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THE EFFECT OF ALLOYING ADDITIVES AND PROCESS PARAMETERS ON THE SOLIDIFICATION CHARACTERISTICS OF THE ALUMINIUM ALLOY AlSi7Mg0.3

VPLIV LEGIRNIH DODATKOV IN PROCESNIH PARAMETROV NA ZNAČILNOSTI STRJEVANJA ALUMINIJEVE ZLITINE AlSi7Mg0,3

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We have investigated the aluminium alloy AlSi7Mg0.3, which is used in the production of castings. Melting was carried out in an electric resistance furnace. Casting was done into measuring cells with thermocouples for simple thermal analysis. Six samples that differed in terms of the additives used for grain refinement and inoculation, with two different holding times, were cast. An alloying material AlTi5B1 was used for grain refinement. The inoculation effect on the solidification was studied with the addition of the inoculant AlSr10. The temperature of the melt in the furnace was 750 °C and the melting time was one hour and ten minutes. Firstly, the samples were cast, the melt was cooled, solidification occurred and finally it was cooled down in the solid state. During the entire cooling process, the temperature as a function of time was measured and the cooling curves were obtained. After the casting, samples for metallographic inspection were prepared. The cooling curves show that in samples with a longer holding time, the additions of either the inoculant or the grain refinement agent, had a better effect. This is evident with a higher maximum liquidus temperature T_{Lmax} in sample Ti10 and lower maximum eutectic temperature T_{E1max} in sample Sr10 in comparison to the basis alloy. The same we can conclude from the microstructure analysis of the samples. The samples with inoculant and with a longer holding time, possessed a finer eutectic structure (α_{Al}+β_{Si}), than the samples with the shorter holding time. The same applies for the samples with grain-refinement additions. The estimated grain sizes were smaller in the sample with the longer holding time.

Keywords: aluminium alloy, AlSi7Mg0.3, grain refining agent, inoculant, simple thermal analysis

V članku avtorji opisujejo preučevanje aluminijeve zlitine AlSi7Mg0,3, ki se uporablja za izdelavo ulitkov. Taljenje je potekalo v elektrouporovni peči. Talina je bila ulita v merilne celice s termočleni za enostavno termično analizo. Odlitih je bilo šest vzorcev, ki so se razlikovali po dodatkih, ki se uporabljajo za udrobnjevanje (zmanjševanje velikosti) kristalnih zrn in modificiranje. Odliti so bili pri dveh različnih časih zadrževanja. Za udrobnjevanje zrn je bila uporabljena predzlitina AlTi5B1. Za preučevanje učinka modificiranja na strjevanje je bilo uporabljeno sredstvo AlSr10. Temperatura taline v peči je bila 750 °C, čas taljenja pa eno uro in deset minut. Najprej so bili vzorci uliti, talina se je ohlajala, prišlo je do strjevanja in nazadnje se je ohladila v trdnem stanju. Med celotnim postopkom ohlajanja so avtorji raziskave merili temperaturo v odvisnosti od časa in pridobili ohlajevalne krivulje. Po litju so bili vzorci pripravljeni za metalografsko analizo. Ohlajevalne krivulje kažejo, da so v vzorcih z daljšim časom zadrževanja dodatki inokulanta ali sredstva za udrobnjevanje bili učinkovitejši. To je razvidno iz višje maksimalne likvidus temperature T_{Lmax} v vzorcu Ti10 in nižje maksimalne temperature evtektika T_{E1max} v vzorcu Sr10 v primerjavi z osnovno zlitino. Enako lahko zaključimo iz mikrostrukturne analize vzorcev. Vzorci z dodatkom inokulanta so v primeru daljšega časa zadrževanja imeli bolj fino evtektsko strukturo (α_{Al}+β_{Si}), kot vzorci s krajšim časom zadrževanja. Enako velja za vzorce z dodatki za udrobnjevanje. Ocenjene velikosti zrn so bile manjše pri vzorcu z daljšim časom zadrževanja.

Ključne besede: aluminijeva zlitina, AlSi7Mg0,3, udrobnilno sredstvo, modifikator, enostavna termična analiza

1 INTRODUCTION

Aluminium is the most frequently used metal among non-ferrous metals. It is used in the form of technically pure aluminium as well as in alloys with Si, Mg, Cu, Mn, Zn, Fe, etc. The addition of the alloying elements influences the mechanical properties, castability, workability and corrosion resistance. Aluminium alloys can be divided into two groups: wrought alloys and casting alloys.[1,2]

Al-Si is a popular aluminium alloy. It is suitable for gravitational casting into moulds that can be made of sand mixtures or steel. It is also used in low-pressure die casting as well as high-pressure die casting.[3] Casting in the sand has lower cooling speeds in comparison with casting into steel dies. Melt quality can be controlled with a cooling curve and a temperature-time diagram (T-t), which is typical output from simple thermal analysis [4,5]. By increasing the cooling speed, undercooling is affected, which has an impact on the alloy microstructure. A higher cooling speed affects the crystallization and as a result the distribution and size of the eutectic phases is finer and dendritically formed grains are smaller. The microstructure of an alloy is also affected by the chemical composition, grain refining, modification of the eutectic and heat treatment.[6,7]

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By adding a grain-refining agent, we introduce heterogeneous nucleus into the melt, that enables crystallization of the primary mixture crystals $\alpha_{Al}{}^{P}$. This causes the formation of fine crystal grains. Adding the inoculant influences the eutectic solidification especially on the formation of β_{Si}. It changes from the coarse β_{Si} phase shape into finer β_{Si} within the eutectic.[8] For grain refining of the primary crystal grains α_{Al}, titanium and boron are used, while agents based on sodium, antimony and strontium are used for the inoculation. Thus, an alloy with additives for grain refining and inoculation has a finer microstructure, including the finer primary mixture crystals $\alpha_{Al}{}^{P}$ and finer eutectic (α_{Al}+β_{Si}), has improved mechanical properties, such as strength, hardness, impact toughness.[9–11]

Depending on the amount of silicon, Al-Si alloys are distributed into three groups: hypoeutectic alloys with 4–7 *w*/% Si, eutectic alloys with 10–13 *w*/% Si and hypereutectic alloys with a ratio of 14–25 *w*/% Si. During the solidification process, primary mixture crystals $\alpha_{Al}{}^{P}$ begin to crystalize from the melt. These crystals are dendrite shaped. During the solidification of primary crystals, the remaining melt is enriched with silicon. This follows a eutectic solidification where the rest of the melt gets solidified into a eutectic (α_{Al}+β_{Si}).[6,12–13]

Al-Si can contain other alloying elements, for example, the hypoeutectic alloy AlSi7Mg0.3. It is mainly used in casting processes. This type of alloy is corrosion resistant, has good castability and welding properties, pressure strength and workability. Chemical composition and alloy's properties are stated in the standard EN 1706: 2010 (ENAC-42100).[14]

Generally, before casting the alloy AlSiMg0.3, enough grain-refining agent and inoculant are added into the melt. The grain-refinement agent increases the number of heterogenous nuclei in the melt. This influences the number of crystal grains formed during the solidification process.[13]

The Mn content in alloy (up to 0.6 *w*/%) disables the formation of phase β-Al_5FeSi, which is undesired because of its acicular shape that can cause fatigue notch factor and inferior mechanical and corrosion properties.[11,13]

The grain refinement of AlSi7Mg0.3, was studied by Anna Knaislová and coworkers.[15] The test required the use of two types of inoculants: inoculant AlTi5B1 and AlTi3B1. Their results show that the finest dendritic structure is in samples that contained a grain-refinement agent of 0.01 *w*/%. The difference between AlTi5B1 and AlTi3B1 in terms of grain-refinement effect is minimal. That means that further increasing of the grain-refinement agent would not contribute to additional beneficial effect on the microstructure of the casting. Grain refining of Al-Si alloys was studied by Agnes and coworkers. They used Al-10 % Ti and Al-4 % B for refining. Research shows that the grain size decreases with increasing Ti concentration. Al-4%B can react with traces of Ti and form TiB_2.[16]

The microstructure of alloy AlSi7Mg0.3 consists of a solid solution of primary crystalline $\alpha_{Al}{}^{P}$, the eutectic (α_{Al}+β_{Si}) and a second eutectic (α+Mg_2Si). The primary aluminium grains have a dendritic form. First eutectic consists of a solid solution of α_{Al} and β_{Si}, which is compressed into a lamellar shape. β_{Si} appears on the plane of the metallographic cut in the acicular shape where the needles are irregular. The form of β_{Si} in the eutectic can also be changed by heat treatment, where we also get a globular eutectic besides the phase α and precipitates.[17–21]

The goal of this research was to study the solidification process of aluminium alloy AlSi7Mg0.3, with additions of a grain-refining agent and an inoculant, during two different holding times, using simple thermal analysis. Then the microstructure of samples was studied with an optical microscope.

2 MATERIALS AND METHODS

The samples were made from a block of aluminium alloy. Each weighed from 355 g to 370 g. This material was put into cups for melting. The melting was carried out in an electric resistance furnace produced by Bosio. The melting time was one hour and ten minutes. The temperature was set up to 750 °C. After the melting process, the oxide layer was removed and then the melt was alloyed with a grain-refinement agent and the inoculant.

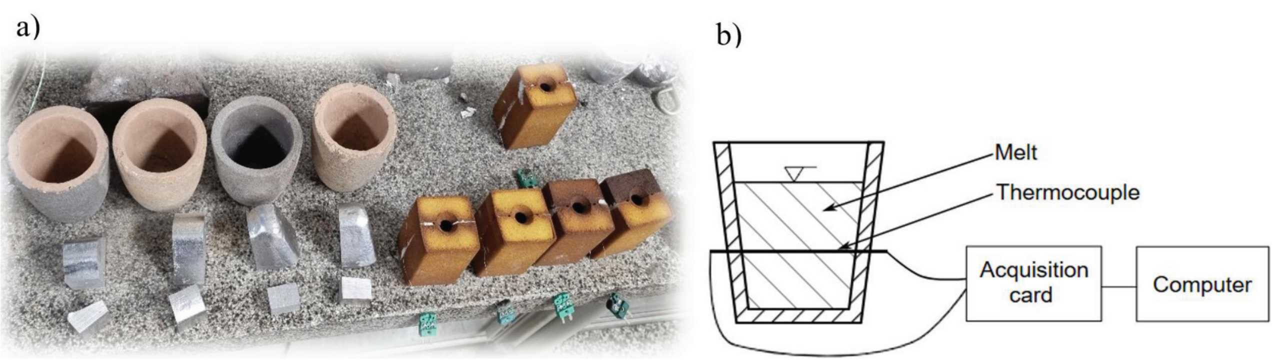

Figure 1: a) Prepared material – cups, measuring cells, weighed pieces of alloy, b) simple thermal analysis schematic view

After the alloying, the melt was stirred with steel rod. After stirring the cups were returned to the furnace for a specific holding time. After a certain holding time the oxide layer was again removed. Next the pouring of melt into the measuring cells followed. There simple thermal analysis was conducted.

Figure 1 shows the prepared material for melting and casting. The picture shows cups, pieces of alloy for each charge, as well as the measuring cells for simple thermal analysis with type K (Ni-NiCr) thermocouples, on which are connectors.

These are connected to the measuring card, which is connected to a computer. The computer follows the temperature in relation to time.

Alloy AlTi5B1 was used for grain refinement and alloy AlSr10 was used as the inoculant. **Table 1** represents the additives, holding times of the tested samples and their designation.

Table 1: Description of tested samples and their designation

Sample designation	Description
B	Primary alloy, AlSi7Mg0.3 (no additions)
Ti1	Addition of AlTi5B1 (1 min holding time)
Ti10	Addition of AlTi5B1 (10 min holding time)
Sr1	Addition of AlSr10 (1 min holding time)
Sr10	Addition of AlSr10 (10 min holding time)
TiSr	Addition of AlTi5B1 and AlSr10 (1 min holding time)

The amount of grain refinement and inoculant additives were determined according to recommendations from the literature.[22] The amount of alloying material used for grain refinement was in all cases 3 g (approximately 0.8 *w*/%), and the inoculant 1 g (approximately 0.3 *w*/%).

Chemical analysis of the primary alloy was carried out with an x-ray fluorescence spectroscopy (XRF analysis) with a device manufactured by ThermoSCIENTIFIC. The model used was Niton XL3. **Table 2** shows a chemical composition of the aluminium alloy before the melting process.

During the solidification process we studied the cooling and solidification process with a simple thermal analysis. For this a type-K thermocouple was used. After the solidification of the castings, some samples were cut to be metallographically analysed. The metallographic analysis was made with an Olympus BX 61 light microscope.

3 RESULTS AND DISCUSSION

This article studies the effect of grain-refinement agent and inoculant on the alloy AlSi7Mg0.3 solidification. Six different samples were produced for the test. They differed in the type of additive with two different holding times in which the refinement agent and inoculant were exposed in the melt. Thus, we prepared a primary melt, a melt with the grain-refinement agent, a melt with the inoculant, and a melt with both grain refinement and inoculant. The tested alloys were cast into the measuring cells for a simple thermal analysis.

After the cooling, cast samples were studied using a simple thermal analysis and observation of the microstructure. Samples for microstructural analysis were taken from the casting of the thermal analysis measuring cells. **Figure 2a** and **2b** shows a cross-section of the measuring cell, and the position of surface for metallographic analysis (**Figure 2c**).

The cut samples were grinded. After the grinding process, the two-step polishing followed. In the first step the samples were polished on a wool base with a 3-μm diamond suspension. In the second step a neoprene base was used and for the polishing agent a suspension of colloid silicon dioxide with grain size of 0.04 μm was added.

The obtained temperatures and times during the solidification process of all the samples were presented as cooling curves using OriginPro. **Figure 3** shows a cooling curve for the base alloy (mark B).

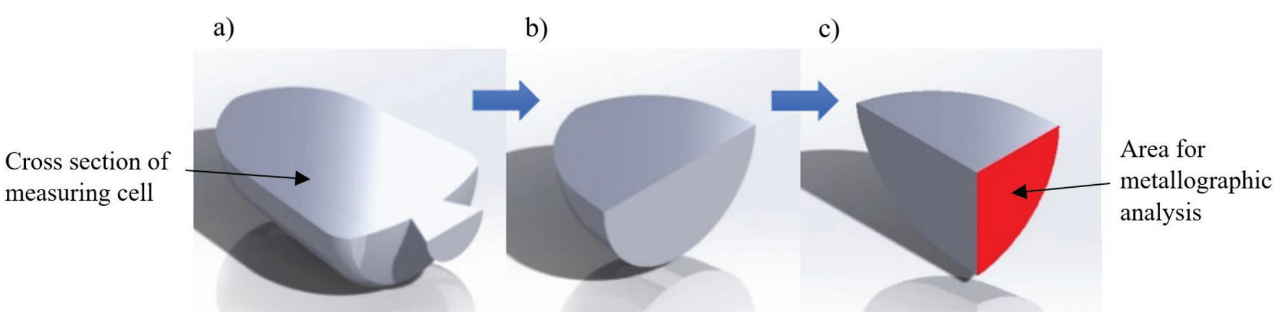

Figure 2: a), b) Presentation of the cut casting of the measuring cell, c) area intended for metallographic analysis

Table 2: Chemical composition of the alloy before melting

Chemical composition (*w*/%)								
Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Al
6.84	0.115	0.011	0.066	0.342	<0.001	<0.001	0.125	rest

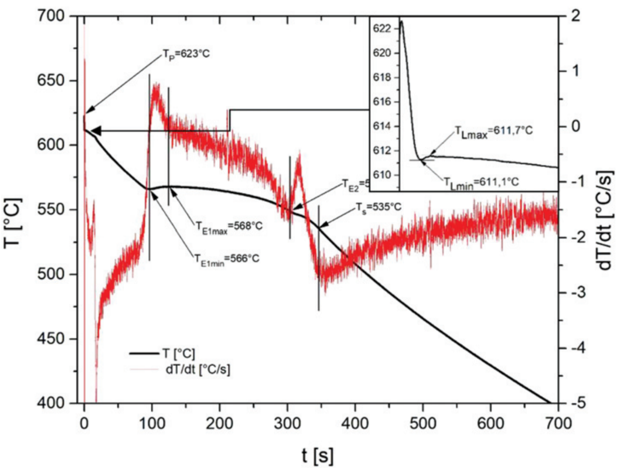

Figure 3: Cooling curve of the sample mark B

Figure 3 shows the casting temperature T_P (623 °C). During the cooling, the melt's temperature decreases. The melt starts to solidify. Important points on the cooling curve are also undercooling T_{Lmin} (611.1 °C), the formation of a nucleus for the growth of crystal mixture α_{Al}, that start with the crystallization of the melt at T_{Lmax} (611.7 °C). The crystals α_{Al} grow to the eutectic temperature T_{E1min} (566 °C) where undercooling occurs and T_{E1max} (568 °C) where the eutectic starts to solidify E_1 (α_{Al}^{E1}+β_{Si}). At temperature T_{E2} (549 °C) the solidification of the next eutectic begins. This is eutectic E_2 (α_{Al}^{E2}+β_{Si}+Mg_2Si). With a solidus temperature T_S (535 °C), the solidification is complete.

Table 3 shows all the collected temperatures of important points from the cooling curves for the analysed samples.

Table 3: Characteristic temperatures from the cooling curves of the tested samples

Temperature	Sample					
(°C)	B	Ti1	Ti10	Sr1	Sr10	TiSr
T_P	623.0	660.0	674.0	691.0	689.0	687.0
T_{Lmin}	611.1	612.2	612.0	609.5	609.5	612.1
T_{Lmax}	611.7	612.7	612.5	611.2	611.0	612.6
T_{E1min}	566.0	567.5	568.4	563.5	562.5	562.0
T_{E1max}	568.0	568.5	569.5	565.5	564.5	565.0
T_{E2}	549.0	549.0	549.5	549.5	549.0	549.0
T_S	535.0	538.0	537.0	537.5	534.5	536.3
ΔT_L	0.6	0.5	0.5	1.7	1.5	0.5
ΔT_{E1}	2.0	1.0	1.1	2.0	2.0	3.0

Figure 4 shows the comparison of the cooling curves of all the samples. The graph presents the cooling curve of the base alloy sample B and alloys with different additives and holding times in the base melt. In sample Ti1 and Ti10 the precipitation of the primary mixture crystals α_{Al}^P starts at higher temperatures than than the one in the base alloy because of the already present heterogeneous germs in the melt.

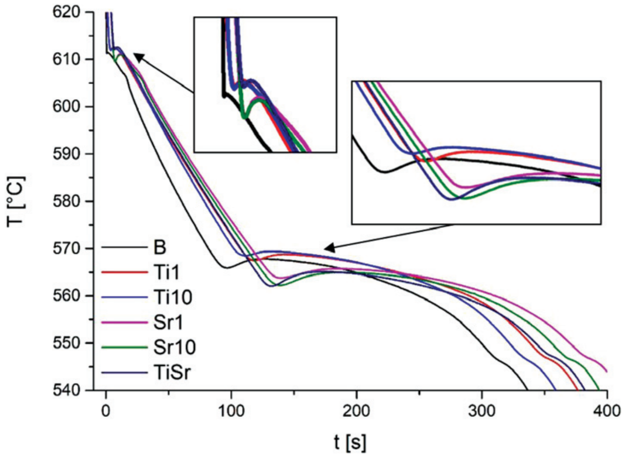

Figure 4: Comparison of the cooling curves of all measured samples

If we compare the base alloy and the sample Sr1 and Sr10, we can conclude that the liquidus and eutectic temperatures are lower than in sample B. Among all the samples, the sample TiSr has the lowest temperature of the first eutectic T_{E1}, which is 562 °C and thus the most ennobled state among the analysed alloys.

The addition of a grain refinement and inoculant affect the curing temperature interval. Thus, by adding the grain refinement AlTi5B1, the interval solidification T_L-T_S increases compared to the base alloy sample B. The interval also increases when an inoculant is added. The temperature difference between T_L-T_S increases the most in the TiSr sample, where both additives are added to the melt (**Figure 5**).

The tested alloys were metallographically studied with an optical microscope. **Figure 6** shows the microstructures of the tested samples. In **Figure 6B** there are larger dendritic shaped α_{Al}^P grains, eutectic E_1 (α_{Al}^{E1}+β_{Si}) and E_2 (α_{Al}^{E2}+β_{Si}+Mg_2Si). The size of α_{Al}^P grains is smaller in the case of samples with the grain-refinement agent addition Ti1 and Ti10 (**Figure 6Ti1** and **6Ti10**). Dendrites α_{Al}^P have decreased in size.

In the samples with added inoculant Sr1 and Sr10 the shape of the eutectic phase β_{Si} (**Figure 6Sr1** and **6Sr10**) changes. It is precipitated in the finer form. The Sr10

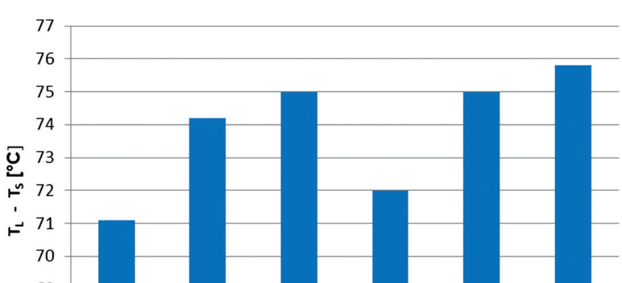

Figure 5: Difference between liquidus and solidus temperature

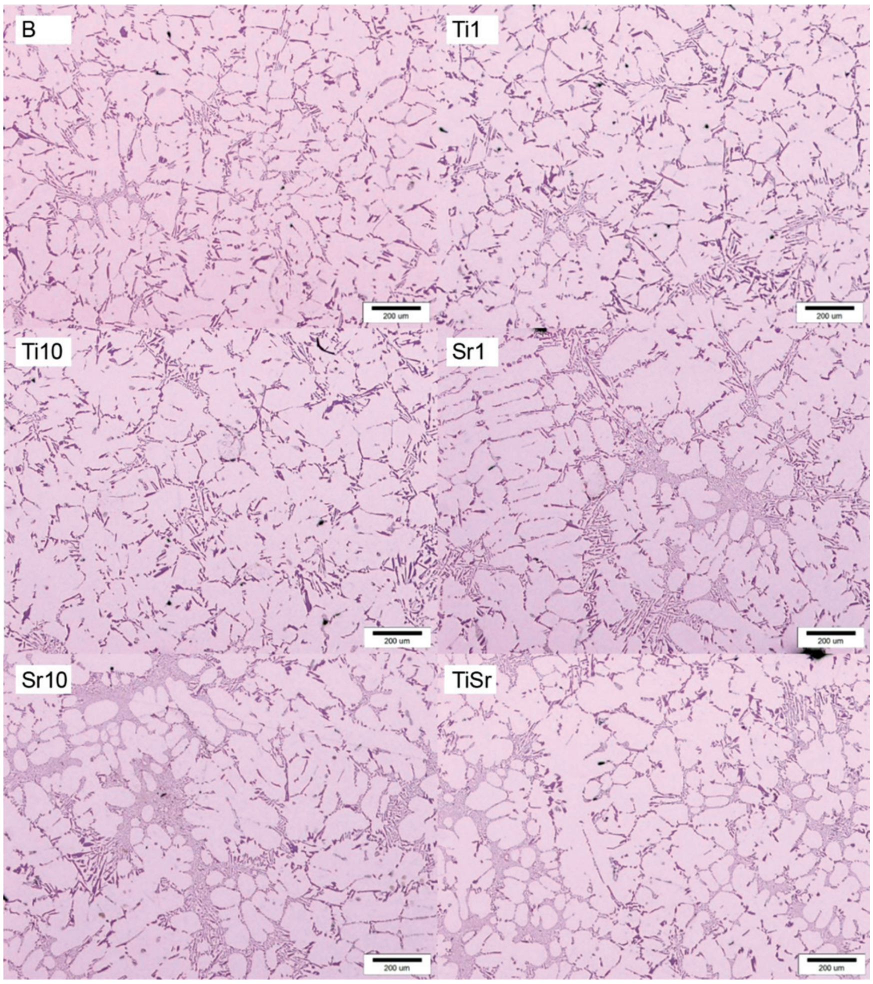

Figure 6: Comparison of microstructures of samples: B, Ti1, Ti10, Sr1, Sr10, TiSr

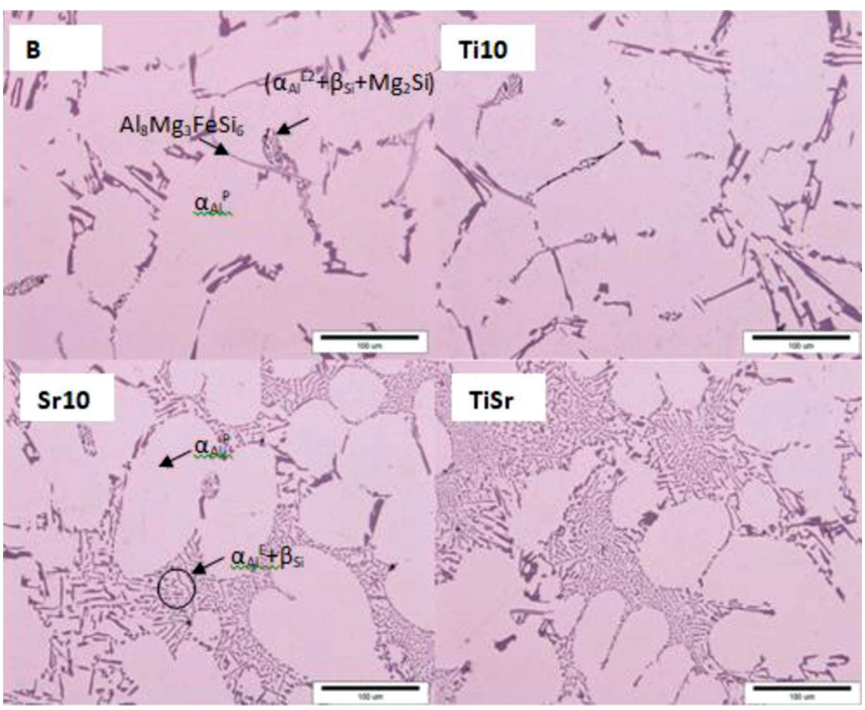

Figure 7: Comparison of samples' microstructures: B, Ti10, Sr10, TiSr

sample in comparison to Sr1, has a higher amount of finer eutectic, which was well modified.

Sample TiSr (**Figure 6TiSr**) was alloyed with both the grain-refinement agent and the inoculant. There are small α_{Al} grains crystalized in the microstructure. β_{Si} in the eutectic has precipitated in a finer globular form in comparison to the base sample B.

Besides the eutectic and primary mixture crystals α_{Al}^{P} dendrites, also a lighter phase can be detected in the microstructure. This phase is based on iron. It is normally formed in sharp forms. In microstructure of base sample B, eutectic phase is in form of rough lamellas, the grain size of the α_{Al}^{P} is around 138 μm (**Figure 7B**). This eutectic is reformed by addition of the inoculant AlSr10 (**Figure 7 Sr10**). By adding the alloy AlTi5B1, the size of primary mixture crystals α_{Al}^{P} crystals (**Figure 7 Ti10**) decreases. In case of sample TiSr, a vivid reduction in lamellas of Si inside the eutectic is shown. The addition of inoculant is seen in the TiSr sample, where we have eliminated the Si phase in fibrous form in the eutectic. Also, smaller dendrites α_{Al}^{P} is visible in **Figure 7 TiSr**, the grain size is around 62 μm.

As can be seen from **Figure 7**, the AlSi7Mg0.3 alloy consists of primary mixed crystals of α_{Al}^{P}, which solidify first. Crystallization of the $Al_8Mg_3FeSi_6$ intermetallic phase follows. The rest of the melt solidifies eutectically. First, the eutectic E_1 (α_{Al}^{E1} + β_{Si}) is formed, and then, within the framework of the ternary eutectic, the remaining melt in the microlast independent solidification areas crystallizes into E_2 (α_{Al}^{E2} + β_{Si} + Mg_2Si).

The addition of a grain refining agent and inoculant are shown in the **Figure 7** marked TiSr, where in the microstructure we have a smaller size of crystalline α_{Al}^{P} and the eutectic Si is precipitated in a spherical form. α_{Al}^{P} crystals are reduced due to the addition of AlTi5B1 refining agent. After the addition of grain refiners AlTi5B1 particles $TiAl_3$ and TiB_2 are formed. The resulting precipitates act as grain growth inhibitors ($TiAl_3$) and as heterogeneous nucleation sites (TiB_2) for the matrix nucleation.[23]

The shape of the precipitated β_{Si} in the eutectic is influenced by the addition of the AlSr10 inoculant. This causes a change in the morphology of β_{Si} in the eutectic phase. The basic mechanism of the added AlSr10 is the adsorption of strontium onto the preferred β_{Si} growth surfaces. Sr is adsorbed on twinning planes. Therefore, crystal growth in preferred directions is inhibited. As a result, β_{Si} grows in the eutectic in a more rounded and fine-grained form.

4 CONCLUSIONS

Aluminium alloys are frequently used in mechanical engineering due to their high strength and low density. The desired mechanical properties can be achieved in the cast state with appropriate melt treatment. To produce the best-possible quality of the castings in the cast state, the quality of the melt has to be monitored during the production process. This can be done by a simple thermal analysis. Thus, we analysed the solidification process of the alloy AlSi7Mg0.3, to which was added the grain-refinement agent in the form of the alloying material AlTi5B1 and the inoculant AlSr10. The results of this study are the following:

- The cooling curves of the simple thermal analysis show that by adding AlTi5B1, the liquidus temperature moves up to higher values, while recalescence decreases with liquidus temperature. The indicator for an effective refinement is thus an absolute value T_{Lmin}, which should be as high as possible at low recalescence ($T_{Lmax} - T_{Lmin}$).
- By adding strontium within the addition of AlSr10 alloying material caused a change (modification) of the eutectic phase β_{Si} in the alloy, which can be observed on the cooling curve where the eutectic temperatures move to lower values.
- The cooling curve also shows that samples with a longer holding time that were added either the inoculant or the grain refinement agent, have a more efficient performance. This is evident with a higher maximum temperature T_{Lmax} in sample Ti10 and lower temperature T_{E1max} in sample Sr10 in comparison to the samples with a shorter holding time.
- In the microstructure are primary mixture crystals α_{Al}^{P} dendrites as well as two eutectics: eutectic 1 (α_{Al}^{E1}+β_{Si}) and eutectic 2 (α_{Al}^{E2}+β_{Si}+Mg_2Si). There was also a lighter phase, which is based on iron and has a long sharp form.
- Based on the microstructures we can see that the estimated size of the primary mixture crystals α_{Al}^{P}, as well as the size, form and distribution of the eutectic silicon β_{Si}, are congruent to the measured cooling curves. This proves the effectiveness of the inoculant and refinement agent, which has a great practical value.

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