The effect of holding time before solidification on the properties of aluminium castings

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Synopsis

The properties of aluminium castings are strongly affected by their inclusion content, particularly double oxide film defects, or bifilms. Such defects have been reported not only to decrease the tensile and fatigue properties of Al casting, but also to increase their variability, making the properties of such alloys unreliable and unreproducible. Earlier research suggested that the bifilm atmosphere might be consumed by reaction with the surrounding melt, which could improve the mechanical properties of the castings. In this work, the effect of holding an Al casting in the liquid state for up to 20 minutes before solidification was studied using a three-level general factorial design of experiments. Two responses were considered, the ultimate tensile strength (UTS) and elongation of the resulting castings. The results showed that the holding treatment had a significant effect on the elongation of the castings produced. In addition, the UTS and elongation peaked at a holding time of 10 minutes. Scanning electron microscopy (SEM) investigation detected many oxide fragments inside pores on the fracture surfaces, reflecting the role of entrained defects in the formation of porosity. The results suggest that two opposing phenomena may take place during the holding treatment. Thus, the consumption of air inside the entrained defects due to reaction with the surrounding molten metal may lead to improvements in mechanical properties, but this may be accompanied by hydrogen passing into the defects, which has a deleterious effect on properties.

Keywords
oxide film, aluminium casting, porosity, defects, hydrogen, design of experiments.

Introduction

The mechanical properties of Al castings are greatly affected by their inclusion contents, especially what is called the double oxide film defect or bifilm (Campbell, 1993; Griffiths, Elsayed, and El-Sayed, 2016; El-Sayed, 2016; El-Sayed, Hassanin, and Essa, 2016a; Basuny *et al.*, 2016). This defect is created due to surface turbulence of the liquid Al, which is a common foundry phenomenon during metal handling, transfer and/or pouring. If liquid aluminium enters a mould cavity with a velocity greater than a critical value, the surface oxide film of the liquid metal will fold over onto itself (but not fuse) and be submerged into the bulk liquid with a volume of air entrapped, creating crevice-like pores with an oxidized interior surface (El-Sayed, 2014, 2018a; El-Sayed and Ghazy, 2017).

Entrained double oxide film defects represent the easiest possible initiating features for cracks and pores, since they are not in atomic contact with the liquid and their dry inner surfaces can be separated with minimal effort (El-Sayed and Griffiths, 2014, El-Sayed, Hassanin, and Essa, 2016b, El-Sayed, 2018b). Also the strength of these features is much lower than the rest of the matrix and the fracture path is expected to preferentially go through them. In addition, double oxide films are considered to be favourable sites for the initiation of pores (Akhtar *et al.*, 2009) and for the nucleation and growth of a wide variety of intermetallic phases (Cao and Campbell, 2003, Asadian Nozari *et al.*, 2015, Samuel *et al.*, 2017). These defects not only reduce the elongation, tensile strength, and fatigue limit of the aluminiumcasting, but also increase their variability (Khaleghifar, Raiszadeh, and Doostmohammadi, 2015; El-Sayed *et al.*, 2011, 2018; El-Sayed, 2012). Oxide films were also suggested to increase the variability in the fluidity of Al melts during the casting process (Timelli and Caliari, 2017).

Nyahumwa, Green, and Campbell (1998) suggested that, due to the transformation of the oxide layer from γ-Al_2O_3 to α-Al_2O_3, (a process thought to take about 5 hours), cracks are introduced into the oxide skin which allow the liquid aluminium to come into contact and react with the atmosphere inside the defect (mainly oxygen and nitrogen). This mechanism could result in the consumption of the atmosphere inside the bifilm and possibly lead to its deactivation (Nyahumwa, Green, and Campbell, 1998b).

This conjecture was further supported by Griffiths and Raiszadeh (2009), who trapped an air bubble (as a proxy for a bifilm) inside liquid Al and monitored its change in volume with time using real-time X-ray radiography. Their results showed that the oxygen in the trapped air should be consumed first, to form Al_2O_3, then the nitrogen would react to form AlN. These reactions started immediately, (with no need for an initiating phase transformation). They also reported an increase in the volume of the bubble when the initial hydrogen content of the melt was higher than the hydrogen equilibrium level associated with the ambient atmosphere, which was suggested to be due to hydrogen diffusion into the bubble (Griffiths and Raiszadeh, 2009). Detailed experiments, using a pore gas analyser to investigate the change in the composition of an air bubble trapped in different Al alloys, demonstrated that hydrogen can diffuse through an oxide layer, showing how a double oxide film defect can grow into a hydrogen-filled pore (El-Sayed *et al*., 2013, 2014; Griffiths, Caden, and El-Sayed; 2014a, 2014b).

Design of experiments (DoE) is a systematic method to help the determination of the relationship between factors affecting a process and the output of that process, and is used to find cause-and-effect relationships. This information is needed to manage process inputs in order to optimize the output. A common experimental design is the three-level design, written as a 3^k factorial design (Croarkin, Tobias, and Zey, 2002). This means that *k* factors are considered, each at three levels. These are (usually) referred to as low, intermediate, and high levels. These levels are numerically expressed as 0, 1, and 2. If there are *k* factors, each at three levels, a full factorial design has 3^k runs.

The purpose of the current study was to explore how the holding time of the casting in the liquid state before solidification (oxide film age) could affect the amount and morphology of bifilm defects, and by implication the tensile properties of the resulting castings. A general factorial design was used for the modelling of the casting process. The results of this investigation could lead to the development of techniques by which oxide film defects might be reduced or eliminated in aluminum castings.

Experimental

Castings from an 1100 Al alloy were produced by gravity casting. To perform the design of experiments, Design-Expert Software Version 7.0.0 (Stat-Ease Inc., Minneapolis, USA) was used and the general factorial design was adopted to analyse the effect of holding time before solidification on the mechanical properties of the Al casting.

The three-level general factorial design was selected because the parameters could be easily varied at three discrete levels and statistically analysed using only three experiments in all, making the DoE easy to regulate and execute due to low complexity. In this work the studied parameter (oxide film age) was varied at ‘0’, ‘1’, and ‘2’ values and its effect on the UTS and elongation was evaluated.

Three experiments were undertaken to produce castings from an 1100 Al alloy containing bifilms of different ages: zero, 10, and 20 minutes. Aluminium ingots were supplied by AluTech - Egyptian Germany Co. The chemical composition of the alloy, certified by the supplier, is given in Table I. The accuracy of the measurements was reported to be ≤0.005 wt.%. In each experiment, about 10 kg of the aluminium was melted in an induction furnace.

Table I

Chemical composition of 1100 Al alloy (wt.%)

Si	Fe	Cu	Cr	Al
0.3	0.6	0.15	0.1	Bal.

The liquid metal was then prepared in such a way as to promote surface turbulence and splashing, by pouring from a height of about 1 m into preheated ceramic shell moulds, and stirring in an induction furnace, using a power setting of 7.5 kW and frequency of 2350 Hz, for one minute. This was intended to create and entrain new bifilm defects.

In one experiment, the casting was allowed to solidify immediately to preserve any bifilm defects created during the melt stirring or mould filling. In the other two experiments, the metal was maintained in the liquid state by placing the filled ceramic shell mould in a resistance-heated furnace for 10 or 20 minutes, then removing it to allow solidification. After solidification, each of the castings was machined into 20 tensile test bars for determination of their ultimate tensile strength and elongation at failure. The tensile tests were conducted with a WDW-100E universal testing machine using a crosshead velocity of 1 $mm.min^{-1}$. To evaluate the H content of the castings, a LECO sample was cut out from the solidified castings from each experiment. The samples were machined to the dimensions of standard Leco samples (8 mm diameter and 49 mm length), and analysed to determine the hydrogen content of the castings from the different experiments. Finally, scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) studies were carried out on the fracture surfaces of the tensile test specimens using a Philips XL-30 scanning electron microscope with Oxford Inca EDS, for the evidence of any bifilms.

Results

The H content of the solidified castings from these experiments was determined to be 0.22, 0.3, and 0.51 cm^3 per 100 g respectively for the castings that contained oxide films aged for zero, 10, and 20 minutes. The error in the hydrogen measurement was shown in an earlier study to be in the range of 0.002 cm^3 per 100 g (El-Sayed and Griffiths, 2014). The values of ultimate tensile strength (UTS) and per cent elongation for specimens cut from the solidified castings are presented in Table II.

Figure 1 presents a normal probability plot of the residual, and a plot of the residual against experimental run order for the UTS values. A normal probability plot of the residual, and a plot of the residual against run order, for the per cent elongation values are shown in Figures 2a and 2b respectively. The observed points on both normal probability plots were distributed relatively near to the straight line. In addition, the residuals *versus* the experimental run order plots showed random scatter without any specific trend. This could suggest that the residuals followed a normal distribution and the observations were independently distributed for the two responses evaluated in this study.

Figures 1 and 2 show a high outlier (out of the 60 values) for the UTS and elongation (62 MPa and 47 respectively), which is indicated by arrows. This is a typical demonstration of the random nature of the bifilm effect on mechanical properties, as by chance a sample from the casting might be free from bifilms with a corresponding highly improved UTS and/or elongation. In an earlier study (Nyahumwa, Green, and Campbell, 1998a), it was reported that Al castings that were free from bifilm defects exhibited up to 100 times higher fatigue lives.

Table II

UTS and per cent elongation of tensile specimens from Al castings held for different periods in the liquid state prior to solidification

Sample No.	Holding time (minutes)					
	Zero		10		20	
	UTS (MPa)	Elongation %	UTS (MPa)	Elongation %	UTS (MPa)	Elongation %
1	48	30	55	35	49	29
2	54	31	54	36	43	22
3	55	33	50	39	57	25
4	46	34	50	36	56	19
5	50	35	56	35	51	23
6	53	27	59	30	54	31
7	56	34	53	34	62	29
8	49	21	55	36	54	23
9	45	30	52	31	47	32
10	57	25	58	31	46	28
11	51	27	54	38	55	27
12	50	39	57	33	53	25
13	47	40	54	32	56	28
14	53	34	53	40	45	29
15	54	32	58	39	50	24
16	55	47	49	36	52	31
17	52	31	53	36	52	31
18	42	28	57	36	51	32
19	56	26	51	37	48	33
20	56	28	48	36	58	30
Average	51.5 ± 4.2	31.6 ± 5.9	53.8 ± 3.1	35.4 ± 2.8	52 ± 4.8	27.6 ± 3.9

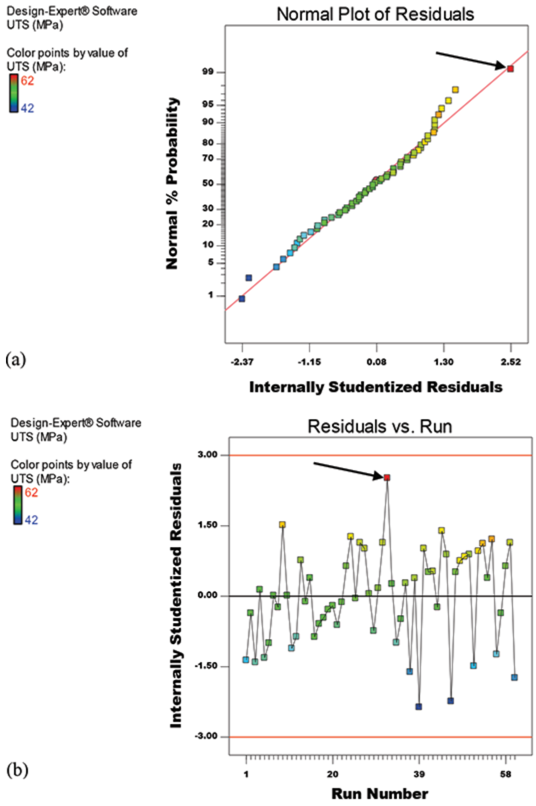

Figure 1—(a) Normal probability plot of the residuals, and (b) residuals against run order, of UTS

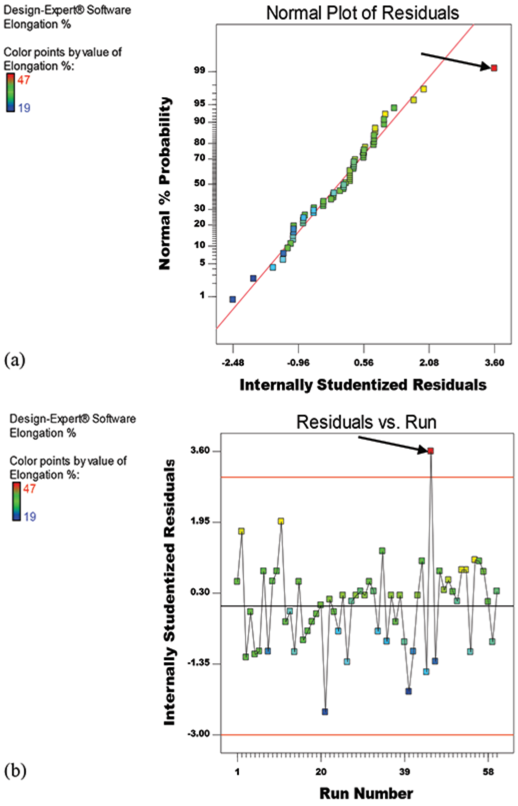

Figure 2—(a) Normal probability plot of the residuals, and (b) residuals against run order, of elongation per cent

Analysis of UTS

Table III shows the analysis of variance results for the UTS values shown in Table II. The 'Model F-value' of 1.81 implies that the model is not significant relative to the noise. In other words, there was a 17.23% chance that a 'Model F-value' this large could occur due to 'noise'. Therefore, the results of the model suggested that the age of the oxide films had no significant effect on the UTS of the castings produced.

Table III

ANOVA results, where values of '*p*-value' less than 0.05 indicate that model terms are extremely significant, and values greater than 0.1 indicate the model terms are not significant

Source variation	Sum of squares	Degrees of freedeom (df)	Mean square	F-value	*p*-value
Oxide Film age	60.90	2	30.45	1.81	0.1723
Error	956.98	57	16.79		
Corresponding Total	1017.89	59			

Table IV

ANOVA results, where values of *p*-values less than 0.05 indicate model terms are extremely significant and values greater than 0.1 indicate the model terms are not significant

Source of variation	Sum of squares	Degrees of freedeom (df)	Mean square	F-value	*p*-value
Oxide Film age	621.15	2	310.58	16.13	<0.0001
Error	1097.54	57	19.26		
Corresponding Total	1718.70	59			

Figure 3 shows the effect of oxide film age on the mean UTS of the test bars cut from different castings (with different holding times before solidification). The average UTS increased from 51.5 ± 4.2 MPa (solidified immediately) to 53.8 ± 3.1 MPa (held in the liquid state for 10 minutes before solidification). However, as the holding period increased to 20 minutes, the mean UTS decreased to 51.9 ± 4.8 MPa. Although this effect was suggested to be statistically insignificant, it might be an indication of a slight improvement in the UTS at a holding time of 10 minutes.

Analysis of elongation

Table IV shows the analysis of variance results for the elongation (%) values shown in Table IV. The Model F-value of 16.13 implies that the model was significant. In other words, there was less than 0.01% chance that a Model F-value this large could occur due to 'noise'. Therefore, the results of the model suggest that the age of the oxide films had a notable on the elongation of the castings produced.

Figure 4 shows the effect of oxide film age on the per cent elongation of the test bars cut from different castings (with different holding times before solidification). The age of oxide films showed an effect on the per cent elongation of the Al castings, with a peak at 10 minutes' holding time. Like the UTS, the mean per cent elongation of the castings maintained at 800°C for 10 minutes prior to cooling was higher than for those solidified immediately or held in the liquid state for 20 minutes before solidification.

SEM examination (Figures 5a–f) of the fracture surfaces of different samples from different conditions (holding times), did not detect oxide films lying on the fracture surfaces. Instead they were often detected inside pores. The identity of the alumina film was confirmed by EDX analyses that detected relatively large amounts of oxygen (indicated by the atomic per cent, which ranged from 20 to 32) at the suspected bifilm layers This is a likely indication that the origins of the pores were primarily bifilm defects, which might have changed their morphologies during the holding treatment and/or solidification of the casting to form pores. This would be an indication of the role played by bifilms in initiating porosity in light metal alloy castings. It should be emphasized that the roughness of the fracture surfaces of test bars might affect the accuracy of EDX analysis carried out in this study.

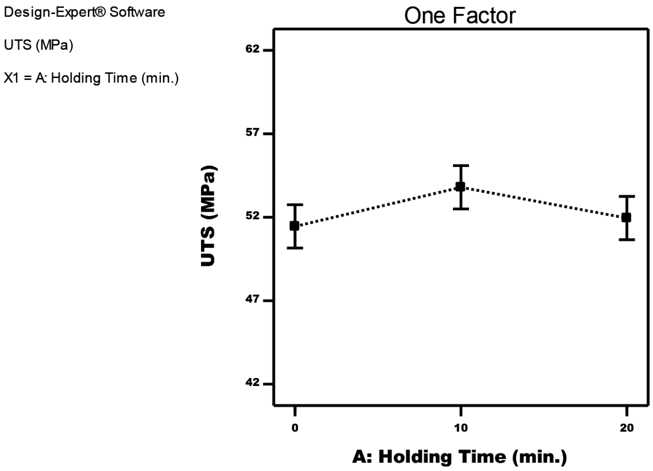

Figure 3—Effect of holding time on the UTS of the Al alloy

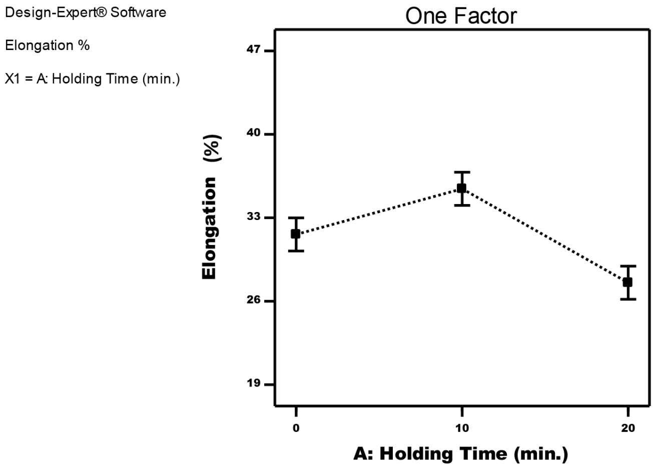

Figure 4—Effect of holding time on the elongation (%) of the Al alloy

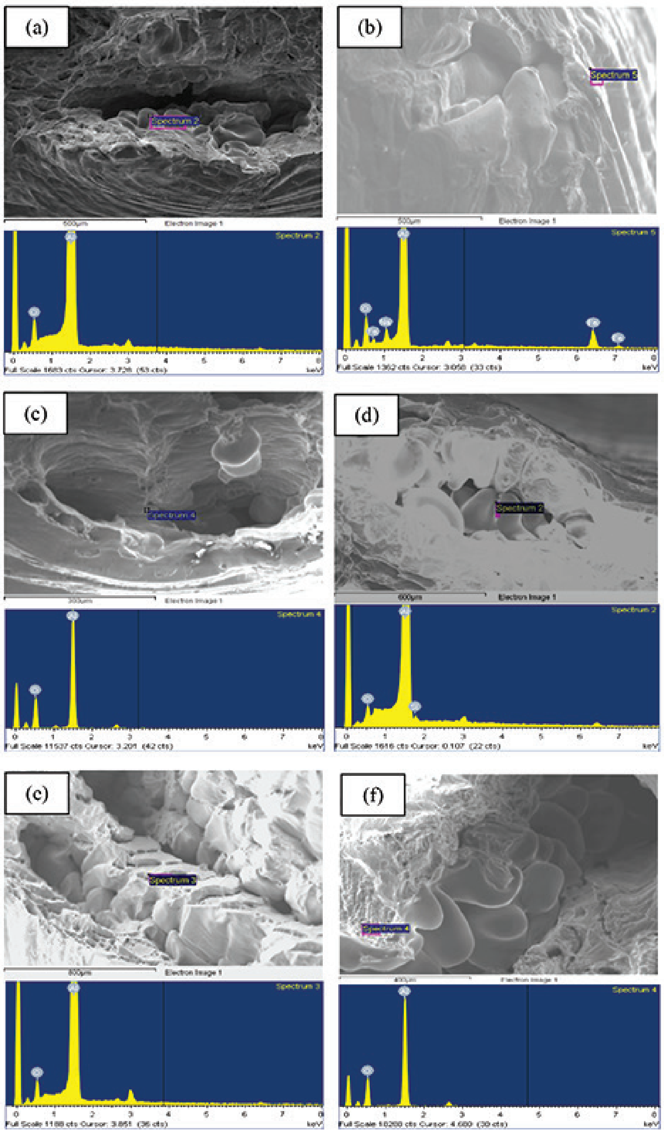

Figure 5—Secondary electron SEM images of pores containing oxide film defects on the fracture surfaces of Al alloy specimens that were held in the liquid state for different periods before solidification, (a) and (b) zero, (c) and (d) 10 minutes, and (e) and (f) 20 minutes

Discussion

The modelled graphs of the holding time *versus* the UTS and elongation (Figures 3 and 4), show that both the mean UTS and mean percentage elongation peaked when the castings were kept in the liquid state for 10 minutes before solidification. The mean values dropped (much more for elongation) when the holding period was increased to 20 minutes. Also, the ANOVA results, presented in Tables III and IV, suggested a significant effect of the holding time on the elongation of the casting produced. Finally, the hydrogen content of the solidified casting was found to increase consistently with the holding time (an increase of about 230% during the 20-minutes holding time).

Raiszadeh and Griffiths (2006) and El-Sayed *et al.* (2013) suggested that the interior atmosphere of a bifilm could be consumed within a few minutes due to reaction with the surrounding liquid Al. This would reduce the size of bifilm defects and hence reduce their deleterious effects on the mechanical properties of the Al castings. In addition, it was also shown that the excess hydrogen that was ejected during solidification might diffuse into the bifilm gap, increasing its size to form a pore which affects the properties adversely (El-Sayed *et al.*, 2014).

The current results suggest that two opposing mechanisms were taking place simultaneously during the holding of an Al casting in the liquid state prior to solidification and/or the solidification process itself (suggested to take about 4 minutes – El-Sayed and Essa, 2018).

i. The first mechanism was related to the reaction between oxygen and nitrogen within the bifilm defect and the contiguous melt, which decreases the bifilm size and in turn improved the mechanical properties.
ii. On the other hand, another mechanism occurs which may be related to the amount of hydrogen picked up by the liquid metal from the surrounding atmosphere (which increased as the molten metal was spending more time in the liquid state before being completely solidified). This hydrogen could diffuse into the bifilms and this would lead to degradation of the overall mechanical properties due to increased porosity.

It could be argued that the mean UTS and elongation values of the castings allowed to solidify immediately after pouring represented the tensile properties of castings with bifilms that were beginning to lose their internal atmosphere, but also had the lowest H levels. During the early stages of holding the first mechanism, consumption of air inside the bifilms, was more prevalent, and there was an improvement in the properties, which reached a maximum at 10 minutes' holding. At this time, bifilm defects may have lost most of their oxygen and nitrogen by reaction with the surrounding liquid Al, while the H content increased by only a small amount. Consequently, the shape of the double oxide films would be expected to be less deleterious to the mechanical properties. However, increasing the holding time to 20 minutes did not cause any further improvement in the mechanical properties as most of the initial atmosphere of bifilms might have already been consumed. On the contrary, the extended holding resulted in a degradation of the properties due to an increasing H content of the melt and hence an increasing H content of the double oxide films and of the porosity size in the final casting.

Therefore, it could be suggested that these two opposing mechanisms during the holding treatment may cancel one another over the 20-minute holding period, hence the average values of the UTS of the castings with zero and 20-minutes holding times were almost the same (51.5 ± 4.2 and 51.9 ± 4.8 MPa, respectively). In addition, the second mechanism, related to H diffusion into the bifilm gap, appeared to have a more harmful effect on the elongation, as the average values of the elongation of the Al castings decreased from 31.6 ± 5.9% (solidified immediately) to 27.6 ± 3.9% (held for 20 minutes before solidification). However, taking the errors into account, these values were relatively close.

SEM detected numerous alumina films, demonstrated by EDX, where such films, were mostly associated with pores (Figure 5). Griffiths and Raiszadeh (2009) suggested that hydrogen in the Al melt could diffuse into the bifilm, causing its expansion into a pore, which might be subsequently torn apart (due to its extreme thinness), leaving only some oxide fragments inside the pore. Therefore, it could be speculated that these oxide-related pores may have been formed due to the diffusion of H from the melt into bifilms. This could be in accordance with the current results, which suggested that the diffusion of hydrogen into the bifilms increases the size of the defects and hence detracts from improvements in the mechanical properties of the castings resulting from the consumption of the bifilm atmosphere.

Since the results of the current study suggested a relationship between bifilms and porosity in Al castings, it is recommended that a quantitative assessment of the size and volume fraction of pores as a function of the holding time be conducted. This could be achieved by determination of a parameter called the ’Bifilm index’, which is defined as the total length of bifilms estimated on the surface of a reduced pressure test (RPT) sample (Dispinar and Campbell, 2006). This might be helpful in the correlation of holding time of the casting in the liquid state before solidification and the amount and size of bifilms and/or pores on the surface of the casting.

Conclusions

1. SEM examination of the fracture surfaces revealed the presence of oxide films, which demonstrated a role for such defects in influencing the failure of Al castings.
2. The tensile properties of Al castings peaked when they were held in the liquid state for 10 minutes before solidification. This might be attributed to a change in the morphology of their entrained bifilms during the holding treatment, with a corresponding change in their harmful influence on the properties.
3. Holding Al castings in the liquid state for 20 minutes before solidification did not cause any significant change in the UTS, while it reduced the elongation of the castings produced by about 12%.

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