Influence of the AlSi12 alloy inoculation on the microstructure and mechanical properties development

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INFLUENCE OF THE AlSi12 ALLOY INOCULATION ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES DEVELOPMENT

Summary

Due to a wide range of favourable properties, aluminium alloys found its application in almost all industrial branches. Potential of improving the usage properties has been recognized through forming of favourable intermetallic in mutual interaction of numerous alloying elements such as silicon, copper or magnesium, along with trace elements such as iron and manganese. Other important elements such as titanium, boron, strontium or sodium are intently added through targeted melt treatment in order to improve solidification path by increasing the nucleation potential and therefore changing morphology. Consideration of the casting technology parameters which influence the cooling / solidification rate also represent the base for changing the microstructure and final quality of the casting. Synergy activity of alloying and/or trace elements and theirs interaction and technological parameters of casting process is of great importance in consideration of alloy applicability.

This investigation deals with EN AC AlSi12 (EN AC 44100) eutectic alloy with narrow solidification interval, intended for rapid cooling/solidification technology such as high pressure casting (HPDC). Effect of different mode of AlSi12 alloy melt treatment on microstructure and mechanical properties was monitored as a quality insurance. Applied melt treatment mode consists of modification of eutectic with addition of AlSr10 master alloy in all investigated cases. The difference in melt treatment mode was in targeted addition of AlTi5B master alloy. Hypothesis of the examination is based on the assumption that targeted melt treatment can influence the solidification manner, development of microstructural characteristics and finally achieving mechanical properties of the alloy in accordance to corresponded casting geometry and/or technology application.

Key words: *AlSi12 alloy, inoculation, modification, microstructure, mechanical properties*

Introduction

In general, aluminum - silicon alloys characterized with low specific weight, possess good castability, relatively low contraction, low production price, good weldability and high corrosion resistance and tensile strength. Low melting point and narrow solidification interval indicates AlSi12 alloy as a most commonly used one due to the uniformly distributed eutectic microstructure indicating superior mechanical and technological properties [1]. Eutectic Al-Si alloy is applied for complex geometry, corrosion resistant and leakage castings. Melt treatment by grain refinement and modification is of essential importance for casting quality.

Investigated AlSi12 alloy (EN AC 44100) belongs to the group of "eutectic alloys" [2]. Silicon is one of the most important alloying elements which comprehend to good castability of aluminium alloys. Addition of silicon improves feeding capability and resistance to hot cracks

[3, 4]. Synergy of alloying and trace elements comprehend to the wide range of intermetallic phases' evolution [5-8].

The influence of Al–Si eutectic morphology is significant for mechanical properties development. Mechanical properties are correlated to fibrous morphology of eutectic achieved either by chemical modification or adjusting solidification i.e. technological parameters [9]. In general Al–Si modification can be achieved through additions of trace impurity elements [10–13] or increased solidification velocity [14-17]. The intention of modification is to change the morphology from lamellar/acicular to fibrous, reduce the size and interphase spacing of eutectic (α_{Al}+ β_{Si}), which all comprehend to the increase of tensile strength.

Grain refining is an important part of melt treatment used also with the aim of improvement of mechanical strength, ductility, homogeneity, feeding during solidification and other desired properties of Al-Si casting alloys [18, 19]. High cooling rate obtained by recent casting technologies (HPDC) enables grain refinement due to rapid solidification process. Grain refinement by inoculation is therefore of essential importance for sand- and permanent mould casting due to the lower cooling rate. Also, near "eutectic" composition alloys are characterized with narrow temperature-time solidification interval, which also comprehends to the development of fine microstructure consists of uniformly distributed eutectic. In general, nucleation potential paradigm offers a number of explanations of grain refinement mechanisms in aluminum alloys. Two well-known models deal with "nucleant paradigm" [20] which is focused to nucleation event and the other "solute element paradigm", which consider the effect of solute elements on grain growth. Both models are very important and should be considered simultaneously [20, 21] by incorporating of solute element influence effect on growth restriction [20] and "free growth model" [18].

This investigation deals with EN AC AlSi12 (EN AC 44100) eutectic alloy intended for high pressure die casting. AlSi12 alloy processed with different mode of melt treatment was monitored for quality insurance. Applied melt treatment mode consists of modification of eutectic with addition of AlSr10 master alloy in all investigated cases. The difference in melt treatment mode was targeted addition of AlTi5B master alloy. Different effect can be expected due to different relationship between melt treatment and casting technology parameters as shown in Fig. 1.

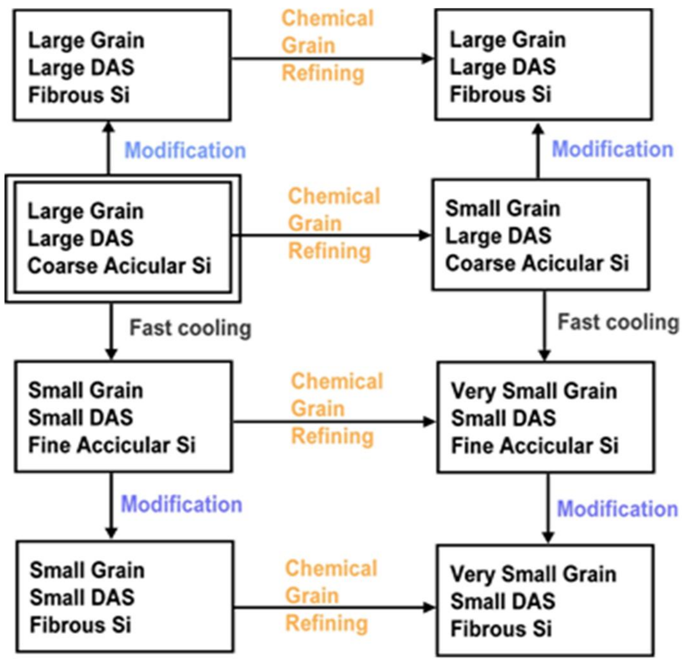


Figure 1. Influence of melt treatment on the alloy microstructure development [22]

Hypothesis of the examination is based on the assumption that targeted melt treatment can influence the solidification manner, development of microstructural characteristics and finally achieving mechanical properties of the alloy in accordance to corresponded casting geometry and/or technology application.

Experimental

Experimental included determination of AlSi12 (EN AC-44100) alloy microstructure and mechanical properties. Investigation methodology comprehends melting and preparation of requested chemical composition according requisition. Chemical composition was determined "in situ" using SPECTROMAXx OES Metals Analyzer prior and after the melt treatment with master alloys.

Melt pre-treatment covered degassing of the melt with the nitrogen (N_2) using a MTS 1500 - Foseco equipment. Melt treatment was also performed through modification with AlSr10 and in second stage with and without grain refinement by inoculation using AlTi5B master alloy.

Experimental included gravity casting of an alloy in standard croning cell and ASTM B108 mould [23]. Simple thermal analysis was performed during cooling/solidification of melt in croning cell for both cases of melt treatment using NI-9211 device equipped with NI cDAQ-9172 module. Numerical simulation of solidification and porosity prediction of ASTM B108 casting was performed using ProCast software.

Mechanical tensile properties investigations were performed on universal testing machine Zwick 50kN, at room temperature T = 20 °C in accordance to EN 10002-1:1998 [24].

Samples for metallographic investigation were prepared by standard metallographic preparation procedure by grinding and polishing, followed by etching in 0.5% HF. Metallographic analysis was performed using optical microscope Olympus GX51 in order to visually identification of particular microstructural constituents. Microstructures were acquired using digital camera Olympus DP70, while the analysis was performed by Analysis®MaterialsResearchLab software. Detail microstructural investigations were performed using scanning electron microscope (SEM) Tescan Vega TS 5136 MM equipped with energy dispersive spectrometer (EDS) Bruker.

Results and discussion

Chemical composition of AlSi12 (EN AC-44100) alloy investigation resulted with values compared with required one [2], as shown in Table 1. Chemical composition of investigated samples differs in melt treatment by addition of inoculant (AlTi5B). The samples are respectively marked:

- M with addition of AlSr10 modifying agent and
- MI with addition of AlSr10 modifying agent and AlTi5B inoculation agent.

Table 1. Chemical composition of AlSi12 (EN AC-44100) alloy

	Si	Fe	Cu	Mn	Mg	Cr	Ni	Zn	Ti	B	Sr	V
[2]	11.93	0.426	0.116	0.465	0.046	0.011	0.007	0.044	0.036	0.0034	0.012	0.012
M	12.49	0.468	0.114	0.455	0.047	0.010	0.006	0.043	0.038	0.0035	0.016	0.012
MI	12.51	0.474	0.114	0.449	0.045	0.010	0.007	0.043	0.064	0.0074	0.017	0.012

The AlSi12 (EN AC-44100) alloy specificity is high ratio *Fe : Mn ≈ 1 : 1*. Chemical composition comparison indicated consistant content of interesting elements except for afterward added Ti and B.

Numerical simulation of AlSi12 (EN AC-44100) alloy solidification in ASTM B108 mould using ProCast software resulted in follow-up of filling and solidification path in characteristic solidification stages as well as prediction of porosity in final casting, as shown in Fig. 2.

(a) 1.6 vol.% solid phase

(b) 2.9 vol. % solid phase

(c) 8.8 vol.% solid phase

(d) 14 vol.% solid phase

(e) Casting porosity ratio

Figure 2. Solidification path and final porosity in casting.

Results of numerical simulation indicate hot spots in lower part of the casting. Although the total time of filling and solidification does not exceed 120s, due to mould preheating (140°C) and adequate thermal coefficient of heat conductivity between melt and mould and as well due to narrow solidification interval of AlSi12 alloy, porosity occurs in hot spots. Simulation does not reveal significant difference in solidification path for M and MI samples.

A thermal analysis performed in croning cell resulted in cooling curves of base M (C1A) and refined MI (C1B) melt, as shown if Figure 3.

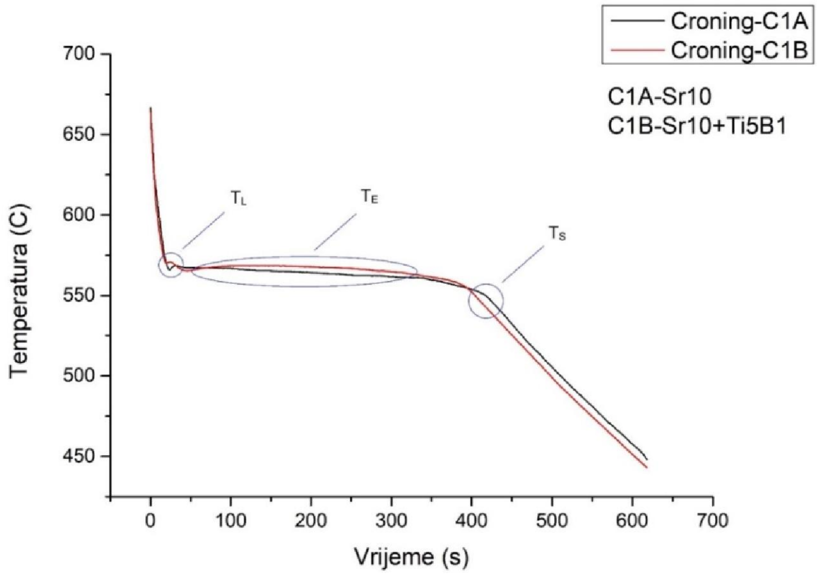


Figure 3. Cooling curves of AlSi12 alloy croning cell casting.

Characteristic temperature-time dependence revealed an influence of inoculant addition in the initial solidification stages by lowering the liquidus undercooling and shortening the solidification time interval.

Microstructural investigation using light and electron microscopy was performed on the same samples from croning cell (Fig. 4) and as well on ASTM B108 samples from test (Fig.5) and inflow part (Fig 6) of the casting after mechanical investigation.

M (C1A) MI (C1B)

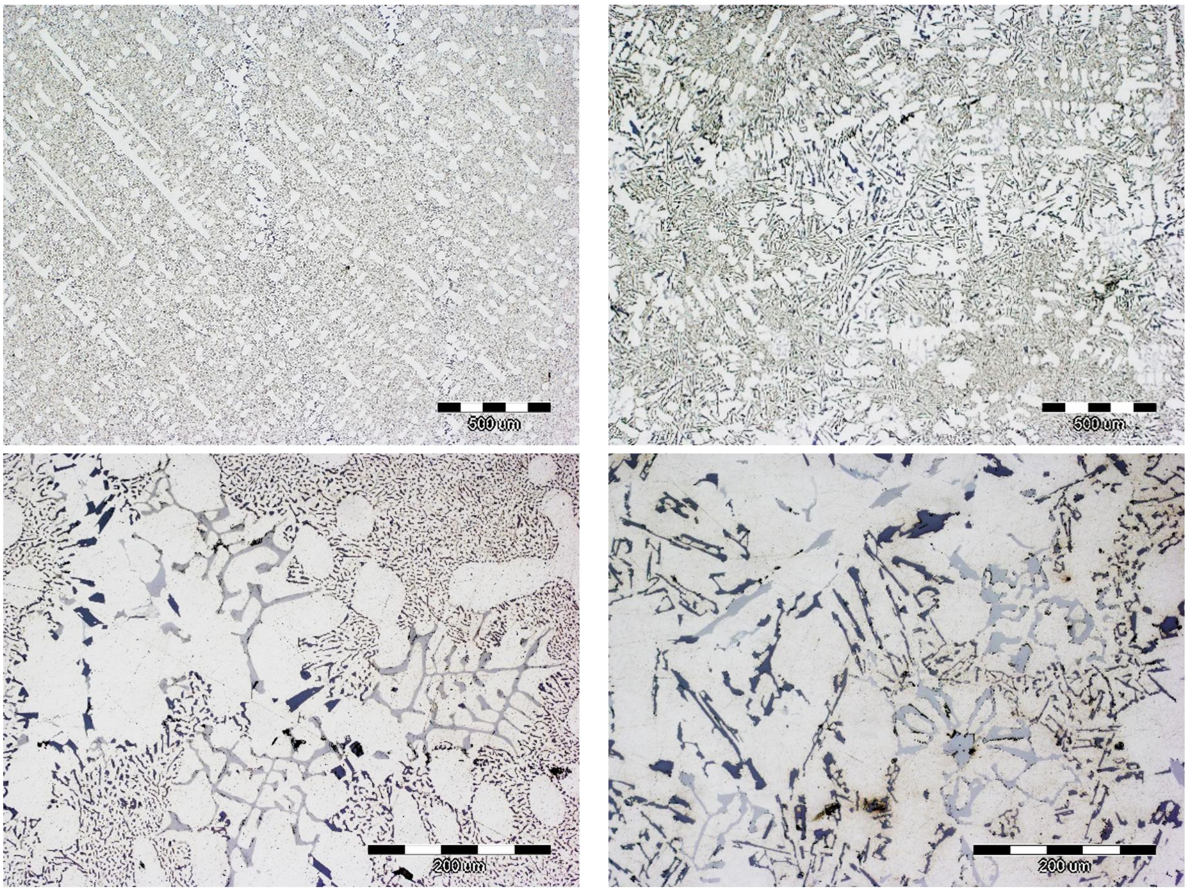
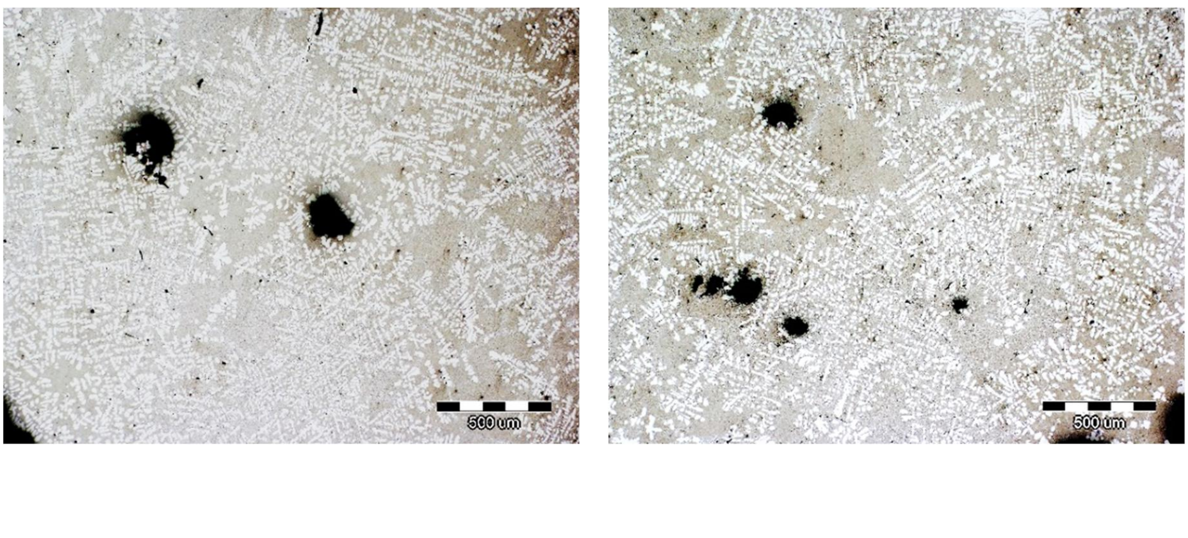


Figure 4. Microstructure of the base (M) and refined (MI) AlSi12 alloy from croning cell

In both cases, dendritic network development has been noticed. In refined sample, a higher ratio of α_{Al} is observed which indicate preferred tendency to primary dendrite development. Higher magnification revealed mixed morphology of eutectic (needle and fibrous) correlated to over-modification. High-temperature iron intermetallic surrounds primary α_{Al}. Their morphology appears as Al_5FeSi in needle-like form due to characteristic low cooling rate in croning cell and $Al_{15}(Fe, Mn)_3Si_2$ in Chinese script form due to high Mn content.

M (A) MI (B)



M (A) MI (B)

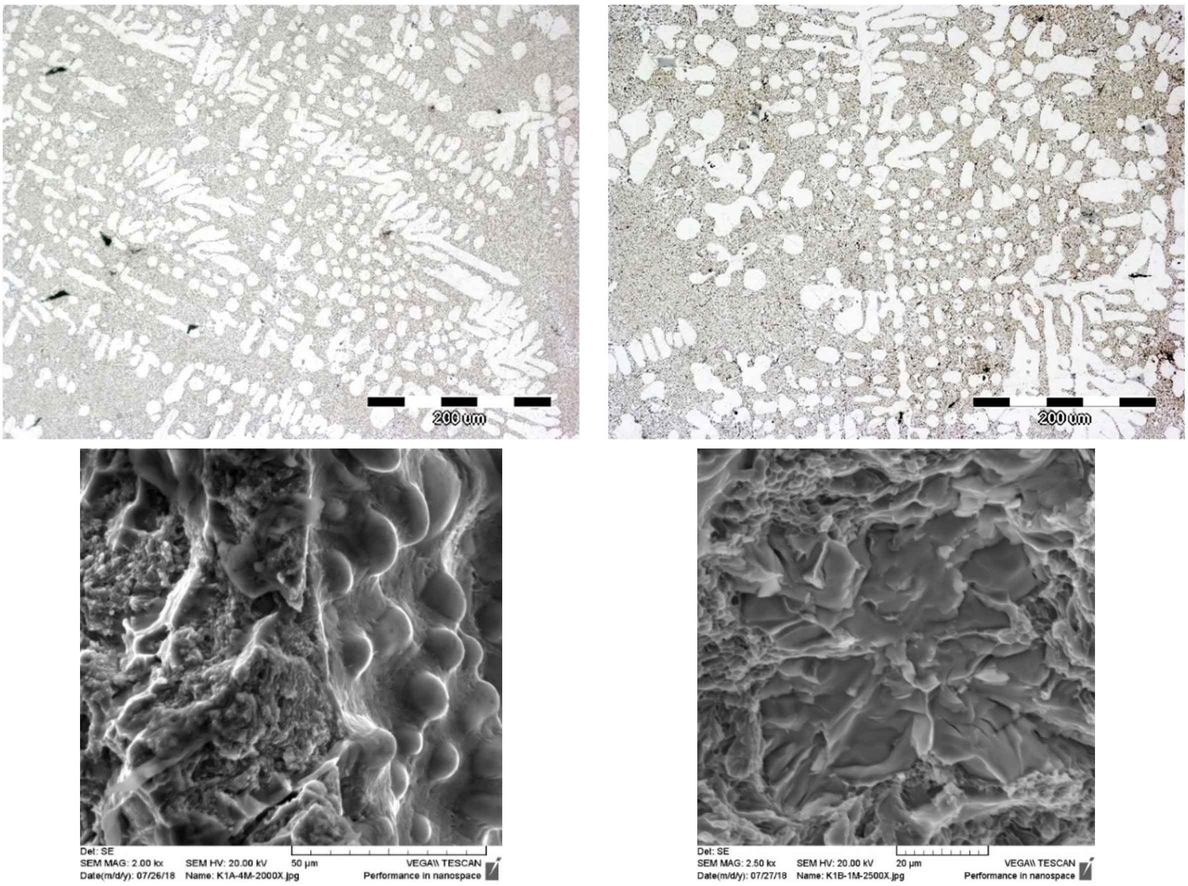


Figure 5. Microstructure of the base (M) and refined (MI) AlSi12 alloy from test part of ASTM B108 mould

Test part of ASTM B108 casting is characterised by edge position in the mould and small diameter in cross section, and therefore higher temperature gradient during solidification. Microstructure observed at position at/near the mechanical investigation fracture occurrence, revealed gas porosity, developed dendrite network and eutectic cells in both cases. Dendrite network is finer and dendrites are fragmented in refined sample (MI-B). Eutectic reveals in fine fibrous and completely modified morphology. Fractography of the base sample M indicate mixed ductile-brittle fracture nature across the primary α_{Al} dendrites and surrounding eutectic. Fractography of refined sample MI reveals completely ductile fracture across the eutectic and Al-Fe-Mn-Si intermetallic.

M (C) MI (D)

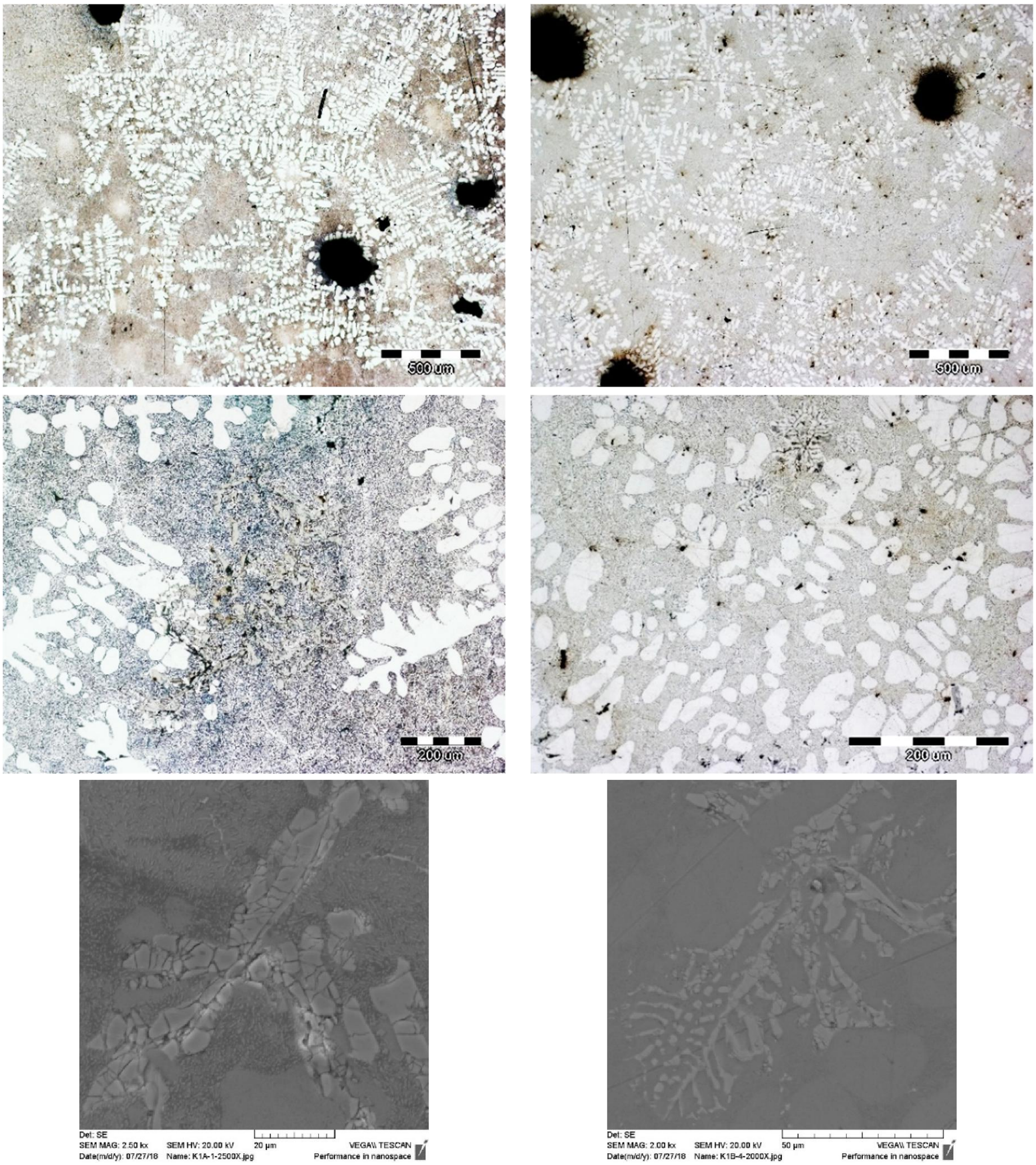


Figure 6. Microstructure of the base (M) and refined (MI) AlSi12 alloy from inflow part of ASTM B108 mould

The inflow part of ASTM B108 casting is closer to the central part of the casting and this area is characterized with the larger diameter in cross section, and therefore lower cooling and solidification rate as predicted by numerical simulation. The technological condition of solidification is confirmed by a rough microstructure that also reveals a highly developed dendritic network which is more uniformly distributed in the refined sample (sample MI). The eutectic is completely modified in fibrous morphology. Large gas porosity can be observed. Porosity represent a weak place suitable for failure. Higher magnification reveals the rougher but fragmented Al-Fe-Mn-Si intermetallic, while the similar one in refined sample is much finer.

Microstructure analysis enables SDAS (secondary dendrite arm spacing) comparison for all investigated samples presented in Fig. 7.

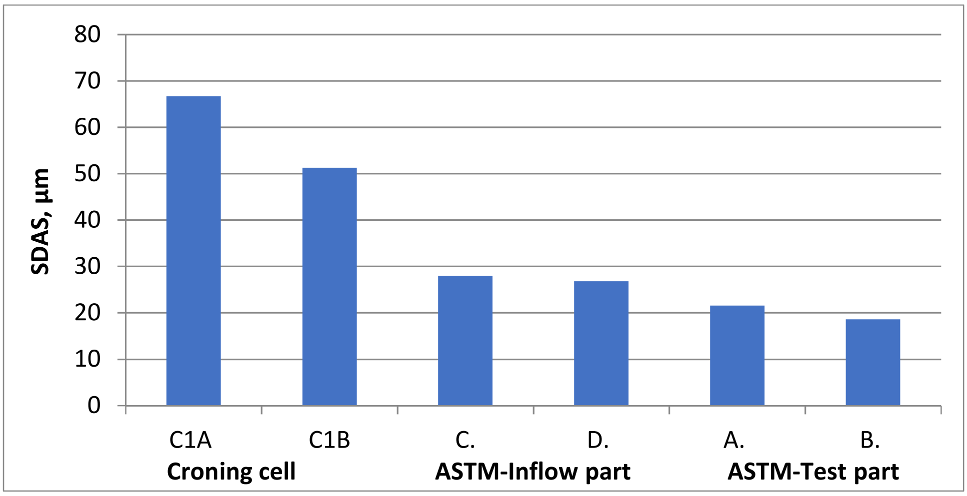


Figure 7. SDAS values

SDAS values confirmed observed changes in microstructure. SDAS is decreasing with the increase of cooling / solidification rate (croning cell → inflow part → test part). It also decreases with applied grain refinement within the same solidification conditions.

Mechanical properties investigation was performed on samples from test and inflow part of the ASTM B108 casting. Obtained tensile strength (R_m) and elongation (A_{50}) are presented in Table 2.

Table 2. Mechanical properties of AlSi12 alloy samples from ASTM B108 mould.

Sample	**Melt treatment**	**R_m, [N/mm^2]**	**A_{50}, [%]**
A. (test part)	M	216.25	2.91
B. (test part)	MI	178.76	1.19
C. (inflow part)	M	198.88	4.36
D. (inflow part)	MI	187.89	3.54

Obtained results correspond to those required by EN 1706 norm. Lower cooling rate (inflow part samples) decreases the tensile strength with simultaneously increasing of elongation due to wider solidification time-temperature interval. Observed porosity also, larger in size in inflow part, also comprehend to the decrease of tensile strength. In general, inoculation did affect the microstructural change toward the uniformly distributed fine dendrite morphology, although it did not have a positive effect on improvement of mechanical properties neither tensile strength nor elongation. On the contrary, higher cooling rate (test part) and applied grain refinement revealed the lowest values of followed mechanical properties.

Correlation of obtained microstructure improvement and tensile strength results, confirmed that eutectic AlSi12 (EN AC 44100) alloy is more suitable for thin wall casting produced with HPDC technology without additional grain refinement.

Conclusions

Targeted melt treatment can influence the solidification manner, development of microstructural features and application finally achieving mechanical properties of the alloy. Obtained results should be correlated to corresponded casting geometry or technology. Investigation of EN AC AlSi12 (EN AC 44100) eutectic alloy with high Fe and Mn content, included different mode of melt treatment: modification of eutectic with addition of AlSr10 and targeted addition of AlTi5B inoculant. Investigation revealed following conclusions:

- Results of numerical simulation indicate hot spots in lower part of the ASTM B108 casting. Technological parameters of casting in combination with narrow solidification interval of AlSi12 alloy comprehends to the porosity occurrence in hot spots which represent a weak place and failure position.
- Grain refinement lowers the liquidus undercooling and shortens the solidification time interval of AlSi12 alloy.
- Microstructural investigation reveals dendrite network development surrounded by eutectic cells. Higher content of Fe and Mn and theirs ratio 1:1 comprehend to the development of Al-Fe-Mn-Si intermetallic. Grain refinement influences on the decrease and/or fragmentation of the microconstituents, in particularly α_{Al}. Inoculation contributes to the uniform distribution of α_{Al} dendrites and lowering the SDAS. Higher cooling rate also comprehend to the grain refinement and SDAS decreasing within the same solidification conditions.
- Although the obtained mechanical properties correspond to those required by EN 1706 norm, grain refinement did not have absolutely positive effect on mechanical properties. Lower cooling rate (inflow part samples) decreases the tensile strength with simultaneously increasing of elongation. Higher cooling rate (test part samples) and grain refinement revealed the lowest values of mechanical properties.

Eutectic AlSi12 (EN AC 44100) alloy with high Fe and Mn content and Fe:Mn=1:1 ratio, according to microstructural and mechanical properties investigation is mostly suitable for thin wall casting (higher cooling rates) without additional grain refinement.

References:

[1] ASM Specialty Handbook: Aluminum and Aluminum Alloys. Ohio: ASM International, Materials Park 1993.

[2] EN 1706:2010 Aluminium and aluminium alloys – Castings – Chemical composition and mechanical properties.

[3] ASM Specialty Handbook: Aluminum and Aluminum Alloys. Ohio: ASM International, Materials Park 1993.

[4] D. Dispinar, J. Campbell, Metal quality studies in secondary remelting of aluminium, J. Inst. Cast Met. Eng. 2004;178:78–86.

[5] V. Raghavan, Al-Fe-Mn-Si (Aluminum-Iron-Manganese-Silicon), Journal of Phase Equilibria and Diffusion, 2007;28:215-17

[6] E. Tillová, M. Chalupová, L. Hurtalová, Evolution of phases in a recycled Al–Si Cast alloy during solution treatment. Phys Scanning Electron Microscopy 2012; 411–438.

[7] T. Tański, K. Labisz, B. Krupińska, M. Krupiński, M. Król, R. Maniara R, Borek W. Analysis of crystallization kinetics of cast aluminum–silicon alloy. J Therm Anal Calorim. 2016;123:63-74.

[8] N. Iqbal, N. H. van Dijk, S. E. Offerman, N. Geerlofs, M. P. Moret, L. Katgerman, G.

J. Kearley, In situ investigation of the crystallization kinetics and the mechanism of grain refinement in aluminum alloys. Mater Sci Eng A Struct. 2006;416(1–2):18–32
[9] T. Hosch, R.E. Napolitano, The effect of the flake to fiber transition in silicon morphology on the tensile properties of Al–Si eutectic alloys, Materials Science and Engineering A 528 (2010) 226–232
[10] M. Garat, G. Laslaz, S. Jacob, P. Meyer, P. H. Guerin, R. Adam, Transactions of the American Foundrymen's Society 100 (1992) 821–832.
[11] S. Z. Lu, A. Hellawell, Metallurgical Transactions A: Physical Metallurgy and Materials Science 18A (10) (1987) 1721.
[12] D. C. Jenkinson, L.M. Hogan, Journal of Crystal Growth 28 (2) (1975) 187.
[13] A. K. Dahle, K. Nogita, S. D. McDonald, C. Dinnis, L. Lu, Materials Science and Engineering A 413–414 (2005) 243–248
[14] T. Hosch, L.G. England, R.E. Napolitano, Journal of Materials Science 44 (18) (2009) 4892–4899
[15] M. G. Day, A. Hellawell, Proceedings Royal Society. Mathematical, Physical and Engineering Sciences 305 (1483), 1968, p. 473.
[16] S. Z. Lu, A. Hellawell, Journal of Crystal Growth 73 (2) (1985) 316.
[17] R. E. Napolitano, L. G. England, Solidification of Aluminum Alloys (2004) 445–451.
[18] A. L. Greer, P.S. Cooper, M.W. Meredith, W. Schneider, P. Schumacher, J. A. Spittle, A. Tronche, Adv. Eng. Mater. 5 (2003) 81–91.
[19] S. A. Kori, B. S. Murty, M. Chakraborty, Mater. Sci. Eng. A 283 (2000) 94–104.
[20] M. Easton, D. St. John, Metall. Mater. Trans. A 30A (1999) 1613–1623.
[21] M. Easton, D. St. John, Metall. Mater. Trans. A 30A (1999) 1625–1633
[22] J. E. Gruzleski, B.M. Closset, The Treatment of Liquid Aluminum – Silicon Alloys, AFS, Illinois, 1999.
[23] ASTM B108, Standard Specification for Aluminum-Alloy Permanent Mold Castings, https://www.astm.org/Standards/B108.htm (26.07.2018.)
[24] EN 10002-1, 2001, Metallic materials – Tensile testing – Part 1: Method of Test at Ambient Temperature