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EFFECT OF ZIRCONIUM ON THE SOLIDIFICATION PATH AND STRUCTURAL PROPERTIES OF COMMERCIAL AlSi10MgCu ALLOYS

Comprehensive understanding of the melt quality is of vital importance for foundry man. The effect of each particular element need to be properly analysed. Therefore, the aim of this paper was to analyse the impact of various content of zirconium on the solidification path and structural characteristics (SDAS, grain size, porosity) of as cast commercial AlSi10MgCu alloys. It has been found that addition of zirconium up to 0.24 wt.% reduce significantly the grains size (from 3.5 mm to 1.2 mm), SDAS (from 57.3 µm to 50.4 µm) and porosity (from 19% to 5%), leading to production of sound cast parts.

Keywords: zirconium, AlSi10MgCu alloy, grain size, SDAS, porosity

1. Introduction

Aluminum-silicon alloys due to their promising metallurgical, mechanical and thermos-physical properties are suitable to be used in several filed of engineering application [1,2]. Last several decades, their application in automotive industry is significantly intensified, being used mostly for production of various engine parts such as: cylinder heads, engine blocks, pistons, intake manifolds and others [3,4]. At the present time, the global automotive industry is going through fundamental changes shifting towards electrification. The shift is mainly triggered by the new environmental regulation. Economical and oil crises, air pollution and global warming are some additional factors that drastically change vehicles production strategy at the beginning of 21st century. In the last few years we are witnesses that most Original Equipment Manufacturers (OEMs) have introduced their Electro Vehicles (EVs) models or prototypes within their product portfolio, even though so far the market accounts for just a very small share of the global vehicle sales, with increasing expectations for the coming years [5]. Based on the OEMs requests for production of E-mobility parts (e.g., battery trays, E housing and others) logically appears the question what kind of alloys can be applied for such production. Commercial aluminum-silicon alloys currently used for production engine parts are firstly consider for such production due to their good fluidity and castability, moderate to high strength (improved by presence of copper and magnesium) as well as excellent corrosion resistivity and dimensional stability (especially by those aluminum alloys with lower copper and magnesium contents). Among all commercially used cast hypoeutectic aluminum-silicon alloys, the AlSi10MgCu alloys have been greatly exploited for production of engine parts. In order to improve properties of theses alloys, especially in as cast conditions, it has been considered their additional alloying with zirconium. Traditionally, zirconium was added to aluminum wrought alloys with aim to alter microstructure (reduce the grain size and inhibit recrystallization) and improve mechanical properties (restrict the dislocation motion) of treated alloys. In the available literatures, there are limited data regarding impact of zirconium on the solidification path, structural, mechanical and thermos-physical properties of as cast and heat treated aluminum-silicon hypoeutectic alloys. According to F. Wang at al. [6] addition of zirconium up to 0.2 wt.% change significantly primary morphology of α-aluminum from columnar to equiaxed grains (from 1150 µm to 500 µm). Any further addition of zirconium up to 0.5 wt.% leads to the gradual decrease of grain size (from 500 µm to 350 µm). According to Elicondo [7], Seperband [8] and Mahmudi [9] minor addition of zirconium to AlSi6Cu4 (319) alloys has beneficial impact on the grain refinement of investigated aluminum alloys. Recently, Voncina at al [10], indicated in their work that addition of 0.202 wt.% zirconium to AlSi9Cu3(Fe) alloy did not considerably refine the

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grain size of this alloy. Thus, it looks that still is not clear what is the optimal added concentration of zirconium to aluminum-silicon alloys necessary to improve their properties. Therefore, the present work was aimed to investigate the influence of small addition of zirconium to commercial hypoeutectic AlSi10MgCu alloy in order to define its solidification paths as well as to get its optimal structural properties.

2. Experimental part

The material use in this work was hypoeutectic AlSi10 MgCu commercial alloy without and with zirconium addition. The chemical composition of alloy is listed in Table 1 (for all elements in wt. %). A series of AlSi10MgCu alloys with increased Zr addition levels (0.0, 0.08, 0.14 and 0.24 wt. %) were cast by adding master Al-10 wt. % Zr alloy into melt. The commercial alloy was melted in 800 kg capacity electric resistance furnace. The melt was degassed for 15 minutes using nitrogen gas. After degassing melt temperature was maintained at 760°C. In order to determine the solidification paths of investigated alloys, the cooling curve analyses have been run. Therefore, the liquid test samples with masses of approximately 250 ± 10 g were poured into preheated steel test cups (high 60 mm, diameter 50 mm) coated with Cillolin. Two calibrated N type thermocouples were inserted into the melt at the constant height, 30 millimeters from the bottom of the crucible and temperatures between 700-400°C were recorded. The National Instrument Data Acquisition System linked to a personal computer was used to collect the data (temperature versus time) during experiments. In total eight experiments have been run for four investigated alloys, (each cooling curve analysis has been performed twice). Standard metallographic techniques (sample preparation, Light Optical Microscope and Scanning Electron Microscope) have been applied in order to analyze the macro and microstructure of collected thermal analysis test samples.

TABLE 1

Chemical composition of investigated AlSi10MgCu alloy (wt.%)

Alloy	Si	Fe	Cu	Mg	Mn	Zn	Ti	Sr	Ni
AlSi10 MgCu+Zr	9.19	0.38	0.27	0.39	0.31	0.13	0.13	0.015	0.008

3. Results and discussions

The microstructure of as cast AlSi10MgCu alloy characterize the presence of moderate amount of primary a-aluminum dendrites (~30%), followed by solidification of primary aluminum-silicon eutectic (~65%) as well as precipitation of small amount of magnesium and copper rich phases.

The Figure 1, shows the light optical micrographs of the as cast AlSi10MgCu (Zr) alloys containing an α-Al dendrits with Al-Si primary eutectic. Small precipitated phases such as magnesium, copper and zirconium rich intermetallics have been not recognized from the Figure 1.

Scanning electron microscope analysis revealed the zirconium rich phase with needle and blocky shape morphology. From the literature [11] is well known that added zirconium into aluminum melt may exist in four different forms; solid solution in matrix, coarse primary Al_3Zr phase, metastable Al_3Zr phase as well as equilibrium Al_3Zr phase. According to EDX analysis, the zirconium added into AlSi10MgCu alloy precipitated in the form of AlZrSiTi rich phase, what is in agreement with results obtained by Voncina and coworkers [10].

The results of cooling curve analysis are summarized in Figure 3. The values of characteristic solidification temperatures (Liquidus and Dendrite Coherency) have been plotted as a function of zirconium content. Increase in the zirconium content up to 0.14 wt. % increase the liquidus temperature from 588.7°C up to 593.6°C as well as the dendrite coherency temperature from 585.5°C up to 587.5°C, allowing earlier precipitation of primary

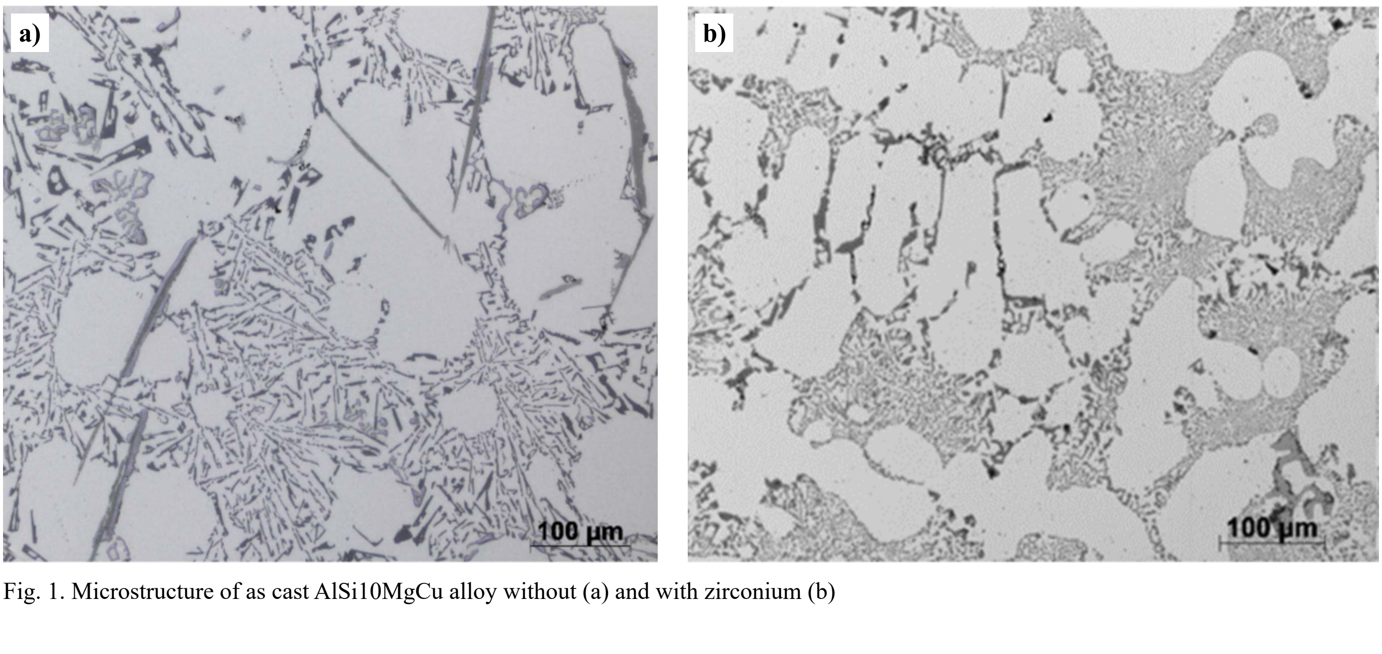

Fig. 1. Microstructure of as cast AlSi10MgCu alloy without (a) and with zirconium (b)

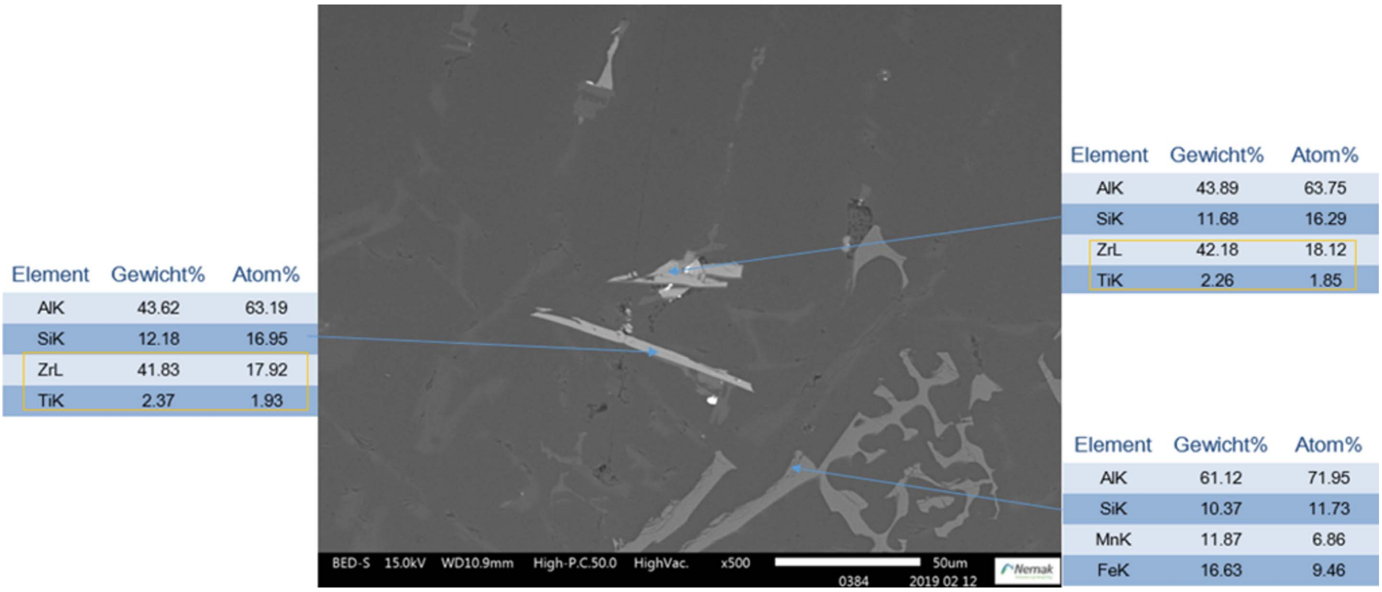

Fig. 2. Scanning electron micrograph with related EDX analysis

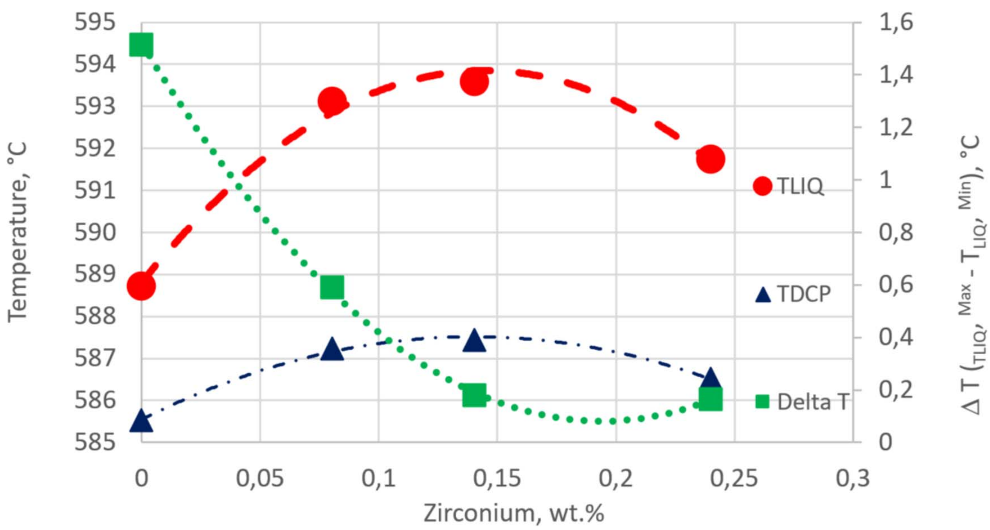

Fig. 3. Impact of Zirconium addition into AlSi10MgCu alloys on the Liquidus and Dendrite coherency temperatures as well as on the undercooling (ΔT) during solidification of primary α-Al dendritic structure

a-aluminum dendrites. Addition of 0.24 wt. % of zirconium leads to decrease of both liquidus and dendrite coherency temperatures down to 591.2°C and 586.5°C respectively. Addition of zirconium into AlSi10MgCu alloy significantly decrease the primary undercooling (already by 0.14 wt. % zirconium ΔT = 0.2°C), indicating strong grain refining potential of zirconium addition.

As cast AlSi10MgCu alloy during primary solidification developed the primary and secondary dendritic structure (SDAS). The SDAS represents the distance between secondary dendrites in the solidifying structure of cast metals and alloys. The cooling rate and alloy compositions are two major factor which determine the size of SDAS. As Figure 4 illustrates, addition of zirconium has impact on the size of SDAS, reducing them significantly from 57.25 µm to 50.37 µm. This effect can be attributed to grain refinement effect of zirconium as well as its small solubility in α-Al matrix.

Hypoeutectic AlSi10MgCu alloy has a small proportion of primary aluminum grains in their microstructure, which needs to be also refine. Generally finer grains are, for most applications, preferable to coarse grains. Grain refinement during solidification can be accomplished by rapid cooling or by addition of certain elements (Ti, B, Zr and so on) to the melt to promote promoting nucleation (chemical grain refinement). Chemical grain refinement is the most widely practiced, and most guaranteed method of grain refinement. According to Figures 3 and 5 it is obvious that addition of zirconium into AlSi10MgCu melt leads to the refinement of as cast structure of this alloy. As can be seen in Figure 5, addition of 0.24 wt.% zirconium decrease the grain size of investigated alloy from 3,5 mm down to 1.17 mm. It can be recognized that first addition of 0.08 wt.% zirconium decrease the grain size by approximately 0.7 mm. Further addition of 0.06 wt. % zirconium (in total 0.14 wt. % zirconium) decrease

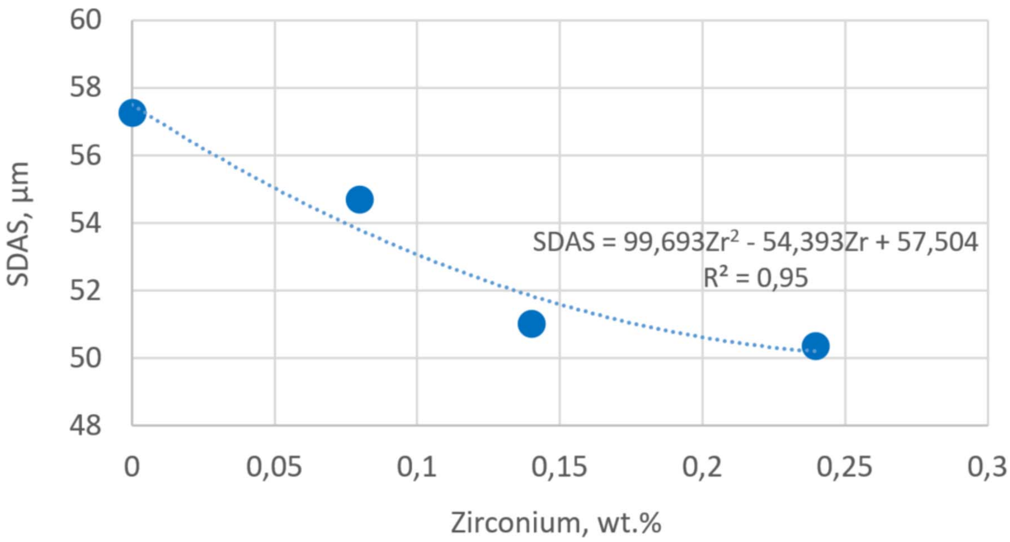

Fig. 4. The effect of the various zirconium content on the size of the SDAS in as cast AlSi10MgCu alloy

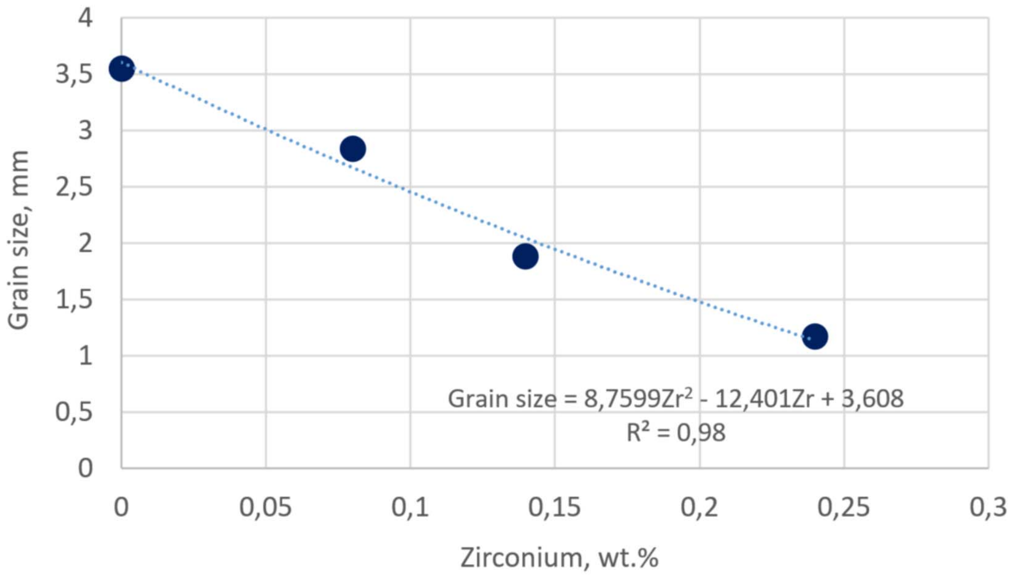

Fig. 5. Impact of Zirconium on the grain size of as cast AlSi10MgCu alloy. The samples for grain size analysis has been taken from the bottom part of thermal analysis test samples (average cooling rate ~0.15°C/s)

the grain size considerably by around 1.6 mm. A finer grains of as cast structure will leads to better feeding and filling ability of this alloy as well as improving its mechanical properties.

For AlSi10MgCu cast alloys, the transformation from liquid to solid state is accompanied by a decrease in volume in the ranges about 5%. In order to compensate the volume deficit, the cast parts during solidification need to be fed with extra volume of liquid melt. Campbell [12] summarized the five characteristic feeding mechanisms that can occur during solidification of aluminum cast alloys. They are liquid feeding, mass feeding, interdendritic feeding, burst feeding and solid feeding. The characteristic solidification temperatures such as Liquidus, Dendrite Coherency, Rigidity and Solidus mark the transition from one to other feeding regions. As Figure 6 indicates, addition of zirconium into commercial AlSi10MgCu alloy has no significant impact on the size of feeding regions. Only some increase in the mass feeding region (area between Liquidus and Dendrite Coherency temperature) can be recognized after 0.08 and 0.14 wt. % zirconium addition, which should lead to better feeding of this alloy.

It is well known that the quality of as cast alloys can be improved by grain refinement, which reduce the size of primary α-Al grains causing at the same time reduction in the amount of porosity. The present results indicate (Fig. 7) that addition of zirconium exceeding a lower limit of about 0.08 wt. % are enough to trigger the reduction of porosity in AlSi10MgCu alloy. The reduction in the amount of porosity seems to be good connected with the reduction of the SDAS and grain size (Figs 4 and 5). Both microstructural parameters are strongly dependent on the presence of zirconium into AlSi10MgCu melt.

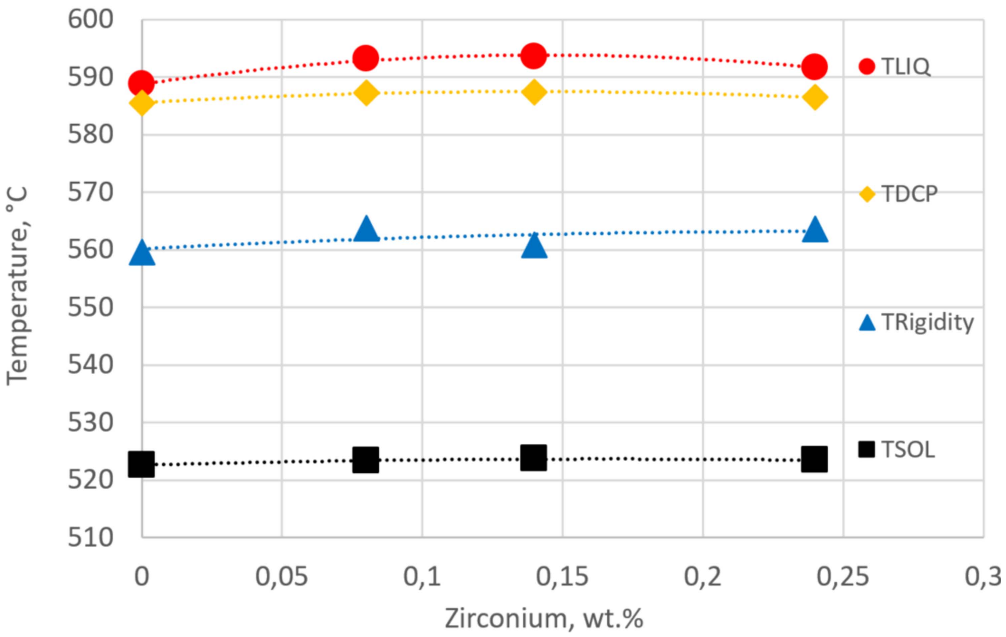

Fig. 6. Impact of Zirconium on the three feeding regions: Mass feeding ($T_{LIQ} – T_{DCP}$), Interdendritic feeding ($T_{DCP} – T_{Rigidity}$) and Burts feeding ($T_{Rigidity} – T_{SOL}$) of commercial AlSi10MgCu alloy

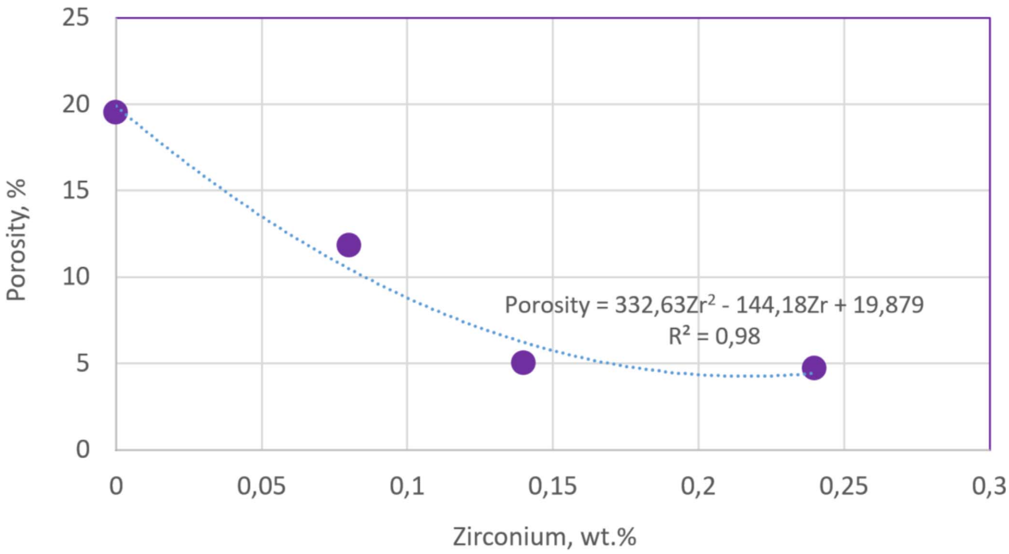

Fig. 7. Impact of Zirconium on the porosity of as cast AlSi10MgCu alloy. The samples for porosity analysis have been taken from vertical cross sections of thermal analysis test samples (average cooling rate ~0.15°C/s)

4. Conclusions

Experiments have been carried out to observe the effect of zirconium additions between 0 and 0.24 wt. % on the structural properties of commercial hypoeutectic AlSi10MgCu as cast alloy. It was found that addition of zirconium up to 0.24 wt. % has significantly change the as cast structure of investigated alloy even though that zirconium precipitated in the form of AlZrSiTi intermetallics. Microstructural parameters such as SDAS, grain size and porosity and strongly influenced by addition of zirconium into AlSi10MgCu alloy. Zirconium added in the AlSi10MgCu melt up to 0.24 wt.% reduce the SDAS for 12%. The main reason for this reduction can be attributed to different solubility of zirconium in liquid and solid phases. Increasing amount of zirconium in the melt will cause the precipitation of finer secondary dendrites. At the same time, zirconium promote grain refinement of aluminium alloys, reducing significantly the grain size of AlSi10MgCu alloy for approximately 67%. Smaller SDAS and grain size are leading to lower porosity of cast parts, improving generally its thermos-physical and mechanical properties. Added zirconium into AlSi10MgCu melt slightly increase the liquids and dendrite coherency temperatures, while the effect of zirconium on other characteristic solidification temperature is insignificant. Small increase in the liquidus and dendrite coherency temperature wide the mass feeding range, may origin better feeding and lees porosity formation in as cast structure.

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REFERENCES

[1] L.H. Cupido, P.L. Żak, N. Mahomed, J. Lelito, G. Piwowarski, P.K. Krajewski, Experimental investigation of modified heat treatment of AK64-type Al-Si-Cu sand cast alloy, Archives of Metallurgy and Materials **60**, 3, 2397-2402 (2015).

[2] B. Dybowski, J. Szymsza, Ł. Poloczek, A. Kielbus, Influence of the chemical composition on electrical conductivity and mechanical properties of the hypoeutectic Al-Si-Mg alloys, Archives of Metallurgy and Materials **61**, 1, 2016, 353-360 (2016).

[3] Peter Hajduch, D. Bolibruchova , Mile B. Djurdjevic, Influence of Molybdenum on the structural properties and micro hardness of AlSi10Mg(Cu) alloy, Archive of Foudnry Engineering **18**, 3, 19-24. (2018).

[4] Par Li Liu, Metallurgical parameters controlling evolution of microstructure in foundry alloys Al-Si-Mg and Al-Si-Cu, PhD Theses, April 2003, University of Quebec, Chicoutimi, Canada.

[5] A. Grauers, S. Sarasini, M. Karlström, Systems Perspectives on Electro mobility, (2013).

[6] F. Wang, D. Qiu, Z.L. Liu, J.A. Taylor, M.A. Easton, M.X. Zhang, Acta Materialia. **30**, 1-10 (2013).

[7] G.H.G. Elizondo, Effect of Ni, Mn, Zr and Sc additoins on the performance of AlSiCuMg. PhD thesis, Université Du Québec À Chicoutimi, April (2016).

[8] P. Seperband, R. Mahmudi, F. Khomamizadeh, Scripta Materialia **52**, 253-257 (2005).

[9] R. Mahmudi, P. Sepehrband, H.M. Ghasemi, Materials Letters **60**, 2606-2610 (2006).

[10] M. Voncina, S. Kores., M. Ernecl, J. Medved, J. Min. Metall. **53** (3)B, 423-428 (2017).

[11] J.-C. Zhang, D.-Y. Ding, W.-L. Zhang, S.-H. Kang, X.-L. Xu, Y.-J. Gao, G.-Z. Chen, W.-G. Chen, X.-H. You, Trans. Nonferrous Met. Soc. **24**, 3872-3878 (2014).

[12] J. Campbel, AFS Cast Metal Research Journal. **5**, 1-8 (1969).