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## **Optimization of the cooling slope casting parameters for producing** aa7075 wrought aluminum alloy thixotropic feedstock

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## ABSTRACT

Thixoforming technology requires a feedstock with a globular microstructure rather than dendritic microstructure used in conventional casting methods. In the present investigation, several AA7075 wrought aluminum alloy feedstock were produced using cooling slope (CS) casting technique at different fabrication conditions. Optimization of the CS castingprocess parameterswas conducted to find out the optimum conditions that achieve the best microstructural characteristics of the feedstock. Moreover, correlations for microstructural characteristics as functions of CS casting process parameters were determined. The results revealed that the optimum values of pouring temperature, cooling length and tilt angle were found to be 650 °C, 350 mm and 45°, respectively. Billets fabricated under such conditions showed the minimum average size as well as the maximum shape factor of  $\alpha$ -Al primary grains. The pouring temperature is the most influential parameter on both the average grain size and shape factor of the primary α-Al grains. The developed empirical correlations were successfully used to predict the average grain size and shape factor of the AA7075 alloy billets produced using CS casting technique. © 2016 Trade Science Inc. - INDIA

### **INTRODUCTION**

Thixoforming is a viable technology for forming alloys in semisolid state to near net-shaped products<sup>[1]</sup>. This method presents a solution to the problems associated with both conventional casting and metal forming processes due to its capability to use temperatures lower than those used in metal casting and a less energy used in metal forming as conventional forging and extrusion processes<sup>[2-4]</sup>. It has been

reported that parts produced by thixoforming has lower defects and shrinkage porosity, higher cross sectional changes, better weldability and longer tool life<sup>[2,3]</sup>. Thixoformed parts are now in everyday use in cars and automotive industry. The thixoforming route consists of three steps; (i) producing a special feedstock with a non-dentritic microstructure, (ii)reheating the metal to the forming temperature, and (iii) forming the metal in a die-casting machine. The success of any thixoforming process depends

## KEYWORDS

Cooling slope casting; Wrought aluminum alloys; Microstructure.

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on the producing of a specialfeedstock that has a fine globular particles of the solid phase surrounded by a continuous film of liquid. Several methods have been developed to produce such feedstocks, for example, magneto-hydrodynamic (MHD) stirring, mechanical stirring, chemical grain refinement and strain induced melt activated (SIMA)<sup>[2,3]</sup> and cooling slope (CS) casting. The latest techniqueis cheap, simple and needs low equipment and running costs<sup>[5-7]</sup>. In the CS casting, amolten alloy with an amountof superheat is cast over an inclined cooling plate. The prepared billetexhibits, when reheated to the semisolid temperaturerange, a non-dendritic, globular microstructure suitablefor thixoforming.

Commercially, thixoformingismainly applied to the cast aluminum alloys such as A356 and A357 due to their good fluidity and "castability"<sup>[5-7]</sup>. However, such alloys do not have as high mechanical properties as the wrought alloys such as AA2024 and AA7075 used widely in aerospace applications. One of the challenges for thixoforming is to process alloys which would otherwise be wrought and have higher performance than the casting alloys. Successful thixoforming of these alloys would serve to strengthen the market potential of semi-solid processing and would address the urgent need in the aerospace industry for near net shape and high strength aluminium products<sup>[8]</sup>. The difficulties in thixoforming of wrought aluminum alloys center on the high sensitivity of the liquid fraction to temperature fluctuation which can lead to hot tearing and make the quality of the slurry unstable<sup>[2,3,8,9]</sup>.

The AA7075 aluminum alloy is a wrought Al-Zn-Cu-Mg alloy that provide highest strength among all aluminum alloys. It has been reported that the AA7075 alloy was successfully formed by semi solid processing<sup>[10,11]</sup>. The aim of the present investigation is to study the significance of the CS casting process parameters, typically, pouring temperature, cooling length and slope angle on the microstructural characteristics of AA7075 wrought aluminum alloy. The 3-levels factorial design of experiment (DOE) and analysis of variance (ANOVA) techniques wereconducted to find out the optimum CS casting process parameters to achieve the best microstructural characteristics for the produced billets. The ANOVA is a statistical technique that quantitatively estimate the relative contribution of each control factor on the overall measured response<sup>[12]</sup>. In the present work, optimization was conducted to find out the best combination of CS casting parameters to achieve the maximum globularity and minimum grain size of primary alpha-aluminum grains.



#### TABLE 1 : Chemical composition of AA7075 aluminum alloys (wt%)

Figure 1 : DSC and liquid weight fraction versus temperature curves for AA7075 wrought Al alloy



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## EXPERIMENTAL PROCEDURES

In the present work, the AA7075 Al-Zn-Cu-Mg wrought alloy was used. Thealloy has a chemical composition shown in TABLE 1. Figure 1 shows the differential scanning calorimetric (DSC) analysis of the AA7075 alloy. The DSC analysis was conducted to determine the solidus and liquidus temperatures of the alloy as well as the variation of melt liquid percent with temperature. The DSC experiments were carried during heating with a heating rate of 5 °C/min. Figure (1) shows that the

liquidus and solidus temperatures of the alloy are 617 and 473 °C, respectively. The figure shows also the curve representing the variation of liquid weight fraction with the temperature. This curve was obtained after integrating the area under DSC curve.

The CS casting process was carried as follows: About 0.9 kg of the AA7075 alloy was melted in graphite crucible in an electric resistance furnace at 680 °C. The molten metal was then degassed by argon gas followed by skimming of the oxides formed. After that, the molten metal was allowed to cool down to the specified pouring temperature. The mol-





Figure 3 : A schematic illustration of theAA7075 wrought Al alloy CS casting billet showing its main dimensions and the positions of the metallographic specimens. (Dimensions in mm)





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TABLE 2 : Independent factors and their levels for DOE of CS casting process



ten metal was poured immediately on an inclined preheated low carbon steel plate at different slope angles with respect to horizontal and different slope lengths, and finally collected into a preheated steel mould. The slope plate is coated with thin layer of hard Chromium in order to avoid sticking of the molten metal on the slope plate and to facilitate a trouble free melt flow. The steel mouldhad a diameter of 50 mm and height of 160 mm with adraft angle of 2° for easy removal of the solidified billet. Figure 2 shows a photograph of the CS casting pro-

cess.

Figure 3 shows a schematic illustration of the sample billet produced from the CS casting process. After solidification, the upper part of the billetcontaining the shrinkage cavity was removed. The remained part in each billet is sectioned horizontally into discs, having 50 mm diameter and 5 mm thick, from the top, middle, and bottom sections as shown in Figure 3. Samples cut from the CS castingbillets were prepared for metallographic examination. The specimens were ground using emery

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Mean of SNratios



Figure 5: The main effect plots of the SC casting parameters on the average (a) size and (b) shape factor of the primary  $\alpha$ -Al grains



Main Effects Plot for SN ratios Of Shape Factor Size Of AA7075 Data Means



Figure 6 : Main effect plot for S/N ratios of average (a) grain size and (b) shape factor

papers of increasing finesse up to 1200 grit then they were polished using 10 mm alumina suspensionb. The specimens were etched using Keller's etchant (2 ml Hydrofluoric acid, 3 ml Hydrochloric acid, 5 ml Nitric acid, and 190 ml distilled water). The time for each specimen was between 5-10 seconds. The metallographic images were taken using optical microscope from the edge zone (radius), mid-radius zone, and center zone of the specimens as shown in

Figure 3. Measurements of the size (GZ) and shape factor (SF) of the primary ±-Al grains were carried out using image-analyzing techniques. The average size of ±-Al grains were measured by the linear intercept method according to the ASTM-E112-9619. The shape factor was determined from the following equation:

$$SF = 4\pi A/P^2$$

(1)

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Where: *P* is the perimeter and *A* is the area of  $\pm$ -Al grain. For a perfect circle, the shape factor would be one.

To explore the effect of the CS casting process parameters on the microstructure of the AA7075 wrought Al alloy, the 3-levels (3<sup>3</sup>) factorial design of experiment (DOE) technique was performed. The  $(3^3)$  factorial design means that there are three factors of interest and each factor is set at three levels (minimum, mean and maximum). In an  $(3^3)$  factorial design, the total number of experiments to be conducted is 27. Factorial design allows for the simultaneous study of the effects that several factors may have on a CS casting process, and also allows for the study of interactions between these factors. An interaction is the failure of the one factor to produce the same effect on the response at different levels of another factor<sup>[13]</sup>. Without the use of factorial experiments, important interactions may remain undetected. Pouring temperature (T), pouring length (L), and slope angle  $(\theta)$  were selected as independent factors. The range of values and coded levels of the factors are given in TABLE 2. The MiniTab commercial software was used to design and analyze the experiments by using analysis of variance (ANOVA) procedure. ANOVA is a useful statistical method in analysis of the effect of CS casting process parameters and their interactions on the microstructure of α-Al grains. ANOVA can exhibit the most and lowest significant process parameter affecting the microstructure. It is clear from Figure 3 that, in the present study, the grain size and shape factor measurements were taken at nine different regions of each AA7075 billet and the average value was calculated. Moreover, the S/N ratio was calculated using the average values by considering the quality characteristics the "larger-the-better" and "smaller-thebetter" for the shape factor and grain size of  $\alpha$ -Al, respectively. The S/N ratio can be calculated using the following equation:

$$\frac{S}{N} = -\mathbf{10} \log \left( \frac{1}{n} \sum_{i=1}^{n} y_i^2 \right)$$

(2)

Where n is the number of measurements (in the present investigation n=9) in a trial and  $y_i$  is the  $i^{ih}$  response value (shape factor and grain size) for each

Materials Science Au Indian Journal noise repetition of measurement and subscript i indicates the number of design parameters in the which is 27.

The polynomial equation (Eq. (2)) was used to predict the response (Y) as a function of independent factors and their interactions. In this work, the number of independent factors is 2, therefore, the response for the quadratic polynomials becomes:  $Y=A_0 + A_1\Sigma X_1 + A_{ii}\Sigma X_1^2 + A_{iii}\Sigma X_1 X_{ii} +$  $A_{iv}\Sigma X_3^2 X_1 + A_v X_2^2 X_3^2$  (3) Where:  $A_0$ ,  $A_{i,} A_{ii,} A_{iii,} A_{iv}$  and  $A_v$  are constant, linear, square and interaction regression coefficient terms, respectively;  $X_1$ ,  $X_2$  and  $X_3$  are the independent factors. The goodness of fit of the model was evaluated by the coefficient of determination ( $\mathbb{R}^2$ ). The closer the R-squared values closer to 1, a better fitting is achieved.

### **RESULTS AND DISCUSSION**

Figure 4 shows typical micrographs of the microstructure of the AA7075 billets produced using different CS cast conditions. The shown microstructure are captured from the bottom and radius positions for billets poured at constant pouring temperature of 650 °C, constant tilt angle of 30° and several cooling lengths. It is clear that then ondentritic  $\alpha$ -Al primary grains are appeared throughout the billet. Variation in the size and shape of  $\alpha$ -Al primary grains in the both radial and axial directionshave been observed in the cast billets. Such variation may attribute to the existence of cooling rate gradient across the billet during solidification in the mild steel mold. The radius positions of the billets showed finer primary  $\alpha$ -Al grains than the mid-radius and center zones. The effects of the CS casting process parameters on both the average size and shape factor of  $\alpha$ -Al primary grains for the AA7075 billets are illustrated in Figure 5. It has been found that increasing the pouring temperature from 630 to 650 °Creduces the average size of the  $\alpha$ -Al grains. Further increase in the pouring temperature above 650 °Cto 670 °C increasing significantly the average grain size. Increasing the tilt angle from 30 to 45° and/or the cooling length from 200 to 350 mmslightly reduce(s) the average size of the  $\alpha$ -Al grains. Further increase in



Source	DF	Seq SS	Adj SS	Adj MS	F	р	Pc
Т	2	7492.33	7492.33	3746.16	203.03	0	67.32
θ	2	34.58	34.58	17.29	0.94	0.393	0.31
L	2	183.99	183.99	92	4.99	0.008	1.65
T×L	4	1216.36	1216.36	304.09	16.48	0	10.93
$T{\times}\theta$	4	640.68	640.68	160.17	8.68	0	5.75
$L \times \theta$	4	547.44	547.44	136.86	7.42	0	4.92
$T\!\!\times\!\!L\!\!\times\!\!\theta$	8	1013.63	1013.63	126.7	6.87	0	9.12
Error	216	3985.4	3985.4	18.45			
Total	242	11129.01	,	,		,	100

TABLE 3 : ANOVA table for the average size of the α-Al primary grains

DF, degrees of freedom; SS, sum of squares; MS, mean square; F, F-test; P, Statistical significance, P, percentage of contribution.

TABLE 4 : ANOVA table for	the average shape facto	or of the α-Al primary	grains
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Source	DF	Seq SS	AdjSS	Adj MS	F	р	Pc
Т	2	0.018729	0.018729	0.009365	74.17	0	47
θ	2	0.001513	0.001513	0.000757	5.99	0.003	3.8
L	2	0.001207	0.001207	0.000604	4.78	0.009	3
T×L	4	0.006041	0.006041	0.00151	11.96	0	15.18
$T{\times}\theta$	4	0.001371	0.001371	0.000343	2.72	0.031	3.45
$L \times \theta$	4	0.003257	0.003257	0.000814	6.45	0	8.18
$T{\times}L{\times}\theta$	8	0.007679	0.007679	0.00096	7.6	0	19.39
Error	216	0.027272	0.027272	0.000126			
Total	242	0.039798					100

DF, degrees of freedom; SS, sum of squares; MS, mean square; F, F-test; P, Statistical significance, P,, percentage of contribution

the tilt angle to 60° and/or the cooling length to 500 increase(s) the average grain size of the primary grains.

The average shape factor of the  $\alpha$ -Al primary grains was found to be increased with increasing the pouring temperature from 630 to 650 °C. Further increase in the pouring temperature from 650 to 670 °C reduces the average shape factor. Similarly, increasing the tilt angle from 30 to 45° and/or the cooling length from 200 to 300 mm increase(s) the average shape factor. Further increase of the tilt angle from 45 to 60 and/or the cooling length from 350 to 500 mm reduce(s) the average shape factor of the  $\alpha$ -Al primary grains. Figure 6 shows the response graphs by the factor level for the S/N ratios of the AA7075 alloy billets produced using CS casting technique. For the grain size, the lower S/N value, the higher performance acceptance, while for the shape factor the higher S/N value, the higher performance acceptance. Based on the analysis of S/N ratios, the optimal microstructural characteristics are obtained at pouring temperature of 650°C, tilt angle of 45°, and cooling length of 350 mm.

TABLES 3 and 4 list the ANOVA results for the average size and shape factor of the  $\alpha$ -Al primary grains. The last column in each table shows the percentage of contribution (P<sub>2</sub>) of each factor on the total variation, indicating the influence of the factors on the results. The higher the value of the P<sub>2</sub>, the more statistical and physical significant the factor is. The results indicated that the pouring temperature has the highest statistical physical significance on the grain size and shape factor of the  $\alpha$ -Al primary grains. The P values for the pouring temperature were about 47% and 67.32% for the average size and shape factor of  $\alpha$ -Al primary grains, respectively. Both of the tilt angle and cooling length parameters exhibited significantly lower values of  $P_c$ . For example, the  $P_c$  values for the tilt angle were about 0.31% and 3.8% for the average size and shape factor of  $\alpha$ -Al primary grains, respectively. The interaction (T×L× $\theta$ ) between the pouring temperature





Figure 7 : Plots of the predicted verses measured (experimental) (a) average size and (b) average shape factor of the α-Al primary grains

(T), tilt angle ( $\theta$ ) and the cooling length (L) exhibited lower exhibited lower statistical and physical significance when compared with the pouring temperature. The P<sub>c</sub> values for the T×L× $\theta$  interaction were about 9.12% and 19.39% for the average size and shape factor of  $\alpha$ -Al primary grains, respectively. The interaction between the pouring temperature (T) and the cooling length (L) exhibited also lower statistical and physical significance when compared with the pouring temperature. The P<sub>c</sub> values for the T×L interaction were about 10.93% and 15.18% for the average size and shape factor of  $\alpha$ -Al primary grains, respectively.

The Empirical expressions of the grain size (GS) and shape factor (SF) were established as functions of the CS casting process parameters; typically, pouring temperature (T), tilt angle ( $\theta$ ) and cooling length (L) is given below:

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 \begin{split} & \mathrm{GS}{=}7415.54 - 23.28 \ \mathrm{T} - 1.73 \ \theta + 0.80 \ \mathrm{L} - 1.25 \times 10^{-3} \\ & \mathrm{T} \ \mathrm{L} + 4.22 \times 10^{-4} \ \theta \ \mathrm{L} + 1.85 \times 10^{-2} \ \mathrm{T}^2 + 1.97 \times 10^{-2} \ \theta^2 - 3.07 \times 10^{-4} \ \mathrm{L}^2 + 3.67 \times 10^{-7} \ \mathrm{L}^2 \ \mathrm{T} + 4.11 \times 10^{-6} \ \mathrm{L}^2 \ \theta \\ & -5.73 \times 10^{-8} \ \theta^2 \ \mathrm{L}^2 \\ & \mathrm{GS}{=} 1.518 - 6.97 \times 10^{-4} \ \mathrm{T} + 4.23 \times 10^{-3} \theta - 1.37 \times 10^{-3} \\ & \mathrm{L} + 3.47 \times 10^{-6} \ \mathrm{T} \ \mathrm{L} - 10^{-5} \ \theta \ \mathrm{L} - 9.38 \times 10^{-7} \ \mathrm{T}^2 - 6.28 \\ & 10^{-6} \ \theta^2 - 3.95 \times 10^{-7} \ \mathrm{L}^2 - 1.096 \times 10^{-9} \ \mathrm{L}_2 \ \mathrm{T} + 2.43 \times 10^{-8} \ \mathrm{L}^2 \ \theta - 4.47 \times 10^{-11} \ \theta^2 \mathrm{L}^2 \end{split}
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The regression analysis revealed that the coefficient of determination ( $R^2$ ) of equations (4) and (5) are 0.991, 0.960, respectively. A comparison of the measured average grain size and average shape fac-

Materials Science An Indian Journal tor (experimental data) against the predicted average grain size and shape factor is shown in Figure 7. A perfect prediction would be when all the plotted points were on the 45° line (the dashed line). The accuracy of equations (4) and (5) can be easily compared by the closeness of the data points to this line. It is clear from Figure 7 that the experimental and predicted values are very close to each other.

### CONCLUSIONS

### Based on the results presented, the following conclusions can be drawn

The optimum values of pouring temperature, cooling length and tilt angle were found to be 650 °C, 350 mm and 45°, respectively. Billets fabricated under such conditions showed the minimum average size as well as the maximum shape factor of  $\alpha$ -Al primary grains.

The pouring temperature has highest statistical and physical significance when compared with the tilt angle and the cooling length on both the average size and shape factor of the primary  $\alpha$ -Al grains.

The correlation coefficients of the developed regression models for the average grain size and average shape factor are 0.991 and 0.960, respectively, which confirms the effectiveness of the developed models.

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