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Effect of semi-solid forming on the microstructure and mechanical properties of the iron containing Al–Si alloys

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A B S T R A C T

Iron is the most common impurity in aluminum casting alloys. The iron-bearing intermetallic compounds have the detrimental effects on the mechanical properties of the alloys. The aim of this research is to study the effects of plastic deformation and semi-solid forming on the morphology and distribution of the ironbearing intermetallics and the microstructure and mechanical properties of the Al–Si alloys. Different amounts of iron and manganese were introduced into the A380 aluminum casting alloys. The alloys were processed through plastic deformation, recrystallisation and partial melting (RAP), and thixoforming. The microstructure and mechanical properties of the thixoformed alloys were investigated. The results showed that the RAP and thixoforming processes promote the formation of the very fine and welldistributed α -Al₁₅(FeMn)3Si2 compounds in the aluminum matrix. The yield and tensile strength as well as elongation of the alloys have been increased considerably by semi-solid forming compared with the as-cast condition.

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1. Introduction

Iron is the most common and detrimental impurity present in aluminum casting alloys. On the other hand, in pressure die-casting alloys, iron is a desirable element that helps to prevent the molten alloys from "soldering" to the casting die. The Al–Si–Fe eutectic composition occurs at about 0.8 wt% Fe. When iron is alloyed to somewhat above this level, the molten metal has little or no tendencies to dissolve die steel while the two materials are in intimate contact. Thus, the higher iron content of the alloy reduces the solution potential for the iron components of the casting machine and die, and for this reason, most aluminum pressure die casters desire that their alloys contain between 0.8 and 1.1 wt% Fe [\[1–5\].](#page-5-0)

Also, the Al–Si alloy should maintain its required mechanical properties at service temperature. Although the copper and magnesium addition can improve the strength of Al alloys substantially by precipitation hardening, the precipitates are not stable at the elevated temperature and result in rapid decrease in strength at high temperature [\[6\].](#page-5-0) Therefore, the addition of alloying elements forming a stable precipitate at high temperature is required. Thermally stable intermetallic compounds can be formed by the addition of transition elements such as Fe to Al alloys.

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Since iron is inevitable and cannot be economically removed from the molten aluminum, strategies have to be developed to neutralize its negative effects. The negative effects of iron are generally associated with the formation of Fe-rich intermetallic phases during solidification [\[2,7–10\].](#page-5-0) The embrittling effect of iron rich intermetallic compounds can be neutralized by the additions of a sufficient level of Mn, modifying the platelet morphology to a less harmful, more compact form [\[2,7,11\].](#page-5-0) However, some technical problems associated with addition of this element to iron containing melts exist and also, addition of manganese generally increases the total volume fraction of intermetallics and their mean diameter size in high level of iron solute and again deteriorates the mechanical properties [\[7,12–14\].](#page-5-0)

Therefore, it becomes important to increase strength and ductility of the alloy and to modify iron intermetallics into the less harmful morphologies. In this study, the plastic deformation and subsequently semisolid forming have been applied to the deformed samples to change the morphologies of the intermetallics. This process is useful to introduce strain which accelerates spheroidization of the dendritic primary α -Al phase and also, to produce uniform distribution of the refined iron-containing intermetallic compounds.

2. Experimental procedure

The aim of this research is to produce the near net shape parts using thixoforming process. Die-casting machine can be used in thixoforming process. The thixoforming is basically the two-steps process, involving the preparation of a feed stock material with good thixotropic characteristics, and then reheating the feed

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stock material to the semisolid temperature to provide the SSM slurry to make the final parts. Cold rolling has been used to cause fragmentation of intermetallic compounds and distribute them well to improve the mechanical properties of the parts.

Alloys with different amounts of iron and manganese were produced by melting appropriate quantity of commercial A380 aluminum alloy ingots. Iron and manganese were added using ALTAB 75 wt% Fe and ALTAB 75 wt% Mn master alloy. The alloys were analyzed using optical mass spectroscopy and their chemical compositions are given in Table 1.

The prepared ingots were heated to 500 ◦C, held for 8 h to homogenize the composition, and then quenched into the boiling water. Ingots were cut in dimensions of 60 mm \times 30 mm \times 20 mm and after machining, they were cold-rolled to achieve 11% reduction in the cross section.

Critical temperatures of the alloys have been determined using thermal analysis in the progressive stages of the solidification process. The temperature variation of the melt has been measured using a calibrated K-type thermocouple and the results have been recorded using a computer aided acquisition system apparatus to obtain cooling curves.

For the thixoforming, specimens were consequently reheated to 578 ◦C in an electrical resistance furnace with ± 1 °C temperature accuracy and held at these temperatures for 25 min. Specimens were then transferred from the furnace to the die on the press and subjected to 30% hot working. A hydraulic press (maximum load of 150 t and maximum, velocity of 200 mm/s) was used for compression. In this experiment, the compression velocity and applied pressure were 200 mm/s and 50 t, respectively. In order to maintain a constant die temperature, the die was heated by the cartridge heaters, which were inserted in the upper and lower dies. The applied pressure was held for 20 s during pressing the specimens. The forged samples were rapidly quenched into the water to investigate their microstructures and mechanical properties.

Samples were prepared for microstructural evaluations. They were etched in 0.5% HF for 15 s. Metallographic examinations were carried out using a Nikon optical microscope (OM) model Epiphoto 300 and a CAMSCAN MV2300 scanning electron microscope (SEM), respectively. The tensile test was conducted according to ASTM E-8M-97 standard tensile sample. All the tests were performed at room temperature using hounsfield tensile test machine, model H50KS with a ±50 kN load cell and a strain rate of 1.0×10^{-3} s⁻¹. Five tensile tests were performed for each sample and the average of them was reported for the mechanical properties.

3. Results and discussion

3.1. As cast microstructure

The aim of this process is to produce different volume fractions of α -Al $_{15}$ (Fe,Mn) $_3$ Si $_2$ intermetallics in the matrix of aluminum alloy by increasing iron and manganese content of the alloy. The volume fraction of intermetallics in the final structure is a function of segregation factor, SF, which is defined as: SF = wt% Fe + 2 wt% Mn [\[10\].](#page-5-0) Fig. 1 shows the variation of intermetallics volume fraction versus SF for the investigated alloys.

The optical micrograph of the alloys in the as-cast condition is shown in [Fig.](#page-2-0) 2. Intermetallics have Chinese script morphology at lower content of iron and manganese [\(Fig.](#page-2-0) 2b). The intermetallics appear as polyhedral at higher iron and manganese content ([Fig.](#page-2-0) 2c and d). All intermetallic compounds formed as primary- α polyhedral or Chinese script morphology and no β -plates were seen in the as-cast microstructure. This is mainly due to the proper ratio of iron to manganese which is kept in 2:1.

Fig. 1. Intermetallic volume fraction versus segregation factor.

[Fig.](#page-2-0) 3 shows more details of intermetallic compounds at higher magnification.As it can be seen, gray phases that marked with letter A, have polyhedral morphology, form in the vicinity of α -Al phase (letter C) and eutectic Si (letter B). Nucleation and growth of eutectic silicon on the intermetallic compounds are clearly observed in the micrographs. In fact intermetallics are suitable nucleation sites for the formation of eutectic silicon [\[15\].](#page-5-0)

EDS microanalysis of polyhedral intermetallic phases (Table 2) demonstrates that the composition of them is α -Al₁₅(Fe,Mn)₃Si₂ [\(Fig.](#page-3-0) 4)a, similar to what reported by others [\[10\].](#page-5-0) Copper-containing phases, $Al₂Cu$, are also detected by EDS microanalysis, because of the presence of Cu in the base alloy ([Fig.](#page-3-0) 4b).

3.2. Deformed as-cast microstructure

[Fig.](#page-3-0) 5 shows the microstructure of the deformed specimens, parallel to the rolling direction. A large amount of brittle phases including silicon particles and α -intermetallics exist in the aluminum matrix. Micro-cracks are clearly observed because of applied stress and low formability of intermetallics in the rolling process. This is because of simultaneous plastic flow of ductile α -Al phase in the vicinity of α -intermetallics which have limited or no ductility and high stress concentration sensibility and therefore would cause cracking of brittle phases. These micro-cracks can propagate in the structure of brittle phases and finally break them into the smaller particles. Because of low ductility of the alloy, high value of reduction in cross section is impossible and therefore fragmented particles cannot be displaced a lot [\(Fig.](#page-3-0) 5d). Therefore, the supplementary processes are needed to compensate these defects and to form the alloy to a near net shape part. Semi-solid forming of the samples can be the solution.

On the other hand, the compressive deformation applied on the as-cast alloys increases the strain energy which can act as a driving force for the recrystallisation in the semi-solid state.

3.3. Microstructure of semi-solid state

Cooling curve and first derivative curve during solidification of the alloys are shown in [Fig.](#page-3-0) 6(a). Variation of liquid volume fraction versus temperature is calculated for the alloy and shown in [Fig.](#page-3-0) 6(b).

Table 2

Fig. 2. Microstructure of the alloys in the as-cast condition. (a) alloy A380, (b) alloy 0.7Fe, (c) alloy 1.5Fe, (d) alloy 3Fe.

As seen in this figure, the volume fraction of liquid phase is about 30–50% at temperatures of 577–579 ◦C and this temperature range is suitable for semisolid forming of the alloys.

Based on the thermal analysis results, the A380 alloy was isothermally heat treated at 578 ◦C for different times. The microstructure of the alloy has been shown in [Fig.](#page-4-0) 7. The recrystallized structure was clearly observed in this figure due to the pre-deformation of the samples before heat treatment.

As seen in ([Fig.](#page-4-0) 7a and b), the sphericity of α -Al globules and contiguity of the eutectic phase are not sufficient with holding times of 15 or 20 min. They were improved in the samples having the holding time of 25 min. The sphericity of α -Al globules was decreased and the volume fraction of eutectic phase was increased with hold-

Fig. 3. Microstructure of the alloy 3Fe (table 1) in the as-cast condition. Letters A, B and C are intermetallic compound, eutectic silicon and α -Al matrix, respectively.

ing time of 30 min. Therefore, the best sphericity and contiguity of the eutectic phase have been obtained in the samples having the holding time of 25 min which is suitable for semisolid forming.

3.4. Microstructure of thixoformed state

Plastic deformation caused fragmentation of iron and manganese-containing intermetallic compounds. Achievement of the proper distribution of intermetallic compounds in the alloy is one of the important objectives to improve the mechanical properties.

The applied plastic strain was not enough to distribute the intermetallic compounds. Therefore, semi-solid forming (thixoforming) is necessary to modify the defects occurred during plastic deformation and to distribute the intermetallics in the matrix.

In fact, in the semi-solid state, samples show very low resistance against shear stresses. As a result, little energy is needed for shaping the samples. The cracked-intermetallic s are disintegrated and all the gaps are filled with the melt.

[Fig.](#page-4-0) 8 shows SEM-BSE micrograph of the thixoformed and ascast alloy 3Fe. In thixoformed sample, the size of intermetallic particles has been decreased considerably and their distribution has been improved. Displacement of these particles has been occurred duo to the movement of the solid constituents into the liquid phase during thixoforming in the semisolid state. The primary particles were uniformly distributed throughout the entire cross-section of the thixoformed sample.

Further microstructural evaluation revealed that there was no entrapped gas in all thixoformed samples, and the total very fine shrinkage porosity which observed occasionally in some samples, was less than 0.7%.

[Fig.](#page-5-0) 9 shows the results of image analysis of the as-cast and thixoformed microstructure of different alloys. Comparing thixo-

Fig. 4. Backscattered electron images of the intermetallic phases. (a) α -Al₁₅ (Fe,Mn)₃Si₂ and (b) Al₂Cu.

Fig. 5. Microstructure of the specimens after plastic deformation parallel to the rolling direction (RD). Showing micro-cracks in the brittle phases containing of (a): silicon particles and (b), (c): intermetallic compounds in alloy 3Fe. (d): micro-cracks in intermetallic compounds in alloy 1.5Fe.

Fig. 6. (a) Cooling curve and first derivative curve (b) variation of liquid volume fraction versus temperature.

Fig. 7. Microstructure of the A380 rolled alloy after isothermal holding in 578 ℃ for (a) 15 min, (b) 20 min, (c) 25 min, (d) 30 min.

Fig. 8. Backscattered SEM microstructure of the alloy 3Fe. (a) as-cast condition and (b) thixoformed condition.

forming results to that of the cast samples, it is seen that the effect of thixoforming on the average size of primary Fe containing compounds is significant. It is worth to note that the average particle size decreased with increasing segregation factor (increasing Fe content) in the thixoformed samples. In thixoforming process, equivalent diameter of intermetallic compounds decreased 39% and 59% when segregation factor was 2.23 and 5.37, respectively. Therefore, it can be expected that with increasing iron content, plastic deformation produces much smaller intermetallics.

3.5. Mechanical properties

Mechanical properties of different alloys are compared in [Fig.](#page-5-0) 10. Thixoforming was effective in improving mechanical properties, particularly in improving the ductility of the alloys. Although the elongation of the thixoformed alloys decreased with increasing Fe contents, but it is worth to mention that the elongation of alloy 3Fe in the thixoformed condition is equal to the elongation of A380 alloy with much lower level of iron content in the as-cast condition.

Fig. 10. Comparison of mechanical properties of different alloys produced via permanent mold casting and thixoforming. (a) elongation, (b) yield strength and (c) ultimate tensile strength.

This is attributed to the size and the morphological modification of the primary Fe containing intermetallic compounds.

Thixoformed samples have substantially higher yield strength and tensile strength compared to the A380 as-cast sample. The yield strength of the thixoformed samples increased and the ultimate strength decreased, with increasing Fe content up to the maximum 2.87 wt% Fe.

The good combination of strength and elongation were obtained in thixoformed samples. The results of this investigation revealed that the mechanical properties of the alloys can be improved by the microstructural uniformity caused by the sphericity of α -Al globules and contiguity of the eutectic phases, reduction of size and well-distribution of Fe-containing intermetallic compounds, reduction of porosity, produced by semi-solid processing.

It is worth to mention that in industrial cases, high pressure die-casting machine (HPDC) can be used for thixoforming process. It is supported with a special cylinder which is surrounded by the electrical resistance elements. The temperature of the feed stock inside the cylinder increases to semi-solid state. The solid fraction of the slurry is controlled by holding temperature and time. Then, the semisolid slurry of the alloy having iron-bearing intermetallics is transferred to the shot chamber of the HPDC machine for the final shaping.

4. Conclusion

Based on the results obtained in this research, the following conclusions can be stated:

- 1. The thixoforming process is effective in modifying the morphology of the primary phases. Applying plastic deformation and isothermal holding of 380 aluminum alloy with excess amounts of iron and manganese, produces fragmented α -Al₁₅ (Fe,Mn)₃Si₂ intermetallics in the globular α -Al matrix surrounded by the low melting point Al–Si eutectic.
- 2. In industrial cases, high pressure die-casting machine (HPDC) can be used for thixoforming process and the semisolid slurry of the alloy having iron-bearing intermetallics is transferred to the shot chamber of the HPDC machine for the final shaping.
- 3. 11% of plastic deformation followed by the isothermal holding of the specimen in 578 ◦C for 25 min has developed a dominant globular structure and contiguity of the eutectic phase.
- 4. The thixoforming process increased the strength and elongation ofthe alloy, compared to the as cast condition. It has been caused by the extremely low porosity, fine and equiaxed morphology of the α -Al grains and uniform distribution of fragmented intermetallic compounds in microstructure of sample.

References

- [1] S. Shankar, D. Apelian, Mater. Trans. 33B (2002) 465–476.
- [2] A. Couture, AFS Int. Cast Met. J. $6(4)(1981)$ 9–17.
[3] J.L. Jorstad, Die Cast. Eng. (November/December)
- J.L. Jorstad, Die Cast. Eng. (November/December) (1986).
- [4] M.R. Ghomashchi, Z. MetaIlkd. 78 (11) (1987) 784–787.
- [5] R. Dunn, Die Cast. Eng. (September) (1965) 8–16.
- [6] T. Hayashi, Y. Takeda, K. Akechi, T. Fujiwara, SAE Technical Paper Series 900407, 1990.
- [7] P.N. Crepeau, AFS Trans. 103 (1995) 361–366.
- [8] T.O. Mbuya, B.O. Odera, S.P. Ng'ang'a, Int. J. Cast Met. Res. 16 (5)(2003) 451–465.
- [9] W. Khalifa, F.H. Samuel, J.E. Gruzleski, Metall. Mater. Trans. A 34A (2003) 807–825.
- [10] L. Backerud, G. Chai, J. Tamminen, Solidification of Characteristics of Aluminium Alloys: vol. 2, Foundry Alloys, AFS/Skanalumium, Des Plaines, IL, 1990, pp. 1–255.
- [11] L.F. Mondolfo, Manganese in Aluminium Alloys, The Manganese Centre, Neuilly sur Seine, France, 1978.
- [12] G.K. Sigworth, S. Shivkumar, D.Apelian, Trans.Am. Foundrymen's Soc 97 (1989) 811.
- [13] S.G. Shabestari, J.E. Gruzleski, Cast Met. 6 (4) (1994) 217–224.
- [14] S.G. Shabestari, M. Mahmudi, M. Emamy, J. Campbell, Int. J. Cast Met. Res. 15 (1) (2002) 17–24.
- [15] S.G. Shabestari, S. Ghodrat, J. Mater. Sci. Eng. A 467 (2007) 150–158.