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The Effect of Cooling Rate on Properties of Intermetallic Phase in a Complex Al-Si Alloy

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Abstract

The cooling rate is one of the main tools available to the process engineer by means of which it is possible to influence the crystallisation process. Imposing a desired microstructure on a casting as early as in the casting solidification phase widens significantly the scope of technological options at disposal in the process of aluminium-silicon alloy parts design and application. By changing the cooling rate it is possible to influence the course of the crystallisation process and thus also the material properties of individual microstructure components. In the study reported in this paper it has been found that the increase of cooling rate within the range of solidification temperatures of a complex aluminium-silicon alloy resulted in a decrease of values of the instrumented indentation hardness (H_{IT}) and the instrumented indentation elastic modulus (E_{IT}) characterising the intermetallic phase occurring in the form of polygons, rich in aluminium, iron, silicon, manganese, and chromium, containing also copper, nickel, and vanadium. Increased cooling rate resulted in supersaturation of the matrix with alloying elements.

Keywords: Al-Si alloy, Intermetallic phase, Cooling rate, Silicon primary crystals and matrix indentation

1. Introduction

Iron, apart from its harmful effect on mechanical properties of aluminium-silicon alloys, is introduced in quantities as high as up to 2% Fe [1] to reduce the tendency to adhere to metal moulds. Iron can be also introduced to aluminium-silicon alloys unintentionally, as a result of dissolving steel in-cast inserts not removed from the circulating scrap used to prepare liquid metal [1]. Some importance has also the economic aspect, as aluminium-silicon alloys contaminated with iron are less expensive. The maximum solubility rate of iron in aluminium at 650°C is 0.05% [2] which results in development of iron-containing intermetallic phases in iron-containing aluminium-silicon alloys, such as e.g.: β — AlFeSi (Al_3FeSi) [3], α — AlFeSi (Al_8Fe_2Si) [2], π — AlFeSi ($Al_8Mg_3FeSi_6$) [4].

Dimensions of iron-containing intermetallic phases increase with increasing content of iron in the alloy and with decreasing cooling rate [5, 6].

According to numerous authors [1, 6–10], shape of iron-containing intermetallic phases is affected by value of the quotient Fe : Mn : Cr (cf. Table 1). At low content of manganese and chromium, these phases are needle-shaped [1]. Increase of manganese content contributes to creation of phases in the form of Chinese script [7]. In alloys containing above 0.40% Mg, one can observe a trend towards precipitation of phases with the Chinese script shape [10]. Increased content of chromium favours creation of phases in the form of polygons. Beryllium introduced in small quantities changes the needle-like shape of phase β into Chinese-script and/or polygonal forms [8, 9].

Table 1.
The effect of chemistry on shape of intermetallic phases — a review of literature

Chemistry	Intermetallic phase shapes
Low Mn & low Cr	Long needles [1]
Fe : Mn = 2:1	Chinese script [7]
At presence of Mg	Chinese script and polygons [3, 10]
Addition of Cr	Polygons [8, 9]
Addition of Be → phase BeSiFe ₂ Al ₈ instead of Al ₅ SiFe	Chinese script and polygons in phase α(Al) [8, 9]

Increase of iron content from 0.5% to 3.0% is connected with a change in morphology of intermetallic phases from lamellar through this resembling Chinese script right to massive polyhedrons [11]. An addition of manganese and chromium and the increase of Al-Si-Fe alloy cooling rate allows to avoid appearance of intermetallic phases with lamellar morphology.

According to authors of [12], an increase of superheating temperature of hypoeutectic aluminium-silicon alloy containing about 1% Fe and near-eutectic alloy containing about 2% Fe from 720°C to 1000°C has a favourable effect on refinement and rounding-off the intermetallic phases which results in improvement of mechanical properties (Table 2).

Table 2.
The effect of superheating temperature on mechanical properties and shape of intermetallic phases [12]

Superheating temperature, °C	R_m , MPa	$R_{0.2}$, MPa	A_5 , %	Intermetallic phase shape
720	140	125	-	Long needles
800	160	140	-	Short needles
950	-	-	-	Fine polygons
1000	225	175	2.3	Fine polygons

An alloy containing: 7.8% Si, 0.4% Mn, 3.5% Cu, 1.12% Fe.

Retention time at superheating temperature – 10 minutes

The shape of intermetallic phases is also influenced by the cooling rate. Authors of paper [13] have found that with the cooling rate increase from 10°C/min to 9 000°C/min, forms of intermetallic phases change from needle-shaped (lamellar) through Chinese script-like, right to polygonal ones (Table 3).

Table 3.
The effect of cooling rate on intermetallic phase precipitate shapes [13]

Cooling rate, °C/min	Intermetallic phase shape
10	long needles
750	Chinese script
9 000	polygons

An alloy containing: 5.94% Si, 0.54% Mn, 0.15% Cu, 1.48% Fe.

The presented review of literature concerning intermetallic phases in iron-containing aluminium-silicon alloys indicates that there is a wide range of options that can be used to influence both chemical composition and morphology of precipitates.

Modern approach to modelling microstructure of alloys requires establishing relationship between chemical composition, morphology of precipitates, and physical properties such as hardness and modulus of elasticity of individual phases, as the

possibility to control microstructure of castings as early as in the crystallisation phase widens significantly the scope of technological options available to designers and then users of aluminium-silicon alloy castings. One of the main tools allowing a process engineer to intervene into the course of crystallisation phenomena is the alloy cooling rate. In attempts to model microstructure of iron-containing aluminium-silicon alloys, to be able to make them highly resistant to abrasive wear, it is important to gain relevant knowledge with respect to silicon precipitates, intermetallic phases rich in iron, and the matrix. In view of the above, the objective of the presented study consisted in determining the effect of cooling rate on material properties of iron-rich intermetallic phase in a complex Al-Si alloy.

2. Research methodology

An aluminium-silicon alloy with the following chemistry: 31.28% Si, 0.55% Mn, 1.41% Cu, 0.56% Cr, 1.10% Ni, 0.47% V, 0.56% Fe, 1.30% Mg, 0.005% Zn, 0.039% Ti, 0.0025% B, 0.05% P, Al to balance, was obtained in an induction furnace with capacity of 5 kg. Modification with copper-phosphorus master alloy was performed at temperature 950°C. In order to differentiate the cooling rate, a water-cooled chill was designed at the metal mould base. To determine the course of the cooling process, two thermocouples were mounted on the mould cavity half-thickness lines (with mould dimensions 400 mm × 120 mm × 40 mm) at casting heights of 10 mm and 90 mm. The temperature vs. time curves were the base on which the cooling rate in the range defined by solidification beginning and end temperature was determined. Liquid metal with temperature 920°C was poured into a metal mould preheated to 300°C. From a plate-shaped casting, at heights 10 mm and 90 mm from the chill base, specimens for tests were cut out and prepared further by means of grinding and polishing technique. In case of specimen no. 1 taken at a distance of 10 mm from the chill face, the cooling rate was 5.1°C/s (this work), while the specimen no. 2 taken at the distance of 90 mm from the chill face was cooled at rate 1.5°C/s. Results of examination concerning the specimen no. 2 are presented in [14]. The specimen no. 1 was subject to examination with the use of VEGA XMH scanning electron microscope (TESCAN), equipped with x-act adapter for chemistry microanalysis (Oxford Instruments) and INCA EDS analysis software, in order to identify the iron-rich intermetallic phase. The specimen was also subject to examination of chemistry of both intermetallic phase and matrix of the alloy. The next step included evaluation of the nanoindentation hardness H_{IT} and nanoindentation elastic modulus E_{IT} with the use of Nanoindentation Tester NHT (CSM Instruments).

3. Research results

Fig. 1 shows microstructure of the alloy cooled at rate 5.1°C/s. Fig. 2 presents example results of studies aimed at disclosure of intermetallic phase rich in iron.

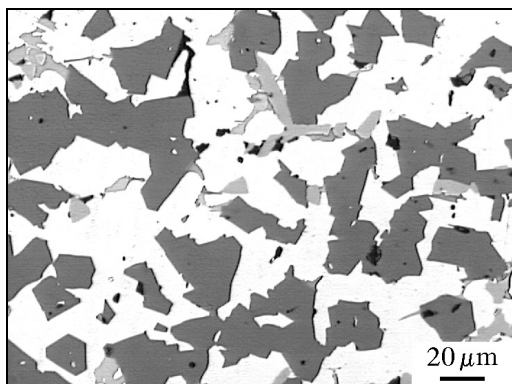


Fig. 1. Microstructure of aluminium-silicon alloy with matrix containing primary silicon, eutectic silicon, and intermetallic phase precipitates with diversified shape, cooled at rate of 5.1°C/s

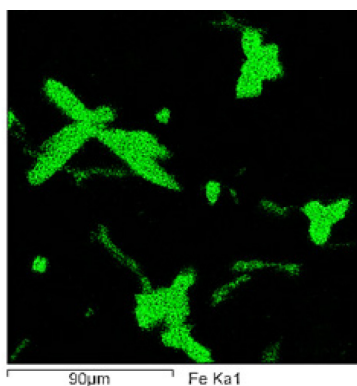


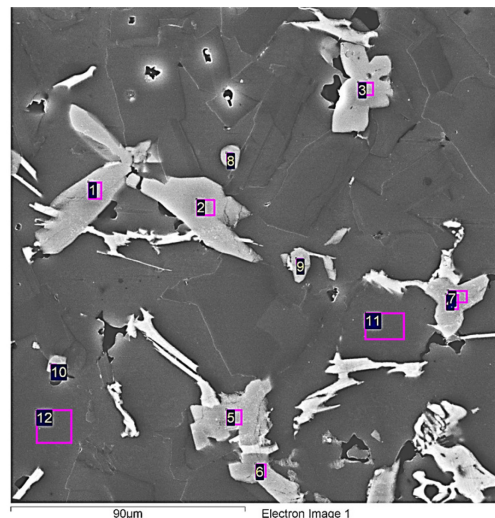
Fig. 2. Results of identification of iron-rich precipitates of intermetallic phase (polygons, isolated and clustered in star-like formations) for the alloy cooled at rate 5.1°C/s

Examination of distribution of alloying elements (mapping) has revealed that the intermetallic phases rich in iron were precipitates in the form of polygons. For a selected precipitate of such iron-rich intermetallic phase, microanalysis of chemistry was performed as shown in Fig. 3.

The obtained results allowed to conclude that the phase contains 12.0% – 14.3% Fe. Further, aluminium and silicon are prevailing components, but also manganese, chromium, copper, nickel, and vanadium are present.

Nanoindentation tests were carried out with the use of a diamond indenter B-L 32 with Berkovich-type tip. The maximum value of load force was 20 mN. The indenter loading and unloading rate for all of the analysed microstructure components was 40 mN/min. The maximum load force application time was 15 s.

The measurements were taken for precipitates of silicone and iron-rich intermetallic phases as well as for the matrix. The results in the form of averages from 3 measurements are given in Table 4.



Point #	Al	Si	Mn	Cu	Cr	Ni	V	Fe	Mg
1	59.6	11.5	4.4	3.5	4.1	2.2	1.0	13.7	–
2	59.3	11.4	4.3	3.2	4.5	2.0	1.0	14.3	–
3	58.3	11.2	4.5	4.3	4.6	1.8	1.0	14.3	–
4	59.5	11.0	3.8	4.7	4.2	2.3	1.0	13.5	–
5	59.1	11.3	4.4	3.3	4.6	2.0	1.0	14.3	–
6	58.8	11.3	4.4	3.9	4.7	2.3	1.1	13.5	–
7	59.9	11.0	4.4	4.9	4.4	2.4	1.0	12.0	–
8	59.1	11.3	4.4	4.2	4.7	1.9	1.1	13.3	–
9	59.0	11.4	4.3	4.8	4.1	1.7	1.0	13.7	–
10	59.1	11.3	4.4	4.3	4.7	2.3	1.0	12.9	–
11	95.9	1.5	–	2.2	–	–	–	–	0.4
12	96.4	1.3	–	1.9	–	–	–	–	0.4

Fig. 3. Results of point-like quantitative analysis of polygon-shaped intermetallic phases (rich in iron) and the matrix for alloy cooled at rate 5.1°C/s

Table 4.

The effect of the cooling rate on material properties (H_{IT} - nanoindentation hardness, E_{IT} - nanoindentation elastic modulus) of individual microstructure components of a complex Al-Si alloy

Intermetallic phase	$v_{cool} = 5.1^\circ\text{C/s}$		$v_{cool} = 1.5^\circ\text{C/s}$ [14]	
	H_{IT} , GPa	E_{IT} , GPa	H_{IT} , GPa	E_{IT} , GPa
Silicon	11.9	158	12.1	161
Intermetallic phase (polygons)	13.8	204	14.2	207
Matrix	1.6	103	1.5	99

4. Summary and conclusions

In a complex aluminium-silicon alloy containing 0.56% of iron, cooled at rate 5.1°C/s, presence of intermetallic phase was found containing 12.0–14.3% Fe, rich in aluminium (58.3–59.9%), silicon (11.0–11.5%), manganese (3.8–4.5%), and chromium (4.1–4.7%), containing also copper (3.2–4.8%), nickel (1.7–2.4%), and vanadium (1.0–1.1%). The phase was characterised, compared to this obtained as a result of cooling at rate 1.5°C/s [14], with a higher content of aluminium, copper, and nickel and lower content of iron, silicon, manganese, chromium,

and vanadium. The effect of these differences consisted in a decrease of values of the analysed properties (H_{IT} - nanoindentation hardness, E_{IT} - nanoindentation elastic modulus). In case of silicon precipitates, the obtained values of the analysed parameters were practically the same, as chemical composition of these precipitates remained virtually unchanged. Slightly lower values can be explained by smaller dimensions of precipitates which enable them to be plunged plastically in the matrix in the course of indentation. As far as the matrix is concerned, it has been found that with increasing alloy cooling rate, the analysed material properties improved, which is a result of increased supersaturation of the matrix with alloying elements.

References

- [1] Wang, L., Apelian, D. & Makhlof, M. (1999). Iron-Bearing Compounds in Al-Si Die-Casting Alloys: Their morphology and conditions under which they form. *AFS Transactions*. 146, 231-238.
- [2] Mondolfo, L.F. (1976). *Aluminium Alloys. Structure and Properties*. Butterworth.
- [3] Taylor, J.A., Schaffer, G.B., & StJohn, D.H. (1999). The Role of iron in the formation of porosity in Al-Si-Cu-based casting alloys: Part III. A microstructural model. *Metallurgical and Materials Transactions A*. 30A, 1657-1662.
- [4] Horng, J.H., Jiang, D.S., Lui, T.S., & Chen, L.H. (2000). The fracture behaviour of A356 alloys with different iron contents under resonant vibration. *Int. J. of Cast Metals Res*. 13(4), 215-222.
- [5] Khalifa, W., Samuel, F.H. & Gruzleski J.E. (2003). Iron intermetallic phases in the Al corner of the Al-Si-Fe system. *Metallurgical and Materials Transactions A*. 34A, 807-825.
- [6] Tang, S.K. & Sritharan, T. (1998) Morphology of β -AlFeSi intermetallic in Al-7Si castings. *Materials Science and Technology*. 14, 738-742.
- [7] Pucella, G., Samuel, A.M. & Samuel F.H. (1999). Sludge formation in Sr modified Al-11.5 wt% Si die casting alloys. *AFS Transactions*. 107, 117-125.
- [8] Murali, S., Raman, K.S. & Murthy, K.S.S. (1995). The formation of β -FeSiAl₃ and Be-Fe phases in Al-7Si-0.3Mg alloy containing Be. *Materials Science and Engineering A*. 190, 165-172.
- [9] Tan, Y.H., Lee, S.L. & Lin, Y.L. (1995). Effects of Be and Fe additions on the microstructure and mechanical properties of A357.0 alloys. *Metallurgical and Materials Transactions A*. 26A, 1195-1205.
- [10] Simensen, C.J., Rolfsen, T.L. (1997). Production of π -AlMgSiFe crystals. *Zeitschrift für Metallkunde*. 88, 142-146.
- [11] Warmuzek, M., Sęk-Sas, G. & Lech, Z. (2002). Microstructure evolution in presence of the transition metals (Fe, Mn, and Cr) in the Al-Si alloys. *Biuletyn Instytutu Odlewnictwa*. 6, 4-11. (in Polish).
- [12] Ahmad, R. & Marshall, R.I. (2003). Effect of superheating on iron-rich plate-type compounds in aluminium-silicon alloys. *Int. J. of Cast Metals Res*. 15, 497-504.
- [13] Narayanan, L.A., Samuel, F.H. & Gruzleski, J.E. (1994). The crystallization behavior of iron - containing intermetallic compounds in 319 aluminum alloys. *Metallurgical and Materials Transactions A*. 25 A, 1761-1773.
- [14] Tupaj, M., Orłowicz, W., Mróz, M. & Trytek, A. (2015). Materials Properties of Iron-rich Intermetallic Phase in a Multicomponent Aluminium-Silicon Alloy. *Archives of Foundry Engineering*. 15(1), 111-114.