical stress–strain curves obtained for top- and bottom-filled specimens. It can be noted that bottom-filled specimens exhibit higher tensile strength, effective elastic modulus and elongation at break. As shown in Fig. 4, the average tensile strength of bottom-filled specimens is 36% superior to that of top-filled specimens. Bottom filling also proved to have a remarkable effect on ductility, which was found to be three times higher as compared with top filling. However, gating design seems to have only a slight influence on yield strength.

In order to evaluate the relative dispersion of mechanical properties, the coefficient of variation (CV) was calculated using the following relation:

$$CV = \frac{s}{\bar{x}}$$

where $s$ is the standard deviation and $\bar{x}$ is the mean value of the considered property. Table 1 gives the coefficient of variation for ultimate tensile strength (UTS), yield strength (YS) and elongation ($\varepsilon$). It can be seen that the coefficient of variation is lower for ultimate tensile strength and elongation in the case of bottom filling, revealing a reduced scatter in these properties. The comparable coefficients of variation for yield strength confirm the weak influence of gating design on this property, in accordance with Campbell [17] and Cáceres et al. [40].

Previous studies on aluminium alloys containing magnesium, which have a relatively high propensity to form oxide films, produced similar results [19,24,25,41,42]. It was reported that, with top-filling running systems, the disruption and the entrainment of oxide films caused by turbulent flow leads to wide scattering of tensile strength and hence low mechanical reliability [19,24,25,41,42]. In this work, the detrimental influence of top filling on ultimate tensile strength, ductility and elastic modulus is also attributed to casting defects.

Fig. 5 shows the visual aspect of cast tensile test specimens. It reveals that top-filled specimens exhibit flow marks on their surface (Fig. 5a), while these features were not visible in the case of bottom-filled specimens (Fig. 5b). Flow marks are surface defects which are usually associated with turbulent flow [43]. With the top-filling gating system, the molten magnesium alloy falls through the mould in a turbulent, uncontrolled way, explaining the occurrence of flow marks.

Fig. 6 shows the cross-section of top- and bottom-filled specimens, which were observed with scanning electron microscope. It reveals the presence of porosity, inclusions and oxide films in all cast parts which were top-filled (Fig. 6a), while these defects were generally not observed in the case of bottom-filled specimens (Fig. 6b). Quantitative porosity measurements and fractographs confirm the presence of casting defects in top-filled specimens. As shown in Fig. 7, bottom-filled specimens are less porous than top-filled specimens. The higher porosity content in top-filled specimens is ascribed to the turbulent flow which entrains gases during the filling of mould. Finally, Fig. 8 shows the SEM fractographs of selected specimens, which revealed features of brittle fracture, with cleavage and quasi-cleavage as the principal fracture modes (Fig. 8a and b). For top-filled specimens, the presence of relatively large round pores was also observed (Fig. 8c), along
with regions containing intact (protruding) dendrites characteristic of shrinkage cavities [44] (Fig. 8d). Porosity was found mostly along interdendritic arms. For bottom-filled specimens, some casting pores were also observed, but intact dendrites were much less visible.

### 3.3 Influence of Casting and Mould Preheating Temperatures

Fig. 9a shows the influence of casting temperature on grain size and secondary dendrite arm spacing in cast specimens. It reveals that, in the range of temperatures studied, SDAS does not vary with casting temperature. A slight increase of grain size with casting temperature was also observed, but it does not significantly influence the ultimate tensile strength and elongation of cast parts, as shown in Fig. 9b. These results are similar to those obtained by Siaminwe and Clegg [45] on Al–Si–Mg alloy. These authors also observed that casting temperature did not influence the microstructure and had little effect on tensile properties.

Fig. 10a shows the influence of mould preheating temperature on grain size and secondary dendrite arm spacing. In the range of temperature studied, a slight increase (20%) of secondary dendrite arm spacing was found as mould preheating temperature was raised, which can be ascribed essentially to the slightly reduced cooling rate during solidification. The influence of mould preheating temperature on ultimate tensile strength, yield strength or ductility was observed, as shown in Fig. 10b, despite the moderate but systematic variation of secondary dendrite arm spacing.

### 3.4 Thickness of Cast Specimen

The tensile properties of top- and bottom-filled specimens are presented in Fig. 11. For section thicknesses above 1.6 mm, mechanical properties of top- and bottom-filled specimens follow a similar tendency. It is observed that ultimate tensile strength and elongation increase as the section thickness decreases, while yield strength remains relatively constant. Below 1.6 mm, for top-filled specimens, it is observed that these properties decrease with a continuing decrease of section thickness although it would be expected that the mechanical properties would further increase with a decrease of section thickness [46,47]. However, a similar trend was observed in the work of Easton et al. [48] and Abbott et al. [49] on die-cast AZ91 for UTS and elongation. The variation in ductility and UTS with section thickness was ascribed to the presence of various defects in the cast parts. The yield strength, in their work, tended to be higher for the thinnest sections and this variation was attributed to the grain size of the specimen.

In this study, the variation of ultimate tensile strength and elongation can also be explained using microstructural considerations. First, for bottom-filled specimens, Fig. 12 shows that grain size and secondary dendrite arm spacing increases with section thickness. This increase of secondary dendrite arm spacing is explained by the reduction of solidification rate, the cooling rate being inversely related to section thickness [27]. Besides, the size of grains closely follows the variation of dendrite arm spacing, longer freezing times promoting coarser microstructure, as shown in Fig. 12.

In Fig. 7, it is shown that the degree of porosity depends on the section thickness of specimens. Within the margin of experimental error, porosity does not vary much between 1.6 and 4.2 mm, but significantly increases when the section thickness decreases to 1.0 mm, this increase being more important for top-filled specimens. Fractographs (Fig. 8) also
revealed that shrinkage porosity, with intact dendrites clearly visible, is all the more present as the section thickness of cast parts decreases. The higher porosity level in thinner sections is ascribed to a more difficult filling.

Finally, as illustrated in Fig. 13a, it was observed that for section thicknesses above 1.6 mm, the microstructure of bottom- and top-filled specimens consists of equiaxed grains, homogeneously distributed within specimens. For section thicknesses below 1.6 mm, bottom-filled specimens still present a uniform microstructure. However, in top-filled specimens, areas with abnormally large grains along with areas containing much smaller grains were observed (Fig. 13b and c). These abnormally large grains are approximately 500 μm in diameter, much larger than those found in the thickest specimens cast in this study. Their presence in the microstructure is detrimental to tensile properties, in agreement with previous studies [50,51]. Their origin is not clear but is likely related to the presence of oxide films which are unfavourable nucleation sites.

As for the yield strength, since the grain size and secondary dendrite arm spacing are simultaneously reduced with the thickness of as-cast specimens, it is expected that it should be improved even more efficiently with a reduction in thickness of specimens, in agreement with a previous study of Couture and Meier [46]. However, it was observed that it does not vary much, except at the lowest section thicknesses in top-filled specimens. The reverse trend observed with thinner specimens in the present work is ascribed to the presence of microshrinkage, to fine particles or to surface defects, which have relatively more importance in thin specimens and reduce the grain size contribution.

3.5. Comparison of Mechanical Properties of Investment Cast Parts with Other Casting Methods

From the different results obtained, it can be seen, in the case of bottom filling, that tensile properties exceed the minimum requirements of ASTM B403 for AZ91C-F magnesium alloy (UTS: 124 MPa, YS (0.2%): 69 MPa, elongation not specified) [52].

For comparison purposes, the typical tensile properties of AZ91D magnesium alloy in as-cast condition for different casting processes are listed in Table 2. Moreover, Fig. 14 compares the yield strength ($\sigma$) of as-cast AZ91 from different sources [46,49,53–59] as a function of the inverse square root of grain size ($d^{-1/2}$). A least square regression was calculated using a model based on the Hall–Petch relationship [60]:

$$\sigma = \sigma_0 + k \cdot d^{-1/2}$$

where $\sigma_0$ and $k$ depend on alloy composition. Values of 379 and 64 were obtained for $\sigma_0$ and $k$ respectively with a $R^2$ of
0.77, based on properties measured in MPa for yield strength and \( \mu \text{m} \) for grain size respectively. Grain size was comprised between 10 and 1100 \( \mu \text{m} \).

As expected, solidification time is shorter in the case of die-casting, resulting in a finer microstructure and higher mechanical properties\[49,55,57,58\]. In the case of permanent mould casting, the results display significant differences\[53–55,58,59\]. The higher values of yield strength obtained by Maltais et al. \[53\] can be explained by considering the secondary dendrite arm spacing. In this work, the SDAS was maintained constant to 16 \( \mu \text{m} \), while it was maintained to 40 \( \mu \text{m} \) in the work of Sasaki et al. \[54\]. The reduction of the effective size of primary phase should even more increase the yield strength. Comparatively to die-casting and permanent mould casting, the solidification time in plaster mould casting

Fig. 9 – Influence of casting temperature (a) on grain size and secondary dendrite arm spacing and (b) on mechanical properties of bottom-filled specimens. Error bars correspond to two standard deviations.

Fig. 10 – Influence of mould preheating temperature (a) on grain size and secondary dendrite arm spacing and (b) on mechanical properties of bottom-filled specimens. Error bars correspond to two standard deviations.

Fig. 11 – Influence of section thickness on mechanical properties (a) bottom filling, (b) top filling. Error bars correspond to two standard deviations.

Fig. 12 – Influence of section thickness on grain size and secondary dendrite arm spacing (bottom filling). Error bars correspond to two standard deviations.
sand casting [46,49] is higher, which leads to a coarser microstructure and lower mechanical properties.

In the present work, the results obtained reveal that the mechanical properties of investment cast AZ91D magnesium alloy are slightly inferior or comparable to those found for sand casting and permanent mould casting. The results are lower than those published by Herrick [6] who found a value of UTS of 198 MPa and an elongation of 2.5% in the as-cast condition in the case of AZ91C alloy cast at 670 °C in a mould at 20 °C. This is probably due to a higher solidification rate provided by a lower mould temperature. However, they are superior to those obtained by Fantetti et al. [61] in the case of foamed plaster casting (without vacuum assistance). This can be explained by the fact that the section thickness used was higher and that foamed plaster is more thermally insulating than dense plaster.

4. Conclusions

In this work, the influence of process parameters on microstructure and mechanical properties of vacuum-assisted solid investment cast AZ91D magnesium alloy was investigated.

In the range of temperature investigated, it was found that casting and mould preheating temperatures do not significantly influence the microstructure and tensile properties of as-cast specimens.

However, the influence of top and bottom filling systems on the properties of AZ91D castings were compared. Top-filling systems promote the formation of microporosity and other casting defects, which appear to adversely affect ultimate tensile strength and elongation of cast AZ91D alloy. Ductility was particularly affected, since an elongation three times lower was obtained, as compared to bottom filling. However, it was also observed that it was not possible to fill section thicknesses inferior to 1.6 mm with bottom filling.

An improvement of tensile strength and elongation with a decrease of section thickness was observed in bottom-filled specimens. It was essentially ascribed to a reduction of the grain size and secondary arm spacing. The thinnest top-filled specimens display decreasing properties. This was ascribed to the presence of porosity and abnormally large grains.

Finally, the ultimate tensile strength and yield strength obtained were higher than the minimum requirements of ASTM B403 for AZ91C-F magnesium alloy. The results were slightly lower than those obtained by sand casting and permanent mould casting, but higher than those obtained by foamed plaster casting.

## Acknowledgements

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### Table 2 – Typical tensile properties of as-cast specimens of AZ91 magnesium alloy prepared using different casting methods

<table>
<thead>
<tr>
<th>Method</th>
<th>UTS (MPa)</th>
<th>YS (MPa)</th>
<th>(\varepsilon_f) (%)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plaster (foamed) casting</td>
<td>103</td>
<td>91</td>
<td>1.0</td>
<td>[61]</td>
</tr>
<tr>
<td>Sand casting</td>
<td>153</td>
<td>104</td>
<td>2.5</td>
<td>[38]</td>
</tr>
<tr>
<td>Permanent mould casting</td>
<td>145</td>
<td>87</td>
<td>2.6</td>
<td>[55]</td>
</tr>
<tr>
<td>100–112</td>
<td>88–95</td>
<td>0.5–0.9</td>
<td>[53]</td>
<td></td>
</tr>
<tr>
<td>170</td>
<td>93</td>
<td>1.4</td>
<td></td>
<td>[59]</td>
</tr>
<tr>
<td>Die-casting</td>
<td>233</td>
<td>163</td>
<td>6.0</td>
<td>[55]</td>
</tr>
</tbody>
</table>
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