ISSN : 0974 - 7486 Volume 14 Issue 12

Materials Science

An Indian Journal

Full Paper

MSAIJ, 14(12), 2016 [494-502]

Effect of grain refining, mould material and pouring temperature on the microstructural characteristics of AA6063 aluminum alloy feed-stocks produced using low superheat casting

A.Abu-El-Fadl, El-Awady Attia, E.H.Mansour, A.M.Gaafer, T.S.Mahmoud*

Mechanical Engineering Department, Shoubra Faculty of Engineering, Benha University, Cairo, (EGYPT)

E-mail: tamer.abdelmagid@feng.bu.edu.eg

ABSTRACT

In the present investigation, feedstocks of AA6063 wrought aluminum alloy, with a thixotropic microstructure, were prepared using low superheat casting (LSC). The effects of pouring temperature, mould material and the grain refining using Al-5%Ti-1%B master alloy on the microstructural characteristics of AA6063 wrought Al alloy were studied. An optimization of the aforementioned parameters was carried out using Taguchi's approach. Moreover, the analysis of variance (ANOVA) statistical approach was carried out to clarify the level of significance of the parameters and their optimum combination. The results revealed that the grain refining has the greatest influence on both the average grain size and shape factor of the primary α-Al grains. Feedstocks poured using the grain refiner exhibited lower size and higher shape factor of α-Al grains than those poured without grain refining. Increasing the pouring temperature slightly increases the average size while reduces the average shape factor of the primary α-Al grains. Feedstocks poured in the copper mould exhibited the lowest average size and highest average shape factor of primary α-Al when compared with feedstocks poured in low carbon steel and stainless steel moulds. The optimum microstructural characteristics suitable for thixoforming was exhibited for grain refined AA6063 feedstocks poured in the copper mould at 665^{o}C.

KEYWORDS

Low superheat casting (LSC);
AA6063 wrought aluminum alloy;
Microstructure.

INTRODUCTION

Thixoforming and thixocasting are semi-solid processing(SSP) technologies combining the near-net-shape capabilities of die-casting and mechanical properties of forging[1-2]. Semi-solid processing has made a significant impact in a number of industries including aerospace,automotive and electronic components[3], due to the high integrity of the castings with excellent mechanical properties and reduced levels of porosity and other solidification defects. While thixoforming offers significant advantages compared with traditional metal forming methods, the process comes with the requirement for a special feedstock[4–8]. Thixoforming uses semisolid slurries with globular solid particles uniformly suspended in a liquid matrix, which can be handled as a solid when at rest and flow like a liquid when sheared during the forming operation[9]. The AA6063 wrought aluminum alloys are heat treatable with moderatelyhigh strength, excellent corrosion resistance and good extrudability. They are regularly used as architectural and structural members and also in automotive industries[10]. Aluminum alloys (6000 series) are widely used for demanding structural applications due to their lightweight with good mechanical properties[11-14]. Unfortunately, it is very difficult to use such alloys in SSP due to their narrow solidification range.

Low superheat casting (LSC) technique is one for reducing energy consumption and environment pollution to produce feedstocks for thixocasting and rehocasting with thixotropic (non-dendritic) microstructure and high mechanical properties[15,16]. This technique has several advantages, for example, simple, cheap, low production cycle time, increased die life, and reduced porosity and solidification shrinkage[15]. In this technique,the moltenalloy with low superheated temperature with addition of grain refiner master alloy above liquidus temperature is poured directly on mould and allowed to solidify. Grain refinement plays a crucial role in improving characteristics and properties of cast and wrought aluminum alloys. The aluminum alloys are grain-refined by addition of a grain refiner (Al-Ti-B master alloy) prior to casting to ensure heterogeneous nucleation. Grain refinement by inoculation involves addition of particles which can act as substrates for heterogeneous nucleation. Inoculation is particularly widely practiced in the aluminum industry[17]. The Al–Ti–B ternary master alloys have been commonlyused as grain refiners for most aluminum alloys[18]. The key processing parameters affecting the final microstructure of the feedstock during LSC process are grain refining, pouring temperature and the mould material. From the industrial point of view, it is essential to find out the best combination of these parameters to attain the best microstructure of the feedstock that is suitable for thixoforming.

The aim of the present investigation is to investigate the effect of grain refining, pouring temperature and mould material on microstructural characteristics of AA6063 aluminum alloy feedstocks produced by low superheat casting. The Taguchi's and the analysis of variance (ANOVA) methods were applied to find out the optimum setting for the aforementioned parameters and their combination to be produced to achieve finest grain size and greater shape factor of the primary α-Al phase.

EXPERIMENTAL PROCEDURES

In this study, the commercial AA6063 wrought aluminum alloy with thechemicalcomposition was used as shown in TABLE 1. To determine the solidus and liquidus temperatures of the alloy the differential scanning calorimetric (DSC) analysis was carried out. The DSC experiments were carried during heating with a heating rate of 5 °C/min. Figure 1 shows the resulted DSC curve of the AA 6063 alloy. It has been found that AA 6063 alloy has solidus and liquidus temperatures of 618~ °C and 655 ~ °C, respectively. Figure 1 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 low superheat casting (LSC) process was carried as follows:about 900 g of AA6063 wrought alloy was melted at 710 °C in a graphite crucible located at a resistance furnace. When complete melting, about 9 g of the Al–5%Ti–1%B master alloy was dispersed into the molten alloy. After that, degassing process was carried out by dry Argon inert gas to remove any undesirable dissolved gases in order to prevent the formation of gas bubbles inside the feedstock. After that the molten alloy was allowed to cool down to the specific pouring temperature, typically at, 665 °C, 675 °C and 685 °C. This gives superheat values of about 10, 20 and 30 °C. Then the molten alloy was poured directly into

TABLE 1 : Chemical composition of AA6063 aluminum alloys (wt.-%)

Alloy	Si	Mg	Fe	Cu	Zn	Mn	Ti	Ca	Al
AA6063	0.535	0.514	0.210	0.001	0.003	0.028	0.011	0.051	Bal.

Full Paper

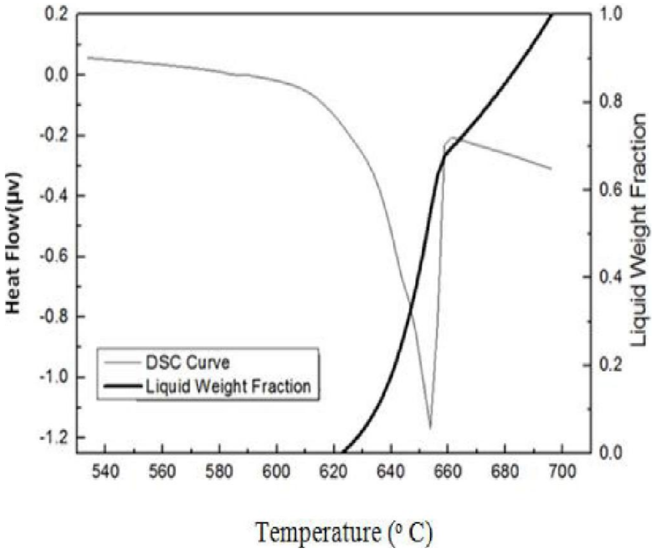


Figure 1 : DSC and liquid weight fraction versus temperature curves forAA6063alloy

from the feedstock. Metallographic samples were sectioned horizontally from the top, middle and bottom of the feedstockas shown in Figure 2b. The samples have 25 mm radius and 5 mm thick. The samples ground and polished using standard metallographic techniques. After that they were etched using chemical solution of Keller's etchant (2 ml hydrofluoric acid (HF), 3 ml hydrochloric (HCL), 5 ml Nitric acid (HNO_3) and 190 ml distilled water). Microstructural examinations were performed using *Olympus*optical microscope. The metallographic images were taken from the radius, mid-radius and center zones of the specimens as shown in Figure 2c.

The microstructural analysis of specimens was carried out using image analysis techniques. The size

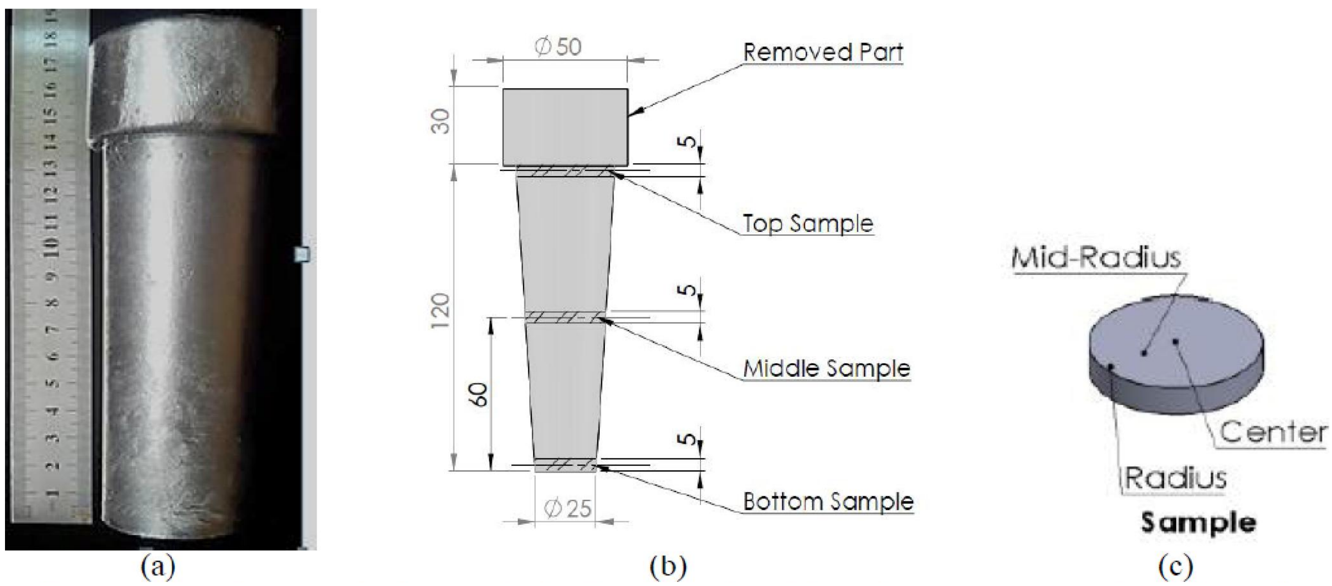


Figure 2 : (a) A photograph of a sample feedstock produced from the LSC process, (b) a schematic illustration of the feedstock showing the main dimensions (in millimeters) and the position of metallographic samples, (c) the positions of metallographic images in radial directions.

the mould. Several moulds having different materials, typically, low carbon steel (C. St.), pure copper (Cu) and 304 stainless steel (304SS) moulds were used. The moulds have the same dimensions of 50 mm diameter and 160 mm height with a draft angle 2^o to remove easily the solidified feedstock. During pouring, the semi-solid metal formed at the start of entering of mold. Figure 2a shows a photograph of a sample feedstock producedfrom the LSC casting process. The upper part of the feedstock with 35 mm height containing the shrinkage cavity wasremoved

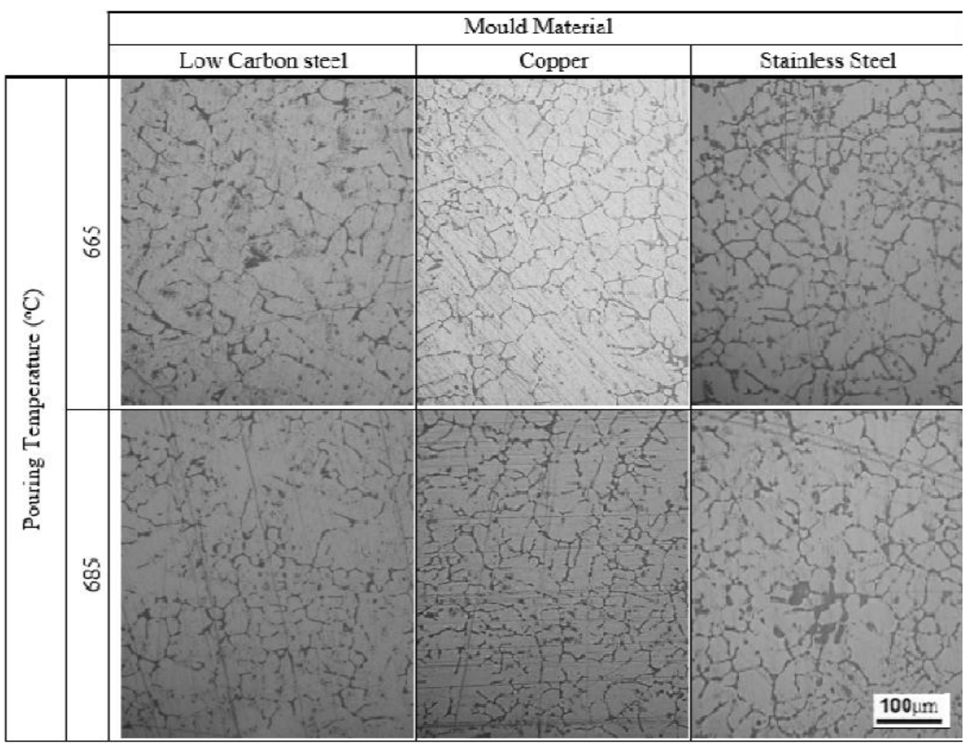
and shape factor of α-Al grains were determined. The size of the α-Al grains was determined using line intercept method. The shape factor of the grains was determined from the following equation[1]:

$$SF = 4\pi A/P^2 \quad (1)$$

Where: P is the perimeter and A is the area of α-Al grain. For a perfect circle, the shape factor would be one. For each feedstock, the average shape factor and average grain size was calculated from the measurements obtained from the top, middle and bottom of feedstocks as well as the radius, mid-ra-

TABLE 2 : L_{18} orthogonal array used in the present work

Exp. No.	LSC Condition		
	Pouring Temp., °C	Mould Material	Grain Refining
1	665	Copper	Without
2	675	Copper	Without
3	685	Copper	Without
4	665	Low Carbon Steel	Without
5	675	Low Carbon Steel	Without
6	685	Low Carbon Steel	Without
7	665	Stainless Steel	Without
8	675	Stainless Steel	Without
9	685	Stainless Steel	Without
10	665	Copper	With
11	675	Copper	With
12	685	Copper	With
13	665	Low Carbon Steel	With
14	675	Low Carbon Steel	With
15	685	Low Carbon Steel	With
16	665	Stainless Steel	With
17	675	Stainless Steel	With
18	685	Stainless Steel	With

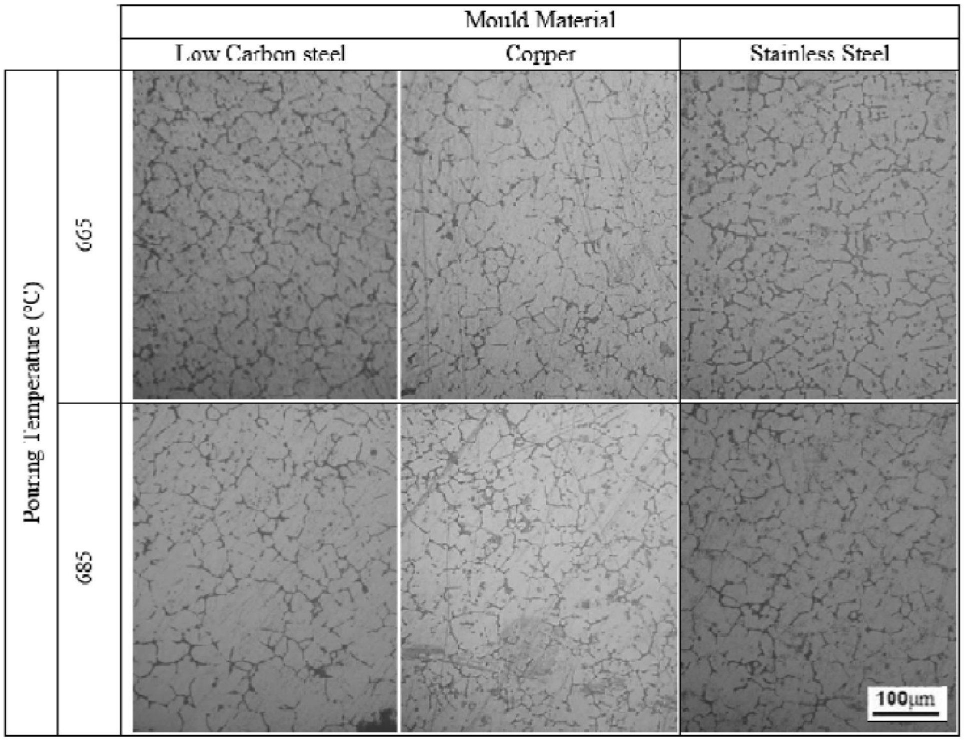


Figures 3 : Typical micrographs of the microstructure at the central positions of the bottom of AA6063 feedstocks produced using several LSC processing conditions without grain refining

dius and center of the specimens.

In the present investigation, the study the effect of LSC process parameters (i.e. grain refining, pouring temperature and mould material) on the different responses (i.e. the size and shape factor of the primary α-Al grains) was carried out using the Taguchi's

Full Paper



Figures 4 : Typical micrographs of the microstructure at the central positions of the bottom of AA6063 feedstocks produced using several LSC processing conditions with grain refining

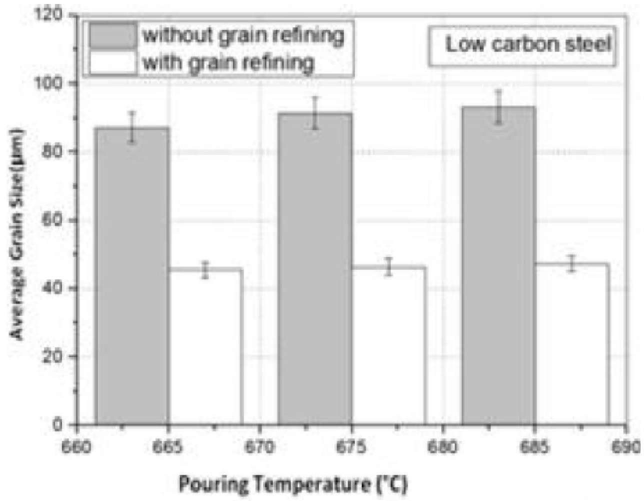
Design of experiment (DOE) optimization technique. The gain refining has two levels, typically, with and without-grain refining. Both of the mould material (C.St., Cu, 304SS) and pouring temperature (665, 675 and 685 °C) have three levels. The standard Taguchi's orthogonal array (OA) L_{18} was chosen in this study. This DOE gives a total of 18 feedstocks as shown in TABLE 2. The analysis of experimental results was carried out using the analysis of variance (ANOVA) statistical approach. From results of ANOVA one can obtain the most and lowest significant parameters. The design of experiments and ANOVA calculations were performed using *Minitab* statistical commercial software.

RESULTS AND DISCUSSION

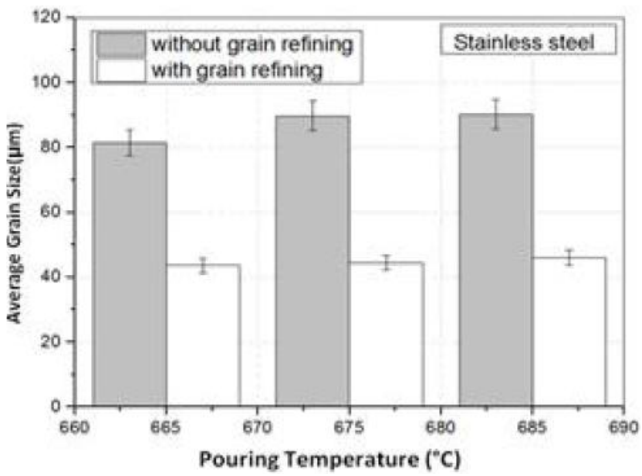
Microstructural investigations

Figures 3 and Figures 4 show typical optical micrographs of the microstructure of the AA6063 feedstocks produced with and without grain refining, respectively, and LSC processing conditions of pouring temperatures and mould materials. The micrographs were captured from the central positions of the bottom of feedstocks.

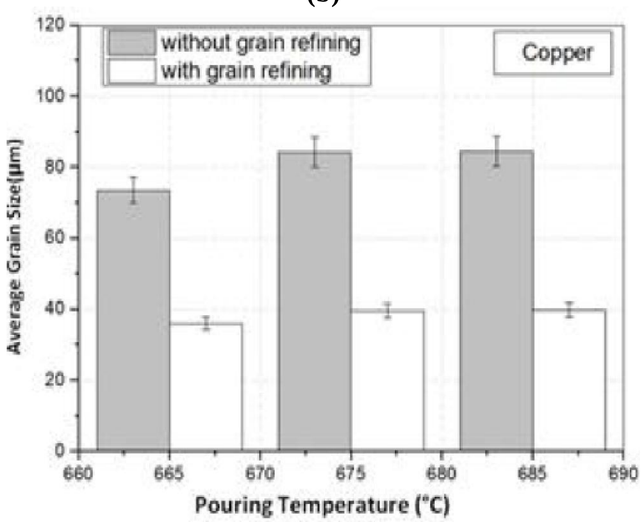
Figures 5 show the variation of the average size of α-Al grains with temperature for feedstocks poured into different moulds with and without grain refining. It has been found that the average grain size varies between 93.25 and 36 μm and the average shape factor varies between 0.79 and 0.65. Feedstocks poured with grain refining exhibited lower grain size and higher shape factor when compared with those poured without grain refining. For example, feedstockspoured into low carbon steel mould at constant temperature of 665°C, with and without grain refining exhibited the average size of 87.12 and 45.44 μm and average shape factor of 0.650 and 0.779, respectively. It is clear from Figure 5 that increasing the pouring temperature slightly increases the average grain size of the LSC feedstocks. For example, for feedstocks poured in the low carbon steel mould without grain refining, increasing the pouring temperature from 665 °C to 685 °C increases the average grain size from 87.19 to 93.25 μm. However, for feedstocks poured in the low carbon steel mould with grain refining, increasing the pouring temperature from 665 °C to 685 °C increases



(a)

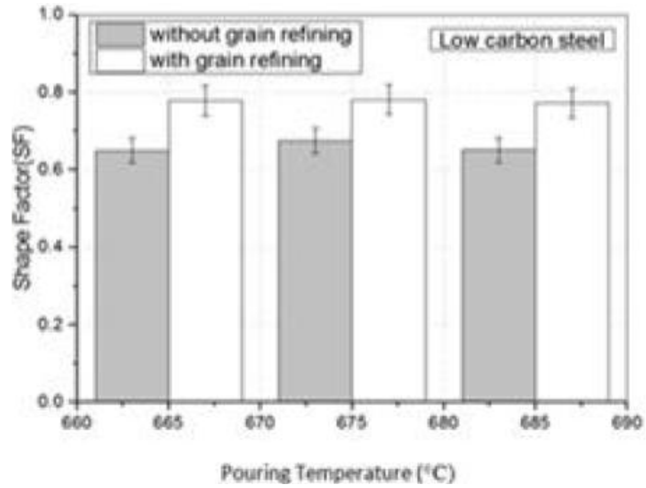


(b)

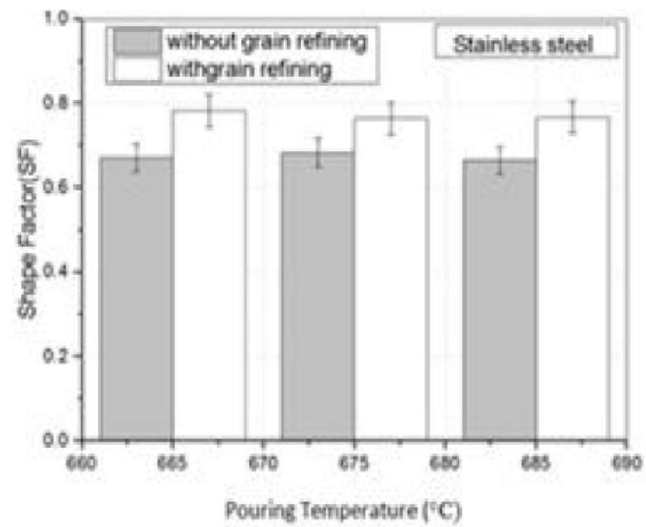


(c)

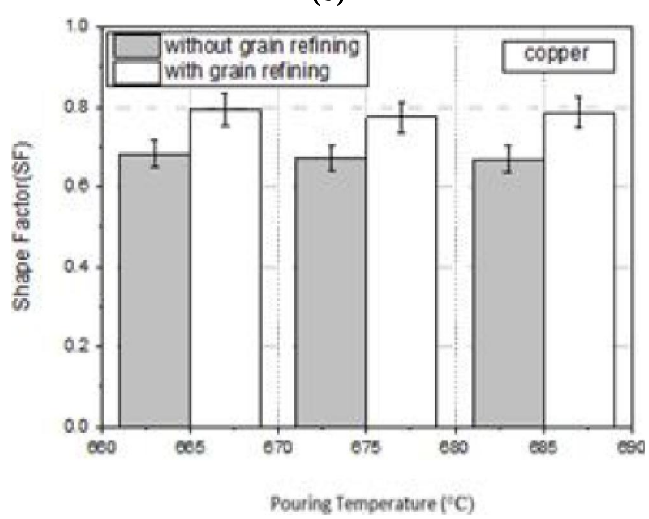
Figures 5 : Variation of the average grain size of α-Al grains with temperature for feedstocks poured with and without grain refining into (a) low carbon steel, (b) stainless steel and (c) copper moulds



(a)



(b)



(c)

Figures 6 : Variation of the average shape factor of α-Al grains with temperature for feedstocks poured with and without grain refining into (a) low carbon steel, (b) stainless steel and (c) copper moulds

the average grain size from increases from 45.44 to 47.26 μm. The results revealed also that increasing the pouring temperature slightly reduces the average shape of the primary α-Al grains.

The feedstocks poured in the copper and low carbon steel moulds exhibited the finest and coarsest average grain size of primary α-Al grains, respectively. This observation was noticed for feedstocks poured with different pouring temperatures and with and without grain refining. For example, grain refined feedstocks pouredat a temperature of 665°C into low carbon steel, stainless steel and copper mouldsshowed average grain sizes of 45.45, 43.59 and 36 μm, respectively. While feedstocks poured without grain refining showedaverage grain sizes of 87.12, 81.41 and 73.57 μm, respectively.

Figures 6 show the variation of the average shape factor of α-Al grains with the pouring temperature for feedstocks poured into different moulds with and without grain refining. Generally, the results revealed that increasing the pouring temperature slightly reduces the average shape of the primary α-Al grains. Such observation was noticed for grain refined and non-grained refined feedstocks. For example, the grain refined feedstocks poured into copper mould at 665 °C and 685 °C exhibited average shape factors of 0.794 and 0.786, respectively. Feedstocks poured with and without-grain refining in the copper mould exhibited the higher average shape factor of primary α-Al when compared with feedstocks poured in low carbon steel and stainless steel moulds. For example, grain refined feedstocks poured at 665 °C into low carbon steel, stainless steel and copper moulds exhibited average shape factors of 0.779, 0.782 and 0.794, respectively. In the present investigation, a minimum average grain size of 36 μm was observed for grain-refined feedstock poured into copper mould at 665°C. While a maximum average shape factor of 0.794 was observed for grain-refined feedstock poured into copper mould at 665°C.

According the aforementioned results, it can be concluded that the addition of Al–5%Ti–1%B grain refinerimproves the microstructural characteristics of LSC feedstocks. The grain refining process assisted in reducing the average size and increasing the average shape factor of the primary α-Al grains. It is believed that Al–5%Ti–1%B grain refiners act as nucleating agents during solidification and control crystal formation by heterogeneous nucleation. Grain refinement can be defined as intentional prevention of columnar grain growth in feedstocks and castings and formation of fine equiaxed grains all over the material[17], which influences the grain size and the shape factor of the primary α-Al grains. In case of pouring without grain refining, the number of crystals nucleated was insufficient to produce fine and spheroidal primary crystals. High pouring temperature leads to decrease in nucleation. Low pouring temperature causes rapid solidification of melt. Increasing of thermal conductivity of mould material (as in case of copper mould) increases the heat transfer through that material which increasing the cooling rate. Increasing the cooling rate influences the grain size and the shape factor of the primary α-Al grains.

ANOVA Results

TABLES 3 and 4 list the ANOVA results for both the average size and the shape factor of the primary α-Al grains, respectively. The last columns in the tables show the percentage of contribution (P_c) of each factor on the total variation indicating the influence of the factors on the results. The higher the value of the P_c, the more statistical and physical significant the factor is. From the analysis of TABLE 3 and TABLE 4, it can be observed that grain refining,mould material and the pouring temperature significantly affect the average grain size and shape factor of the primary α-Al grains, respectively. The grain refining exhibited the highest statistical and physical significance on both the grain size and shape factor of the primary α-Al grains. The mould material and pouring temperature exhibited much lower statistical and physical significance when compared with the grain refining. The grain refining exhibited P_c values of 97.90% and99.70 % for the grain size and shape factor of the primary α-Al grains, respectively. While, the pouring temperature and mould material exhibited (P_c) values of 1.46%and 0.64%, and 0.096% and0.21%for the size and shape factor of the primary α-Al grains, respectively. From TABLES 3 and 4, it is clear that the residuals are

TABLE 3 : TheANOVA results for the grain size of the α-Al primary grains

Source of variation	DF	SS	MS	F	P	P_c
Grain Refining	1	8334.4	8334.4	1815.55	0.000	97.90023
Mould Material (M)	2	109.2	54.6	11.90	0.001	0.64168
Temperature (T)	2	248.2	124.1	27.04	0.000	1.45809
Residual	17	55.1	4.6			
Total		8746.9				100
			R^2 = 99.37%			

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

TABLE 4 : TheANOVA results for the shape factor of the α-Al primary grains

Source of variation	DF	SS	MS	F	P	P_c
Grain Refining	1	0.057148	0.057148	299.80	0.000	99.69738
Mould Material (M)	2	0.000235	0.000118	0.62	0.556	0.2061787
Temperature (T)	2	0.000112	0.00056	0.29	0.750	0.096441
Residual	12	0.002287	0.000191			
Total	17	0.059782				100
			R^2 = 96.17%			

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

less than 2%, which indicates that there are no interactions between grain refining, mould material and pouring temperature.

CONCLUSIONS

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

Grain refined feedstocks produced using Low superheat casting (LSC) exhibited better microstructural characteristics than those produced without grain refinement. The addition of Al–5%Ti–1%B grain refiner has asignificant effect on reducing the size or/and increasing shape factor of the primary α-Al grains. The grain refining exhibited the highest statistical and physical significance on both the grain size and shape factor of the primary α-Al grains.

Increasing the pouring temperature slightly increasesthe average grain size of the primary α-Al grains. In contrast, increasing the pouring temperature reduces the average shape factor of primary α-Al grains.

The AA6063 feedstocks poured in the copper mould exhibited finer average size and higher average shape factor of primary α-Al when compared with feedstocks poured in low carbon steel and stainless steel moulds.

ACKNOWLEDGMENTS

The authors are thankful to Benha University-Shoubra Faculty of Engineeringfor providing financial support and facilities for carrying out this work.

REFERENCES

[1] F.Taghavi, A.Ghassemi; "Study on the effects of the length and angle of inclined plate on the thixotropic microstructure of A356 aluminum alloy", Technical Report, Materials and Design, **30(5)**, 1762-1767 **(2009)**.

[2] C.G.Kang, P.K.Seo, M.D.Lim; "Rheo- and thixo-die casting for automobile suspension parts", In Proceedings of the 8th International Conference on Semi-Solid Processing of Alloys and Composites, Cyprus, **(2004)**.

[3] H.V.Atkinson; "Modelling the semisolid processing of metallic alloys", Progress in Materials Science, **50**, 341–412 **(2005)**.

[4] E.Tzimas, A.Zavaliangos; "A comparative characterization of nearequiaxed microstructures as produced by spray casting, magnetohydrodynamic casting and the stress induced, melt activated process", Material Science and Engineering, **A289**, 217–227

(2000).

[5] J.C.Choi, H.J.Park; "Microstructural characteristics of aluminum 2024 by cold working in the SIMA process", Journal of Materials Processing Technology, **82**, 107–116 **(1998)**.

[6] S.Y.Lee, S.Oh; "Thixoforming characteristics of thermo-mechanically treated AA 6061 alloy for suspension parts of electric vehicles", Journal of Materials Processing Technology, **130–13**, 587–593 **(2002)**.

[7] D.Liu, H.V.Atkinson, P.Kapranos, W.Jirattiticharoean, H.Jones; "Microstructural evolution and tensile mechanical properties of thixoformed high performance aluminium alloys", Material Science and Engineering, **A361**, 213–224 **(2003)**.

[8] Sang L.Yong, Jung L.Hwan, Young L.Seon; "Characterization of Al 7075 alloys after cold working and heating in the semi-solid range", Journal of Materials Processing Technology, **111**, 42–47 **(2001)**.

[9] E.Tzimas, A.Zavaliangos; "Evolution of near-equiaxed microstructure in the semisolid state", Material Science and Engineering, **A289**, 228–240 **(2000)**.

[10] Villalobos C.J.Gutierrez, Gedler G.E.Chacon, La Barbera J.G.Sosa, A.Piñeiro, M.H.Staia, J.Lesage et al.; "Fatigue and corrosion fatigue behavior of an AA6063-T6 aluminum alloy coated with a WC–10Co–4Cr alloy deposited by HVOF thermal spraying", Surface and Coatings Technology, **202**, 4572–4577 **(2008)**.

[11] D.J.Chakrabarti, D.E.Laughlin; "Phase relations and precipitation in Al–Mg–Si alloys with Cu additions", Progress in Materials Science, **49**, 389–410 **(2004)**.

[12] A.K.Gupta, D.J.Lloyd, S.A.Court; "Precipitation hardening processes in an Al– 0.4%Mg–1.3%Si–0.25%Fe aluminum alloy", Material Science and Engineering:A, **301**, 140–6 **(2001)**.

[13] C.S.Tsao, C.Y.Chen, U.S.Jeng, T.Y.Kuo; "Precipitation kinetics and transformation of metastable phases in Al–Mg–Si alloys", Acta Materiala, **54**, 4621–4631 **(2006)**.

[14] S.Esmaeili, S.X.Wang, D.J.Lloyd, W.J.Poole; "On the precipitation-hardening behavior of the Al–Mg–Si–Cu alloy AA6111", Metallurgicaland MaterialsTransactions A, **34A**, 751–63 **(2003)**.

[15] H.M.Guo, X.J.Yang, B.Hu; "Rheocasting of A356 alloy by low superheat pouring with a shearing field", Acta Metallurgica Sinica (English Letters), **19(5)**, 328-334 **(2006)**.

[16] M.A.Easton, H.Kaufmann, W.Fragner; "The effect of chemical grain refinement and low superheat pouring on the structure of NRC castings of aluminium alloy Al–7Si–0.4Mg", Material Science and Engineering: A, **420(1–2)**, 135–143 **(2006)**.

[17] D.G.McCartney; "Grain refining of aluminium and its alloys using inoculants", International Materials Reviews, **34**, 247-260 **(1989)**.

[18] M.A.Easton, D.H.Stjohn; "A model of grain refinementincorporating alloy constitution and potency ofheterogeneous nucleant particles", Acta Materurgica el Materiala, **49**, 1867–1878 **(2001)**.