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Microstructure formation and grain refinement of AI-Mg-Si-Mn casting alloys

Kształtowanie mikrostruktury i rozdrobnienie ziaren odlewniczych stopów Al-Mg-Si-Mn

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Abstract

Together with development of casting technology for AI-Si-Mg alloys, new groups of casting materials are undergoes its implementation into foundry practice. Al-Mg-Si casting alloys possessed several advantages such as good strength in as-cast state combined with high ductility, good corrosion resistance and castability. In both Al-Si-Mg and Al-Mg-Si systems, the range of the eutectic crystallization occurs: $L \rightarrow \alpha_{Al} + \beta_{Si}$ and $L \rightarrow \alpha_{Al} + Mg_2Si$, respectively. In the hypoeutectic alloys of both system as a primary phase - dendrites of the solid solution $\alpha_{_{\!A\!I}}$ solidify. The transition elements - Ti, Zr, Sc, which provides efficient grain refinement can dissolve in this solid solution α_{a} causing precipitation strengthening effect. In the article the present state of the researches on the development of AI-Mq-Si casting alloys is considered together with the results of the examinations on the effect of Ti addition on the microstructure of the AIMg5Si2Mn alloy. These researches results were discussed at the annual conference on the casting of non-ferrous metals "Science and Technology" (2018) and initially presented in an shortened form in the article [1].

<u>Keywords</u>: aluminium alloys, Al-Mg-Si system, alloying addition, grain refiners, eutectic modifiers, precipitation, casting

Streszczenie

Wraz z rozwojem technologii odlewniczych stopów Al-Si-Mg, także kolejne grupy materiałów znajdują zasto-

sowanie w odlewnictwie. Stopy odlewnicze Al-Mg-Si charakteryzują się korzystnymi właściwościami nie tylko technologicznymi, jak np. dobra lejność, ale także odpowiednio wysoką wytrzymałością i plastycznością w stanie lanym czy też odpornością na korozję. W układach równowagi Al-Si-Mg i Al-Mg-Si występują odpowiednio obszary krystalizacji eutektycznej $L \rightarrow \alpha_{a_l} + \beta_{s_i}$ oraz $L \rightarrow \alpha_{a_l} + Mg_2Si$. W stopach podeutektycznych metale przejściowe, takie jak: Ti, Zr, Sc, dodawane przede wszystkim w celu rozdrobnienia pierwotnej struktury ziarn dendrytycznych, mogą rozpuszczać się w pierwotnej fazie α_{a_i} i powodować jej umocnienie wydzieleniowe. W pracy został omówiony aktualny stan badań nad odlewniczymi stopami Al-Mg-Si. Przedstawiono wyniki badań własnych dotyczących wpływu dodatku Ti na mikrostrukturę stopu AIMg5Si2Mn, które były dyskutowane w ramach konferencji "Science and Technology" (2018) oraz wstępnie omówione w publikacji [1].

<u>Słowa kluczowe</u>: stopy aluminium, Al-Mg-Si układ równowagi, dodatki stopowe, modyfikatory eutektyki, wydzielanie, odlewanie

1. Introduction

1.1. Historical remarks

The aluminium alloys belong to a group of casting materials that are in tonnage terms the second most popular after ferrous castings. The dominant group of Al-Si foundry alloys contains between 5 and 25 wt.%

Si, with Cu and Mg additions. Considerable advantages of this system are the relatively high strength (ultimate tensile strength up to 350 MPa in T6 condition), good corrosion resistance together with good castability [2].

In the car body structure, both wrought and cast aluminium alloys are essential for aluminium-intensive passenger cars. Their importance has abruptly increased simultaneously with the increase in the number of electric vehicles. The most common wrought aluminium alloys are based on the AI-Mg-Si system (6xxx) and in T6 condition they possessed the mechanical properties in the range of 120 to 240 MPa of yield strength (YS), 270–320 MPa of ultimate tensile strength (UTS), and 10–30% of elongation [2].

In order to maximize the benefits of aluminium-intensive car body structure, the die castings need to have comparable mechanical properties with the components made with aluminium sheet. However, the mechanical properties of currently available die casting alloys are not competitive and cannot satisfy the industrial requirement. In particular, the ductility is not sufficient in manufacturing and in application. Therefore, the cast alloy needs to be specially developed for car body structure and similar applications where elongation is one of the crucial characteristic.

Over the past 20 years, several new Al-based foundry alloys have been developed where composition and, subsequently, phase equilibria shifted from Al-Si-Mg to the Mg-rich part of the Al-Mg-Si phase diagram.

The main similarities and differences in the chemical composition of the alloys of both systems are shown in Figure 1.

The comparison of typical microstructures of Al-Si-Mg-Mn and Al-Mg-Si-Mn alloys in the initial state and after the modification of the eutectic are presented in Figure 2.

Similarly to Al-Si-Mg casting alloys, the alloys of the Al-Mg-Si system demonstrate the strong sensitivity to the cooling rate (Fig. 3).

Several alloys with nominal composition AlMg3-5Si1-2-Mn0.6 were developed and successfully introduced into the foundry practice [4]. The properties of a die casting alloy depend firstly on its composition. Currently, the die casting aluminium alloys include Al-Si, Al-Si-Cu and Al-Mg, Al-Mg-Si alloys. Al-Si and Al-Si-Cu (AlSi9Cu3) alloys are the most popular die casting alloys that offer a good combination of strength, castability and processability, but less ductility.

Al-Mg-Si alloy can provide a much higher ductility. The specification of available commercial Al-Mg-Si die casting alloys varies in different countries and manufacturers. The information on Al-Mg-Si casting alloys collected from the internet pages of different manufacturers is summarised in Table 1 in comparison to the grade designation of these alloys in different countries which is represented in Table 2. The alloys provide mechanical properties of 90–120 MPa of YS, 170–320 MPa of UTS and 3–18% of elongation and strongly depend on the alloying additions and applied casting techniques.

One of the first attempts to characterize the structure and elements distribution in Al-Mg-Si casting alloy such as AIMg5Si (Hydronalium 511) casting alloy was performed by Zalar and Pirš [6]. They subjected alloy having nominal composition AIMg5Si used for the production of the 6-stroke air-cooled Diesel engine to the EDX and WDX analysis. Specimens were cast into the permanent mold (PM) and heat treated. It has been found that the alloy with highest Mg content was characterized by highest mechanical properties. Addition of 5.58 wt. % Mg caused an increase of the tensile strength up to the 298 MPa, in comparison to alloy containing 4.51 wt.% Mg of tensile strength 268 MPa. Results of EDX microanalysis shown that the $\alpha_{\mbox{\tiny Al}}$ matrix was saturated mostly with Mg while Si was built in Mg₂Si intermetallics. It is worth noting that the Ti content (0.4 wt.%) in the alloy was much higher than that of peritectic composition (0.15 wt. % Ti). Therefore, the primary Al, Ti properitectic phase should form at the beginning of solidification and these crystals should be observed in the field of $\alpha_{_{\rm AI}}$ matrix. However, authors [6] do not observe such type of primary Al₃Ti crystals. Based on the results of Auger spectroscopy concluded that the Ti bound to oxygen.

Five years later in 1996 on TMS Annual meeting researchers from Aluminium Rheinfelden GmbH (Germany) reported the development of a new Al-Mg-Si alloy with the nominal composition already mentioned above and represented it under brand name Magsimal-59. According to the concept of alloy design [7], the main alloying elements are magnesium, silicon, and manganese. The Mg/Si ratio is also important in obtaining the desired 40–50% volume fraction of eutectic (α_{a_1} + Mg₂Si). This, in turn, favors sufficient castability and feeding during solidification. The free silicon crystals cannot be present in the microstructure to provide outstanding corrosion behavior. The last requirement is somewhat controversial because in absence of silicon, there were no building material for precipitates to form and, subsequently, matrix will be strengthened only by solid solution mechanism.

According to the investigation of Martin Hartlieb [8], who interviewed more than 150 specialists in HPDC in America and Canada in 2013 Magsimal-59, ranked 3 in terms of brand recognition among the o nline respondents with 20%, although it is scarcely used in North America (unlike Europe). AuralTM – $\frac{2}{3}$ ranked 4 with only 17.5%. The Japanese ADC3SF alloy jointly developed by Ryobi Ltd. and Honda Motor Co. was only recognized by 8.5% of the respondents and it is mainly used in Japan and Japanese companies such as Ryobi in North America. Results questionnaire clearly specified that the industry knowledge about Al-Mg-Si alloy group is rather limited. Following the success of Magsimal-59, every large manufacturer



^{*)}Both systems find application in all casting methods. ^{**)}Pabel T., Schumacher P.: Method for the refining and structure modification of Al-Mg-Si alloys. US Patent 9,279,170 B2, March 8, 2016.

Fig. 1. General characteristics of chemical composition of casting alloys of Al-Si-Mg and Al-Mg-Si systems Rys. 1. Ogólna charakterystyka składu chemicznego odlewniczych stopów układu Al-Si-Mg i Al-Mg-Si



Fig. 2. Microstructure of AI-Si-Mg-Mn and AI-Mg-Si-Mn alloys in the initial state and after chemical modification of eutectic (developed based on: www.alurheinfelden.com)

Rys. 2. Mikrostruktura stopów Al-Si-Mg-Mn i Al-Mg-Si-Mn w stanie wyjściowym oraz po chemicznej modyfikacji eutektyki (opracowana na podstawie: www.alurheinfelden.com)



Fig. 3. Microstructures AIMg5.5Si2.3Mn0.6 alloy (Magsimal-59) solidified at a different cooling rate [3]

Rys. 3. Mikrostruktury stopu AIMg5,5Si2,3Mn0,6 (Magsimal-59) krzepnącego z różną szybkością [3]

 Table 1. Chemical composition of commercial AI-Mg-Si-Mn casting alloys used by different manufacturers

 Tabela 1. Skład chemiczny przemysłowych stopów odlewniczych AI-Mg-Si-Mn stosowanych przez różnych producentów

Manufacturer/	Elements content, wt.% (Al-balance) / Zawartość pierwiastków, % wag. (pozostałe Al)							
Producent	Si	Mg	Fe	Mn	Cu	Ti	Other/Inne	
Salzburger Aluminium Group	1.80-2.60	5.00-6.00	0.45-0.90	0.50-0.80	0.08	0.05-0.15	Be	
Salzburger Aluminium Group	2.20-3.00	5.60-6.30	0.20	0.60-0.80	0.02	0.05-0.15	Cr, Be, RE*	
Salzburger Aluminium Group	8.50-9.50	0.30-0.50	0.15	0.10	0.01	0.15	Zn 9.00- 10.00, Be	
Aluminium Rheinfelden GmbH	1.80-2.60	5.00-6.00	0.20	0.50-0.80	0.03	0.20	Be	
Aluminium Rheinfelden GmbH	0.15	0.90-1.40	0.10-0.40	0.90-1.40	-	0.20	Со	
Rio Tinto Alcan	1.80-2.20	4.00-5.00	0.15-0.22	0.50-0.60	0.03	0.04-0.08	-	
Hydro Aluminium Metal Products	0.20-2.00	1.00-5.00	-	0.70-1.30	_	-	-	
	Manufacturer/ Producent Salzburger Aluminium Group Salzburger Aluminium Group Salzburger Aluminium Group Aluminium Rheinfelden GmbH Aluminium Rheinfelden GmbH Rio Tinto Alcan Hydro Aluminium Metal Products	Manufacturer/ ProducentSiSalzburger Aluminium Group1.80-2.60Salzburger Aluminium Group2.20-3.00Salzburger Aluminium Group8.50-9.50Aluminium Group1.80-2.60Aluminium Rheinfelden GmbH0.15Rio Tinto Alcan1.80-2.20Hydro Aluminium Metal Products0.20-2.00	Manufacturer/ ProducentEl ZawartProducentSiMgSalzburger Aluminium Group1.80–2.605.00–6.00Salzburger Aluminium Group2.20–3.005.60–6.30Salzburger Aluminium Group8.50–9.500.30–0.50Aluminium Group Aluminium Group1.80–2.605.00–6.00Aluminium Group Aluminium Rheinfelden GmbH0.150.90–1.40Rio Tinto Alcan1.80–2.204.00–5.00Hydro Aluminium Metal Products0.20–2.001.00–5.00	Manufacturer/ ProducentElements con Zawartość pierwiaSalzburger Aluminium Group1.80-2.605.00-6.000.45-0.90Salzburger Aluminium Group2.20-3.005.60-6.300.20Salzburger Aluminium Group8.50-9.500.30-0.500.15Aluminium Group Aluminium Group1.80-2.605.00-6.000.20Salzburger Aluminium Group0.150.30-0.500.15Aluminium Group Aluminium Rheinfelden GmbH1.80-2.605.00-6.000.20Rio Tinto Alcan1.80-2.204.00-5.000.15-0.22Hydro Aluminium Metal Products0.20-2.001.00-5.00-	Manufacturer/ Producent Elements content, wt.% (Zawartość pierwiastków, % w Si Mg Fe Mn Salzburger Aluminium Group 1.80-2.60 5.00-6.00 0.45-0.90 0.50-0.80 Salzburger Aluminium Group 2.20-3.00 5.60-6.30 0.20 0.60-0.80 Salzburger Aluminium Group 8.50-9.50 0.30-0.50 0.15 0.10 Aluminium Group 1.80-2.60 5.00-6.00 0.20 0.50-0.80 Manuinium Group 0.15 0.30-0.50 0.15 0.10 Aluminium Group 1.80-2.60 5.00-6.00 0.20 0.50-0.80 Aluminium Rheinfelden GmbH 0.15 0.90-1.40 0.10-0.40 0.90-1.40 Rio Tinto Alcan 1.80-2.20 4.00-5.00 0.15-0.22 0.50-0.60 Hydro Aluminium Metal Products 0.20-2.00 1.00-5.00 - 0.70-1.30	Manufacturer/ Producent Elements content, wt.% (Al-balance Zawartość pierwiastków, % wag. (pozos Si Mg Fe Mn Cu Salzburger Aluminium Group 1.80-2.60 5.00-6.00 0.45-0.90 0.50-0.80 0.08 Salzburger Aluminium Group 2.20-3.00 5.60-6.30 0.20 0.60-0.80 0.02 Salzburger Aluminium Group 8.50-9.50 0.30-0.50 0.15 0.10 0.01 Manufacturger 8.50-9.50 0.30-0.50 0.15 0.10 0.01 Aluminium Group 1.80-2.60 5.00-6.00 0.20 0.50-0.80 0.03 Aluminium Rheinfelden GmbH 1.80-2.20 5.00-6.00 0.20 0.50-0.80 0.03 Aluminium Rheinfelden GmbH 0.15 0.90-1.40 0.10-0.40 0.90-1.40 - Rio Tinto Alcan 1.80-2.20 4.00-5.00 0.15-0.22 0.50-0.60 0.03 Hydro Aluminium Metal Products 0.20-2.00 1.00-5.00 - 0.70-1.30 -	Manufacturer/ Producent Si Mg Fe Mn Cu Ti Salzburger Aluminium Group 1.80-2.60 5.00-6.00 0.45-0.90 0.50-0.80 0.08 0.05-0.15 Salzburger Aluminium Group 2.20-3.00 5.60-6.30 0.20 0.60-0.80 0.02 0.05-0.15 Salzburger Aluminium Group 8.50-9.50 0.30-0.50 0.15 0.10 0.01 0.15 Manufacturger 8.50-9.50 0.30-0.50 0.15 0.10 0.01 0.15 Aluminium Group 1.80-2.60 5.00-6.00 0.20 0.50-0.80 0.03 0.20 Aluminium Rheinfelden GmbH 1.80-2.60 5.00-6.00 0.20 0.50-0.80 0.03 0.20 Rio Tinto Alcan 1.80-2.20 4.00-5.00 0.15-0.22 0.50-0.60 0.03 0.04-0.08 Hydro Aluminium Metal Products 0.20-2.00 1.00-5.00 - 0.70-1.30 - -	

Table 2. Chemical composition of Al-Mg-Si-Mn-type casting alloys designed in different countries [5]

 Tabela 2. Skład chemiczny stopów typu Al-Mg-Si-Mn opracowanych w różnych krajach [5]

Country/Kraj	Allov/Stop	Element's content, wt.% (Al-balance) / Zawartość pierwiastków, % wag. (pozostałe Al)						
	, mogretop	Si	Mg	Mn	Fe	Cu	Zn	Ti
UK (BS1490)	LM5	0.30	3.00-6.00	0.30-0.70	0.80	0.10	0.10	0.05
China (GB/15115)	YL302	0.80-1.30	4.50-5.50	0.10-0.40	≤1.20	≤0.10	≤0.20	≤0.20
Germany (DIN)	GK-AlMg5Si	0.90-1.50	4.60-5.50	≤0.40	0.40	0.03	0.10	0.001
France (NFA57-105)	AG66	0.40	5.00-7.00	0.50	0.50	0.10	0.20	0.20
Russia (FOCT 1583-93)	AMr4K1,5M	1.30-1.70	4.50-5.20	0.60-0.90	0.40	0.70-1.00	-	0.1-0.25
Japan (JIS H2212)	ADC6	≤1.00	2.60-4.00	0.40-0.60	≤0.60	≤0.10	≤0.40	-
USA (ASTM B179)	516.0	0.30-1.50	2.50-4.00	0.15-0.40	0.35-1.00	0.30	0.20	0.10-0.20
Sweden	4163	0.50-1.50	4.00-6.00	0.50	0.50	0.10	0.20	0.20

		,		
Phase/Faza	Density, 10 ³ kg m ⁻³ / Gęstość, 10 ³ kg m ⁻³	TEC, 10 ⁻⁶ K ⁻¹ / α, 10 ⁻⁶ K ⁻¹	Elastic modulus, GPa / Moduł sprężystości, GPa	Melting point, °C / Punkt topnienia, °C
Mg ₂ Si	1.88	7.5	120	1085
Si	2.33	3.6	112	1411
				*

Table 3. Physical and mechanical properties of Mg₂Si and Si [12] Tabela 3. Fizykomechaniczne właściwości Mg₂Si i Si [12]

TEC – Thermal Expansivity Coefficient / α – współczynnik rozszerzalności cieplnej

of aluminium castings worldwide designed at least one casting alloy with a similar composition containing five to six weight percent Mg, two percent of Si, and manganese. In Table 1, the average composition of the recently developed by different manufacturers Al-Mg-Si casting alloys is represented.

In the most general sense, the attractiveness of Al-Mg-Si casting alloys can be summarized as follows:

- High mechanical properties. It was established a) by different researchers that the ultimate tensile strength (UTS) and yield strength (YS) of the AIMg5Si2Mn alloy subjected to high pressure die casting strongly depend on the wall thickness. For 2-3 mm wall thickness UTS can rich 330 MPa and YS 220. Further enlargement of wall thickness results in drastic decreases of both characteristics [5,7]. Similar behaviour has been observed for elongation to fracture. Maximum elongation was observed in castings having the wall thickness of about 3 mm (18%) and it falls down to 8% with the wall thickness of 12 mm. The mechanical properties also depend on the Mg content in the alloy. Highest UTS and YS were detected for the alloy containing 5.5 wt. % Mg [5]. It was also found that the UTS and YS vary with applied casting techniques. Highest values can be achieved for HPDC and lowest for permanent mould casting (PM).
- b) **Good fluidity**, which provides the ability to fill the thinnest sections of a mold cavity. According to Di Sabatino et al., the length of the spiral obtained by pouring the AIMg5Si2Mn alloy into a sand mould was 540 ±30 mm [9]. This is due to the presence of the (α_{AI} + Mg₂Si) eutectic.
- c) **Good feeding behavior**, because of the relatively high fraction of eutectic (α_{AI} + Mg₂Si). According to [12], the eutectic volume fraction in the AIMg5Si2Mn alloy is about 30%. Based on Thermo-Calc calculations, Otarawanna et al. [10] reported that the fraction of the eutectic is 38 wt.%.
- d) **Good corrosion resistance** and stress corrosion cracking [11].

In parallel to the advances in understanding of mechanical behavior of alloys subjected to HPDC Al-Mg-Si system is considered as a base to design *in situ* Al-Mg₂Si composites for different types of casting techniques. According to Georgatis et al. [12] Al-Mg₂Si composites constitute a new category of superlight materials attracting significant interest for potential aerospace, automotive, and other applications. The Mg₂Si intermetallic with its low density, high melting point, and high Young modulus (Table 3) allow expecting great advantages in the final material as an attractive substitute of conventional Al-Si alloys for automotive or other applications.

1.2. Effect of alloying elements

There were numerous experimental data related to the effect of alloying elements on the structure formation and mechanical properties of Al-Mg-Si casting alloy as well as *in situ* composites.

The major alloying elements in Al-Mg-Si alloys are Mg and Si. Investigation of Boyko V. et al. [13] showed that largest part of Si is bounded into Mg_2Si eutectic lamellas and few goes into AlFeMnSi primary intermetallic. Therefore, the Mg content in the alloy is the first factor affecting its mechanical properties.

Fan Z. et al. [14] reported that the maximum UTS has been obtained for an alloy containing as high as 8.8 wt. % Mg and it continuously rise with increasing Mg addition. However, the elongation of the alloy with such a high Mg content drastically decreases from 17 wt. % down to 8 wt.% in comparison to the alloy containing 5.5 wt.% Mg and 1.5 wt.% Si. Hence for HPDC, the optimum composition of an alloy is 5.0–5.5 wt.% Mg and 1.5–2.0 wt.% Si. Mn content has low effect on the mechanical properties and its addition is usually keeping on the level of 0.6 wt.%.

For AIMg5Si2Mn alloy subjected to HPDC it was found that the addition of Mn, Cu, and Zn leads to the considerable increasing of UTS up to 350 MPa after addition of 0.6 wt. % Mn [14]. The significant decrease of elongation was observed (down to 2.0%) while UTS was maintained on the level of 350 MPa. Alloying the Al-Mg-Si alloy with Zn promotes the formation of primary Zn containing intermetallic phase identified as Al₁₃Mg₅Zn. Its particles were located at the boundaries of the α_{AI} solid solution and inside (α_{AI} + Mg₂Si) eutectic cells. It was also reported by Korzhova et al. [15] that the Zn addition to the alloy cast into the permanent mold (PM) after heat treatment caused some increase of mechanical properties. The authors explained it by hardening of α_{AI} solid solution with dispersive precipitates of η -MgZn₂. It has been also established that in the presence of 0.2 at.% Cu further increase in the mechanical properties took place.

SAG reported about design of the special alloy containing Cr, Be and rare-earth elements, which offers maximum strength and ductile yield properties already after casting, making heat treatment unnecessary (see Table 1). It was reported by Eigenfeld et al. [16] that the alloy AIMg3Si1 alloyed with (Sc + Zr) is suitable for application above 250°C as its mechanical properties are excellent.

Titanium is used in wrought aluminium alloys as a grain refiner. The experimental results represented by Fan et al. [17] confirm that Ti could significantly increase the elongation of the die casting Al-Mg-Si alloy at a very low level. The elongation was at a level of 11% at 0.015 wt.% Ti and it increased to 18% at 0.15 wt.% Ti. It is more significant because Ti could also increase the YS and the UTS. The increase in strength was up to 10% when Ti content increased to 0.2 wt.%. Therefore, Ti is an essential element for ductile die casting aluminium alloys. However, structural investigations were not performed and the role of Ti was not adequately explained.

Thus one of the aims of this work was to analyse and discuss the microstructural effect of Ti addition into Al-Mg-Si alloy.

2. Experimental procedure

Three alloys of chemical compositions presented in Table 4 (defined by X-ray fluorescence elemental analysis) were melted in the laboratory furnace. The first one denoted as B is the alloy containing nominally 5.5 wt.% Mg, 2.0 wt.% Si and 0.6 wt.% Mn, of 0.04 wt.%. Ti content, practically on the level of the impurity. Two alloys were melted with Ti content of 0.09 (denoted as T1) and 0.22 wt.% Ti (denoted as T2).

Alloys were prepared in an electric resistant furnace using graphite crucibles. As master alloys, AIMg50, AlSi25, AlMn26, AlTi6 and high purity aluminium (A99.98) were used. Pure aluminium was charged into preheated to 720°C crucible. After superheating up to 720°C, preheated to 350°C master alloys were added to the melt. After complete dissolution of the master alloys, the melt was heated up again to the 720 ±5°C and its surface was covered by flux powder. As a flux powder, carnallite KMgCl, 6(H,O) was used. Prior to addition, it was dried for 24 hours at 250°C. Between different castings, the flux powder was stored in the preheated furnace to avoid its interaction with air and moisturizing. Mg was added then in the following manner: pieces of Mg-containing master alloys were wrapped in aluminium foil and added in small increments by plunging them under the melt surface using a titanium instrument. After addition of Mg, argon fluxing was used to remove non-metallic impurities and soluble gases from the melt. Fluxing time was 10 min and kept constant for all melts. After fluxing the melt surface was skimmed to remove dross and after reaching the temperature of 700°C, the alloys were cast into a steel mold (see Fig. 4). The mold temperature was 25°C for all casting to produce similar cooling conditions for all alloys. Preliminary tests showed that such conditions result in a cooling rate of 2 K·s⁻¹ prior to solidification.

The obtained ingots were cut on two halves. One was used for macroetching and the other one for cutting the specimens for heat treatment and metallographic examinations.

The specimens for metallographic examinations were cut from the center of round part of the ingots and prepared using standard metallographic procedures. An optical microscope Zeiss Axioskop with a MR MC80 digital camera with the software program AxioVision Rel. 4.7 was used for metallographic analysis. The SEM's used in this work were Zeiss EVO and ULTRA. All microscopes were equipped with Energy Dispersive Spectrometry microanalyser (EDS). The quantitative SEM/EDX analysis was performed under 5 and 10 kV accelerating voltage.

Table 4.	Cł	nemica	l compositi	on of studi	ed alloys
Tabela	4.	Skład	chemiczny	badanych	stopów

Specimen/Próbka	Element content, wt.% / Zawartość pierwiastków, % wag.							
	AI	Si	Mn	Cu	Mg	Ti	Fe	
B (base alloy)	91.15	2.21	0.73	0.002	5.42	0.04	0.18	
T1	91.54	2.14	0.68	0.004	5.36	0.09	0.19	
T2	91.59	2.23	0.58	0.002	5.54	0.22	0.17	



Fig. 4. Mold after casting (a) and specimens obtained from alloys subjected to research program (b) Rys. 4. Forma odlewnicza po odlewaniu (a) oraz próbki otrzymane ze stopów doświadczalnych (b)

3. Results and discussion

The microstructure of the base alloy (B) and those after addition of Ti (T1 and T2), are shown in Figures 5–7, respectively. The alloy phase constituents can be clearly visible, namely:

- α_{AI} solid solution,
- Mg₂Si,
- Al(Mn,Fe)Si.

The α_{AI} solid solution solidified in form of a dendritic grains with well-developed primary and secondary dendrite arms. The lamellar (α_{AI} + Mg₂Si) eutectic was distributed in the interdendritic areas. In the alloy B the primary Mg₂Si crystals of a regular polyhedral shape were situated in the centres of eutectic colonies.

Microstructure of T2 specimen is presented in Figure 6. The morphology of the particle observed in the α_{AI} solid solution grain centre is very close to that typical for Al₃Ti crystals commonly identified in the grain refined by Ti aluminium alloys. EDX microanalysis results (Fig. 6d) confirm that this particle was enriched with Ti. The small content of V was detected too and it was ascribed to AITi6 master alloy. Thus, this particle could be identified as primary Al₃Ti, acting as the nucleus for α_{AI} solid solution grain.

Further examination of the microstructure showed that the similar particles are situated in the center of the Mg_2Si primary crystals. As can be seen from Figure 7 in the center of the primary crystal light grey particle is visible. The EDX microanalysis results revealed the presence of Ti in this particle (see Fig. 7b). Simultaneously with Ti in the analysed microregions the oxygen was also detected. However, it is not obvious that this

intensity rises from the complex compound containing Al, Ti, O_2 , and/or Mg and Si. The previous study of Al-Mg-Si casting alloys showed that their surface is very sensitive to oxidation during and after polishing, especially when oxide polishing suspension was applied.

Presence of Ti-containing particles in the centers of primary Mg₂Si crystals and α_{AI} solid solution grains displayed the role of Ti addition in the structure formation of Al-Mg-Si casting alloys. The Ti-rich particle inside the Mg₂Si crystals gives additional evidence of multistep nucleation hypothesis, which in this case can be described in the following manner. When the temperature of the melt is close to peritectic temperature for Al-Ti system (665°C) the primary Al₃Ti intermetallic particles are formed. Later in the temperature range between 620 and 595°C, primary Mg₂Si crystals start to form on the existing Al₃Ti/L interface. Simultaneously some fraction of the Al₃Ti particles is used as nuclei of the α_{AI} grains. At a temperature of 595°C (α_{AI} + Mg₂Si) eutectic starts to grow in the residual liquid at α_{AI}/L interface.

Together with the identified role of Ti as powerful grain refiner not only grains of a solid solution but primary Mg₂Si crystals, the negative side effects have to be taken into account (alloy T2, Fig. 8). Formation of the large Al₃Ti intermetallic particles, unevenly distributed, as it represented in Figure 8, do not play any role in the nucleation of both α -grains and primary Mg₂Si crystals. Presence of such crystals in alloy T2 shows that the addition of 0.22 wt.% Ti is too high and crosses the optimum level accepted for the practical purpose. Thus, to achieve a desired nucleating effect provided through the Ti addition in Al-Mg-Si alloy optimum addition level should be kept close to 0.15 wt.% mentioned previously for avoiding the formation of a very large primary intermetallic crystals.





Fig. 5. Microstructure of alloy B Rys. 5. Mikrostruktura stopu B









d)

Ti M

Fig. 6. Equiaxed grain of α_{AI} solid solution in alloy T2 (a), particles observed in the grain center (b), enlarged view of the particle in grain center (c) and mapping of Ti, V, Mg and Si concentration (d)

Rys. 6. Równoosiowe ziarna α_{AI} roztworu stałego w stopie T2 (a), cząstki obserwowane w centrum ziaren (b), powiększony widok cząstki w centrum ziarna (c) oraz mapy rozkładu stężenia dla Ti, V, Mg i Si (d)





Rys. 7. Morfologia pierwotnych wydzieleń Mg₂Si, cząsteczki zarodków wewnątrz Mg₂Si oraz rozkład stężenia Ti (zielony) wewnątrz kryształu Mg₂Si (brązowy) (b)



Fig. 8. Distribution and morphology of primary Al₃Ti particles observed in alloy T2 Rys. 8. Rozmieszczenia i morfologia pierwotnych cząsteczek Al₃Ti występujących w stopie T2

4. Summary

Obtained results can be summarized as follows:

- Addition of Ti to Al-Mg-Si alloys can produce double effect on the structure formation. During cooling the Al₃Ti particles are formed in the melt and act as nucleation particles for formation of solid solution grains, thus could act effectively as the grain refiner. Simultaneously these particles act as nucleation substrate for primary Mg₂Si crystals.
- Amount of Ti added to the Al-Mg-Si alloys should be optimized to avoid the formation of undesired large Al₃Ti crystals (<0.15 wt.%.) and, subsequently, to improve mechanical properties of alloys.

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