

Effect of the grain refinement, modification and the cooling rate on microstructure of the AlSi10Mg alloy

Vpliv udrobnevanja, modificiranja in ohlajevalne hitrosti na mikrostrukturo zlitine AlSi10Mg

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Abstract: This paper describes the influence of grain refiners, modifiers and cooling rate on microstructure of AlSi10Mg alloy. Solidification of this alloy has been investigated. Investigations have been made by computer simulations on program MAGMASOFT, »in situ« thermal analysis, simultaneous thermal analysis (STA) and metallographic analysis. Investigated alloy AlSi10Mg has been grain refined and modified. By cooling curves at different cooling rates the undercooling and recalescence of primary and eutectic crystallization have been detected and related with largeness of primary crystals of α_{Al} and eutectic phase β_{Si} from eutectic ($\alpha_{Al} + \beta_{Si}$). Also the relation between largeness of microstructural constituents and cooling rate was determined.

Izveček: Ta članek opisuje vpliv udrobnilnih in modificirnih sredstev ter ohlajevalne hitrosti na mikrostrukturo zlitine AlSi10Mg. Preiskano je bilo strjevanje te zlitine. Preiskava je potekala s pomočjo različnih metod, kot so: računalniška simulacija polnjenja in strjevanja s programom MAGMASOFT, »in situ« termična analiza, simultana termična analiza (STA) in metalografska analiza. Preiskovano zlitino smo udrobnjevali in modificirali. Iz ohlajevalnih krivulj posnetih pri različnih ohlajevalnih hitrostih smo določili podhladitve in recalescence za primarno in evtektsko strjevanje katere smo povezali z velikostjo primarnih kristalnih zrn α_{Al} in evtektске faze β_{Si} iz evtektika ($\alpha_{Al} + \beta_{Si}$). Prav tako je bila opredeljena povezava med velikostjo mikrostrukturnih sestavin in ohlajevalno hitrostjo.

Key words: grain refinement, modification, cooling rate and microstructure

Ključne besede: udrobnevanje, modificiranje, ohlajevalna hitrost in mikrostruktura

INTRODUCTION

AlSiX casting alloys are today commonly used because of their properties, as are good castability, mechanical properties and corrosion resistance. Microstructure, which is formed during the solidification, has a great

influence on mechanical properties. That is the reasons for examination of the relations between melt and the mould.

During casting of aluminium alloys, defects on castings are common. These defects are usually micro porosity, shrinkage porosity

and cracks. Reasons for these defects are usually not well-prepared melt and mistakes in the casting technology. To improve soundness of castings one can use grain-refining agents and modifying agents, which improves soundness of castings, causes better feeding and better chemical and mechanical treatment^[1,2].

Thermal analysis and chemical analysis are commonly used in foundry to control the state of a melt. With thermal analysis we are monitoring the temperature during solidification. From the cooling curves of the thermal analysis we can predict solidification and microstructure. During crystallization of phases the heat is liberated what is seen on the cooling curves. From characteristic shape of the cooling curves we can identify formation of different phases and predict microstructure even before the casting. If necessary we can modify and grain refine the alloy. It is also possible to predict the level of grain refining and modification from the shape of the cooling curve, undercoolings

and recalescences at primary solidification of α_{Al} and eutectic solidification ($\alpha_{Al} + \beta_{Si}$). Unmodified and modified cooling curve are shown in Figure 1^[3]. The first peak on cooling curve is showing that the liquidus temperature is higher and the recalescence is lower in well grain refined alloy. In a modified alloy eutectic temperature is lower and recalescence is higher^[4] (second peak).

EXPERIMENTAL

In present research we used standard blocks of AlSi10Mg alloy from WAV-IMCO producer. The alloy was investigated by several methods, which were: the chemical analysis, computer simulation of cooling rates and cooling curves, simple thermal analysis and metallographic analysis.

Three samples were investigated. First sample was the basic alloy, the second sample was grain refined with B (master alloy of AlB3) and modified with Sr (master alloy of

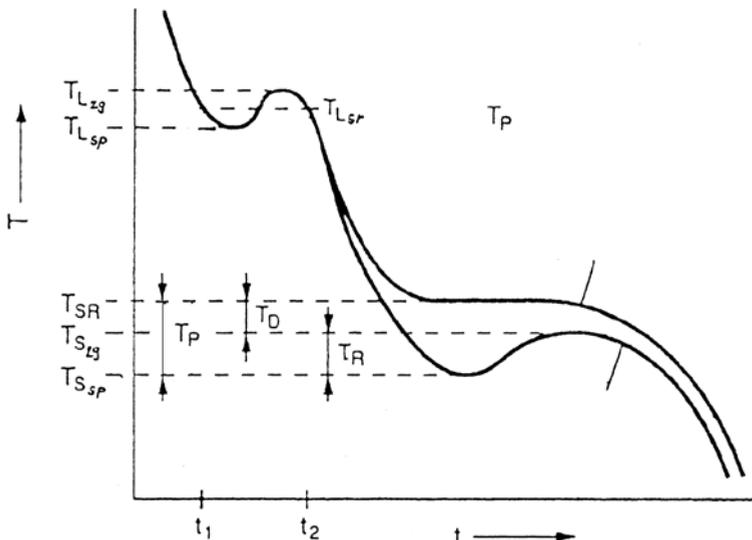


Figure 1. Cooling curve of hypoeutectic Al-Si alloy^[3]

Table 1. Labels of castings and additions of grain refiners and modifiers

Lable of casting	Grain refiner	Modifier
Vz1	-	-
Vz4	AlB3 – (0.05%B)	AlSr10 – (0.015%Sr)
Vz11	AlTi5B1 – (0.03%Ti)	Silutal 20 – (3%)

AlSr10) and the third one was grain refined with Ti (master alloy of AlTi5B1) and modified with Na (salt mixture of NaCl and NaF). Additions of the grain refiners and modifiers are shown in Table 1. Alloy was melted in the graphite crucibles in an induction furnace. As temperature of 700 °C was reached the grain refiners and modifiers were added and after a minute it was poured into measuring cells. At the addition of the salt mixture the temperature has reached 740 °C, after the salt was melted it was stirred into the melt and after three minutes it was poured into measuring cells.

Simple thermal analysis has been carried out in two measuring cells. The first one was made by Croning process and the second one was newly developed measuring cell called the cone probe made of the grey iron (Figure 2). This measuring cell has a continuous

change of cooling rates on overall intersection. K-type thermocouples were used and placed in the center of Croning measuring cell and in the center and top of the cone probe respectively as is shown in Figure 2. Thermocouples were connected to the National Instruments DAQPad-MI0-16XE-50 measuring card and this one further to the personal computer, where the measured values were collected with LabVIEW 5.0 program. Cooling curves were plotted using the Origin 7.0 program.

The cooling curves and the cooling rates were also calculated with computer simulation program MAGMASOFT and chemical compositions were analyzed too respectively.

The specimens for metallographic analyses were cut from the castings of the measur-

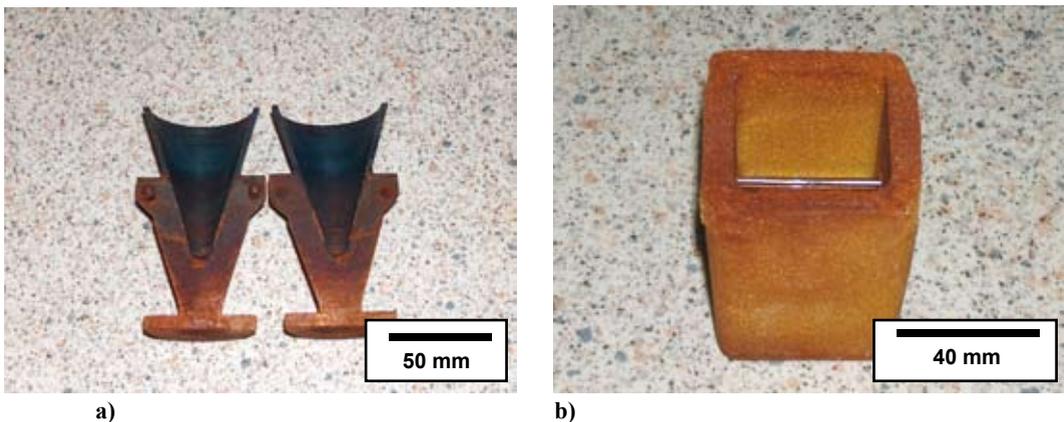


Figure 2. Measuring cells: measuring cell from the grey iron (a), the standard quick cup probe made by Croning process (b)

ing cells. The samples were prepared by the standard metallographic procedure for optical microscopy and by the anodic oxidation for observation in polarized light. Anodized samples were used for determining

the grain size and the others were used for determining the length of the eutectic silicon particles. Grain sizes and the grain size numbers (G) were determined by intercept counting method from ASTM standard with

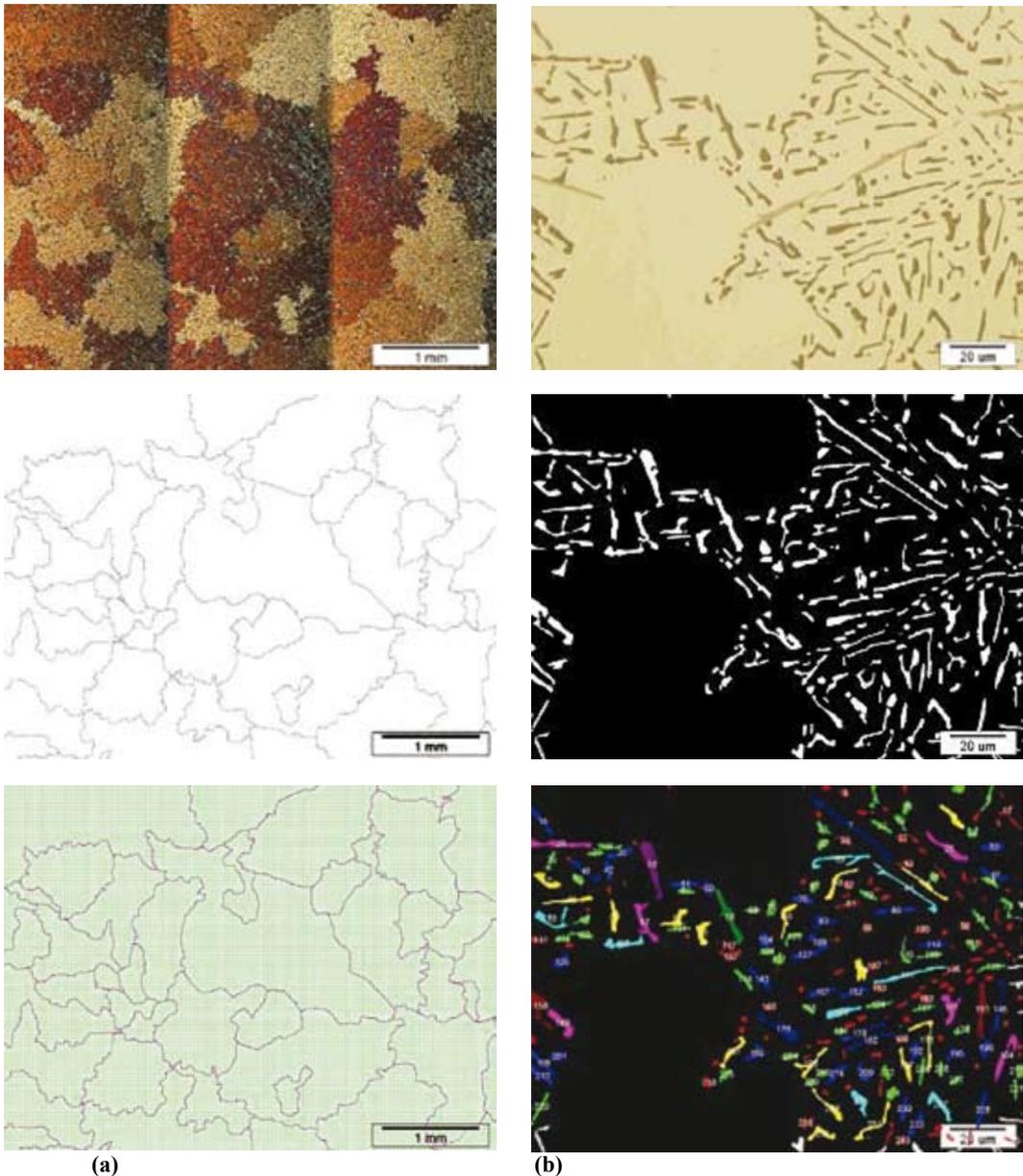


Figure 3. Presentation of the grain size determination (a) and the particle lengths (b)

analySIS 5.0 computer program. Lengths and ratios between the length and the width of the silicon particles were also determined by the analySIS 5.0 computer program. Figure 3 is showing procedures for determining grain size and particle lengths.

RESULTS AND DISCUSSION

AlSi10Mg alloy was used at present research. This alloy contains about 10 mass % Si, 0,4 mass % Mg. Tables 2 and 3 show chemical compositions of the alloy before and after the experiment. One can see that composition is changed after the experiment, because of the addition of the grain refining and the modifying agents.

The filling, solidification, cooling rates and cooling curves for the sample in the cone probe were calculated with the computer simulation program of MAGMASOFT. Figure 4 is showing the cooling rates in overall section off the cone probe. The cooling curves were calculated in the spots shown

in Figure 4 and they are similar to experimentally determined cooling curves. Figure 5 is presenting the calculated and the experimentally determined cooling curves from the cone probe for a basic alloy (Vz 1).

The calculated cooling rates are: 19 K/s on the spot A, 38 K/s on spot B and 176 K/s on spot C. Measured cooling rates are similar and in average are: 20 K/s on spot A and 43 K/s on spot B. On the spot C the measurement was impossible because the cooling rate was too high. Cooling rate in quick cup of the measuring cell was the lowest. It was 6,3K/s in average. Cooling curves of all three specimens and the derivate curve of the specimen Vz.1 are shown on Figure 6 respectively.

Microstructure of AlSi10Mg alloy is composed of the dendrite crystals of α_{Al} and of the eutectic ($\alpha_{Al} + \beta_{Si}$). It is better for mechanical properties if microstructural constituents of α_{Al} and of the eutectic ($\alpha_{Al} + \beta_{Si}$) are finer. For grain refining we used master alloys of AlTi5B1 and AlB3. With this master al-

Table 2. Chemical composition of the basic alloy

Element	Si	Fe	Mn	Mg	Zn	Ti
Mas. %	10.815	0.663	0.066	0.334	0.037	0.052
Cr	Ni	Pb	Sn	Ca	Cu	Al
0,008	0.005	0.005	0.002	0.004	0.066	87.943

Table 3. Chemical compositions of the castings after investigations

Element (Mas. %)	Si	Fe	Cu	Mn	Mg	Cr	Ni
Vz. 1	9,981	0,678	0,068	0,076	0,348	0,009	0,006
Vz. 4	9,811	0,698	0,081	0,076	0,383	0,011	0,007
Vz. 11	10,456	0,649	0,075	0,077	0,183	0,010	0,009
Element (Mas. %)	Zn	Ti	Ag	B	Na	Sr	Al
Vz 1	0.053	0.047	0.006	0.00016	<0.0001	<0.0003	Ostalo
Vz 4	0.034	0.063	0.006	0.0948	<0.0001	0.0173	Ostalo
Vz 11	0.038	0.081	0.006	0.0059	0.014	<0.0003	Ostalo

loys we introduce nuclei for heterogeneous nucleation into the melt^[3,5]. At the primary crystallization the undercooling is lower and α_{Al} crystals are smaller^[6].

For modification of the eutectic silicon we used master alloy of AlSr10 and the mixture of salts containing sodium. These two agents are the reason for modification of silicon from lamellar structure to fibrous structure^[7] (particles become rounder). This affects on the cooling curve as depression of the eutectic temperature and the recalescence^[8] (Figure 1).

The characteristic temperatures such as liquidus, eutectic and the solidus were determined from the cooling curves obtained

on the quick cup measuring cell (Figure 6). The temperatures are shown in Table 4. Unmodified alloy has the liquidus temperature of 581.9 °C and the recalescence of 2.5 °C. The eutectic temperature is 570 °C and the recalescence about 0.5 °C. The specimen grain refines and modified with B and Sr has the highest liquidus temperature at 593.3 °C and there is no recalescence. The eutectic temperature is at 563.9 °C and the higher recalescence 2.5 °C respectively. Lower recalescence at the primary solidification and the higher one at the eutectic solidification is a good sign of well grain refined and well modified alloy. Similarly as for the specimen 4 is for the specimen 11 grain refined and modified with Ti and Na. Liquidus temperature is at 587.6 °C, the recalescence is 0.3 °C.

Ne razumem popravka!

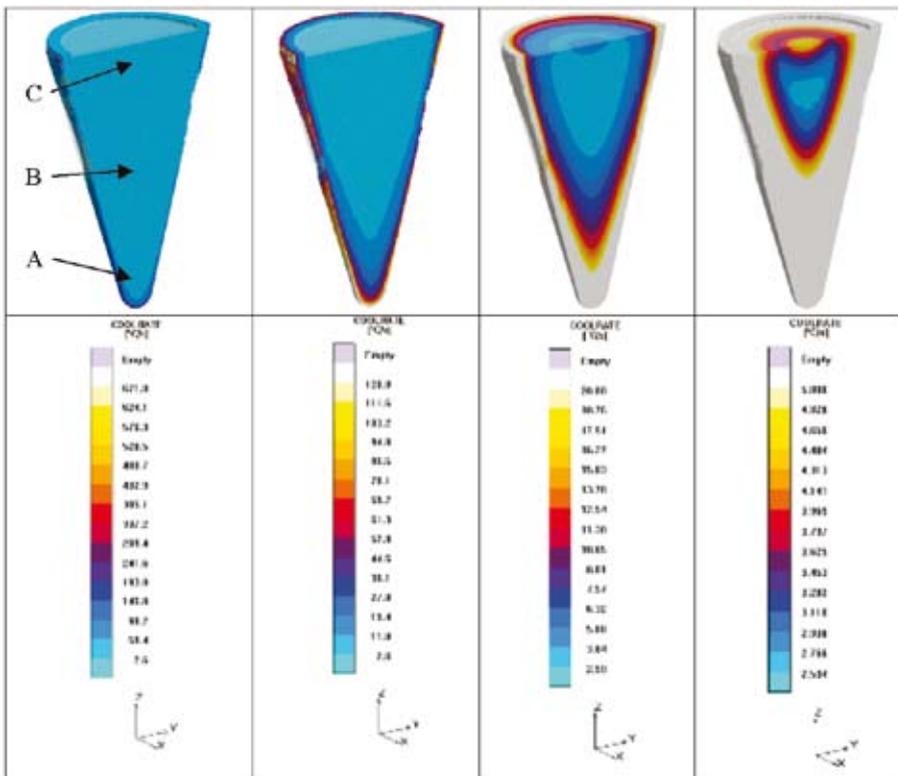


Figure 4. Cooling rates on the overall section of the cone probe

Table 4. Typical temperatures on the cooling curves

	V_{cool} [K/s]	T_{pour} [°C]	T_L^{min} [°C]	T_L^{max} [°C]	T_E^{min} [°C]	T_E^{max} [°C]	T_{E2}^{Mg2Si} [°C]	T_S [°C]	dT_L [°C]	dT_E [°C]
Vz. 1	7.2	680	582.0	584.5	570.0	570.5	549.2	527.0	2.5	0.5
Vz. 4	6.8	688	593.3	593.3	563.9	566.7	548.9	533.7	0	2.8
Vz. 11	4.7	731	587.6	587.9	562.7	564.5	550.2	537.4	0.3	1.8

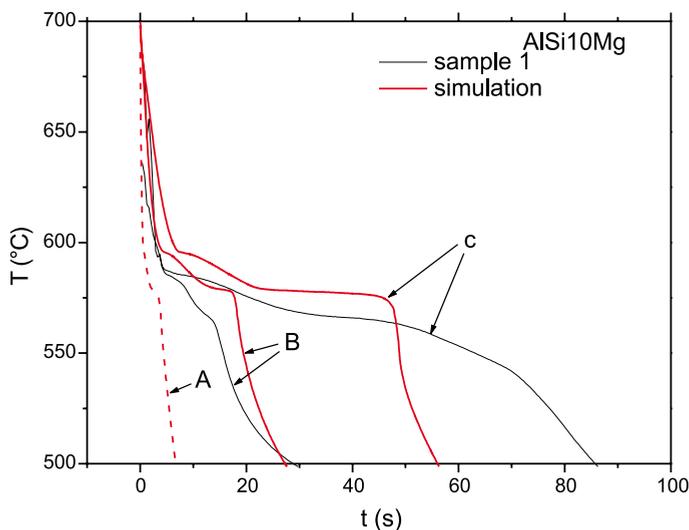
At the eutectic temperature of 562.3 °C the recalescence is 1.8 °C.

We determined grain sizes and grain size numbers (G) for the all specimens. The largest crystals were in specimen of the basic alloy from quick cup measuring cell where was the lowest cooling rate. With the higher cooling rate the grain size dropped from 2079 μm to 310.8 μm . In the sample of Vz.4

the grain refined with B is the smallest grain size. At the lowest cooling rate of 6.3 K/s it is 184.9 μm and at the cooling rate of 176 K/s it is only 96.2 μm . In the sample of Vz.11 the grain size is 881.3 μm at the lowest cooling rate and 271.6 μm at highest cooling rate respectively. All the data are collected in Table 5 and the microstructures are presented in Figures 7, 8 and 9.

Table 5. Grain sizes and the grain size numbers for all specimens on the different measuring spots

Sample	Vz.1		Vz.4		Vz.11	
	G	l_{sr} [μm]	G	l_{sr} [μm]	G	l_{sr} [μm]
Quick cup	-5.4	2079	1.6	184.9	-2.9	881.3
C	-2	637.8	2.1	153.7	-1.8	595.9
B	0.1	329.9	3.3	101.5	0.5	267.5
A	0.1	310.8	3.5	96.2	0.5	271.6

**Figure 5.** Calculated cooling curves and the measured cooling curves of the sample Vz.1

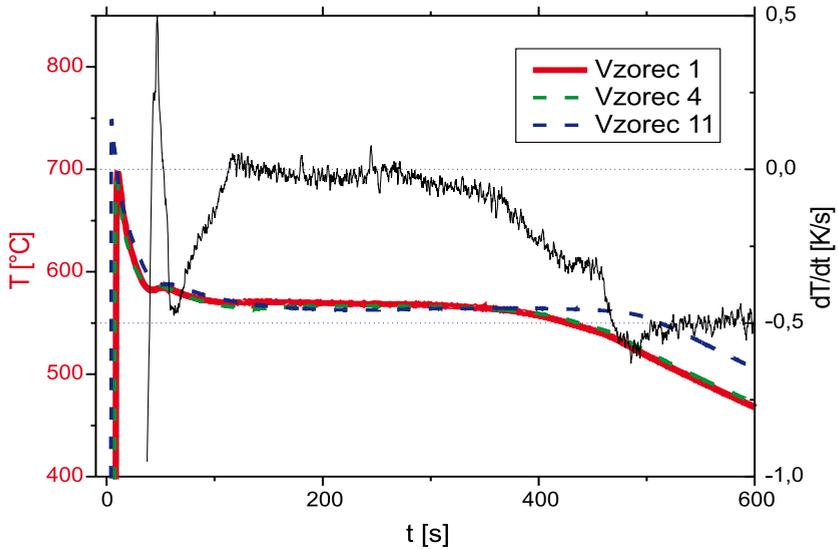
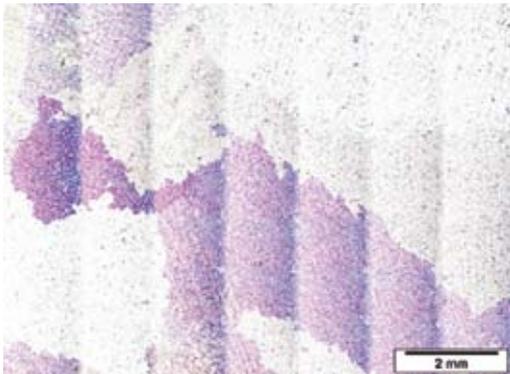
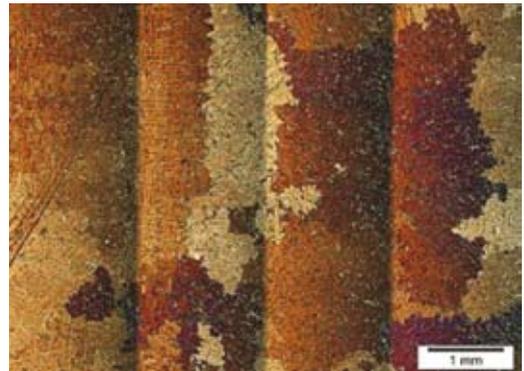


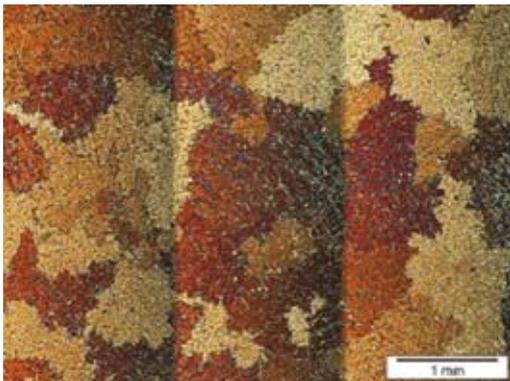
Figure 6. Cooling curve and the derivative curve of the specimen Vz.1 and the cooling curves of specimens Vz.4 and Vz.11



(a) Quick cup (6.3 K/s)



(b) Cone probe – C (19 K/s)

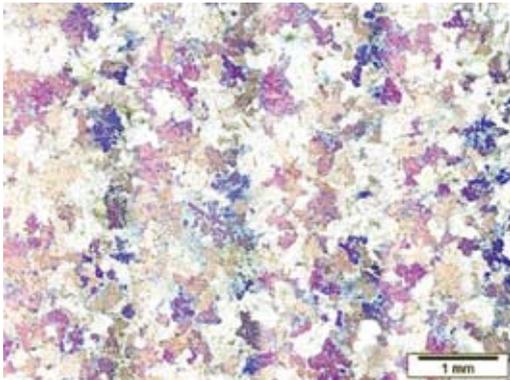


(c) Cone probe – B (38 K/s)

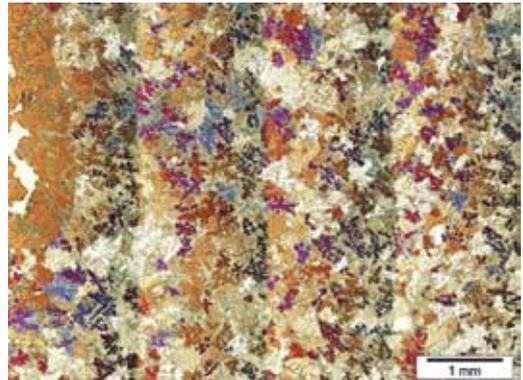


(d) Cone probe – A (176 K/s)

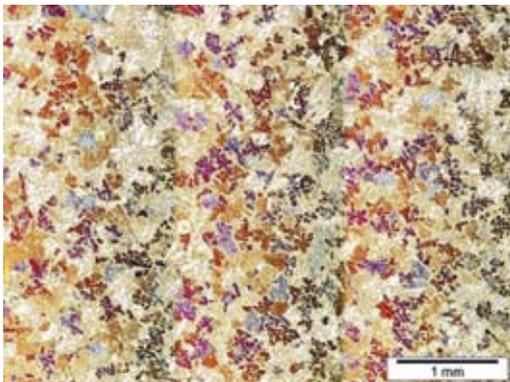
Figure 7. Microstructure of the sample Vz.1 (basic alloy) in the polarised light



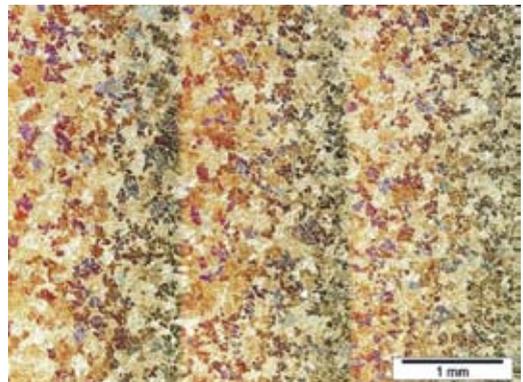
(a) Quick cup (6,3 K/s)



(b) Cone probe – C (19 K/s)

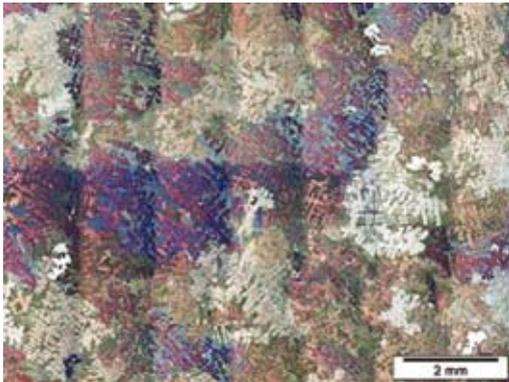


(c) Cone probe – B (38 K/s)



(d) Cone probe – A (176 K/s)

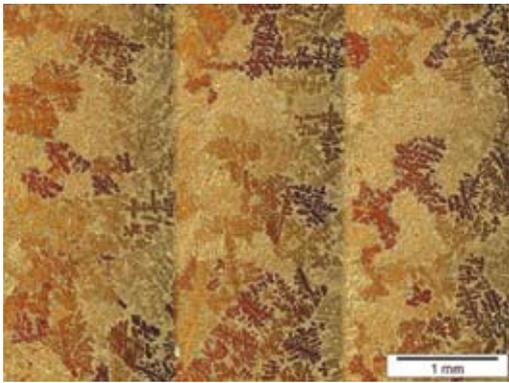
Figure 8. Microstructure of specimen Vz.4 (grain refined with B, modified with Sr) in the polarised light



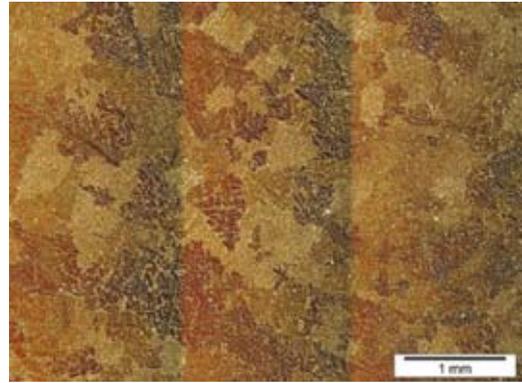
(a) Quick cup (6.3 K/s)



(b) Cone probe – C (19 K/s)



(c) Cone probe – B (38 K/s)



(d) Cone probe – A (176 K/s)

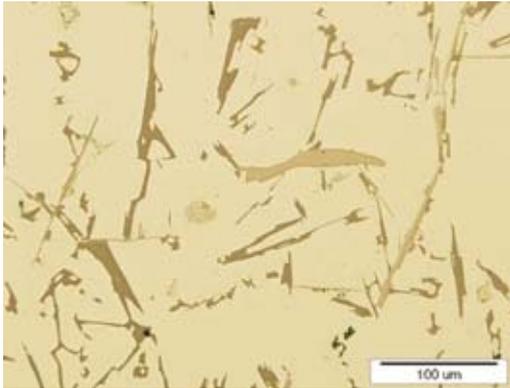
Figure 9. Microstructure of the specimen Vz.11 (grain refined with Ti, modified with Na) in the polarised light

The lengths of the silicon particles were determined too. The largest particles were in the unmodified alloy (sample Vz.1). At cooling rate of 6.7 K/s the average length was 15.644 μm . Particle length is also decreasing with the higher cooling rate and it reached 4.951 μm at the highest one. The length of the particles of the sample of Vz.4 modified with Sr became shorter and rounder. At the

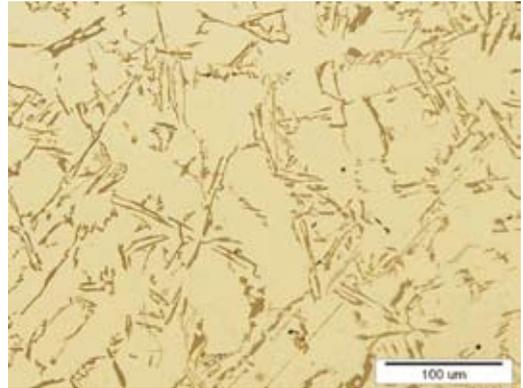
lowest cooling rate the length is 4.959 μm and at highest one it is only 0.507 μm . In the sample modified with Na the results are the best. Particles are only 1.190 μm long at the lowest cooling rate and 0.578 μm at the highest cooling rate. Table 6 is showing average lengths of the particles and ratios between length and width. Microstructures are presented in Figures 10, 11 and 12.

Table 6. The silicon particle lengths and ratios for all the specimens on the different measuring spots

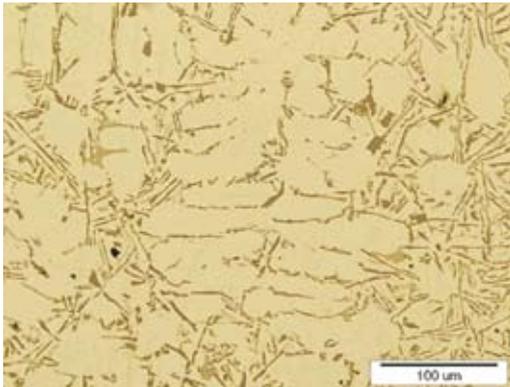
Sample	Vz.1		Vz.4		Vz.11	
	l [μm]	l/d	l [μm]	l/d	l [μm]	l/d
Quick cup	15.644	3.7	4.959	3.6	1 190	1.9
C	6.354	3.3	1.619	2.0	1.120	1.9
B	6.157	3.3	0.682	2.1	0.626	1.7
A	4.951	2.4	0.507	1.8	0.578	2.3



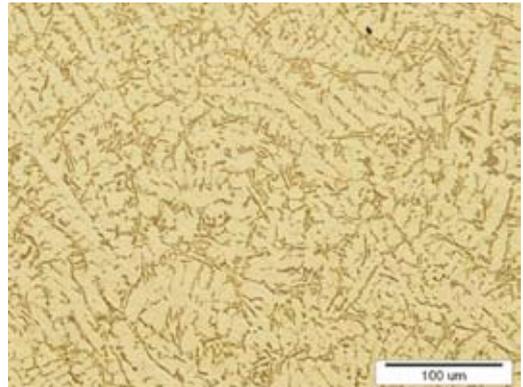
(a) Quick cup (6.3 K/s)



(b) Cone probe – C (19 K/s)



(c) Cone probe – B (38 K/s)



(d) Cone probe – A (176 K/s)

Figure 10. Microstructure of the sample Vz.1 (basic alloy)

As a result two diagrams are showing the relationship between largeness of the microstructural constituents and the cooling rate for the range of 6 K/s to 176 K/s. Figures 12 and 13 are showing relationship between largeness of the crystals and of the eutectic silicon particles as the function of the cooling

rate. We also calculated the physical model for the grain size reduction and the eutectic silicon particle reduction as the function of the cooling rate. Fitted exponential curves are also shown on Figures 13 and 14. Equations and parameters of the fitted curves are collected in Table 7.

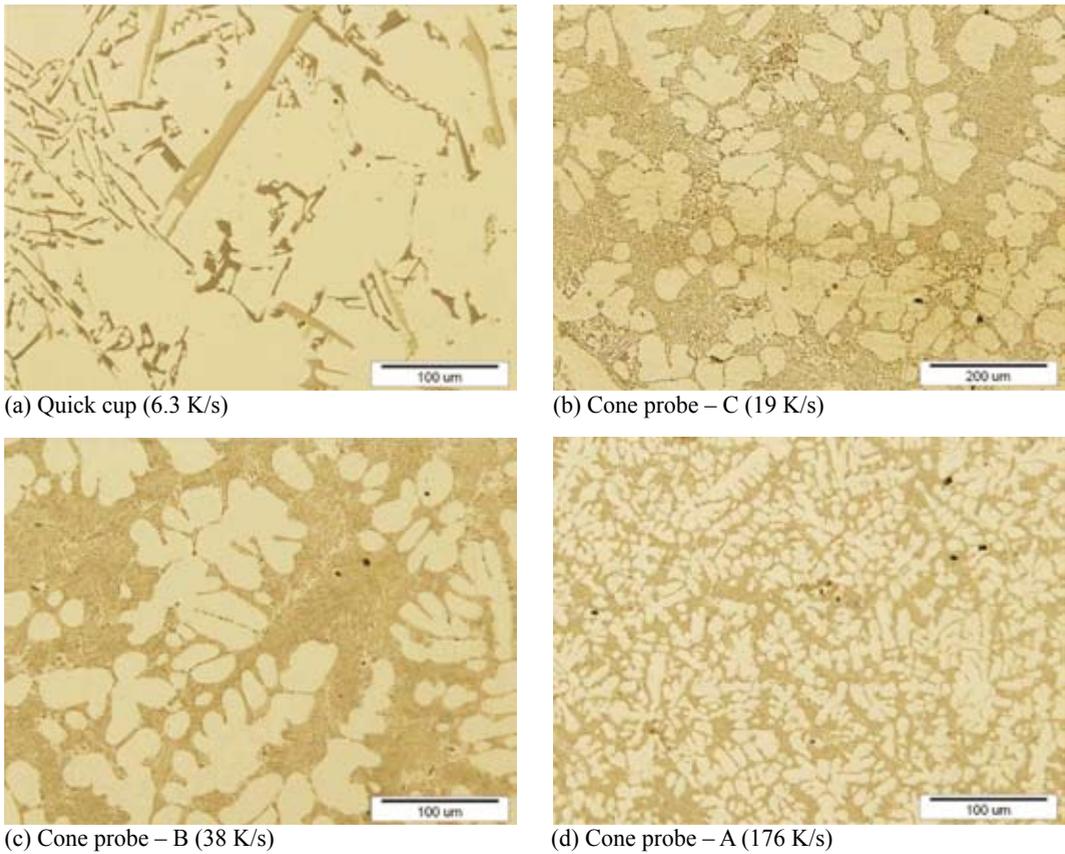


Figure 11. Microstructure of the specimen Vz.4 (grain refined with B, modified with Sr)

Table 7. Equations and parameters of the calculated physical models

Grain size	Model	R ²	y0	A1	t1
Vz.1	$y=A1\exp(-x/t1)+y0$	0.99999	307.41226 +/-4.21092	4080.72927 +/-40.58991	7.55081 +/-0.09232
Vz.4		0.97071	97.10749 +/-0.47917	142.64657 +/-4.3781	17.38183 +/-0.63481
Vz.11		0.97292	262.866 +/-1.97866	1079.5082 +/-24.6731	14.59254 +/-0.35247
Particle length					
Vz.1	$y=A1\exp(-x/t1)+y0$	0.99044	5.52465 +/-0.61155	34.20882 +/-19.42654	5.17144 +/-2.49776
Vz.4		0.99994	0.52289 +/-0.02266	8.84548 +/-0.13965	9.12575 +/-0.20775
Vz.11		0.86737	0.54676 +/-0.20373	0.89092 +/-0.37989	25.70911 +/-22.90963

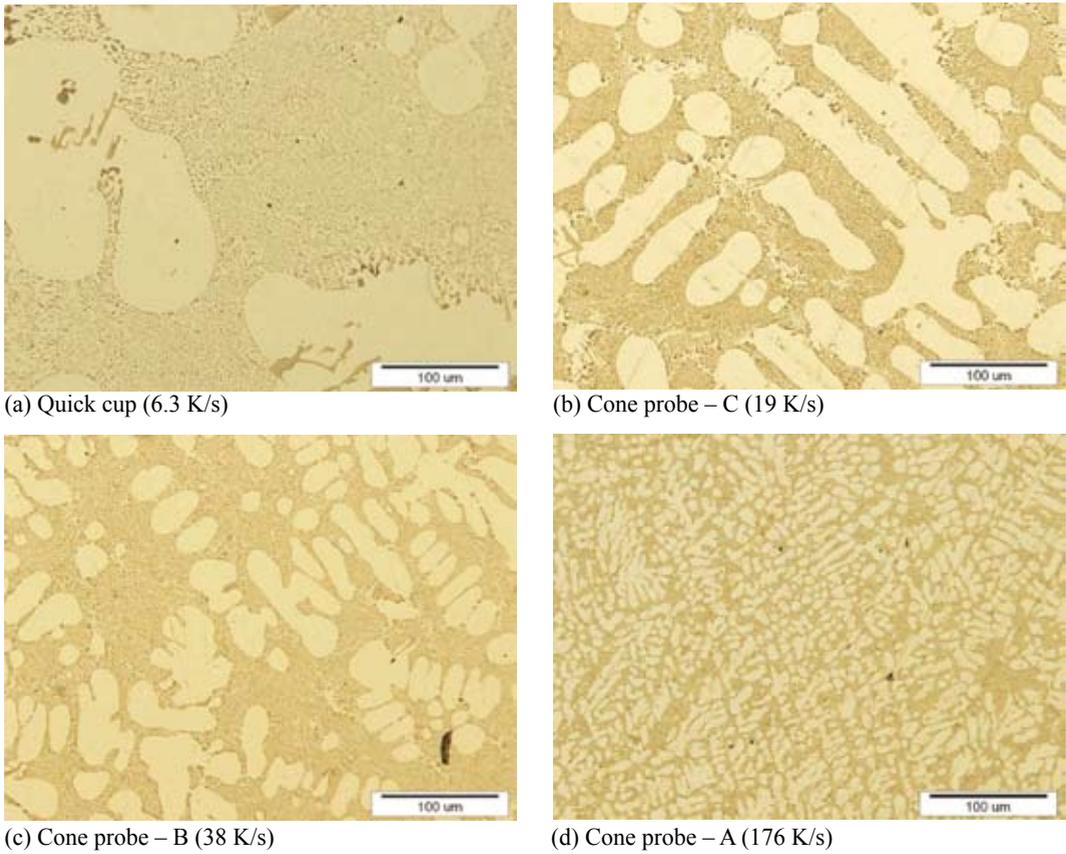


Figure 12. Microstructure of the specimen Vz.11 (grain refined with Ti, modified with Na)

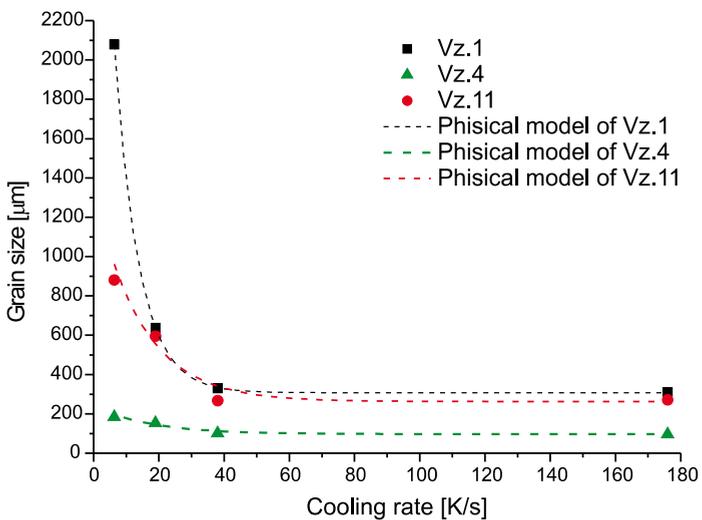


Figure 13. Grain size of the primary α_{Al} crystals as the function of the cooling rate at the solidification and the calculated physical models

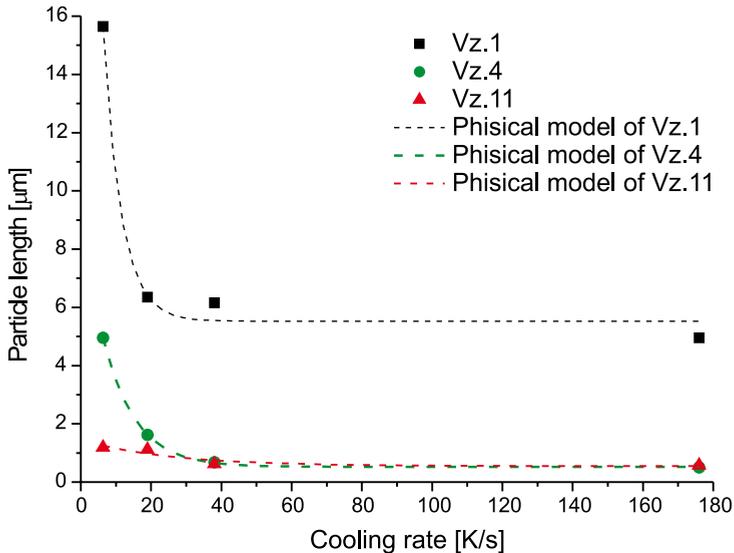


Figure 14. Silicon particle length as the function of the cooling rate and the calculated physical models

CONCLUSIONS

»In situ« thermal analysis is very good and simple tool to predict the microstructure of the Al-Si alloys. This method gives also the informations of the nucleating potential of an alloy and is measured from the liquidus and the solidus temperature on the cooling curve after the thermal analysis. Both criterions show the levels of the grain refinement and modification.

From measurement in quick cup measuring cell we determined liquidus temperatures and the recalescences at the primary solidification and the eutectic temperatures and the recalescences at eutectic solidification. Liquidus temperature in the specimen grain refined and modified with B and Sr is about 11 °C higher than in unmodified alloy and there is no recalescence. At specimen grain refined and modified with Ti and Sr the liquidus temperature is 5.5 °C higher than in unmodified

alloy and recalescence of 0.3 °C is occurring. Higher liquid temperature and the lower recalescence at the primary solidification is a good sign of well done grain refinement.

In modified alloys the eutectic temperature is usually lower and the higher recalescence is occurred. In the specimen grain refined and modified with B and Sr the eutectic temperature is 6 °C lower than in unmodified alloy and the recalescence is 2.8 °C. In the unmodified specimen the recalescence is only 0.5 °C. In the specimen grain refined and modified with Ti and Na the liquidus temperature is 7 °C lower and recalescence is 1.8 °C. These are also good signs of a well modified alloy.

These findings are also confirmed by the microstructural analysis. The biggest grain size is in the unmodified alloy. It is in range from 2.1 mm at lowest cooling rate to 330 µm at highest one. In the grain refined specimen

with B the grain size is from 185 μm to 96 μm and in the grain refined specimen with Ti the grain size is from 881 μm at lowest cooling rate to 271 μm at highest one.

Similar results are with length of the eutectic silicon. The length has shortened from 15 μm in the quick cup measuring cell of the unmodified alloy to 0.5 μm in the Sr modified alloy at highest cooling rate.

This research is applicable in industry at the high-pressure die-casting and at the gravity die-casting where the thickness of the castings is not unique and the cooling rates are different. In thick parts of the castings the largeness of the eutectic silicon particles and the grain size are bigger so it would be necessary to use the grain refinement and modification. From the calculated diagrams and the fitted physical models it is possible to determine the level of the grain refinement and modification.

POVZETEK

Vpliv udrobnevanja, modificiranja in ohlajevalne hitrosti na mikrostrukturo zlitine AlSi10Mg

Namen preiskave je bil ugotoviti učinkovitost različnih udrobnilnih sredstev in modificirnih sredstev za pridobitev finejših zmesnih kristalov α_{Al} in vlaknatega evtektkega silicija. Preiskave smo izvedli na podevtektski zlitini AlSi10Mg, pri čemer smo oplemenitenje oziroma modificiranje (udrobnitev zmesnih kristalov α_{Al} in modificiranje evtektika ($\alpha_{\text{Al}} + \beta_{\text{Si}}$) izvedli z različnimi udrobnilnimi in/ali modificirnimi sredstvi. Zlitino smo talili v indukcijski peči, nato pa

smo dodajali različne količine udrobnilnih in modificirnih sredstev.

Za oceno udrobnitve primarnih zmesnih zrn α_{Al} in modificiranja evtektkega silicija smo uporabili naslednje analize: enostavno termično analizo in metalografsko analizo, pri čemer smo vzorce opazovali v svetlem polju in polarizirani svetlobi.

Analizirali smo tri vzorce. Prvi je bil osnovna, neoplemenitena zlitina, drugi je bil modificiran z borom in stroncijem in tretji s titanom in natrijem.

Vzorce smo ulili v dve merilni celici. Prva je bila narejena po postopku Croning, druga pa je bila tako imenovana stožčasta proba iz sive litine. Med strjevanjem smo izvedli »in situ« enostavno termično analizo in iz zbranih podatkov dobili ohlajevalne krivulje in izračunali ohlajevalne hitrosti.

S pomočjo računalniške simulacije smo izračunali polnjenje livne votline stožčaste probe in strjevanje litine. Prav tako smo izračunali tudi ohlajevalne hitrosti na mestih meritve. Izračunani in eksperimentalno dobljeni podatki se dobro ujemajo. Izračunane ohlajevalne hitrosti so bile na mestu meritve A znašajo 176 K/s, na mestu B 38 K/s in na mestu C 19 K/s.

Pri opisu makro in mikrostrukture smo določili velikosti izločenih primarnih zmesnih kristalov α_{Al} in delcev evtektkega silicija. To smo izvedli s pomočjo svetlobnega mikroskopa in računalniškega programa *analySIS 5.0* z integriranim standardom ASTM E 112 – 96. Za določevanje velikosti primarnih zrn smo vzorce opazovali v polarizirani svetlobi, za določevanje ve-

likosti delcev evtektskega silicija pa smo vzorce opazovali v svetlem polju.

Iz podatkov smo izdelali dva diagrama, ki podajata odnos med ohlajevalno hitrostjo in velikostjo zmesnih kristalov in delcev evtektskega silicija. Na podlago teh podatkov in dobljenih krivulj smo izračunali še fizikalni model, ki podaja eksponentno padajočo odvisnost med velikostjo kristalnih zrn in delcev evtektskega silicija ter ohlajevalno hitrostjo. Iz njih je razvidno, da so v primeru neoplmenitene zlitine (vzorec Vz.1) pri počasnem ohlajanju (6,3 K/s), zrna v povprečju velika približno 2 mm. Pri zlitini udrobneni z B (vzorec Vz.4) so pri enaki ohlajevalni hitrosti zrna stokrat manjša in pri vzorcu udrobnenem s Ti (vzorec Vz.11) so zrna velika približno 880 μm . Z večjo ohlajevalno hitrostjo se velikost zrn v vseh primerih zmanjša, največji učinek pa ima leta pri osnovni zlitini. Pri največji ohlajevalni hitrosti (167 K/s) so ta velika cca. 310 μm , v vzorcu Vz.4 so velika samo 96 μm in v vzorcu Vz.11 le 270 μm . Iz teh podatkov je jasno, da je zelo dobro udrobnilno sredstvo Ti, prav tako tudi B, slaba stran slednjega pa je, da talina postane nagnjena k poroznosti. Učinek udrobnenja teh sredstev se še poveča z večjo ohlajevalno hitrostjo.

Na velikost in obliko izločanja evtektskega silicija prav tako vplivajo ohlajevalna hitrost in modificirna sredstva. Z večjo ohlajevalno hitrostjo in z dodatkom modificirnih sredstev se delci evtektskega silicija β_{Si} izločajo bolj fino in zaobljeno. Pri vzorcu osnovne, neoplemenitene zlitine (vzorec Vz.1), se pri počasnem ohlajanju lamele izločajo s povprečno dolžino cca. 15 μm in razmerjem med dolžino in širino 3,7. Pri najvišji ohlajevalni hitrosti pa je dolžina delcev 5 μm razmerje med dolžino in širino 2,4. Vzorec modificiran s Sr (vzorec Vz.4) ima dolžino lamel pri majhni ohlajevalni hitrosti cca. 5 μm in razmerje med dolžino in širino 3,6, pri najvišji ohlajevalni hitrosti pa 0,2 μm in razmerje 1,8. Pri vzorcu modificiranem z Na (vzorec Vz.11) so delci evtektskega silicija najmanjši pri vseh ohlajevalnih hitrostih. Delci so pri najmanjših ohlajevalnih hitrostih dolgi le 0,9 μm in razmerje med dolžino in širino 1,8. Pri velikih ohlajevalnih hitrostih pa delci dosežajo 0,2 μm razmerje pa se malce poveča zaradi prečne usmerjenosti vlaken evtektskega silicija na mestu meritve.

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