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Grain Refinement of AlSi7Mg0,3 Alloy Cast by Rheocasting SEED

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Abstract

The paper concerns experimental work studying chemical composition, structures and selected mechanical properties of castings produced by rheocasting method SEED. After previous experiments, which showed inclusions in the primary phase α(Al) when observing structures, hypothesis of external nuclei was taken. The main goal of the work was to determine the influence of inoculation by various additions of titanium/boron based inoculant on the structure and properties of AlSi7Mg0,3 alloy. The master alloy AlTi5B1 was added in amounts of 0,05, 0,1, 0,15, 0,2 wt %. Metallographic observation by light and SEM microscopy was used for analysing the structures. Measurements of grain size were realised and evaluated. Brinell hardness measurements were performed. Chemical composition was measured by GDS analysis. Undertaken experiments did not prove the effect of inoculation of combined AlTi5B1 master alloy on castings made of AlSi7Mg0,3 alloy made by rheocasting SEED at given amounts and conditions.

Keywords: Theory of crystallization, Innovative foundry technologies and materials, Solidification process, Metallography, Rheocasting

1. Introduction

The thixotropic properties were discovered more than 30 years ago. The possible advantages of applying these properties to process material in a semi-solid state were soon recognized and two different routes were proposed: thixocasting and rheocasting.

There is presently a renewed interest in the semi-solid processing associated with the rheocasting route. However, the difficulty in obtaining a high-quality semi-solid material, together with the lack of a procedure for in situ measuring the rheological properties of the semi-solid slurry, has created some hurdles for the widespread use of the semi-solid casting technologies [1].

The SEED process (Swirled Equilibrium Enthalpy Device) is one of those rheocasting processes in industrial production of semi-solid castings. The SEED process is based on achieving thermal equilibrium between the metallic crucible and the bulk of metal by swirling. Morphology and size of the solid phase and the subsequent rheological properties of the semi-solid slurry are dependent upon the selected process parameters, including the pouring temperature and time of swirling in relation to the metal volume. The special rheological properties of the semi-solid alloys are linked to a globular morphology of the solid phase, fundamental to achieving good quality final products. The key features of SEED method are quality improvements, such as production of high integrity shape complex parts with good inner quality suitable for structural applications, possibility of heat treatment of castings (blister free), parts are weldable, near-netshape, thin and even thick wall pressure tight parts with geometrical flexibility, enhanced mechanical properties. There are also technological aspects, such as possible productivity improvement due to faster cycle rate, reduced total heat load on tooling, resulting in longer die life as well as lower filling velocities, returns can be fully recycled in the foundry. The SEED method principle is described in Figure 1. Melted alloy is poured to special crucible, where is eccentrically swirled for accurately determined time to reach the semisolid state. Than is the slurry moved and demoulded to the cold chamber and than starts the piston movement as well as for standard HPDC process.

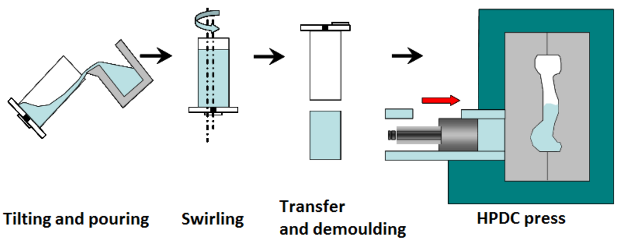

Fig. 1. SEED rheocasting method principle – phases [2]

For semi-solid methods, alloys with wide two-phase interval, both hypo- and hyper eutectic are suitable [3]. Practically, hypoeutectic alloy AlSi7Mg0.3 is one of the most applied alloys. Compare to alloys Al-Si commonly used for HPDC, this alloy has less silicon and, what is important, less iron content, which contribute to enhanced mechanical properties.

There are several theories of spheroidal microstructure formation during rheocasting process. The hypothesis of rheocasting method using stirring for preparation of semisolid slurry is that dendrites of primary phase α(Al) crumble into small particles, which then join and create round globular grains of primary aluminum [4]. Other work specifies the mechanism of crumbling; it supposes that due to high shear forces dendritic arms are teared off from the central part of dendrite, or they are melted out from it [5].

With certainty, it could be said that the growth of primary grains is influenced by flowing of the melt. With increasing speed of stirring, the shear rate is increasing and the mechanism of grain growth is changing from dendritic to globular [5]. The speed of flowing is increasing and the grains separate from the crucible wall faster. Then the grains are drifting into the melt and nucleation of new grains takes place on the walls of crucible. But at certain stirring rate, the rate of grain breakage from the walls does not increase. The size of primary grains is decreasing with increasing stirring rate, but also up to certain speed of stirring/flowing, after the grain size does not decrease. Existing grains continue to grow already at stirring rate 1000 rpm, at higher stirring rate grains separate from walls and new grains nucleates preferably [4]. The grain size of solid phase and the viscosity of mushy metal are influenced by sheer stress and by cooling rate [6].

1.1. Hypothesis

There were made several experiments before with the same alloy [7, 8, 9] and during studying of structures, inclusions were found (see Fig. 2 – 9). Interesting are hard inclusions in primary phase α(Al) which can be seen in Fig. 4 –5. These inclusions were observed in many samples. Detailed analysing by electron microscopy and elemental mapping (Spectrum 2 in Fig. 6) showed high content of silicon and oxygen. There was an assumption about particles SiO_2 to serve as inoculation germs for nucleation of primary phase α(Al). This could bring the explanation of quite coarse structure and big grain size of primary phase α(Al): if there are several inclusions of SiO_2 in the melt with melting temperature 1100 °C, they could cause preferential growth of the primary phase α(Al) leading to bigger grain size and smaller amount of grains.

But generally, oxides have very weak wettability. There are some works discussing and experimenting oxides as possible inoculants [such as 10], but those oxides are endogenous oxides of the base metal created by oxidation. There is possibility of reaction between Al and SiO_2, but this is usually realised at higher temperatures. Also the origin of SiO_2 is not satisfactorily explained.

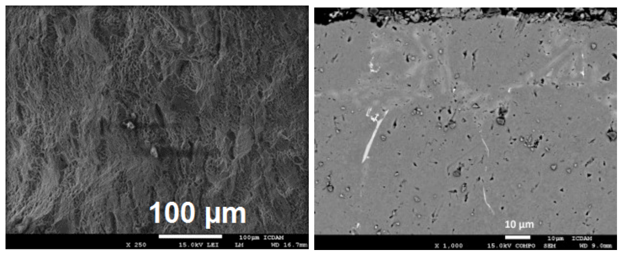

Fig. 2, 3. Inclusions in structure observed by electron microscopy

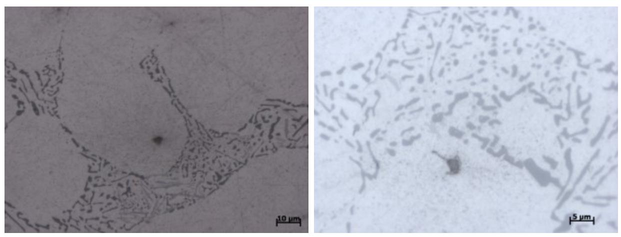

Fig. 4, 5. Inclusions in primary α(Al) phase observed by light microscopy

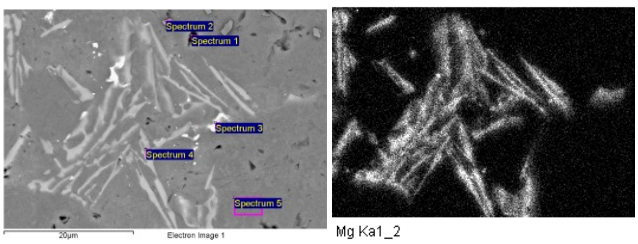

Fig. 6, 7. Studied structure (left) and mapping for magnesium (right)

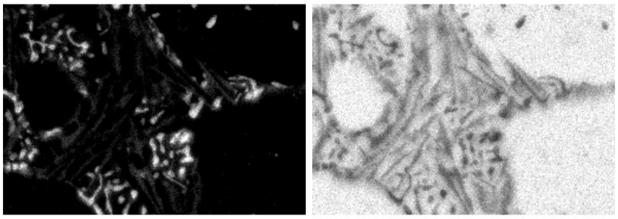

Fig. 8, 9. Mapping for silicon (left) and aluminium (right)

Anyway, this experience lead to the hypothesis, that the targeted inoculation can help with decreasing of grain size of primary phase α(Al). There was a presumption that the liquid metal can nucleate not only exogenously on the crucible wall, but also endogenously by nucleation on the external inoculation germs homogenously dissipated in the melt due to the local overcooling. The swirling would keep the distribution of nucleating grains homogenous within volume. Calculated amount of inoculant addition would ensure sufficient number of germs, which, in cooperation with swirling and the cooling effect of the crucible, could cause the growth of fine grains with equilibrium enthalpy.

Good effect of inoculation of primary phase α(Al) have several elements: titanium, boron and zirconium. Mainly used are titanium in combination with boron. These elements create intermetallic phases type Al – Ti – B such as $TiAl_3$, AlB_2 and TiB_2, which have the best properties for creating crystallisation nuclei. Some other elements giving inoculation effect are known or experimented such as carbon and niobium [11].

2. Experiment

In this study, aluminum alloy EN-AC AlSi7Mg0.3 was used to produce semi-solid castings with the SEED process. Chemical composition of the alloy is in Table 1.

Table 1.
Chemical composition of experimental alloy EN-AC AlSi7Mg0,3 according EN 1706:2010 (weight %)

Si	Fe	Cu	Mn	Mg	Zn	Ti	Others: each	total
6,5 – 7,5	0,19	0,05	0,10	0,25 – 0,45	0,07	0,25	0,03	0,10

Samples were inoculated by commercial master alloy AlTi5B1 in the form of wire (Ø 10 mm). The addition was calculated for amount of 0,05 %, 0,1 %, 0,15 %, 0,2 % (wt.) of AlTi5B1. The master alloy was added to the pouring ladle to dissolute and then poured in the holding furnace with temperature 645 °C. Reference samples without inoculation were used.

Rheocasting process SEED was applied with standard parameters. Castings were cut and metallographic samples were prepared (Fig. 10, 11).

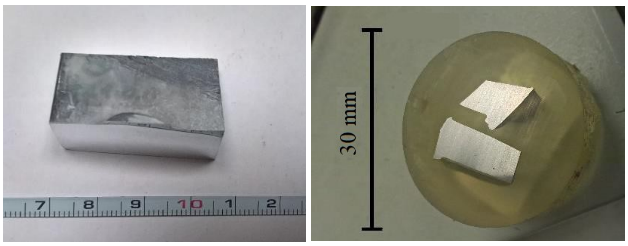

Fig. 10, 11. Preparation of samples for light microscopy

According the standard EN ISO 6506-1 sintered carbide (WC) ball with diameter 2,5 mm was applied as indentor for measuring of Brinell hardness with load of 62,5 kg. The hardness was measured 10x for every sample, in Fig. 29 there are arithmetic means. Results are listed in Table 2.

For verification of chemical composition, GDS analysis using Horiba JobinYvon GD Profiler II. was used. Results are listed in Table 3.

For observing structures and for measurements, light microscope Olympus PME3 was used. Measurement of grain size and statistical evaluation was carried out. For each sample (not inoculated, inoculated by 0,05, 0,1, 0,15, 0,2 % AlTi5B1) the structure was observed and 100 of primary grains were measured by perpendicular diameters d1 and d2 (small grains created by secondary crystallization were not included), see Fig. 22. These data were analysed and the average value of grain size for the sample was calculated (Fig. 23 – 27, 28). Also, standard deviation was calculated (Tab. 2).

Table 2.
Grain size evaluation and hardness HBW

Sample [% AlTi5B1]	Average d [μm]	Hardness HBW
0,0	94	71
0,05	100	71
0,1	106	69
0,15	96	73
0,2	90	70
Average d	97	71
σ	6	1,5

3. Results and discussion

3.1. Observation and analysing of structures

Structures of not inoculated (Fig. 12, 13) and inoculated (Fig. 14 – 21) samples contain globular to dendritic formations of primary phase α(Al) (white) within which foreign particles occur (black). These inclusions are detected in all samples, both not inoculated and inoculated, and they are similar to previously observed inclusions (see Fig. 4, 5 and compare to Fig. 12 – 21). Primary phase is surrounded by eutectic β (grey) which consists of fine particles of silicon growing up from aluminum matrix. Also, small grains of phase α(Al) created by secondary crystallization are found. Secondary crystallization takes place already in the mold from the residual liquid phase.

Table 3.
Chemical composition after inoculation (weight %)

% AlTi1B5	Si	Fe	Cu	Mn	Mg	Zn	Ti	B (ppm)	others
0,0	6,1	0,2	0,002	0,04	0,4	1,7	0,12	0,4	0,00
0,05	6,6	0,2	0,01	0,04	0,4	0,7	0,11	0,6	0,04
0,10	5,3	0,2	0,01	0,04	0,3	2,1	0,13	0,6	0,22
0,15	4,3	0,2	0,01	0,04	0,3	2,3	0,14	0,8	0,01
0,2	5,3	0,2	0,01	0,03	0,3	0,6	0,13	0,7	0,03

There are no significant differences between analysed structures. This observation is confirmed by statistical measurement of primary grains size – see Fig. 23 – 27 and Table 2. Comparison of grain sizes resulting from Fig. 23 - 27 is in Fig. 28; the growing trend from not inoculated sample up to 0,1 % of AlTi5B1 master alloy addition can be seen, then the grain size decreases again and for the inoculation by 0,2 % AlTi5B1 the value is the lowest, lower than for not inoculated sample. This may seem like the effect of innoculation is starting at 0,2 % AlTi5B1. But the experiment including higher amount of AlTi5B1 was not performed, and the differences between achieved grain sizes are not statistically signifficant to responsibly confirm the effect of inoculation.

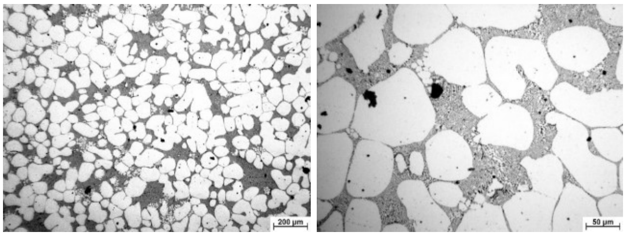

Fig. 12, 13. As cast structure without inoculation

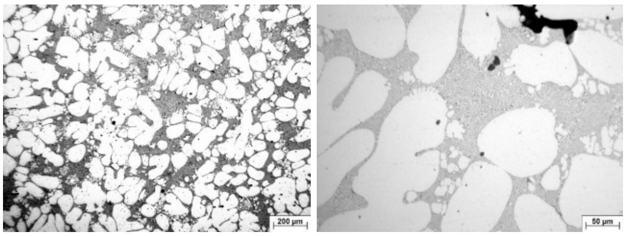

Fig. 14, 15. Structure inoculated by 0,05 % AlTi5B1

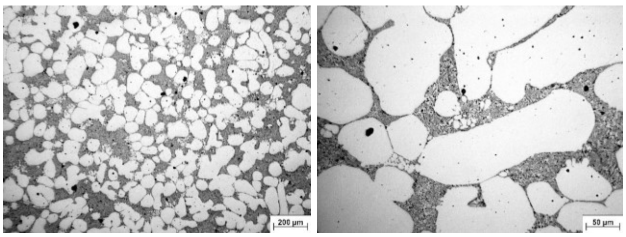

Fig. 16, 17. Structure inoculated by 0,1 % AlTi5B1

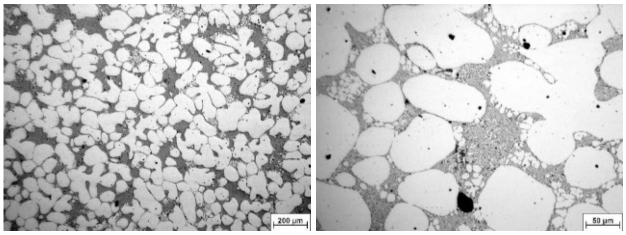

Fig. 18, 19. Structure inoculated by 0,15 % AlTi5B1

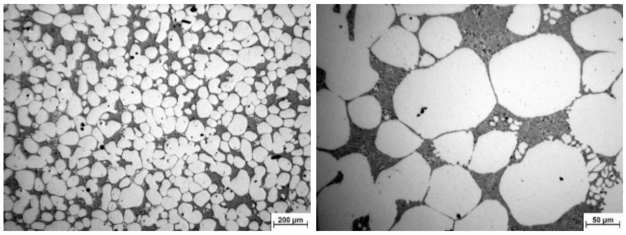

Fig. 20, 21. Structure inoculated by 0,2 % AlTi5B1

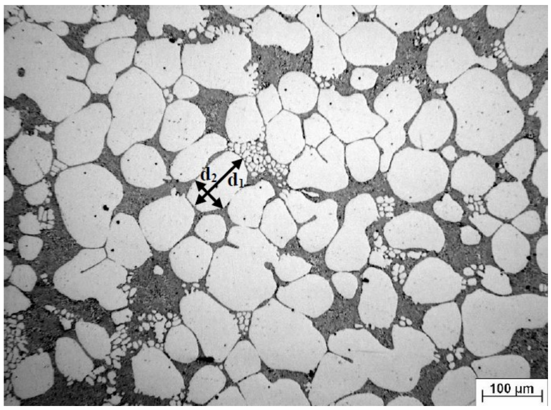

Fig. 22. Measuring of grain size

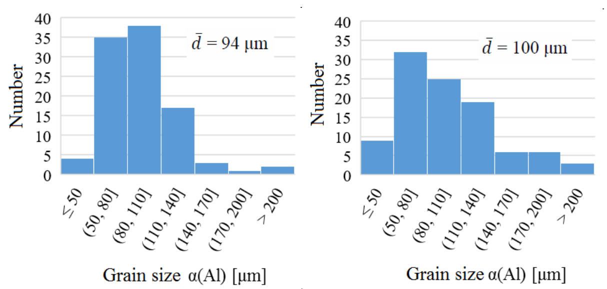

Fig. 23, 24. Average grain size – no inoculation (left), 0,05 % AlTi5B1 (right)

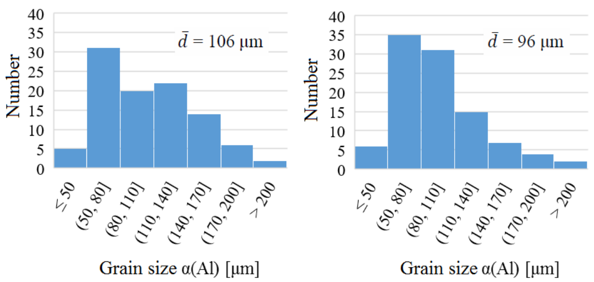

Fig. 25, 26. Average grain size – 0,1 % AlTi5B1 (right), 0,15 % AlTi5B1 (right)

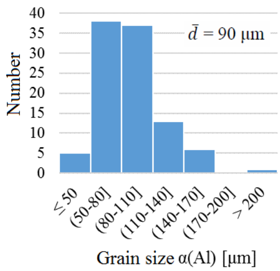

Fig. 27. Average grain size - 0,2 % AlTi5B1

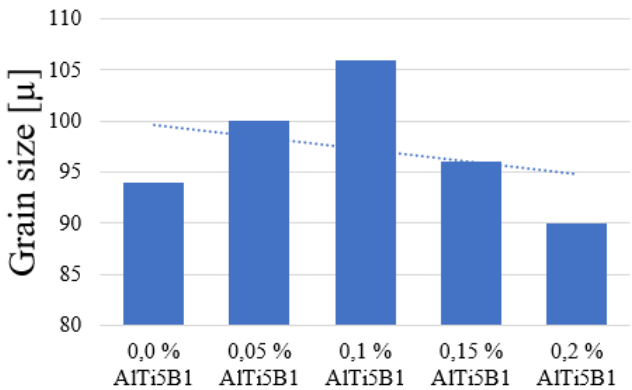

Fig. 28. Average grain size

3.2. Hardness and chemical composition measurements

Measurements of Brinell hardness also did not show any satisfying results. It varies around average value 71 HBW with very small standard deviation (1,5 – see Table 2). This leads to statement, that inoculation does not have significant effect on hardness. On the other hand, the lowest value of the hardness was reached for 0,1 % AlTi5B1 and it corresponds with the worst achieved result for the grain size – both results are out of the standard deviation range. Unfortunately, even the chemical composition (Table 3) does not bring satisfying explanation, because contents of elements particularly influencing the hardness are similar.

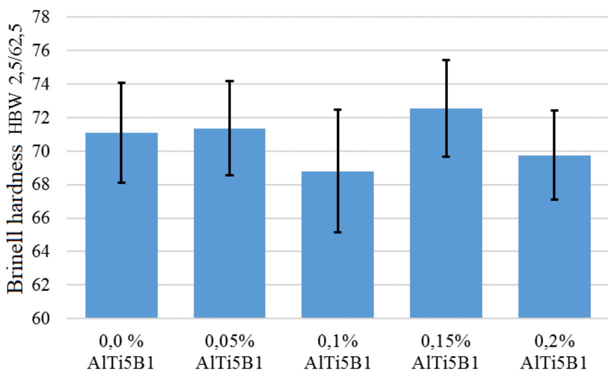

Fig. 29. Brinell hardness HBW

The amount of other measured elements can be summarized as follows. Except value of sample inoculated by 0,05 % AlTi5B1, all samples have low content of silicon. All samples have the same content of iron, which is higher than the standard. Copper fits the standard and is lower for all cases, manganese as well. Magnesium content corresponds to the standard. Zinc content is significantly higher for an unknown reason. Measured content of titanium fits to the standard.

The measured amount of titanium does not show up anything about the inoculation. The measured amount of boron show much lower content than it should be present after inoculation. After analysing of these results, there is suspicion that the inoculation master alloy was not dissolved completely and not the whole content of elements in added master alloy was used for nuclei creation.

4. Conclusions

An experiment was performed using master alloy AlTi5B1 in different amount to achieve the grain refinement due to addition of external nuclei of phases $TiAl_3$, TiB_2 or TiB_2. The added amount was 0,05 %, 0,1 %, 0,15 %, and 0,2 % of AlTi5B1 which after conversion corresponds to 25, 50, 75 and 100 ppm of titanium and 5, 10, 15 and 20 ppm of boron. This was compared with sample of alloy without inoculation. Grain size measurements, hardness measurements and chemical composition measurements were performed.

The results show the best values of grain size for addition of 0,2 % AlTi5B1, but the differences between individual grain size values of all samples are statistically minor.

The hardness measurements show the best results for addition of 0,15 % AlTi5B1, but also here the differences between achieved values are statistically not significant. Chemical evaluation confirmed presence of both titanium and boron in the alloy, but does not show significant differences.

Even the results of the experiment did not confirm the hypothesis, it cannot be stated that the inoculation does not have the effect. It is necessary to consider the conditions of the experiment. There are several factors which could influence the results:

The temperature of the melted alloy in the holding furnace is quite low. The technological regulation for holding temperature is

645 °C due to preparation of adequate melt to achieve the proper semi-solid state in the following step – the melt swirling. Due the thermal balance between the crucible and the melt, exact temperature gradient is required. The higher temperature of the holding would disrupt this balance.

Due to the real foundry – not laboratory – process, the chemical composition of the prime alloy is varying and the exact amount of added and dissolved inoculation elements cannot be evaluated and compared.

There is the suspicion that (due to previous statements) the master alloy was not dissolved.

For further experiments it is recommended to measure the chemical composition of the melt before the inoculation and stir the melt properly to make sure the master alloy was dissolved completely and it is not sedimentated on the bottom of the furnace. Also, the higher amount of the master alloy should be experimented to show the trend of the grain size change. The addition could vary up to 0,4 % AlTi5B1, which is a limit amount for boron. Above 40 ppm of boron, clusters of borides can be created and they can sediment, grow up on the crucible wall or inclusions in the casting. Also for titanium higher amounts are recommended: 0,03 – 0,05 % Ti (as TiBAl 5:1) for low silicon content in Al-Si alloy (according to some works, e.g. [15]).

The other recommendation is to use thermal analysis for evaluation of inoculation effect.

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