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Morphology prediction of intermetallics formed in 4xxx type of aluminium alloy

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Materials

ABSTRACT

Purpose: It is intention of this paper to present results of studies of AlSi5Cu2Mg cast alloy in T6 condition with emphasis on composition of intermetallic phases. It was revealed that the microstructure of the alloy is characterized by three different intermatallic phases. Their morphology depends on the base metal and crystallization parameters applied.
Design/methodology/approach: Microstructural examination was conducted for the specimens using optical metallography, scanning (SEM) and transmission (TEM) electron microscopy as a complement to light optical investigation. Differential scanning calorymetry was utilized to assist in intermetallics identification. Phase identification was done by electron diffraction spectrometry microanalyser EDS using X-ray mapping technique on representative area for the structure of the material and a quantitative analysis was conducted for the large particles, which are large enough in size for individual analysis.
Findings: Three types of intermetallic α–AlFeMnSi phase precipitates vary on shapes and chemical compositions were determined. Their morphology was mainly influenced by the parameters of crystallization process. The intermetallics can precipitate in form of needle or “Chinese script”. The shape of phases determined can also be modified by adjusted modifying heat treatment process of the alloy.
Research limitations/implications: It is recommended to perform further analysis of the alloy by using more sophisticated technique for micrsoctructure observation - TEM in order to clarify a formation model and provide characteristic of the refined phases. These dispersed, smaller in size intermetallics compared to these characterized in this paper, have a considerable impact on the mechanical properties of the alloy.
Practical implications: It is important to develop a. predictive understanding of intermetallics formation through crystallization and heat treatment process since they play the most significant role in development of final properties of a material designed for utilitarian application.
Originality/value: This work is a contribution to the understanding of precipitation hardening behavior of the intermetallic phases and clarifying their formation mechanism by determination of their chemistry and occurring sequence in the microstructure during crystallization and heat treatment processes.
Keywords: Metallic alloys; Electron microscopy; Microstructure; Intermetallic phase

1. Introduction

The progress of material engineering control the improvement of materials development for elements operating under severe working conditions. Improvements in efficiency through increased pressure and temperature have resulted in a more pressing need for improved materials. The need to develop light materials for manufacturing constructional elements for aircraft and helicopter application has been a matter of investigation during the last decade [1-13]. AlSi5Cu2Mg was among the candidate 4xxx group of casting alloys. These elements are usually made from this alloy as they face temperature exceeding

270°C, in some circumstances. Corrosion resistance, thermal conduction, low density, good castability and mechanical properties (relative mechanical strength R_m/δ in particular) are the most important properties, which fulfill the requirements of that sort of service conditions [1-7,13-22]. Practically all aluminium alloys used in practice depend on finely dispersed intermetallic particles for their strength. The major problem in developing a material with good mechanical properties and ductility is to predict morphology, relative volume and composition of the phase particles forming through casting and heat treatment process. The type of intermetallics present is also of importance. AlSi5Cu2Mg alloy seems to be a good candidate, meeting those requirements. This aluminium alloy possess a unique ability to develop high strength with relatively simple heat-treatments, without the loss of ductility and corrosion resistance associated with other aluminium alloys of comparable strength levels [8-13,18-22].

Coarse and medium in size particles of intermetallics have a considerable influence on the microstructure and consequently mechanical properties of commercial aluminium alloys. Dispersoids can influence the fracture behaviour, control grain size providing stability to the substructure and finally may limit strain localization during deformation. However, the coarse particles of intermetallic phases can affect recrystallization, surface and corrosion performance. Thus, particle characterization is important not only to decide what sort of processing courses should be applied, but also for designing optimized chemical composition of a material.

2. Materials and methodology

Table 1 shows the chemical analysis of the type of aluminium alloy used throughout this study. AlSi5CuMg casting alloy was delivered in the T6 condition - solution heat treated at 515°C for 5h followed by water cooling and artificially aging at 225°C for 5h followed by air cooling.

Table 1.
Composition of AlSi5Cu1Mg alloy (% mas.)

Si	Cu	Mn	Mg	Fe	Al
5	2.1	0.8	0.4	0.2	rest

Calorymetry analyses were performed using a Setaram Labsys model DSC unit by placing the sample disc in the sample pan and a super purity aluminum of equal mass in the reference pan of the cell. The cell was heated to 600°C at 4°C min in a dynamic argon atmosphere. The heat effects associated with precipitation/dissolution reactions were then obtained by subtracting a super purity Al baseline run from a given heat flow curve. The DSC curves thus obtained were highly reproducible. The observed signals in the DSC curve allow to perform heat treatment of the examined alloys. The alloy has been subjected to 20 minutes isothermal heating at the temperatures characteristic for the peaks noted during calorimetric analysis (518, 568, 580, 619 and 629°C) followed by quick water quench cooling.

The properties of the intermetallic phases were explained through observation of the microstructure using standard optical and advanced scanning electron microscopy technique. Metallographic microscope Nikon 300 has been utilized on the specimen polished and etched by the reagent of the following composition: 2ml HF + 3ml HCl + 20ml HNO_3 + 175ml H_2O).

Hitachi 3400 scanning electron microscope equipped with Thermo – Noran X-ray energy-dispersive spectroscopy (EDS) system operated at 20 kV was used.

3. Results and discussion

In this paper attempts were made to determine the nature, amount, location and composition of the intermetallic phases within the alloy. Characterization of the phases was initially made using optical microscopy.

Figs. 1 and 2. show the microstructure by optical metallography of the considered alloy in T6 state/as delivery condition. The structure consists of different precipitates showed variations in brightness and color. From Figs. 1-2, we can see that the precipitates present are: fine dispersed (I), rounded (II), rod-like (III) and "Chinese script" (IV). The characteristics of these phases are presented in the Table 2.

Fig. 3 shows the X-ray intensity distribution of elements; Al, Mn, Fe and Si chosen area for X-ray mapping, which is, at first approximation, proportional to the weight fraction.

a) b)

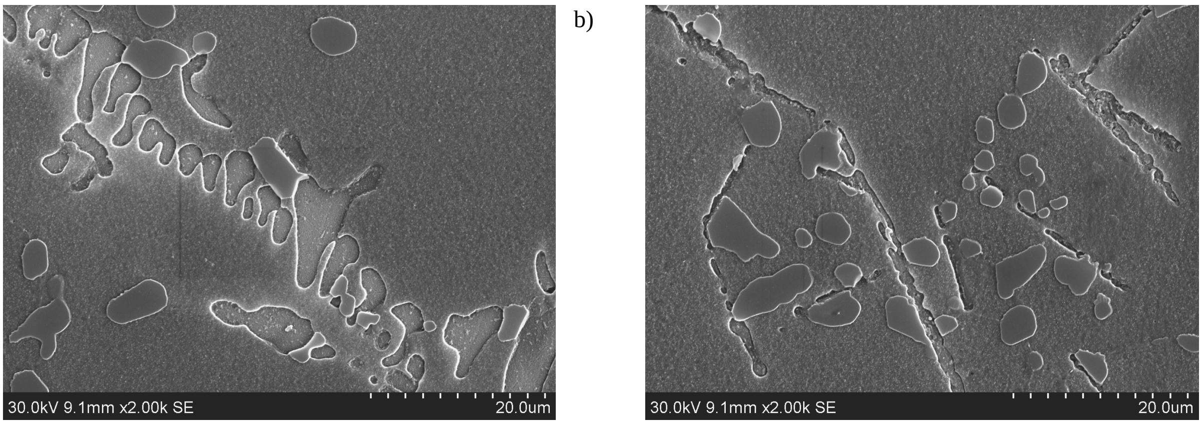


Fig. 1. Metallography pictures of the *AlSi5Cu2Mg* alloy in the T6 condition a) b) images from scanning electron microscope (SEM)

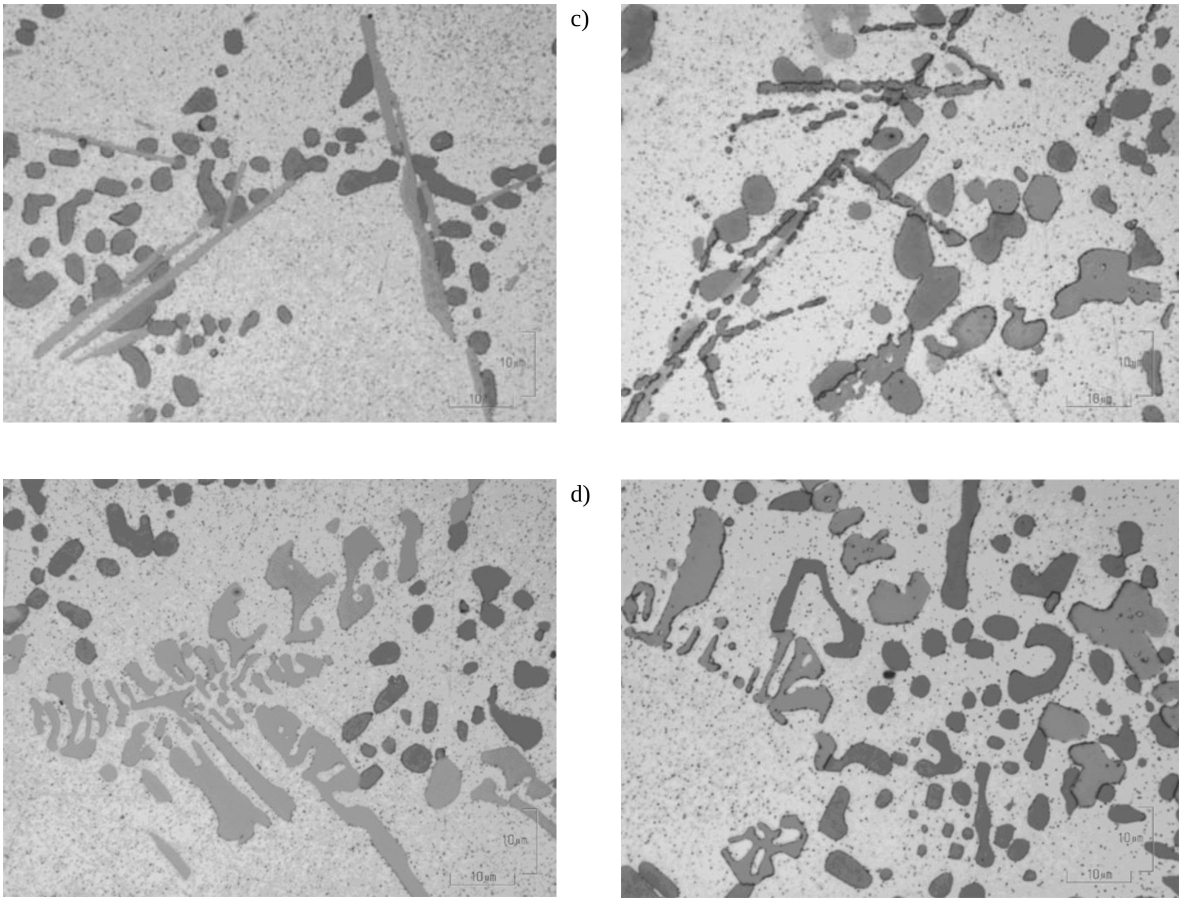


Fig. 2. Optical metallography pictures of the AlSi5Cu2Mg alloy in the T6 condition a, b) unetched; c, d) etched microstructure - images from light microscope (LM)

Table 2.
The characteristics of the phases in the AlSi5Cu2Mg alloy

The characteristics		The phase number			
		I	II	III	IV
Color	Unetched	-	Dark gray	Fair gray	Fair gray
	Etched	-	No changes observed	Well shaped edges, the color changes into dark gray	Well shaped edges, the color changes into dark gray
Shape			Rounded	Rod-like	„Chinese script”
Distribution		Uniformly in the whole alloy	In the interdendritic areas of the α-Al alloy	In the interdendritic areas of the α-Al alloy and forming clusters	In the interdendritic areas of the α-Al alloy

The results in Fig. 4 are displayed in colors to enhance the higher intensity values of given elements. Black represents the lowest concentration, while glaring colors represents the maximum concentration. The presence of Al, Fe and Mg is evidently highest in the matrix, while Si, Mn and Fe are at their maximum levels on the large "chinese script" shaped light precipitates.

The EDS analysis revealed, that rounded inclusions (II) are the eutectic silicon ones, whereas the rod-like (III) and “Chinese script” shaped (IV), are inclusions of the phase, consisting of *Al*, *Si*, *Mn* and *Fe* (Figs. 3-5 and Table 3).

Fig. 5 shows similar results of the surface distribution of the alloying elements taken from the other part of the analyzed sample.

Table 3.
The chemical composition if the intermetallic inclusions in the AlSi5Cu2Mg alloy

	Chemical composition (%wt)		
	II	III	IV
	Si 85-95.0	Si: 7.3-9.8 Mn: 13.5-21.5 Fe: 4.2-6.0	Si: 7.3-9.8 Mn: 13.5-21.5 Fe: 4.2-0.0
Phase	Si	α-AlFeMnSi	α-AlFeMnSi

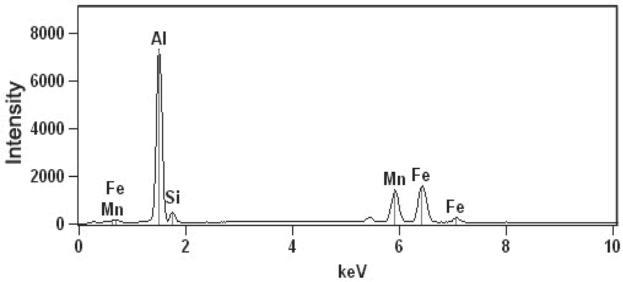


Fig. 3. EDS analysis of the area shown in Fig. 4

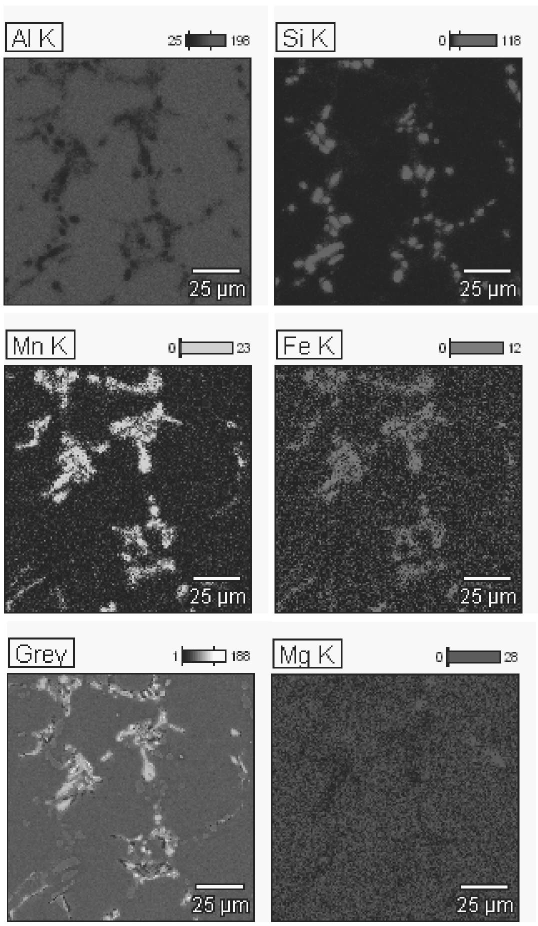
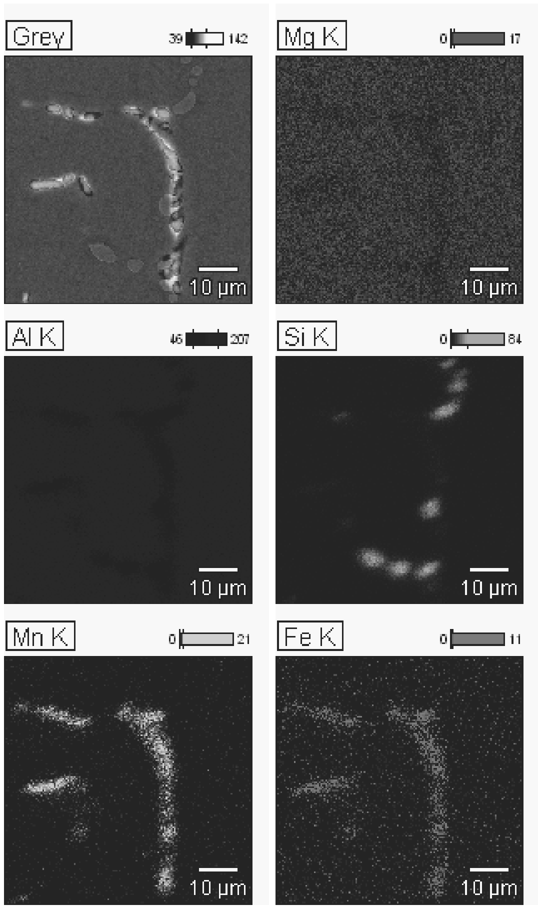


Fig. 4. The surface distribution of the alloying elements in the area of Si and “Chinese script” shaped inclusions occurrence

a)



b)

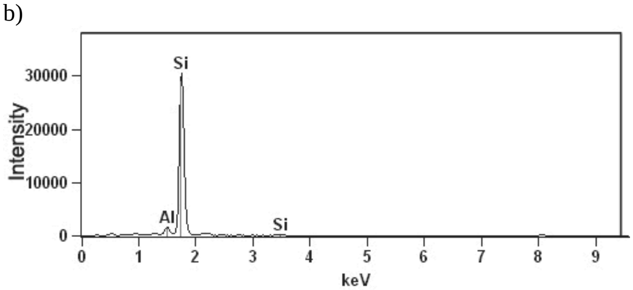


Fig. 5. a) The surface distribution of the alloying elements in the area of Si and “Chinese script” shaped inclusions occurrence; b) EDS X-ray spectra of the local analysis of rounded in shape particles

The DSC curve of *AiSi5Cu2Mg* alloy revealed two enthalpic effect (Fig. 5). Both of them were endothermic. In fact, one should expect a more peaks in the DSC thermogram because of many phases precipitates within the analyzed range of temperature. It shows an endothermic peak doublet with the first low temperature peak having the maximum at 561°C and the second large peak at 619°C. Since no extra endo peaks was observed in Fig. 6 it was decided to carry out an isothermal heating at the temperature of peaks and the other characteristic points observed in DSC curve with the intention of more accurate analysis of the transformation.

Examination of microstructure showed, that during 20 minutes annealing at the temperature 518°C, the rod-like shaped *α–AlFeMnSi* inclusions (III Table 2) begin to coagulate to form rounded particles preserving the same distribution (Figure 7a), whereas "Chinese script" shaped *α–AlFeMnSi* inclusions (IV Table 1) were partially melted (Fig. 7b).

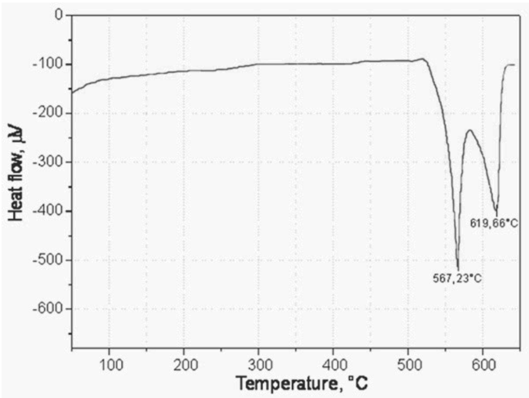
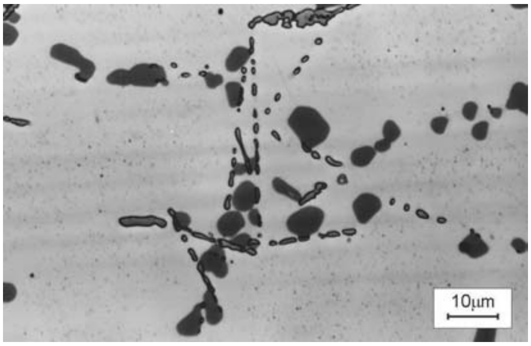


Fig.6. DSC curve of the AlSi5Cu2Mg alloy in the T6 conditions obtained by heating at the rate of 4°C/min to 625°C

a)



b)

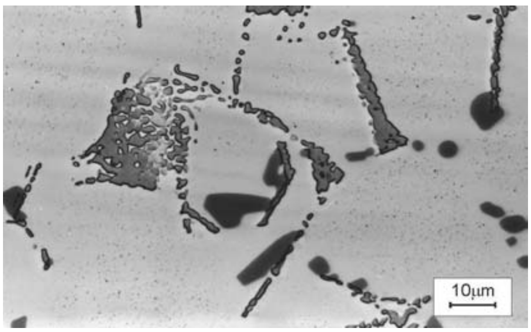
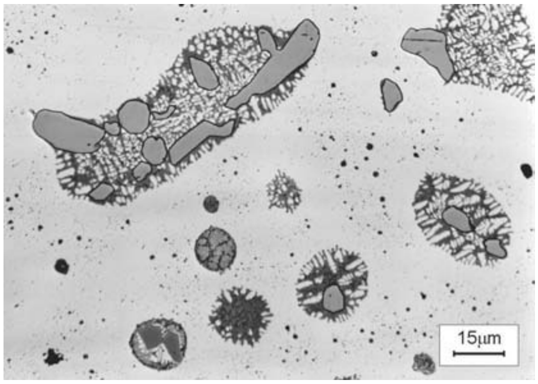


Fig. 7. The AlSi5Cu2Mg alloy microstructure after 20 minutes annealing at 518°C

Annealing at the temperature 568°C for 20 minutes caused the partial dissolution of both kinds of the *α–AlFeMnSi* phase, that is the rod-like (III) and "Chinese script" (IV), in the solid solution *α–Al*. During cooling, the *α–AlFeMnSi* (III) phase precipitated as a rounded microeutectic inside the *α–Al* dendrites and on their boundaries (Fig. 8a), whereas the *α–AlFeMnSi* (IV) phase precipitated in the interdendritic regions forming the microeutectic (Fig. 8b).

a)



b)

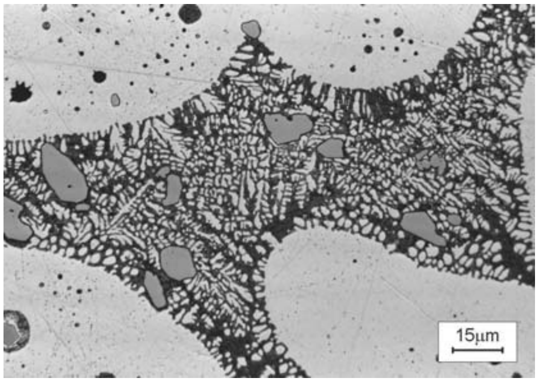


Fig.8. Optical micrograph of the AlSi5Cu2Mg alloy subjected to annealing at 568°C for 20 minutes

The complete dissolution of the *α–AlFeMnSi* (III and IV) phase was observed after 20 minutes annealing at higher temperature of 580°C. The rounded in shape microeutectics inside dendrites *α–Al* were not observed (fig.7a), but *α–AlFeMnSi* phase occurred as ultra fine eutectic in the interdendritic regions (Fig. 9b). Furthermore, the *α–Al* dendrites partial melting was observed (Fig. 9).

The examined aluminium alloy annealing at 619°C for 20 minutes resulted in the complete melting of the *α–AlFeMnSi* (III and IV) phase particles, followed by their precipitation as the microeutectic in the interdendritic areas of the α – Al solution.

The complete melting of the *α–AlFeMnSi* (III and IV) phase particles and the *α–Al* solution took place after 20 minutes annealing at 629°C. Subsequently, the fine, uniform dendritic structure of the *α–Al* solution was formed (Fig. 10).

a)

b)

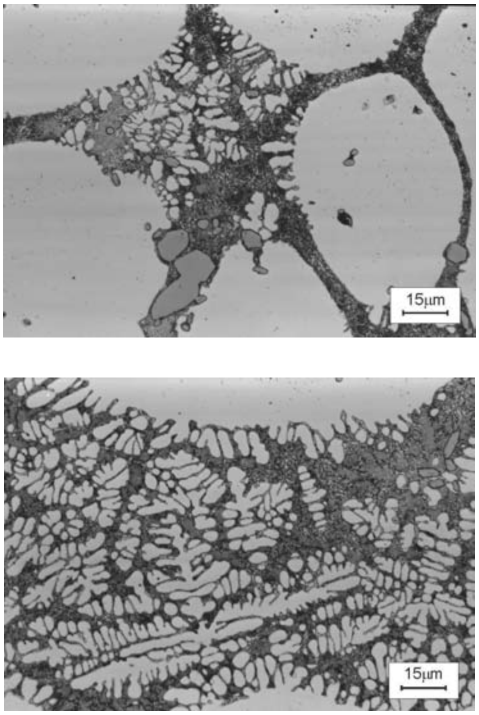


Fig. 9. The alloy microstructure after 20 min annealing at 580°C

a)

b)

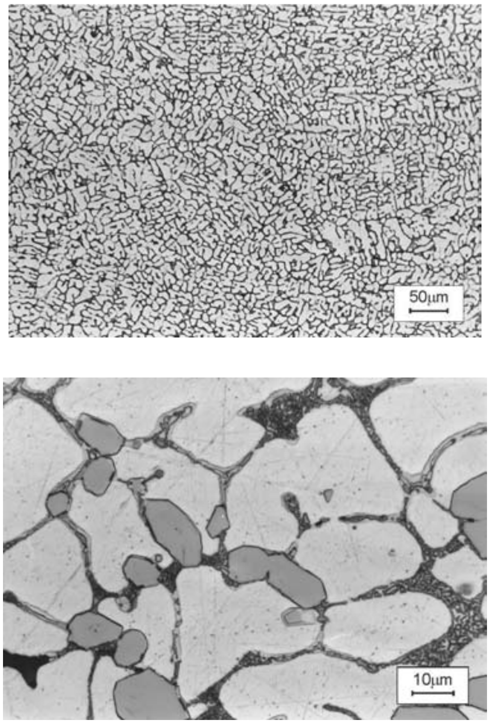


Fig. 10. The AlSi5Cu2Mg alloy microstructure after 20 minutes annealing at 629°C

The microeutectic of *α–Al* + *α–AlFeMnSi* (III and IV) and *α–Al* + *S*i came to existence in the interdendritic areas (Fig. 9b). The shape and morphology of the *Si* crystals remained the same in the whole range of the annealing temperatures (519–629°C), except the insignificant melting at the edges (Figs. 7-10).

4. Conclusions

The electron energy-dispersive X -ray spectrometry (EDS) analysis technique evidenced that *AlSi5Cu2Mg* alloy microstructure is composed of only one type of the intermetallic phase, the *α–AlFeMnSi* phase. Since the EDS method is considered as a basic one, the thorough investigation by the use of the X -ray method is carried on and the results will be published soon. Nonetheless, even the preliminary analysis showed, that the chemical composition, morphology, shape (rod-like or "Chinese script") and distribution of the intermetallic phase depend on the parameters of the heat treatment and crystallization process. Therefore, it is possible to control their morphology and what follows final properties of the alloy by modification of the crystallization and heat treatment parameters.

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