Observation on the Formation of β-Al_5FeSi Phase Depending on the Content of Fe in Aluminium Cast Alloy

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This work examines the effect on the formation of second Fe phase group Al-Fe-Si: Al_5FeSi in aluminium alloy containing the iron of 0.123 wt. %, 0.454 wt. % and 0.655 wt. %. Studies were focused on the size and content of Al_5FeSi changes and their effect on the porosity formation and mechanical properties in AlSi7Mg0.3 cast alloys. One of the key problems of these phases is their harmful morphology effect on to properties (mechanical, fracture, fatigue and so on) of aluminium casts. The increasing usage of recycled metals for production new casts is the main factor which influences content of iron in composition of aluminium cast alloys. The observation with using scanning electron microscope (SEM) and energy dispersive X-ray analysis (EDX), optical microscopy and quantitative analysis software confirmes enlarging of the β-needles phases particles and size of pores with increasing of iron content.

Keywords: β-Al_5FeSi formation, iron in aluminium cast alloys, porosity of aluminium cast alloys,

1 Introduction

Regulations and directives from instances such as EU are sanctioned with the aim to both decrease the amount of toxic gasses released to the atmosphere and to utilise used metal scrap. The choice of whether to use alloys based on primary aluminium (low Fe levels, high cost) or secondary aluminium (moderate/high Fe levels, cheaper) for a cast product is often a commercial decision. However, a compromise may be necessary between the desire for reduced metal cost and the need to maximise casting productivity via reduced defect formation and to minimise the deleterious effect of iron on mechanical properties [1-6]. Therefore is important to study this type of microstructural components.

The iron (Fe), as major impurity element in Al-alloys is frequently acquired for non-high pressure die casting operations usually at levels ranging from ~ 0.25 to 0.8 wt. %, being most common with values around 0.4-0.8 wt. % and for high pressure die casting operations usually contain up to 1.5 wt. % of Fe from Al scrap during the recycling proces [4,7].

Liu et al. [8] reported that solubility of iron is approximately 1.8 wt. % at 655 °C in liquid pure Al but only 0.0052 wt. % at 450 °C in solid pure Al. Therefore, almost all the iron will precipitate from liquid Al alloys in the form of iron-rich intermetallics. The iron-rich intermetallic phases are groupen into three major morphology: polyhedral or star-like, Chinese script or skeleton like and polyhedral [1,2,4-6,8,9]. Šerák et al. [10] describes principles of reduction the deleterious effect of iron on the properties of Al-Si cast alloys. First principle is change in crystallization of iron-rich phases. This is possible by the mean of addition of some elements into the melt: Mn, Co, Cr, Be, Ni. This addition causes changes of harmfull morphology of phases to less harmfull especially Chinese script or skeleton like. Second principle is heat treatment of alloy, but here are two possibilities: heat treatment of the melt or of the casting. Both of the methods lead to fragmentation of selected iron-rich phases into smaller particles. Third principle present decrease in iron content into the acceptable level or full removing of iron in ideal case. This method uses high difference between the iron content in upper and bottom regions of crucible [10,11]. In secondary aluminium alloys is iron separated from molten aluminum, but not always it is economical. Among various Fe-rich intermetallic phases in Al-Si cast alloys (α-Al_8Fe_2Si; $Al_{15}(FeMn)_3Si_2$; β- Al_5FeSi; π-$Al_8Mg_3FeSi_6$ and δ-Al_4FeSi_2), the β-Al_5FeSi needles (platelets) are considered as the most deleterious compounds since their edges act as stress concentration sites, facilitating crack iniciation in the matrix [1,2,9,12-13]. The formation of Fe-rich intermetallic phase's group β is connecting with two possibilities of decreasing the properties of aluminium alloys. First is the brittleness of Fe rich phases and the second porosity, whose reduces the ductility of Al-Si alloys. The brittleness led to formation of cracks in Fe-rich phase's region. Cracks are caused with existing gas or shrinkage problems arising during solidification. If there are cracks the gas go deep into the dendritic mesh and the porosity is forming [14,15]. Therefore porosity is a leading cause in the reduction of mechanical properties, particularly elongation and fatigue resistance, as well as a loss of pressure tightness and a degradation of the surface appearance in cast parts [3,16,17]. A lot of work shows that the ultimate tensile strength of aluminium alloys did not relate to the average volume fraction of porosity, but with the length, shape or the size of the pore in the cross-section surface [17]. Also the harmfull effect of the Al_5FeSi phases morphology (2D brittle needles and 3D long platelets) was investigated in a lot of works, but the quantitatively understanding of the size and distribution of the Fe-phases are of paramount importance in the physical metallurgy of recycling Al alloys, and in the manufacturing of high-quality components for the transportation industry [18]. Therefore this study describes effect of different iron content on formation of Fe-needles phases without special solidification, casting or other condition and their effect on to porosity and mechnaical properties in secondary AlSi7Mg0.3 cat alloys.

2 Experimental procedure

The chemical compositions of the secondary AlSi7Mg0.3 alloys prepared for present study are reported in Table 1; they were characterized by the different conents of the Fe (0.123 wt. % – material A, 0.454 wt. % – material B and 0.655 wt. % - material C). Experimental bars with dimensions 300 mm length and diameter 20 mm were casted into the sand mould (Fig. 1a). From these bars were made samples for mechanical test (Fig. 1b). The experimental materials were not modified, grain refined or heat treated. The adition of Fe were performed in order to study influence of higher Fe content in aluminium alloys, which may occur mainly during use of scrap for production of cast components especially casts in mechanical, electrical engineering, automotive and aerospace industries for shipbuilding, and so on [17].

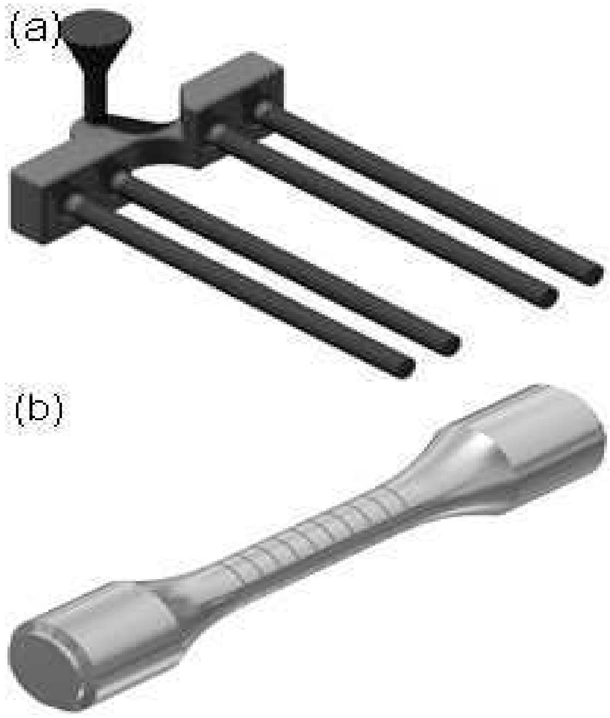

Fig. 1 *The experimental material (a) bars; (b) testing specimens*

Tab. 1 *The chemical compositions of experimental materials (in wt. %)*

Material	Al	Si	Fe	Cu	Mn	Mg	Zn	Ti
A	92.253	7.028	0.123	0.013	0.009	0.354	0.036	0.123
B	91.673	7.340	0.454	0.021	0.009	0.302	0.020	0.118
C	91.486	7.315	0.655	0.030	0.010	0.292	0.028	0.120

The amount of Fe is higher for experimental materials, and is expected the effect of this element on mechanical properties and formation of porosity. It is well known that this amount leads to the formation of a Fe-rich intermetallic phase's group called "beta or β-needles phase" and these phases deteriorate the properties too, because of their morphology (sharp - edged needles). The formation of intermetallic phases and porosity in experimental material was studied by using Thermo-calc software, light microscope NEOPHOT 32 with quantitative analyser software NIS Element 4.0 and scanning electron microscope VEGA LMU II with EDX analysis. Metallographic samples have been selected from the bars (Fig. 1a), which were standard prepared for metallographic observations such as – wet ground on SiC papers, DP polished with 3 µm diamond paste followed by Struers Op-S, etched by 0.5 % HF and H_2SO_4 for the highlighting of Fe-intermetallic phases. The results of quantitative analysis represent the measured values on about twenty-five field of each set of samples.

3 Experimental results

The investigation of the basic microstructure showed that microstructure of each experimental material (A, B, C) consists of α-phase dendrites, eutectic (it is mechanical mixture of α-phase and eutectic silicon) and different intermetallic phases. The termocals prediction (Fig. 2) of phase's formation and EDX analysis (Fig. 3 - point and mapping) confirmed presence of these intermetallic phases in microstructure of experimental material:

- Fe-rich intermetallic phases in needles form: Al_5FeSi,
- Fe-rich intermetalic phases in form of skeleton-like: $Al_{15}(FeMg)_2Si_2$
- Mg-rich intermetallic phases in form of script-like: $Al_5Fe_2Mg_2Si_6$ and $Al_8FeMg3Si_6$

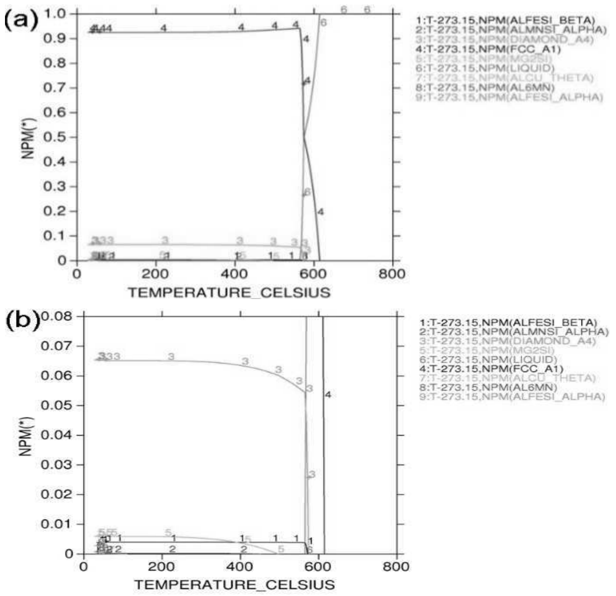

Fig. 2 *Thermo-calc prediction of phase formation in AlSi7Mg0.3 cast alloys.*

The β-phases crystallizes as thin plates that look like needles in the cross section. This phase is mostly associated with iron levels greater than 1 wt. %. The authors [19] examined that enough iron segregation occurs during solidification to cause and the β-phase is to form, even when the iron level is much less than 1wt. %.

The experimental results of metallography observation (Fig.4) and quantitative analysis (Table 2) demonstrated that iron phases content increase with increasing content of wt. % Fe. Also were obtained that iron content determines the shape of these needles. This needles are longer with sharper ending and curved shape (Fig. 4b) and also wider (Fig. 4b, c). These results relate with findings of authors [19] which confirmed that the higher the iron content is, the longer and wider are the needles. In any case, results show that higher Fe content leads to growing length of Fe-rich needles intermetallic phases which is not good with regard to properties. The removing of Fe from the melts is a very costly process [20,21]. Therefore it is important to study how this elements influence the properties of aluminum materials. The importance for this study was porosity formation and mechanical properties changes.

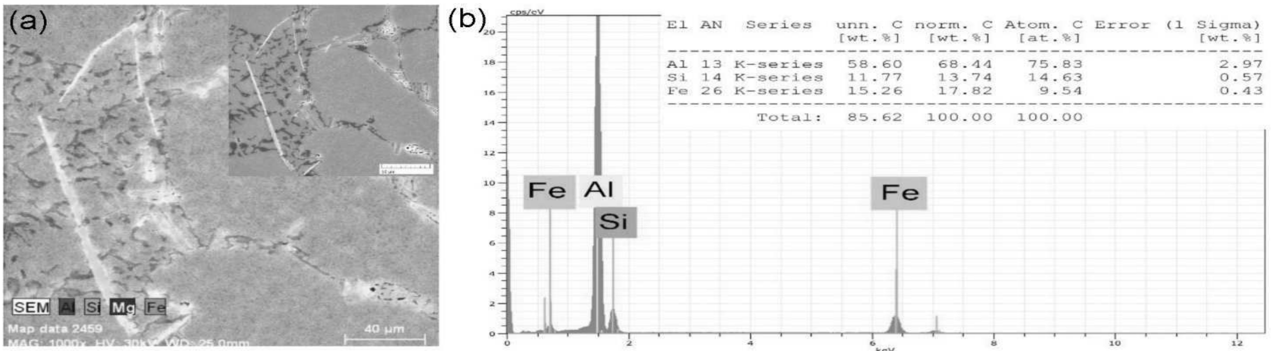

Fig. 3 *Microstructure evaluation by using SEM observation, etch. 0.5 % HF. (a) mapping analysis; (b) point analysis of Fe needles.*

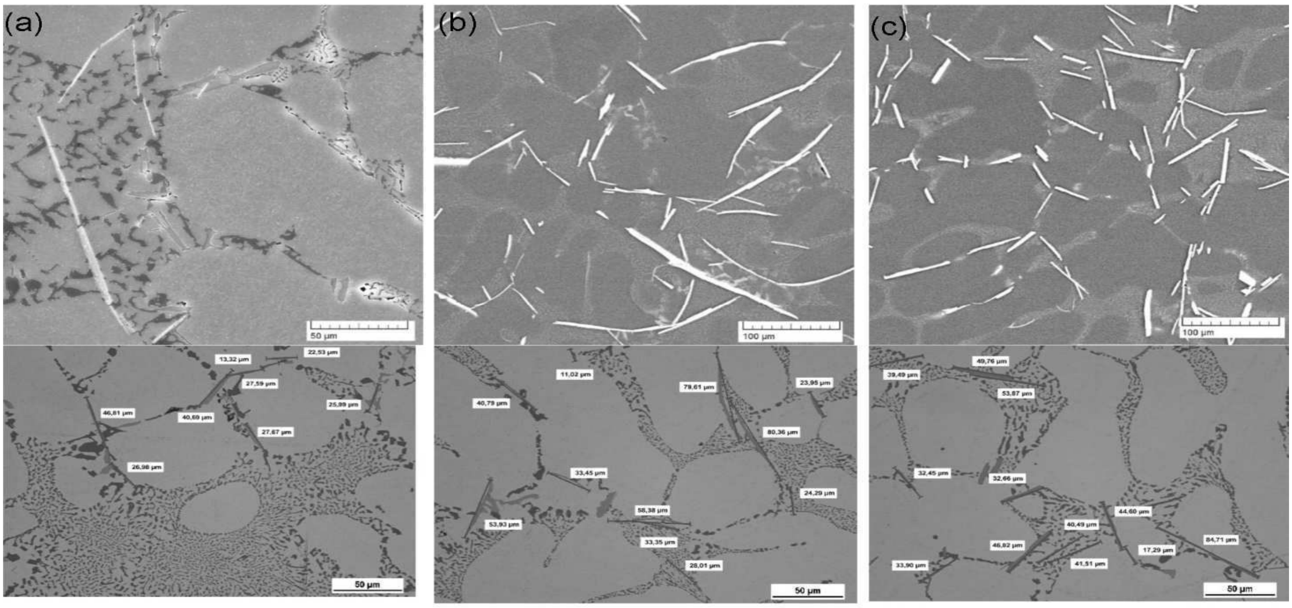

Fig. 4 *The Al5FeSi phases formation and their quantitative assesment in experimental amterials. (a) alloy A; (b) alloy B; (c) alloy C.*

Tab. 2 *The quantitative analysis of Al_5FeSi phase*

Material	A	B	C
The average length of Al_5FeSi [μm^2]	24.49	35.43	41.93
The average volume fraction of Al_5FeSi [%]	1	2.3	2.6

Tab. 3 *The quantitative analysis of pores*

Material	A	B	C
The max. area size of pores [μm^2]	125723.4	125066.4	219627.1
The max. volume fraction of pores [%]	3.8	5.6	3.2

Sacinty et al. [22] examined that the β platelets grow twice as big when the Fe concentration is doubled and this, in turn, increases shrinkage porosity and leads to a 3-fold decrease in the tensile elongation values. To examine the effect of the Fe content on the porosity formation in experimental materials were specimens subject to quantitative analysis of area size and volume fraction of porosity (Fig. 4 and Table 3). The results point that the biggest area size of porosity is in material C which declare bigger influence of Fe-rich needles on to formation of porosity in experimental material. The maximum volume fraction shows that the biggest content of porosity is in materia B (Table 3 and Fig. 4). These results probably related with morphology of Fe needles. These are more sharpened as in as cast state which relate with higher stress concentration.

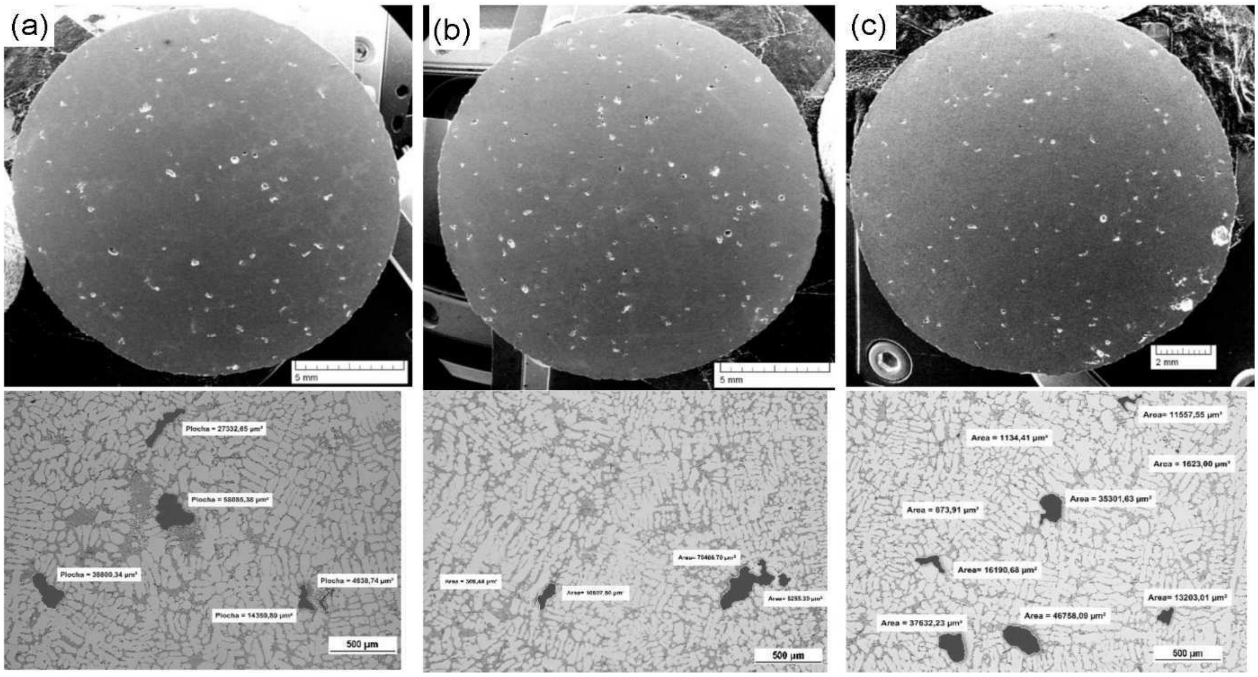

Fig. 5 *The assesment of porosity formation in experiemtnal materials. (a) alloys A; (b) alloy B; (c) alloy C.*

The mechanical proeprties results demonstrated changes depending on Fe content too (Table 4). While these structural changes are reflected by a modest improvement in the ductility, ultimate tensile strength (UTS) and Brinell hardness (HBW 5/250/15) are comparable for all material (A,B,C). The differences were about 5 or 7 % whch is negligible. The authors [22] mentoined in their work that hence, limiting the Fe content of the primary AlSi7Mg0.3 alloy to 0.12 wt. % is worthwhile and pays off with superior microstructural features and mechanical properties. According this experimental study is possible to demonstrated that increasing amout of Fe have negative effect to greater formation of needles iron rich phases, but mechanical properties are not significantly affected.

Tab. 4 *The mechanical properties of experimental materials*

Material	**A**	**B**	**C**
UTS [MPa]	140	150	147
HBW 5/250/15	52	55	54
Ductility [%]	1.45	1.91	1.58

4 Conclusions

The results presented in this paper refer about influence of different Fe content on formation of Fe needles phase and porosity in the secondary AlSi7Mg0.3 cast alloy. There have been observed these changes during experimental work:

- The microstructure of experimental material is created with α-matrix, eutectic and intermetallic phases: Al_5FeSi, $Al_{15}(FeMg)_2Si_2$, $Al_5Fe_2Mg_2Si_6$ and $Al_8FeMg3Si_6$. The higher amount of Fe in chemical composition lead to formation rather Al_5FeSi then $Al_{15}(FeMg)_2Si_2$ phase.
- The metallography observation and quantitative analysis show that with increasing Fe content increase the length of Fe needles phases and their volume fraction. The quantitative assessment of porosity shows that 3.7 times higher amount of Fe (0.454 wt. % - alloy B) caused formation smalles pores with their higher volume. The 5.3 times higher amount of Fe (0.655 wt. % - alloy C) lead to formation larger pores with small volume on cross section. In any case, results show that higher Fe content leads to growing length of Fe-rich needles intermetallic phases and creating of larger pores which is not desirable.
- The results of mechanical tests demonstrated on to maximum properties in state (B) with 0.454 % of Fe (UTS = 150 MPa, HBW = 55, and A = 1.91 %). It relate with smallest size of porosity despite the fact that there is a large volume of porosity. The material A has the lowest properties, but differences were very small.

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References

[1] CAO, X., CAMPBELL, J. (2006). Morphology of β-Al_5FeSi Phase in Al-Si Cast Alloys. In: *Materials Transactions*, Vol. 47, No. 5, pp. 1303 - 1312.

[2] SHOUXUN, J., WENCHAO, Y., FENG, G., DOUGLAS, W., ZHONGYUN, F. (2013). Effect of iron on the microstructure and mechanical property of Al–Mg–Si–Mn and Al–Mg–Si die cast alloys. In: *Materials Science & Engineering*, Vol. A 564, pp. 130–139.

[3] FINTOVA, S., KONSTANTOVA, V., KONEČNA, R., NICOLETTO, G. (2008). Experimental study of porosity and fatigue behaviour of cast Al–Si alloys. In: *Proceedings of Metal 2008*, pp. 1-8.

[4] TAYLOR, A. J. (2012). Iron-containing intermetallic phases in Al-Si based casting alloys. In: *Procedia Materials Science*, Vol. 1, pp. 19 – 33.

[5] SLAGSVOLD, M. (2011). Effect of Fe and Si content in Aluminium Alloys as a result of increased recycling. Ph.D. thesis, Norwegian University of Science Technology.

[6] LAGO, J., RIDKÝ, O., BOKUVKA, O., NOVÝ, F. (2016). Qualitative evaluations of the AlSi7Mg0.3 microstructure by the X-Ray diffractometry. In: *Manufacturing Technology*, Vol. 16, No. 6, pp. 1284-1291.

[7] PUNCREOBUTR, C., PHILLION, A.B., FIFE, J.L., ROCKETT, P., HORSFIELD, A.P., LEE. P.D. (2014). In situ quantification of the nucleation and growth of Fe-rich intermetallics during Al alloy solidification. In: *Acta Materialia*, Vol, 79, pp. 292–303.

[8] LIU, K., CAO, X., CHEN, X. G. (2011). Solidification of Iron-Rich Intermetallic Phases in Al-4.5Cu-0.3Fe Cast Alloy. In: Metallurgical and materials transactions, Vol. 42A, pp. 2004-2016.

[9] HURTALOVÁ, L., TILLOVÁ, E. (2013). Elimination of the negative effect of Fe-rich intermetallic phases in secondary (recycled) aluminium cast alloy. In: *Manufacturing Technology*. Vol. 13, No. 1, pp. 44- 50.

[10] ŠERÁK, J., VOJTĚCH, D., NOVÁK, P., DÁM, K., JANOUŠEK, T. (2009). The influence of annealing conditions and microstructure of Al-SiCuMgFe alloys. In: *Proceedings of Metal,* pp. 1-8.

[11] SHABESTARI S. G. (2004). Effects of ageing parameters on the quality of 413-type commercial alloys. In: *Materials and Science Engineering,* Vol. 383A, pp. 289-298.

[12] FABRIZI, A., FERRARO, S., TIMELLI, G. (2014). The influence of Fe, Mn and Cr additions on the formation of iron-rich intermetallic phases in Al-Si die-casting alloy. In: *TMS: Joutnal the Minerals, Metals and Materials Society, Shape casting*, edited by: Murat Tiryakioglu, John Campbell, Glenn Byczynski. pp. 277-291.

[13] LU, L., DAHLE, A.K. (2005). Iron-Rich Intermetallic Phases and Their Role in Casting Defect Formation in Hypoeutectic Al-Si Alloys. In: Metallurgical and materials transactions, Vol. 36A, pp. 819-835.

[14] MAHTA, M., EMAMY, M., CAO, X., CAMPBELL, J. (2008). Overview of β-Al□FeSi phase in Al-Si alloys. In: *Materials Science Research Trends*, pp. 251-271.

[15] KUČERA, V., DALIBOR, V. (2018). Processing of Al-Si Waste Contaminated with Iron by Powder Metallurgy. In: *Manufacturing technology*, Vol. 18, No.:1, pp. 60-65.

[16] MAYER, H., PAPAKYRIACOU, M., ZETTL, B., STANZL-TSCHEGG, S.E. (2003). Influence of porosity on the fatigue limit of die cast magnesium and aluminium alloys. In: *International Journal of Fatigue*, Vol. 25, pp. 245–256.

[17] BANGYIKHAN, K. (2005). Effects of Oxide film, Fe-rich phase, Porosity and their Interactions on Tensile Properties of Cast Al-Si-Mg Alloys. *Ph.D. thesis*, University of Birmingham.

[18] ZHAO, Y., DU, W., KOE, B., CONNOLLEY, T., IRVINE, S., ALLAN, P.K., SCHLEPÜTZ, C.M., ZHANG, W., WANG, F., ESKIN, D.G., MI. J. (2018). 3D characterisation of the Fe-rich intermetallic phases in recycled Al alloys by synchrotron X-ray microtomography and skeletonisation. In: *Scripta Materialia*, Vol. 146, pp. 321–326.

[19] HAGHDADI, N., ZAREI-HANZAKI, A, ABEDI, H.R., ABOU-RAS, D., KAWASAKI, M., ZHILYAEV, A.P. (2016). Evolution of microstructure and mechanical properties in a hypoeutectic Al-Si-Mg alloy processed by accumulative back extrusion. In: *Materials science and engineering*, Vol. 651A, pp. 269-279.

[20] KARAMOUZ, M., AZARBARMAS, M., EMAMY, M., ALIPOUR, M. (2013). Microstructure, hardness and tensile properties of A380 aluminum alloy with and without Li additions. In: *Materials science and engineering*, Vol. 582A, pp. 409-414.

[21] SREEJA-KUMARI, S. S., PILLAI, R. M., NOGITA, K., DAHLE, A. K., PAI, B. C. (2006). Influence of calcium on the microstructure and properties of an Al-7Si-0.3Mg-*x*Fe alloy. In: *Metallurgical and materials transactions*, Vol. 37 A, pp. 2581-2587.

[22] SACINTI, M., CUBUKLUSU, E., BIROL, Y. (2017). Effect of iron on microstructure and mechanical properties of primary AlSi7Mg0.3 alloy. In: *International journal of cast metals research*, Vol. 30, No. 2, pp. 96-102.

10.21062/ujep/147.2018/a/1213-2489/MT/18/4/611