Effect of oxide film defects generated during mould filling on mechanical strength and reliability of magnesium alloy castings (AZ91)

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The influence of new oxide film defects generated during mould filling on the mechanical strength and reliability of magnesium alloy castings (AZ91) has been investigated. The filling behaviour was evaluated using an optical video camera. Three point bend tests were conducted on specimens cut from the cast plates. The Weibull statistical method was employed to quantify the scatter of mechanical properties. Scanning electron microscopy and energy dispersive X-ray techniques were used to observe features of the oxide films on the fracture surfaces. Results indicated that there is a critical in-gate velocity that lies between 0·25 and 0·35 m s^{-1}. Thin and folded surface oxide films were observed at the fracture surfaces. These films are likely to be distributed throughout the casting due to the random nature of the entrainment process. It was found that the presence of new oxide films strongly affects the scatter of the mechanical properties of the castings.

Keywords: AZ91 alloy, Filling behaviour, In-gate velocity, New oxide films, Mechanical properties and reliability

Introduction

Magnesium cast alloys with their low density and excellent properties are receiving much attention for applications in automobile, communications and aerospace industries.[1] However, castings with greater reproducibility and property reliability are expected, as their current use is limited due to high scatter of mechanical properties. Recent investigations have revealed that a major cause of failure of aluminium castings is defects that initiate from double oxide films that become entrained into the bulk of melt by a folding mechanism arising from surface turbulence during melting, transfer and casting processes.[2–6]

The concept of the double oxide film and its effect on mechanical properties of aluminium alloy castings has been championed by Campbell.[7] He concludes that the minimisation of oxide films generated in mould filling plays a very significant role in the attainment of high quality and reliable mechanical properties of castings. It has been shown that the entrapped oxide films are frequently accompanied by different casting defects such as shrinkage porosity,[8] cracks and dross[9] in solidified castings. It has also been demonstrated that to avoid such deleterious effects, a stable meniscus front during mould filling needs to be maintained and that in order to achieve this stability, it is necessary to control the liquid velocity to below $\sim$0·5 m s^{-1}, as confirmed by Runyoro *et al.*[10] for Al alloys, and by Bahreinian *et al.*[11] for Mg alloy ZK51.

A few studies have been carried out on the morphology and features of double oxide films in Mg alloy castings. Owing to the high affinity of magnesium alloys for oxygen and the high vapour pressure of Mg, a loose and porous oxide film layer tends to form and this is unable to prevent further oxidation of the melt.[12] It has been found that the features of surface oxide films, such as morphology, thickness, etc., are affected by the chemical composition of the molten metal[13,14] and by the composition of the protective atmosphere used during melting and casting operations.[15]

Griffiths and Lai[16] investigated the nature of folded oxide film defects in pure magnesium in the as cast condition. They showed that a poor running system design was associated with greater entrainment of oxide films during mould filling and that this produced a greater spread in ultimate tensile strength than castings produced with a good running system design. Mirak *et al.*[17] revealed the characteristics of new oxide films in AZ91 alloy where the oxidation was induced by the impingement of bubbles within the solidifying casting. Wang *et al.*[18] identified some of the mechanisms of microporosity formation in magnesium alloy AZ91 and showed the existence of abundant oxide film defects, similar to those observed in aluminium alloys. Although the deleterious effect of double oxide films on castings has been known for some time, the relationship between entrainment defects and the scatter of properties of castings in Mg alloys has not been clarified so far.

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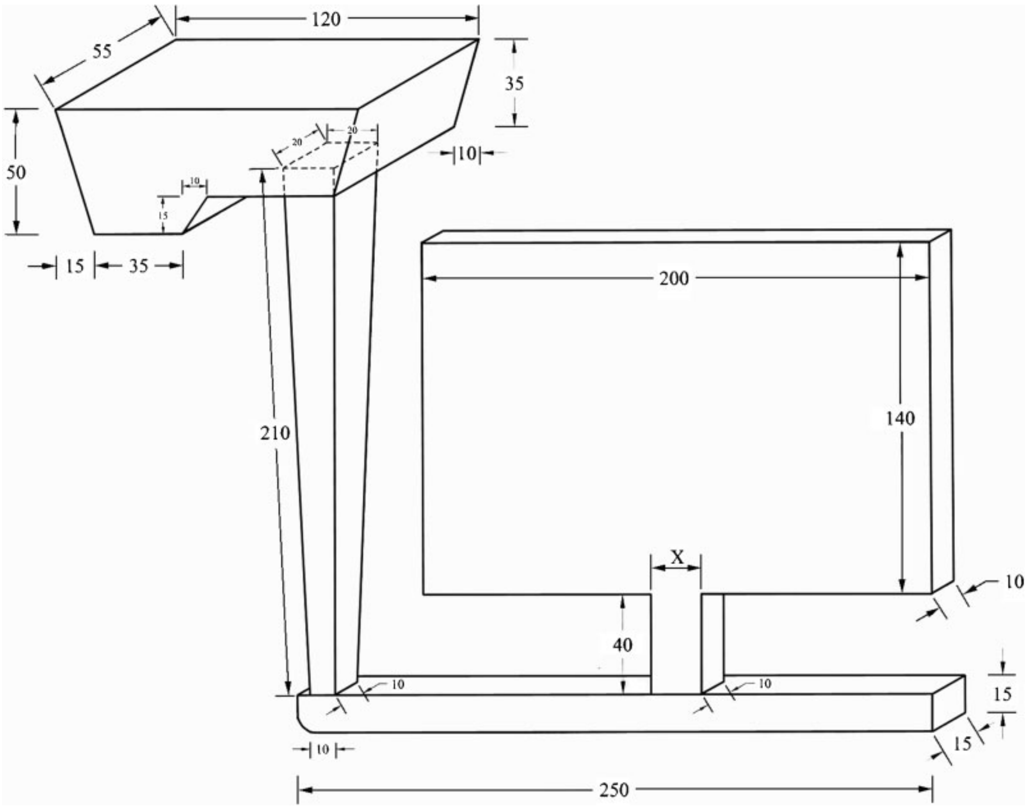


1 Schematic diagram of plate casting with bottom gate system used in this work

In this work, we concentrate on the effect of filling behaviour of magnesium alloy (AZ91) on the mechanical properties of castings. The study identifies the morphology of double oxide films related to the surface turbulence during mould filling. A Weibull statistical model is employed to quantify the effects on the strength and reliability of the castings produced with different in-gate velocities.

Experimental

The chemical composition of the AZ91 alloy used in this work is Mg–8·7Al–0·5Mn–0·9Zn (wt-%). Silica sand (containing 2% sulphur as an inhibitor), and sodium silicate binder cured with carbon dioxide were used as the moulding material. Melting was carried out in a steel crucible using an electrical induction furnace. A protective atmosphere of SO_2 gas was used during both melting and pouring. The melt was prepared and held at $700\pm5^{\circ}C$ and all of the castings were poured from this temperature. Experimental work was based on 10 mm thick plate castings with a bottom gated running system, as shown in Fig. 1. The in-gate velocity was changed by varying the cross-sectional area of the gates, i.e. 100, 200, 250, 300 and 400 mm^2. A double layer glass window (6 mm thickness) was fixed to one side of the mould covering the mould cavity and the in-gate. By using this technique, it was possible to view and study the filling behaviour of the liquid metal as it filled the mould at different in-gate velocities. The fillings were recorded by a video camera at 25 frames per second, corresponding to 0·04 s between frames. Real in-gate velocity was measured from the height/time data, providing a best fit parabola, and finding the slope at zero height by a small backwards extrapolation as described by Runyoro *et al.*[10]

Three point bend testing using an Instron Universal Testing Machine was carried out on samples cut from cast plates made with different in-gate velocities to characterise the casting properties. Test specimens were prepared by vertically sectioning the cast plates into testpieces $100\times10\times10$ mm. The reason for using the bend test is that a greater volume of material is uniformly stressed, so that the strength results are more representative of the bulk material. For each condition, at least 30 samples from the plate castings were tested.

The fracture surfaces of the bending test specimens were studied to characterise the oxide film morphology, using an XL30 Philips scanning electron microscope. Energy dispersive X-ray (EDX) microanalysis was performed to determine the composition of the oxide layers.

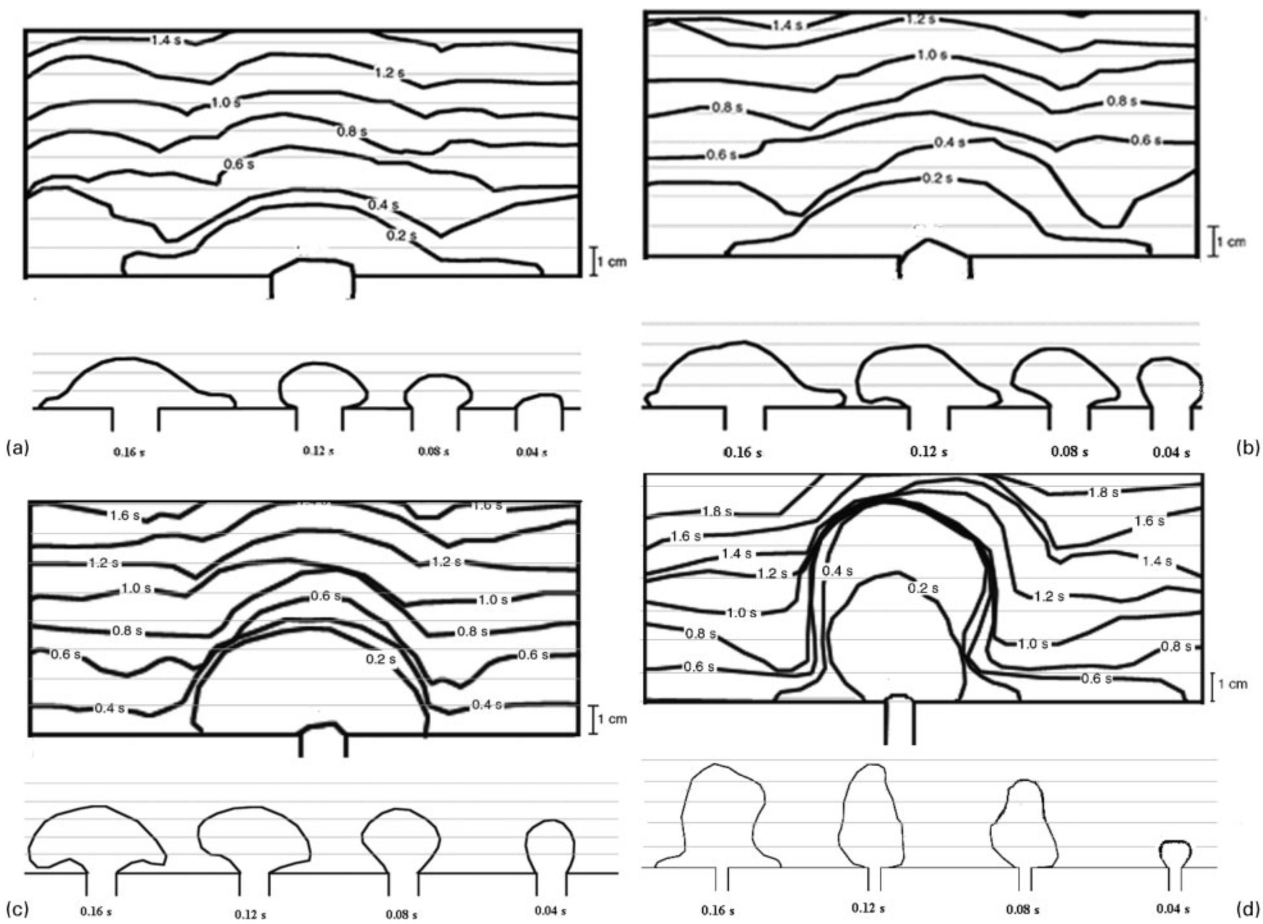
Weibull statistical analysis

For the study of the distribution of mechanical properties of castings, it has been shown that the Weibull distribution can be used to reasonably describe the probability of fracture.[19–21] The Weibull distribution is given as

$$P_i = 1 - \exp\left[-\left(\frac{x}{\eta}\right)^{\beta}\right] \quad (1)$$

where P_i is the probability of failure at a value of x, x is the variable being measured, i.e. ultimate bend strength (UBS), η is the characteristic stress at which $\sim$63·23% of the specimens have failed and β is the Weibull modulus. Taking the natural logarithm of equation (1) twice gives

$$\ln\left[\ln\left(\frac{1}{1-P_i}\right)\right] = \beta\ln x - \beta\ln\eta \quad (2)$$



a 0·25 m s^{-1}; *b* 0·35 m s^{-1}; *c* 0·5 m s^{-1}; *d* 1 m s^{-1}

2 Schematic diagrams of morphology of melt entry and filling front behaviour for AZ91 alloy plate castings at different in-gate velocities: in-gate velocities are changed by altering in-gate cross-sectional area

If ln x is plotted against

$$\ln\left[\ln\left(\frac{1}{1-P_{\rm i}}\right)\right]$$

a straight line is produced with slope β and intercept $-\beta\ln\eta$. The slope β physically represents the Weibull modulus of the casting and is clearly a measure of the spread of the distribution. As suggested by Dai *et al.*[2] the failure probability $P_{\rm i}$ can be estimated using the following relationship[2]

$$P_{\rm i}=\left(\frac{i-0{\cdot}3}{n+0{\cdot}4}\right) \qquad (3)$$

where i is the rank position of a result when all results are ordered in an ascending fashion and n is the total number of results.

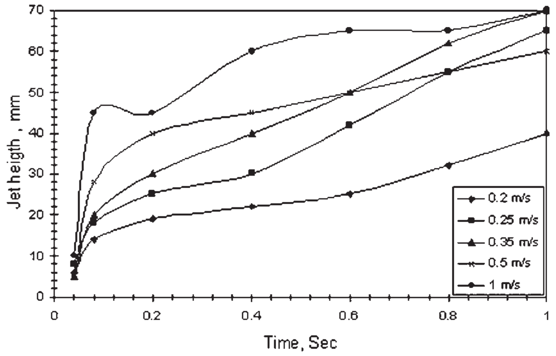
A series of UBS values with a higher Weibull modulus reveals a lower scatter of properties and is taken as indicative of a casting operation associated with a low number of defects in the castings and greater reproducibility of properties.

Results

The experimental flow patterns of molten metal resulting from captured video images for several in-gate speeds are shown in Fig. 2. Figure 2*a* shows the filling front profiles at increasing times after the melt enters the cavity at a gate speed of 0·25 m s^{-1}. At this speed, the melt emerges and expands smoothly across the mould surface without any evidence of significant surface turbulence. It can be seen that the melt fills the mould in advancing layers of slightly varying thickness across the casting width. Figure 2*b* shows the filling front profiles observed a gate speed of 0·35 m s^{-1}. At this speed, the melt has a clearly formed central mushroom shape that allows some mixing of the melt surface into the bulk. Figure 2*c* shows the filling front profiles for a gate speed of 0·5 m s^{-1} with surface turbulence clearly well developed. Figure 2*d* shows the filling profiles for an in-gate velocity of 1 m s^{-1}. At this speed, the front jets upwards with a fountain shaped morphology, and it is evident that the upper layers of the melt fold over the lower layers with considerable intermingling of the surface into the bulk melt occurring. Figure 3 shows the heights of the molten metal jet as it enters into the mould cavity as a function of time for different in-gate velocities.

Table 1 shows UBS versus in-gate velocity. It can be seen that there is a sharp reduction of UBS in the in-gate velocity range of 0·25–0·35 m s^{-1}. The bending strength decreases from ~400 MPa at 0·25 m s^{-1} to ~270 MPa close to 0·35 m s^{-1}. The bending stress continues to fall reaching a value of ~180 MPa at an in-gate velocity of 1 m s^{-1}.

The frequency histogram plots of measured bending strength of the multiple samples for each of various in-gate velocities are shown in Fig. 4. Weibull distribution



3 Graph showing molten metal jet height versus filling time for different in-gate velocities

plots that summarise all of data for each of the four in-gate velocities are shown in Fig. 5, where σ is the measured bend strength of an individual samples. The calculated Weibull parameter for each set of castings is shown in Table 2. The parameter β is the resulting slope of the lines for each set.

Scanning electron micrographs of selected fracture surfaces of bend test samples (at various magnifications) are shown in Figs. 6–8. The EDX spectrum shown in Fig. 9 is taken from the wrinkled oxide region observed in Fig. 8.

Discussion

Critical in-gate velocity

The effect of in-gate velocity on flow behaviour of molten metal during mould filling is significant. As can be seen in the flow pattern of molten Mg alloy at different in-gate velocities (Fig. 2), there is a morphological change of the filling front from smooth and uniform meniscus at 0·25 m s^{-1} to mushroom shaped at 0·35 m s^{-1} and then at a higher in-gate velocity close to 1 m s^{-1} a change to fountain shaped (Fig. 2*d*). A schematic drawing showing the outlines of the liquid magnesium profiles at different in-gate velocities that are taken from the first frames of Fig. 2 (i.e. at <0·1 s) is given in Fig. 10. It can be seen at the highest in-gate velocity that the jet rises up to a high level in the mould and then falls back over itself, forming a folded surface that collapses under the influence of gravity. Surface turbulence is clearly well developed and the surface oxide skin is in danger of being entrained into the bulk of the melt. It is therefore proposed that there is a critical in-gate velocity for magnesium alloy (AZ91) casting in the range of 0·25–0·35 m s^{-1} in a 10 mm thick plate.

It has been demonstrated that the in-gate velocities of molten metals are dependent on both inertial pressure ρV^2 and the restraining pressure $2\gamma/r$, giving the critical velocity when these two parameters are in balance, as follows

Table 1 Ultimate bending strength versus in-gate velocity

In-gate velocity/m s^{-1}	Ultimate bend strength/MPa	Error/%
0·25	400	0·9
0·35	275	1.5
0·5	200	7·5
1	180	9·7

$$V=\left(\frac{2\gamma}{\rho r}\right)^{1/2} \qquad (4)$$

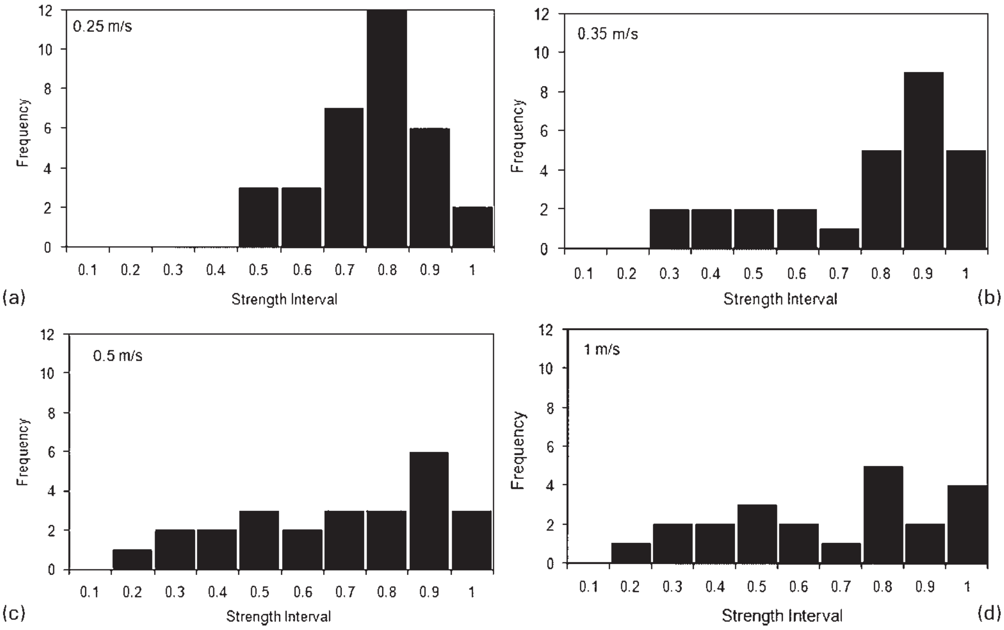
where V is the local flow velocity, γ is the surface tension, ρ is the density and r is the arc radius of the melt front that for 10 mm thick plate is assumed to be between 0·005 and 0·10 m. For liquid magnesium, $\gamma\approx0{\cdot}6$ N m^{-1} and $\rho\approx1700$ kg m^{-3}, giving a calculated critical velocity in the range of 0·27–0·37 m s^{-1}. It can be seen that there is a good coincidence between experimental results and theoretical calculation.

The flow of liquid metal during the filling is basically a transient free surface flow. Lai *et al.*[22] indicated that the free surface area of the melt is a function of liquid velocity during filling. The higher the in-gate velocity, the higher the free surface front of melt and this led to more oxidation. It is likely that due to the folding and random break-up of the melt through the central region of the casting that the folded surface oxide films (i.e. double oxide films) enter into the bulk of the melt and are subsequently able to behave as open cracks when the melt solidifies, as shown schematically in Fig. 11. However, by controlling the chaotic behaviour of liquid flow into the mould and by dissipating its kinetic energy, it is suggested that mould filling without any surface turbulence is likely to reduce the probability of such entrainment defects.

Mechanical properties and reliability

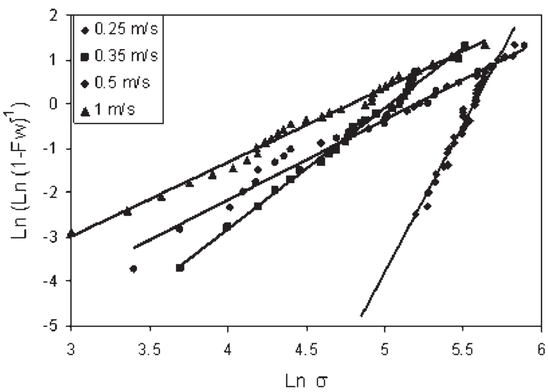
Table 1 shows a sharp reduction of UBS from 400 MPa at 0·25 m s^{-1} to 275 MPa at 0·35 m s^{-1}. This corresponds to a sharp reduction in mechanical properties as the surface turbulence starts (*see* flow patterns in Fig. 2). It seems that there is a relationship between the degree of surface turbulence during filling and the mechanical properties of castings. As can be seen from histogram plots in Fig. 4, the castings that suffered surface turbulence during filling had a higher degree of scatter of properties. It is clearly evident from the frequency distribution of bending strength at 0·25 m s^{-1} that the distribution of strength is skewed, with the majority of failures occurring close to the maximum (Fig. 4*a*). As the in-gate velocity increases towards 1 m s^{-1}, the distribution of strength becomes less skewed and more random with a tail of low strength castings (Fig. 4*d*). This is reflected in the Weibull plot of Fig. 5, where the lower the reproducibility of the casting strength, the more shallow the slope of the line (i.e. lower Weibull modulus). The lowest Weibull modulus was 1·6 for the most turbulent castings (in-gate velocity 1 m s^{-1}), while the highest modulus was found to be 6·6 for samples produced at the lowest turbulence condition (i.e. <0·25 m s^{-1}).

It is clear that castings that were filled under surface turbulence conditions displayed a high scatter of mechanical properties. This is most likely due to formation of large numbers of oxide films (most likely folded and behaving as cracks) generated during filling. The scatter is indicative of the randomness of oxide formation and entrainment process. At lower in-gate velocities (i.e. 0·35 m s^{-1} and below), where there is some control of the liquid meniscus and surface disruption as shown in Fig. 2*a* and *b*, the source of oxide films is drastically removed.

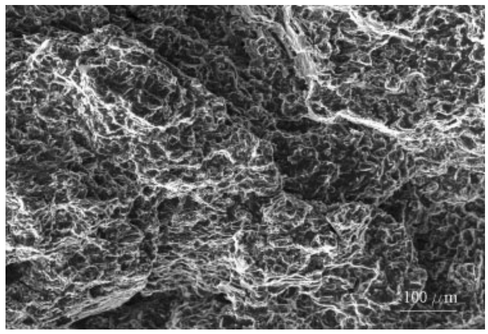


a 0·25 m s^{-1}; *b* 0·35 m s^{-1}; *c* 0·5 m s^{-1}; *d* 1 m s^{-1}

4 Frequency histograms showing distributions of bending strength among samples (in terms of intervals expressed as fraction of maximum recorded UBS value) for different in-gate velocities



5 Weibull plots of bending strength data (*see* text for details)



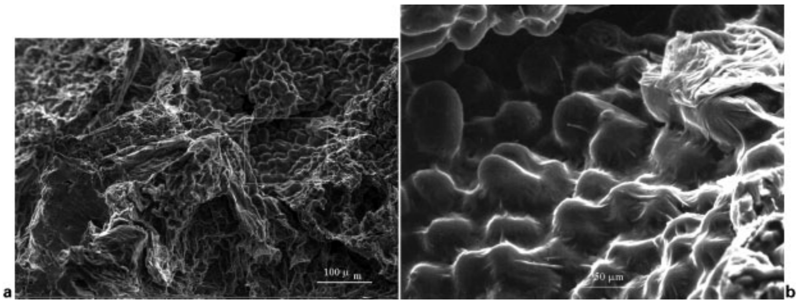
6 Image (SEM) of typical morphology observed at normal fracture surface of casting that suffered little or no surface turbulence during filling: it shows ductile dimpled fracture surface

Oxide film structure

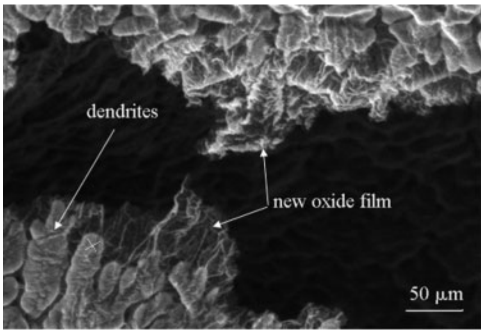
The surface oxide film can clearly be distinguished from the general fracture structure of the samples. Figure 6 shows a typical matrix morphology taken from a normal ductile fracture surface of a sample that had suffered little or no surface turbulence during filling (0·25 m s^{-1}). Figure 7*a* shows the typical mixed fractured structure (i.e. some brittle characteristics) including shrinkage porosity of a sample produced with turbulent flow. A closer view shows secondary dendrite arms covered with new oxide films identified by their folded, wrinkled appearance and very thin thickness (Fig. 7*b*). The rough and wrinkled morphology of oxide film in Mg based alloys has been reported before.[17] The time scale for the formation of these oxide films is in the order of seconds or less since they were most likely formed during mould filling. If, as assumed, these are folded double oxide films, they may opened when subjected to the internal pressure of released hydrogen gas, or a reduction in pressure as a result of shrinkage forces. As they expand apart, the two sides the films are drawn back into the dendritic mesh and thus form an open complex film form. In Fig. 8 it is possible to see where a very thin film has been torn and ruptured. Energy dispersive X-ray examination of the above region (Fig. 9) shows the structure contains four main elements: magnesium, aluminium, oxygen and zinc, with MgO most probably the dominant phase in surface layer.

Table 2 Weibull distribution best fit parameters for each set of castings

In-gate velocity/m s^{-1}	Weibull modulus/β	$-\beta\ln\eta$	R^2
0·25	6·6	36·9	0·96
0·35	2·72	13·7	0·99
0·5	1·8	9·3	0·98
1	1·67	8·01	0·99



7 Images (SEM) of fracture surface of sample that suffered significant surface turbulence during filling: *a* general view and *b* close-up view of marked area from Fig. 7*a* showing shrinkage porosity with secondary dendrite arms covered with fresh (young) oxide film



8 Image (SEM) of fracture surface from casting that suffered significant surface turbulence during filling showing fracture of transparent wrinkled oxide film

Recent investigations have demonstrated that the thickness and morphology of oxide films are affected by chemical composition[11,12] and by the composition of the protective atmosphere used during melting and casting operations.[13] The thickness of the oxide film seems to be an important factor in the formation of many defects in castings. Owing to the formation of a thick and rough oxide film in Mg alloy,[15–17] it can be speculated that the microscopic and macroscopic folds would be expected to retain air and then enter the bulk of the melt by a folding mechanism, so the possibility of porosity formation would be high by forming excellent sites for the precipitation of gas porosity. It is clear that such thick and loose films are likely to cause more damage during preparation of Mg alloy castings compared to the problems caused by films in Al based alloy castings where a dense and protective film forms on the melt surface.

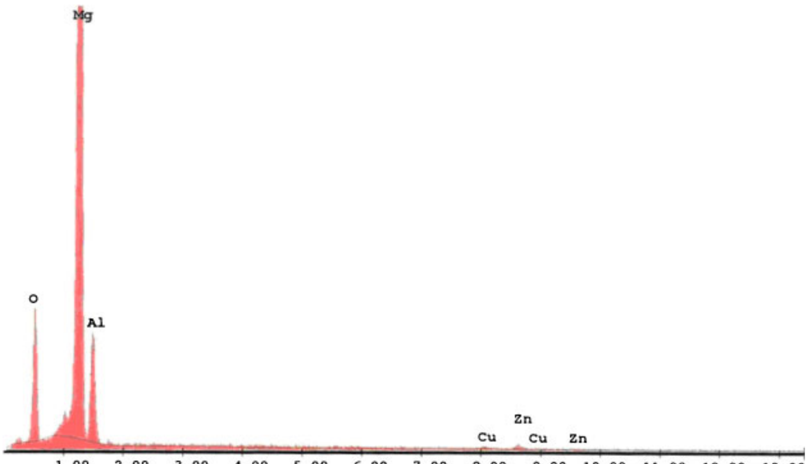
Table 3 shows the Weibull modulus values for Mg and Al alloys using the same method of casting but at slightly different in-gate velocities. As can be seen, the Weibull modulus values of Mg alloys are less than those of comparable Al based alloy castings at the similar in-gate velocities. It is therefore argued that surface turbulence in Mg alloy castings is more destructive to mechanical properties than it is in aluminium alloy castings.

These results confirm the expectation that the formation of new fresh oxide films is closely related to the surface turbulence experienced during filling. A reduction in surface turbulence will reduce the possibility of free surface break-up and the accompanying entrapment of oxide films into the bulk of the melt. It seems that the more deleterious effect in magnesium alloys compared to Al alloys results from the differences in the morphology and probably also the distribution of the entrained oxide films.

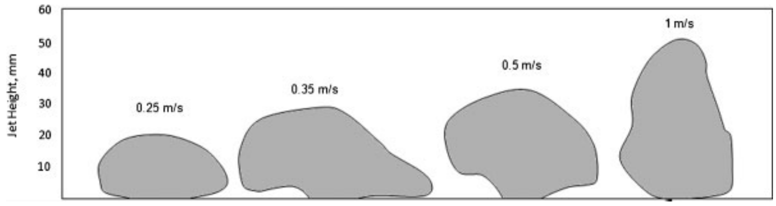
Conclusions

1. There appears to be a critical in-gate velocity for AZ91 alloy in the range of 0·25–0·35 m s^{-1} that reflects a morphological change of the entrance melt front through the in-gate into the mould from a smooth front to a mushroom shape in a 10 mm thick plate casting as shown in Fig. 2.

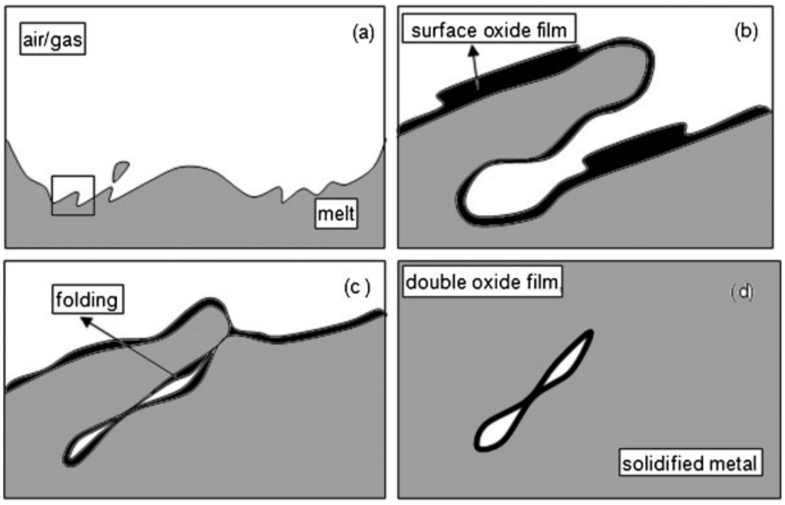
2. The UBS of cast samples reduces sharply in the range of 0·25–0·35 m s^{-1} in-gate velocity. It seems that there is a strong relationship between the degree of surface turbulence during filling and the mechanical properties achieved, in accordance with the flow pattern change in the above range.



9 Typical EDX spectrum taken from area marked in Fig. 8: full scale 4703 cts (1·23 keV 4105 cts)



10 Profiles of liquid magnesium alloy (AZ91) filling fronts at 0·2 s after entry for different in-gate velocities



11 Schematic diagram of entrainment mechanism of double oxide film into bulk of melt

Table 3 Comparison of Weibull moduli of mechanical properties of Al alloy castings[2] and Mg alloy castings (this work) for comparable casting situations

Alloy	In-gate velocity/m s^{-1}	Weibull modulus
Al	0·4	12·4 [2]
	0·7	8·8 [2]
Mg	0·25	6·6
	1	1·6

3. From the frequency histograms and the subsequent Weibull analyses of bending strength of samples, it is clear that as the in-gate velocity increases, the Weibull modulus decreases, meaning increased scatter of mechanical properties and a reduction of reliability of the castings.

4. Fresh young oxide films can clearly be distinguished in the general fracture structure of the samples. These young oxide films are indicated by folds or wrinkles on fracture surfaces that otherwise appear as a homogeneous ductile fracture.

5. Energy dispersive X-ray examination shows that the structure contains four main alloy elements: magnesium, aluminium, oxygen and zinc. It is likely that MgO is the dominant phase in the surface layer.

6. Owing to the formation of thick, folded and wrinkled entrained oxide films in Mg alloys compared to a thin and uniform film in Al based alloy, it seems that they may exert a more deleterious effect on mechanical properties and reliability.

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