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Intermetallic Phases Examination in Cast AlSi5Cu1Mg and AlCu4Ni2Mg2 Aluminium Alloys in As-Cast and T6 Condition

Grażyna Mrówka-Nowotnik

*Rzeszów University of Technology, Department of Materials Science
Poland*

1. Introduction

Cast Al-Si-Cu-Mg and Al-Cu-Ni-Mg alloys have a widespread application, especially in the marine structures, automotive and aircraft industry due to their excellent properties. The main alloying elements – Si, Cu, Mg and Ni, partly dissolve in the primary α -Al matrix, and to some extent present in the form of intermetallic phases. A range of different intermetallic phases may form during solidification, depending upon the overall alloy composition and crystallization condition. Their relative volume fraction, chemical composition and morphology exert significant influence on a technological properties of the alloys (Mrówka-Nowotnik G., at al., 2005; Zajac S., at al., 2002; Warmuzek M., at al. 2003). Therefore the examination of microstructure of aluminium and its alloys is one of the principal means to evaluate the evolution of phases in the materials and final products in order to determine the effect of chemical composition, fabrication, heat treatments and deformation process on the final mechanical properties, and last but not least, to evaluate the effects of new procedures of their fabrication and analyze the cause of failures (Christian, 1995; Hatch, 1984; Karabay et al., 2004). Development of morphological structures that become apparent with the examination of aluminium alloys microstructure arise simultaneously with the freezing, homogenization, preheat, hot or cold reduction, annealing, solution and precipitation heat treatment of the aluminium alloys. Therefore, the identification of intermetallic phases in aluminium alloys is very important part of complex investigation. These phases are the consequence of equilibrium and nonequilibrium reactions occurred during casting of aluminium alloy. It worth to mention that good interpretation of microstructure relies on heaving a complete history of the samples for analysis.

Commercial aluminium alloys contains a number of second-phase particles, some of which are present because of deliberate alloying additions and others arising from common impurity elements and their interactions. Coarse intermetallic particles are formed during solidification - in the interdendritic regions, or whilst the alloy is at a relatively high temperature in the solid state, for example, during homogenization, solution treatment or recrystallization (Cabibbo at al., 2003; Gupta at al., 2001; Gustafsson at al., 1998; Griger at al., 1996; Polmear, 1995; Zhen at al., 1998). They usually contain Fe and other alloying elements

and/or impurities. In the aluminium alloys besides the alloying elements, transition metals such as Fe, Mn and Cr are always present. Even small amount of these impurities causes the formation of a new phase component. The exact composition of an alloy and the casting condition will directly influence the amount and type of intermetallic phases (Dobrzański at al., 2007; Warmuzek at al. 2004, Zajac at al., 2002). Depending on the composition, a material may contain CuAl_2 , Mg_2Si , CuMgAl_2 , and Si as well as Al(Fe,M)Si particles, where M denotes such elements as Mn, V, Cr, Mo, W or Cu. During homogenization or annealing, most of the as-cast soluble particles from the major alloying additions such as Mg, Si and Cu dissolve in the matrix and they form intermediate-sized 0.1 to 1 μm dispersoids of the AlCuMgSi type. Dispersoids can also result from the precipitation of Mn-, Cr-, or Zr-containing phases. A size and distribution of these various dispersoids depend on the time and temperature of the homogenization and/or annealing processes. Fine intermetallic particles ($<1 \mu\text{m}$) form during artificial aging of alloys and they are more uniformly distributed than constituent particles or dispersoids. Dimensions, shape and distribution of these particles may have also important influence on the ductility of the alloys. Therefore, a systematic research is necessary regarding their formation, structure and composition. For example, the coarse particles can have a significant influence on a recrystallization process, fracture, surface and corrosion, while the dispersoids control grain size and provide stability to the metallurgical structure. Dispersoids can also have a large affect on the fracture performance and may limit strain localization during deformation. The formation of particles drains solute from the matrix and, consequently, changes the mechanical properties of the material. This is particularly relevant to the heat-treatable alloys, where depletion in Cu, Mg, and Si can significantly change the metastable precipitation processes and age hardenability of the material (Garcia-Hinojosa at al., 2003; Gupta at al., 2001; Sato at al., 1985). Therefore, the particle characterization is essential not only for choosing the best processing routes, but also for designing the optimized alloy composition (Mrówka-Nowotnik at al., 2007; Wierzbińska at al., 2008, Zajac at al., 2002; Zhen at al., 1998).

The main objective of this study was to analyze a morphology and composition of the complex microstructure of intermetallic phases in AlSi5Cu1Mg and AlCu4Ni2Mg2 aluminium alloys in as-cast and T6 condition and recommend accordingly, the best experimental techniques for analysis of the intermetallic phases occurring in the aluminium alloys.

2. Material and methodology

The investigation was carried out on the AlCu4Ni2Mg2 and AlSi5Cu1Mg casting aluminium alloys. The chemical composition of the alloys is indicated in Table 1.

Alloy	Cu	Mg	Si	Fe	Ni	Zn	Ti
AlSi5Cu1Mg	1.3	0.5	5.2	0.2	-	<0.3	0.18
AlCu4Ni2Mg2	4.3	1.5	0.1	0.1	2.1	0.3	-

Table 1. Chemical composition of investigated AlCu4Ni2Mg2 and AlSi5Cu1Mg aluminium alloys, Al bal (wt%)

Microstructure analysis was carried out on the as-cast and in T6 condition aluminium alloys. The alloys were subjected to T6 heat treatment: solution heat treated at 520°C for 5 h followed by water cooling and aging at 250°C for 5 h followed by air cooling. The

microstructure of examined alloy was observed using an optical microscope on the polished sections etched in Keller solution (0.5 % HF in 50ml H₂O). The observation of specimens morphology was performed on a scanning electron microscope (SEM), operating at 6-10 kV in a conventional back-scattered electron mode and a transmission electron microscopes (TEM) operated at 120, 180 and 200kV. The thin foils were prepared by the electrochemical polishing in: 260 ml CH₃OH + 35 ml glycerol + 5 ml HClO₄. The chemical composition of the intermetallics was made by energy dispersive spectroscopy (EDS) attached to the SEM.

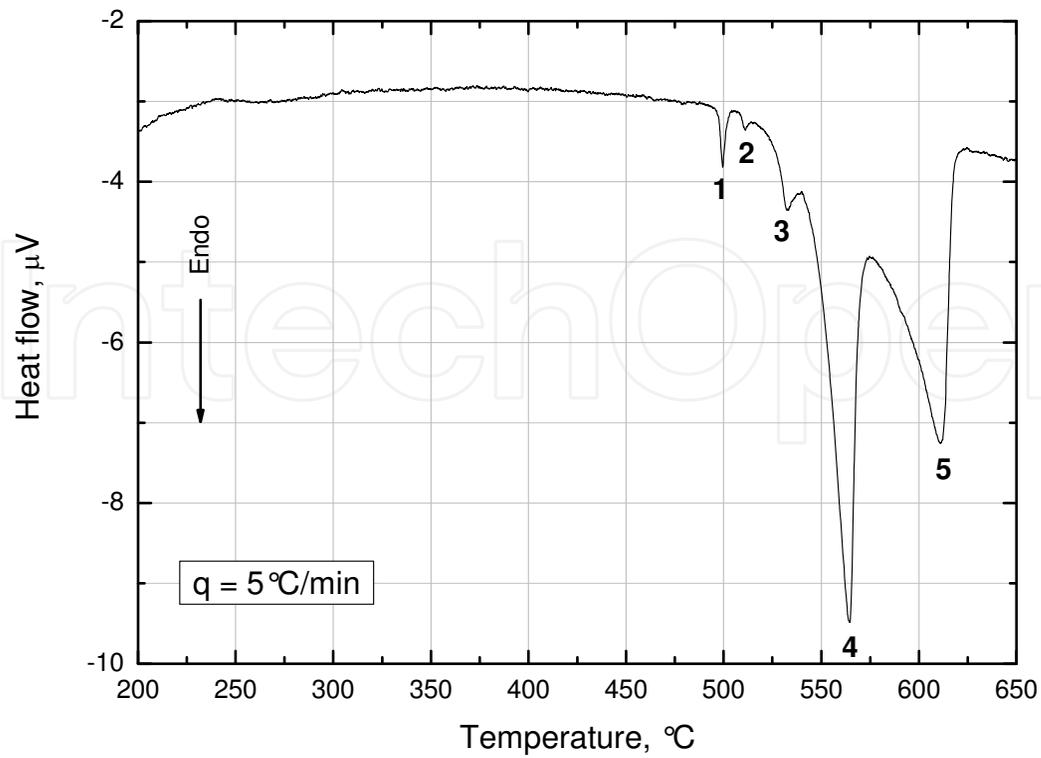
The intermetallic particles from investigated AlCu4Ni2Mg2 and AlSi5Cu1Mg alloys in T6 condition were extracted chemically in phenol. The samples in the form of disc were cut out from the rods of Ø12 mm diameter. Then ~0.8 mm thick discs were prepared by two-sided grinding to a final thickness of approximately 0.35 mm. The isolation of phases was performed according to following procedure: 1.625 g of the sample to be dissolved was placed in a 300 ml flask containing 120 mm of boiling phenol (182°C). The process continued until the complete dissolution of the sample occurred ~10 min. The phenolic solution containing the residue was treated with 100 ml benzyl alcohol and cooled to the room temperature. The residue was separated by centrifuging a couple of times in benzyl alcohol and then twice more in the methanol. The dried residue was refined in the mortar. After sieving of residue ~0.2 g isolate was obtained. The intermetallic particles from the powder extract were identified by using X-ray diffraction analysis. The X-ray diffraction analysis of the powder was performed using a diffractometer - Cu K α radiation at 40kV.

DSC measurements were performed using a calorimeter with a sample weight of approximately 80-90 mg. Temperature scans were made from room temperature ~25°C to 800°C with constants heating rates of 5°C in a dynamic argon atmosphere. The heat effects associated with the transformation (dissolution/precipitation) reactions were obtained by subtracting a super purity Al baseline run and recorded.

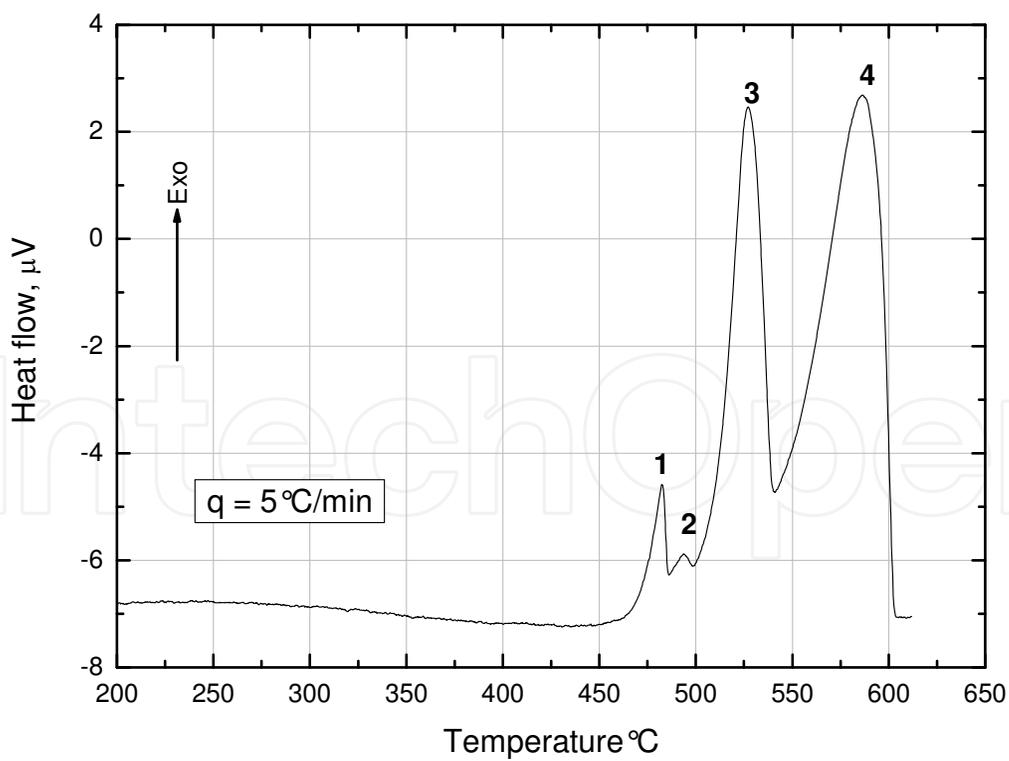
3. Results and discussion

DSC curves obtained by heating (Fig. 1a) and cooling (Fig. 1b) as-cast specimens of the examined AlSi5Cu1Mg alloy are shown in Fig. 1. DSC curves demonstrate precisely each reactions during heating and solidification process of as-cast AlSi5Cu1Mg alloy. One can see from the figures that during cooling the reactions occurred at lower temperatures (Fig. 1b) compared to the values recorded during heating of the same alloy (Fig. 1a). Solidification process of this alloy is quite complex (Fig. 1) and starts from formation of aluminum rich (α -Al) dendrites. Additional alloying elements such as: Mg, Cu, as well as impurities: Mn, Fe, leads to more complex solidification reaction. Therefore, as-cast microstructure of AlSi5Cu1Mg alloy presents a mixture of intermetallic phases (Fig. 2). The solidification reactions (the exact value of temperature) obtained during DSC investigation were compared with the literature data (Bäckerud et al., 1992; Li, et al., 2004) and presented in Table 2. Results obtained in this work very well corresponding to the (Bäckerud et al., 1992; Li, et al., 2004; Dobrzański et al., 2007).

Fig. 2 shows as-cast microstructure of AlSi5Cu1Mg alloy. The analyzed microstructure contains of primary aluminium dendrites and substantial amount of different intermetallic phases constituents varied in shape, (i.e.: needle, plate-like, block or "Chinese script"), size and distribution. They are located at the grain boundaries of α -Al and form dendritic network structure (Fig. 2).



(a)



(b)

Fig. 1. DSC thermograms of as-cast specimens of AlSi5Cu1Mg alloy, obtained during a) heating and b) cooling at rate of $5^{\circ}\text{C}/\text{min}$

Bäckerud et al.	Temp., °C	Li, Samuel et al.	Temp., °C	This work
$L \rightarrow (Al)$ dendrite network	609	(Al) dendrite network	610	610
$L \rightarrow (Al) + Al_{15}Mn_3Si_2 + (Al_5FeSi)$	590			
$L \rightarrow (Al) + Si + Al_5FeSi$	575	Precipitation of eutectic Si	562	564
$L \rightarrow (Al) + Si + AlMnFeSi$	558	Precipitation of $Al_6Mg_3FeSi_6 + Mg_2Si$	554	532
$L \rightarrow (Al) + Al_2Cu + Al_5FeSi$	525	Precipitation of Al_2Cu	510	510
$L \rightarrow (Al) + Al_2Cu + Si + Al_5Mg_8Cu_2Si_6$	507	Precipitation of $Al_5Mg_8Cu_2Si_6$	490	499

Table 2. Reactions occurring during the solidification of the AlSi5Cu1Mg alloy according to (Bäckerud et al., 1992; Li, Samuel et al., 2004)

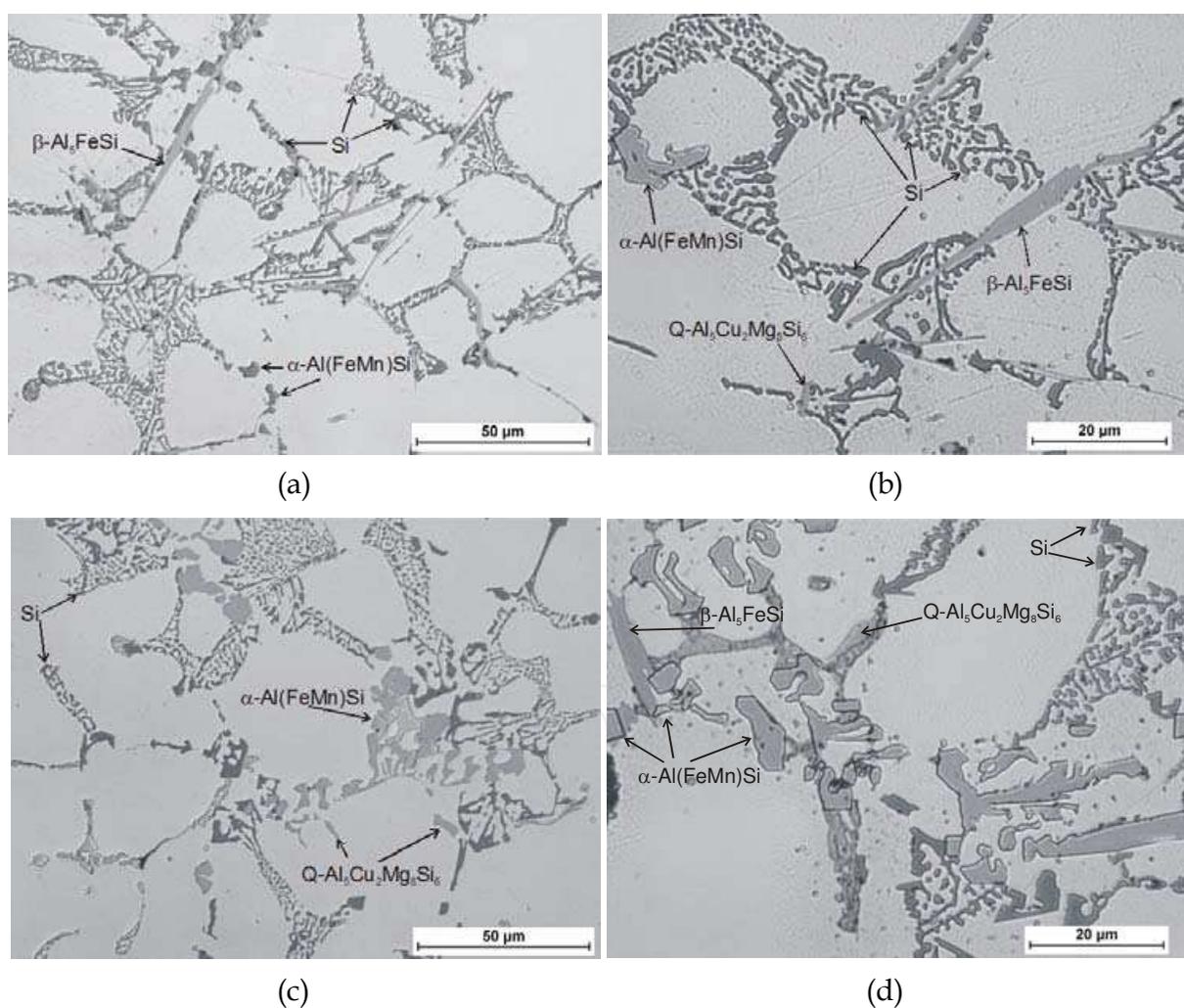


Fig. 2. Morphology of AlSi5Cu1Mg alloy in the as-cast state: (a,c) unetched and (b,d) etched

In order to identify the intermetallic phases in the examined alloy, series of elemental maps were performed for the elements line Al-K, Mg-K, Fe-K, Si-K, Cu-K and Mn-K (Fig. 3 and 4). The maximum pixel spectrum clearly shows the presence of Al, Mg, Fe, Si, Cu and Mn in the

scanned microstructure. In order to identify the presence of the elements in the observed phases, characteristic regions of the mapped phase with high Mg, Fe, Si, Cu and Mn concentration were marked and their spectra evaluated (Fig. 5).

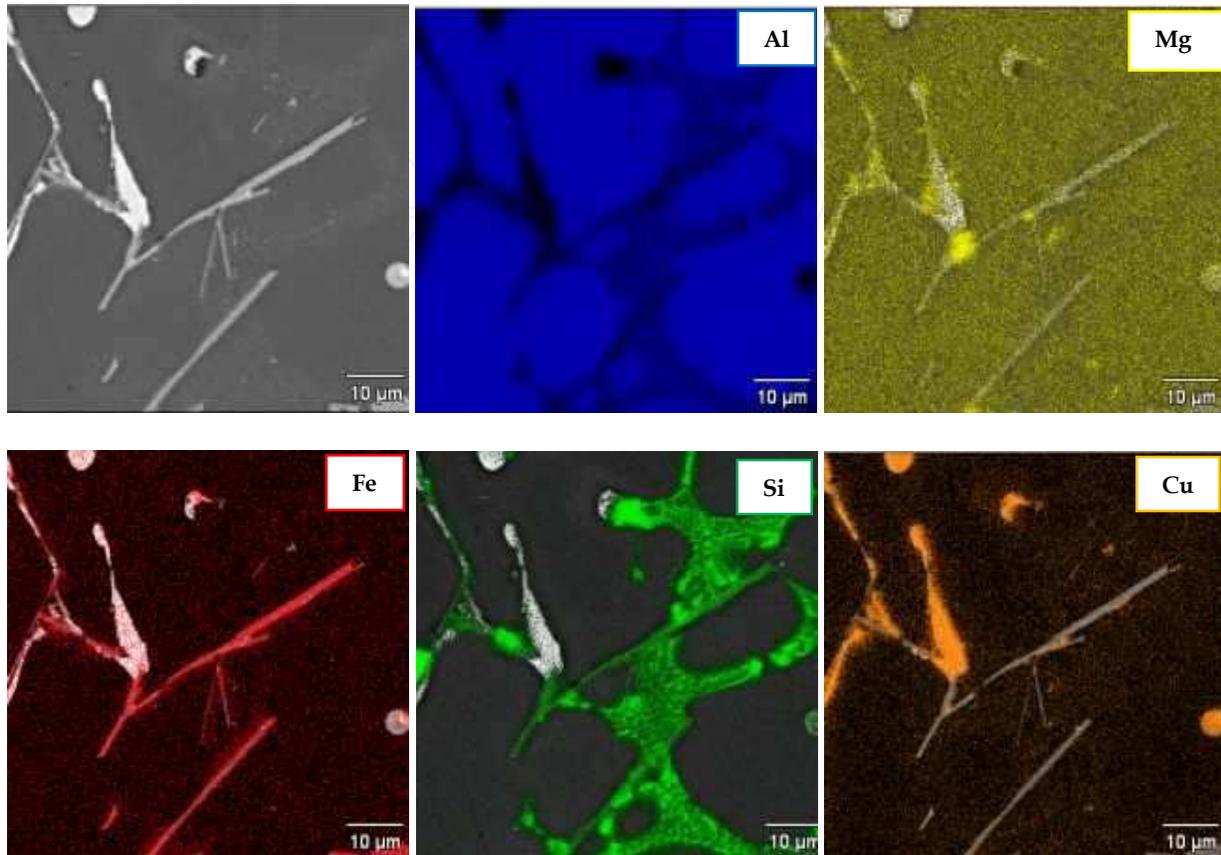


Fig. 3. SEM image of the AlSi5Cu1Mg alloy and corresponding elemental maps of: Al, Mg, Fe, Si and Cu

Fig. 5 shows the SEM micrographs with corresponding EDS-spectra of intermetallics observed in the as-cast AlSi5Cu1Mg alloy. The EDS analysis indicate that the oval particles are Al_2Cu (Fig. 5a). Besides Al_2Cu phase, another Cu containing phase $\text{Al}_5\text{Mg}_8\text{Cu}_2\text{Si}_6$ was observed (Fig. 4,5). In addition the Cu-containing intermetallics nucleating as dark grey rod, primary eutectic Si particles with “Chinese script” morphology were also observed. Fe has a very low solid solubility in Al alloy (maximum 0.05% at equilibrium) (Mondolfo, 1976), and most of Fe in aluminium alloys form a wide variety of Fe-containing intermetallics depending on the alloy composition and its solidification conditions (Ji et al., 2008). In the investigated as-cast AlSi5Cu1Mg alloy Fe-containing intermetallics such as light grey needle like $\beta\text{-Al}_5\text{FeSi}$ (Fig. 5a) and blockly phase consisting of Al, Si, Mn and Fe (Fig. 5a) were observed. On the basis of literature date (Liu Y.L. et al., 1999; Mrówka-Nowotnik et al., a,b, 2007; Wierzbińska et al., 2008) and EDS results (Fig. 5 and Tab. 3) this particles were identified as $\alpha\text{-Al(FeMn)Si}$ phase.

Fig. 5 shows SEM micrographs with corresponding EDS-spectra of intermetallics observed in as-cast AlSi5Cu1Mg alloy. The EDS spectra indicate that the oval particles are Al_2Cu (Fig. 5a). Besides Al_2Cu phase, another Cu containing phase AlCuMgSi is observed (Fig 5b). The results of EDS analysis are summarized in Tab. 3 versus the results obtained by earlier investigators.

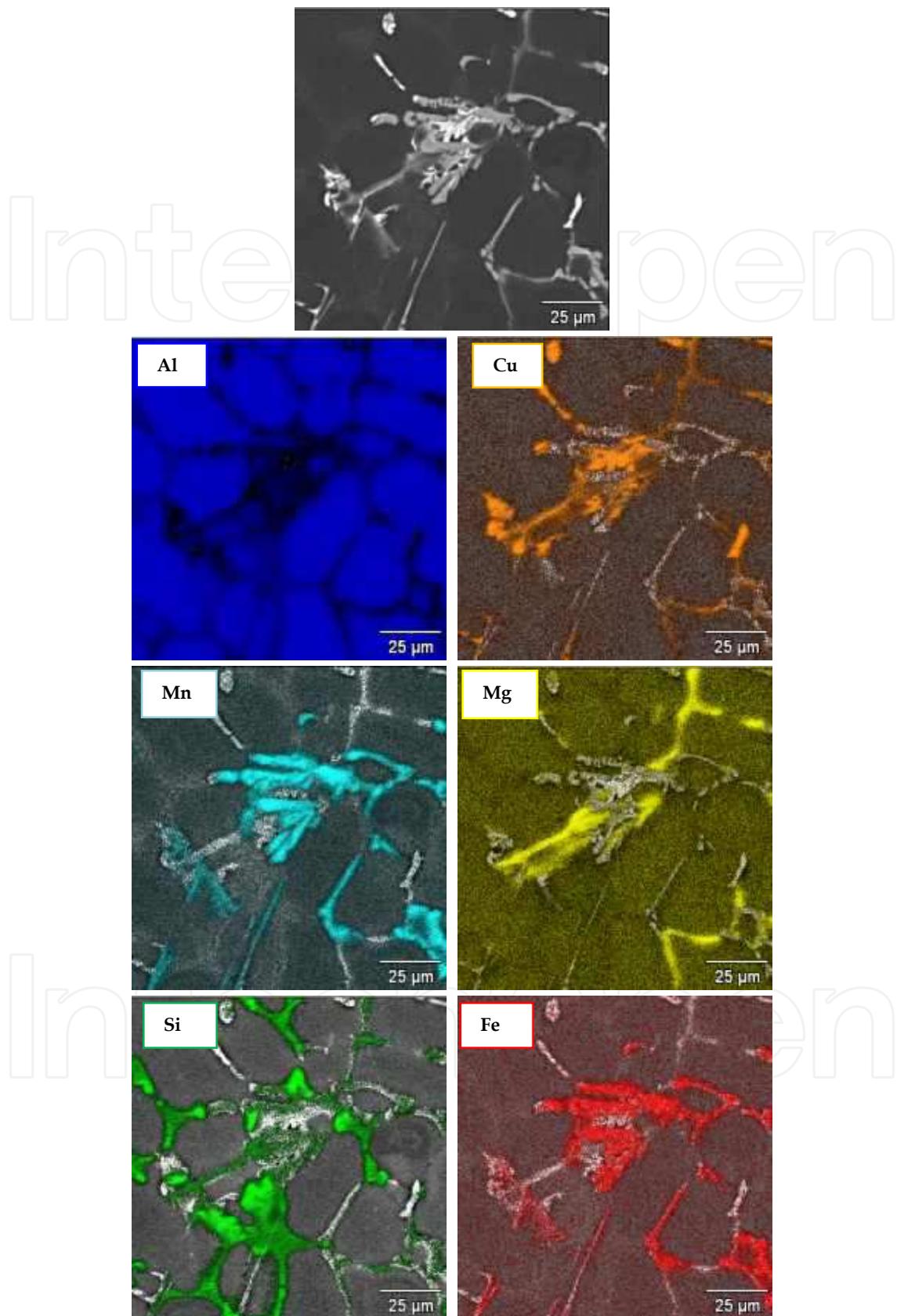


Fig. 4. SEM image of the AlSi5Cu1Mg alloy and corresponding elemental maps of: Al, Mn, Mg, Fe, Si and Cu

The following phases were identified in the as-cast AlSi5Cu1Mg alloy based on DSC results and microstructure - LM and SEM observations (Tab. 2 and 3, Fig. 1-5): Si, β -Al₅FeSi, Al₅Cu₂Mg₈Si₆, Al₂Cu, α -Al(FeMn)Si. These results can suggest, that in this alloys occur five solidification reactions (Tab. 4). The data presented in Tab. 4 shows that the solidification sequence of AlSi5Cu1Mg alloy differ only slightly from this obtained by Backerud and Li (Tab. 2).

No. of analyzed particles	Suggested type of phases	Chemical composition of determined intermetallic phases, (% wt)					References
		Si	Cu	Mg	Fe	Mn	
20	Al ₅ Cu ₂ Mg ₈ Si ₆	19.2 15.2 17.97	31.1 26.9 27.48	33 29.22 28.49			Ji, 2008 Lodgaard, (2000) This work
25	β -Al ₅ FeSi	12-15 12.2 14.59 13-16			25-30 25 27.75 23-26		Mondolfo, 1976 Warmuzek, 2005 Liu, 1999 This work
12	α -Al ₁₂ (FeMn) ₃ Si	10-12 5.5-6.5 5-7 8-12			10-15 5.1-28 10-13 11-13	15-20 14-24 19-23 14-20	Mondolfo, 1976 Warmuzek, 2006 Liu, 1999 This work
10	Al ₂ Cu		52.5 49.51				Belov, 2005 This work
25	Si	85-95					This work

Table 3. The chemical composition of the intermetallic phases in AlSi5Cu1Mg alloy in the as-cast state



Fig. 5. a) SEM micrographs of the AlSi5Cu1Mg alloy in the as-cast state

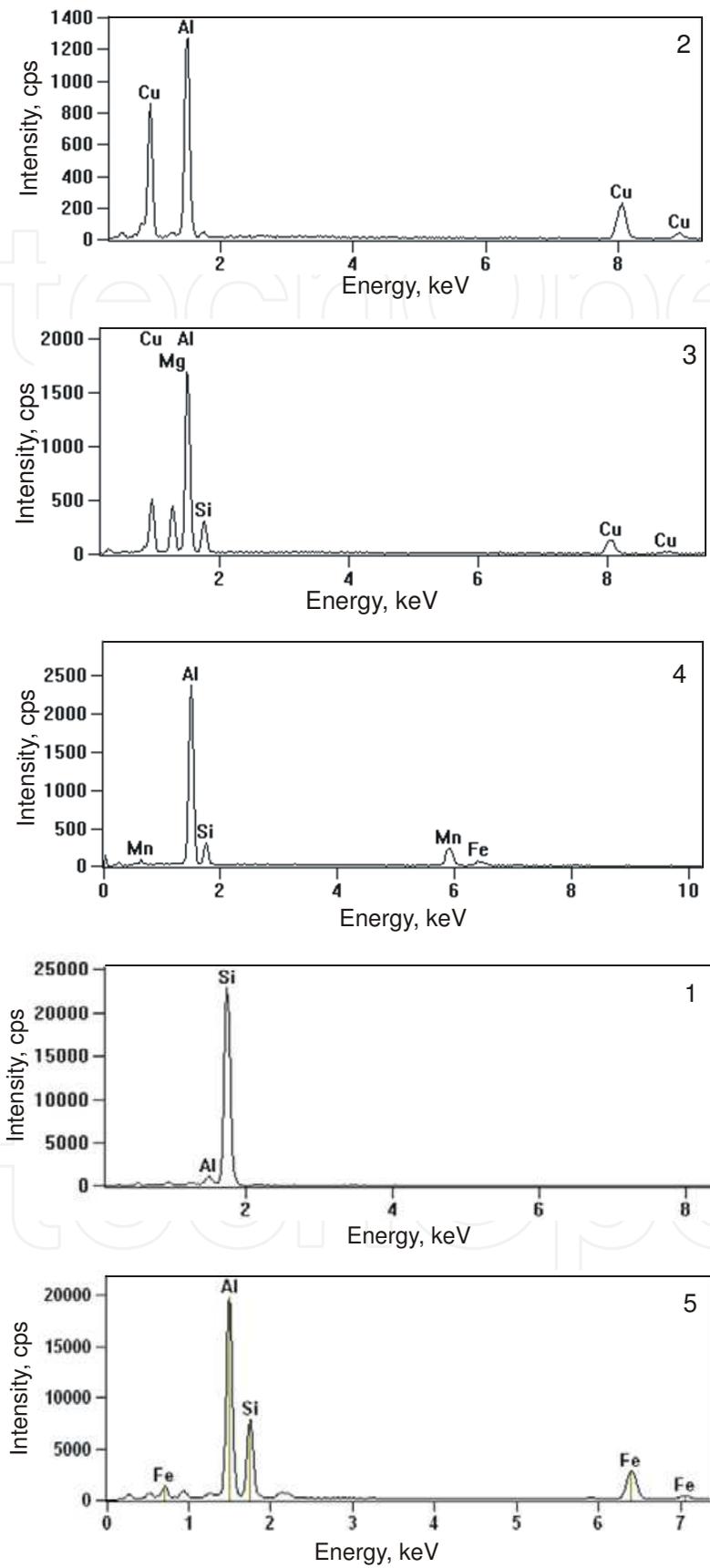


Fig. 5. b) The corresponding EDS-spectra acquired in positions indicated by the number 1-5

Reactions	Temperature, °C
$L \rightarrow (Al)$ dendrite network	610
$L \rightarrow (Al) + Si + Al_5FeSi$	564
$L \rightarrow (Al) + Si + AlMnFeSi$	532
$L \rightarrow (Al) + Al_2Cu + Al_5FeSi$	510
$L \rightarrow (Al) + Al_2Cu + Si + Al_5Cu_2Mg_8Si_6$	499

Table 4. Solidification reactions during nonequilibrium conditions in the investigated AlSi5Cu1Mg alloy, heating rate was 5°C/min

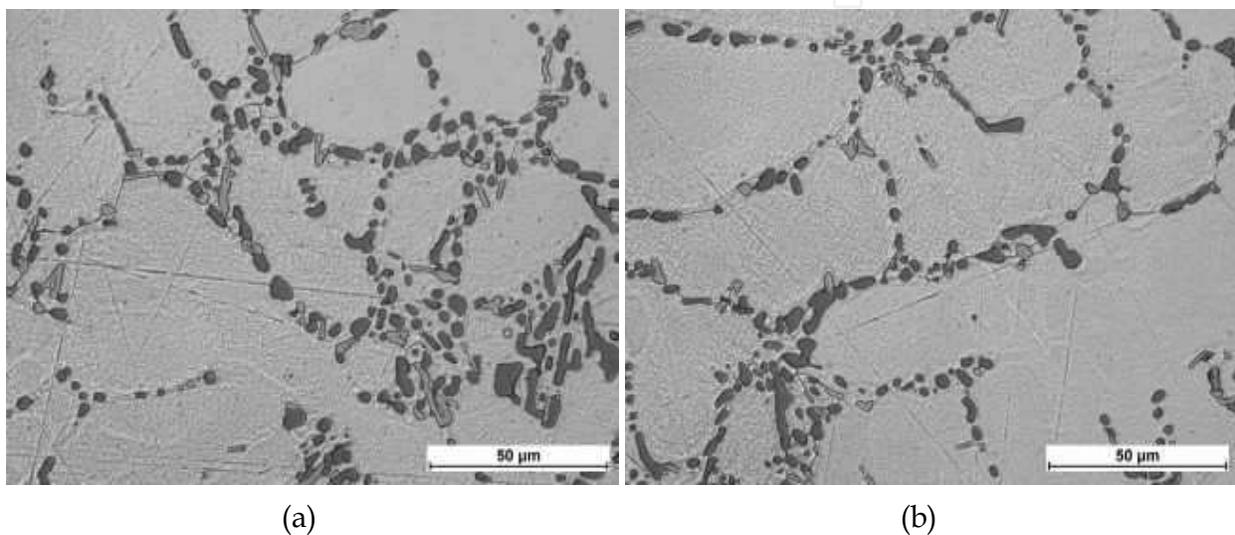


Fig. 6. The microstructure of AlSi5Cu1Mg alloy in the T6 condition (a,b)

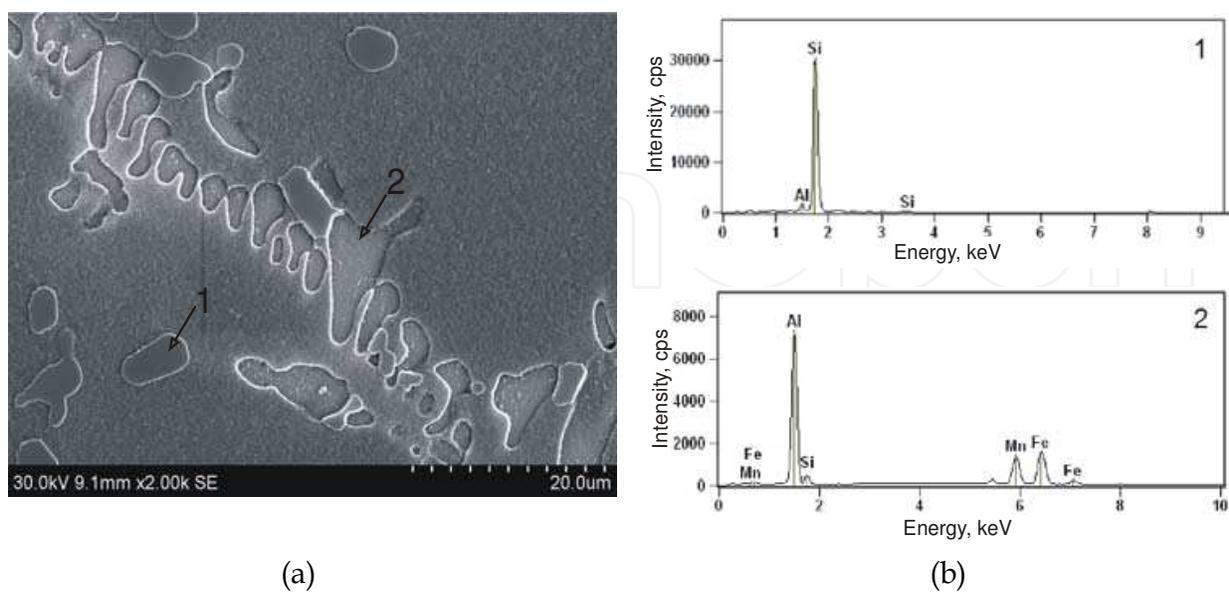


Fig. 7. a) SEM micrographs of the AlSi5Cu1Mg alloy in the T6 condition; b) The corresponding EDS-spectra acquired in the positions indicated by the number 1 and 2

Microstructure of AlSi5Cu1Mg alloy in T6 condition is presented in Fig. 6. Analyzing the micrographs of the alloy after heat treatment at 520°C for 5h it had been found that during solution heat treatment the morphology of primary eutectic Si changes from relatively large needle like structure to the more refined “Chinese script” and spherical in shape particles. Most of the needle like particles of β -Al₅FeSi phase transform into spherical-like α -Al(FeMn)Si (Kuijpers et al., 2002; Liu et al., 1999; Christian, 1995) as shown in Figure 6 and 7. It has been found that Al₂Cu and Al₅Cu₂Mg₈Si₆ phases dissolve in the α -Al matrix during solution heat treatment. The subsequent aging heat treatment at 250°C for 5 leads to formation of the supersaturated solid solution fine intermetallic strengthening particles of Al₂Cu (<1 μ m).

Fig. 7 shows scanning electron micrographs and EDS analysis of particles in the investigated AlSi5Cu1Mg alloy in T6 condition. The EDS analysis performed on the phases presented in microstructure of the alloy revealed, that spherical in shape inclusions are the eutectic

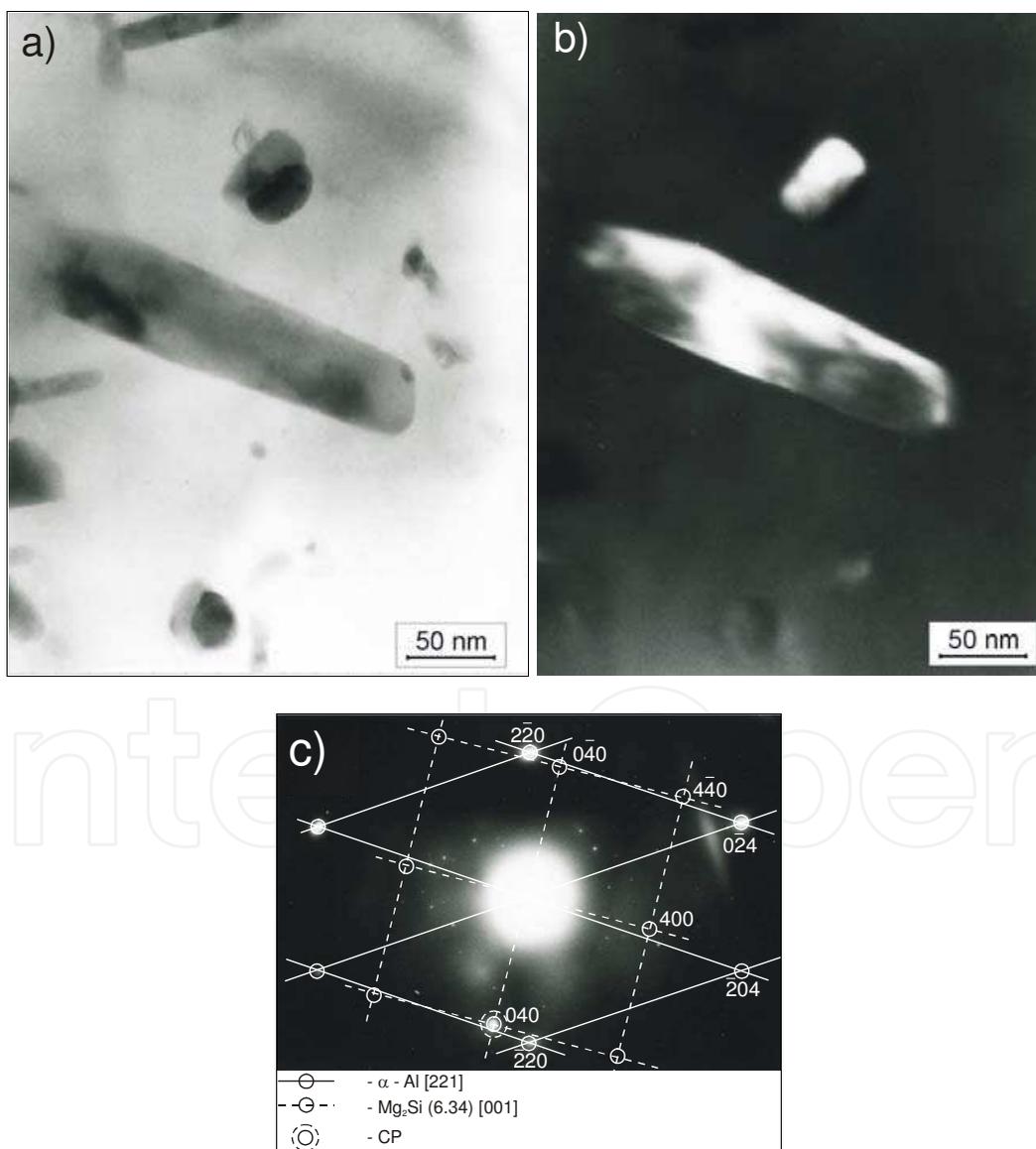


Fig. 8. TEM micrograph of AlSi5Cu1Mg alloy in T6 conditions showing the precipitate of the β -Mg₂Si phase (a,b), and corresponding electron diffraction pattern (c)

silicon ones, whereas the rod-like and “Chinese script” shaped, are inclusions of the phase consisting of Al, Si, Mn and Fe (Fig. 2,7 and Tab. 3).

Since it is rather difficult to produce detailed identification of intermetallic using only one method (e.g. microscopic examination) therefore XRD and TEM techniques was utilized to provide confidence in the results of phase classification based on metallographic study. The microstructure of the examined alloy AlSi5Cu1Mg in T6 state consists of the primary precipitates of intermetallic phases combined with the highly dispersed particles of hardening phases. The TEM micrographs and the selected area electron diffraction patterns analysis proved that the dispersed precipitates shown in Figure 8 and 9 were the precipitates of hardening phase β -Mg₂Si (Fig. 8) and θ' -Al₂Cu (Fig. 9).

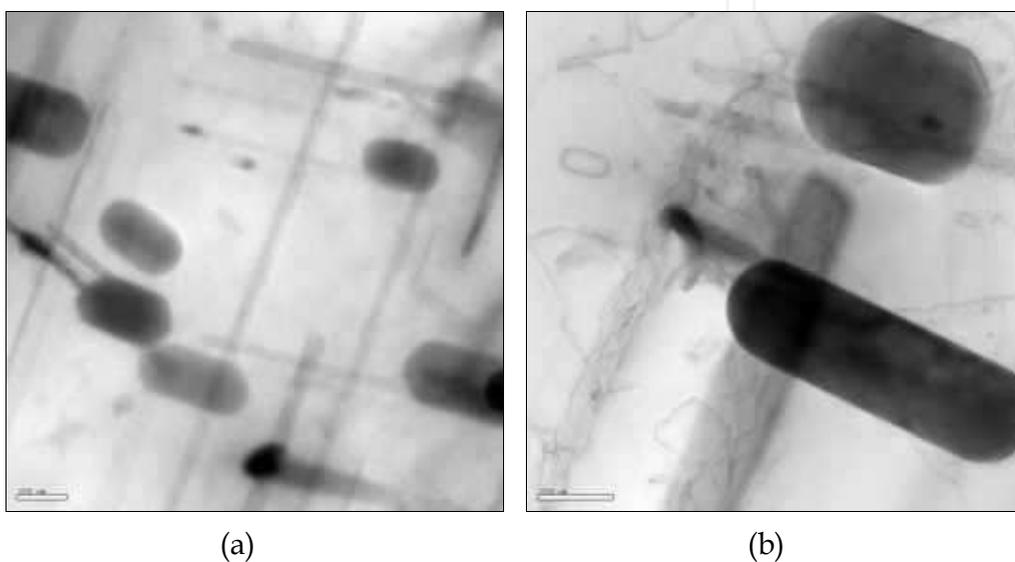


Fig. 9. Precipitation of strengthening β -Mg₂Si i θ' -Al₂Cu phases in AlSi5Cu1Mg (a,b) – TEM

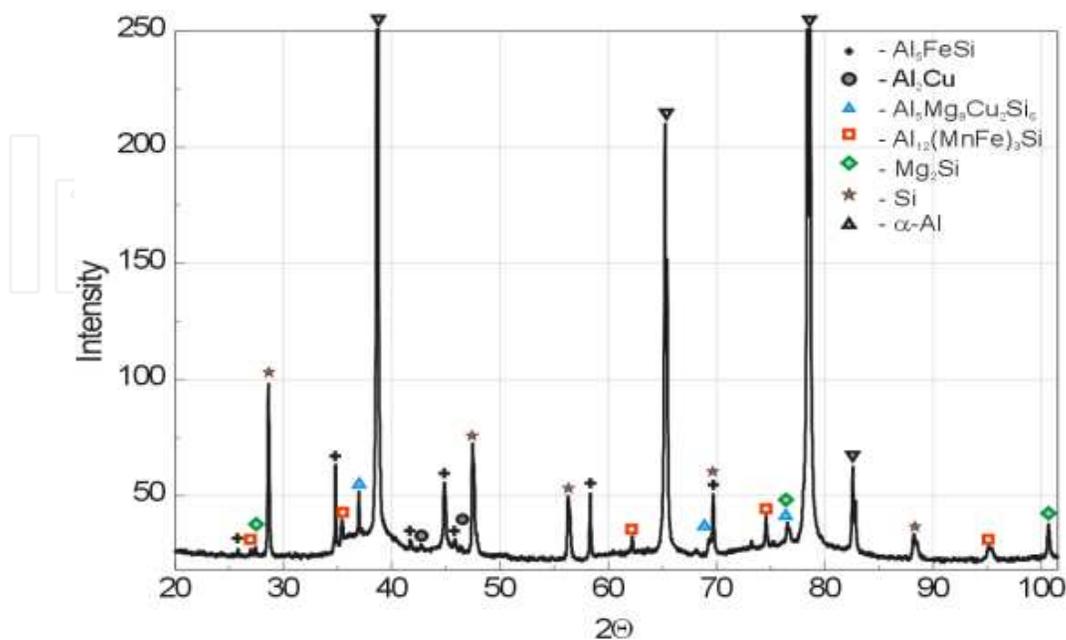


Fig. 10. X-diffraction pattern of AlSi5Cu1Mg alloy

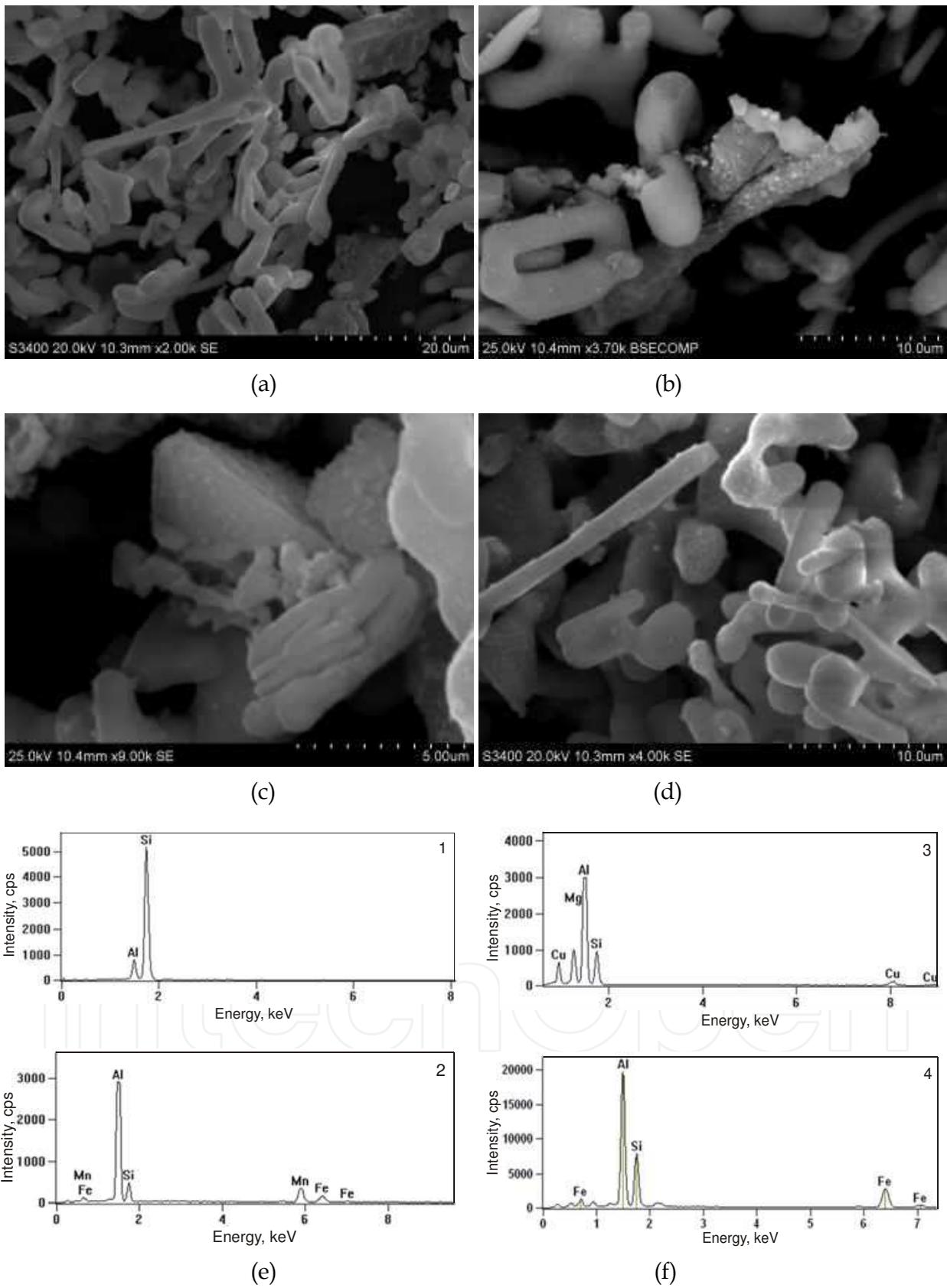
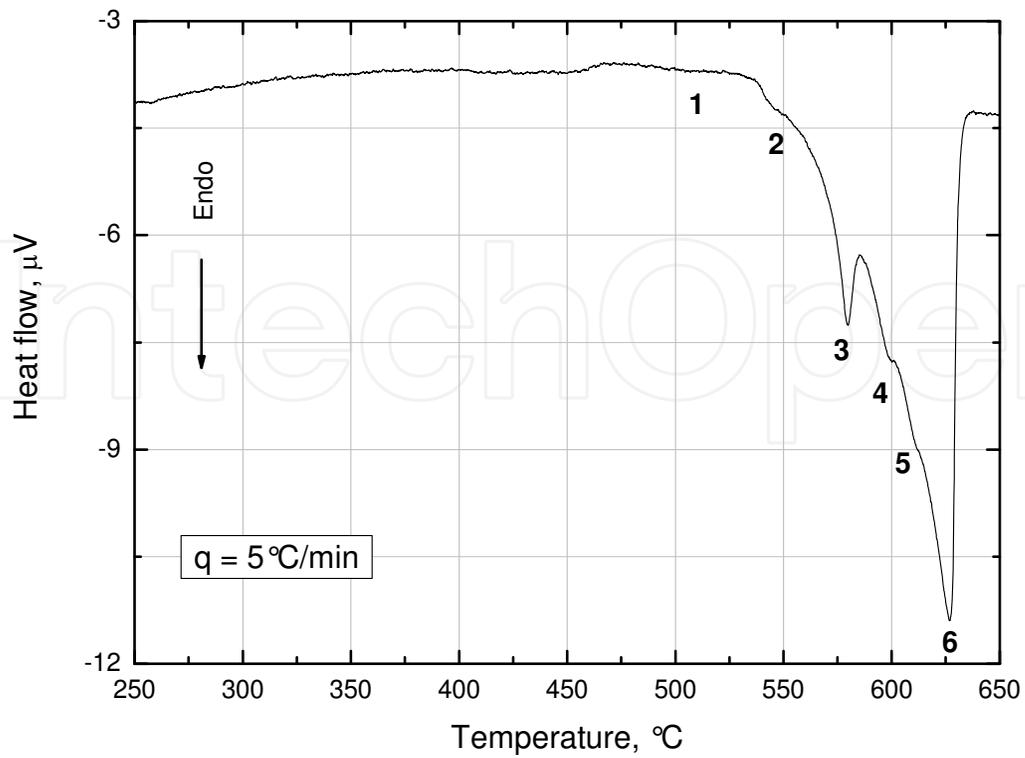
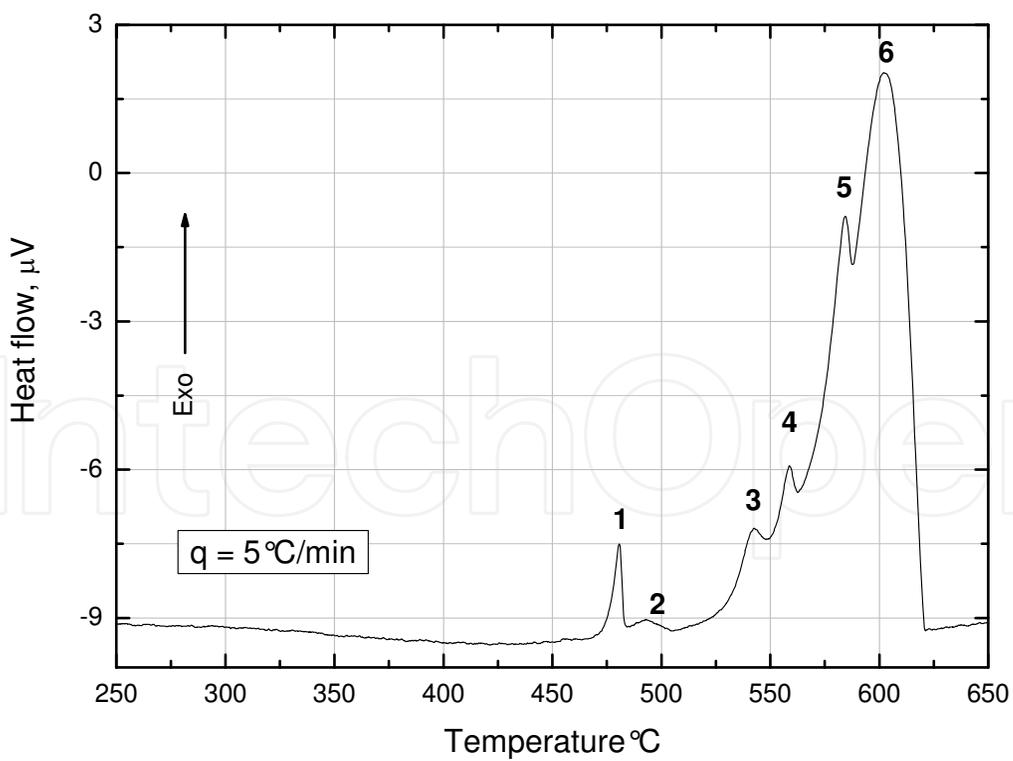


Fig. 11. SEM micrographs (a-d) of the particles extracted from the AlSi5Cu1Mg alloy and EDS spectra (e,f)



(a)



(b)

Fig. 12. DSC thermograms of as-cast specimens of $\text{AlCu}_4\text{Ni}_2\text{Mg}_2$ alloy, obtained during a) heating and b) cooling at a rate of $5^{\circ}\text{C}/\text{min}$

The results of XRD investigation are shown in Fig. 8. X-ray diffraction analysis of AlSi1MgMn alloy confirmed metallographic observation. Additionally the presented above results were compared to the analysis of the particles extracted from the AlSi5Cu1Mg alloy using phenolic dissolution technique (Fig. 11). The EDS spectra revealed the presence of Al, Mg, Mn, Si, Fe and Cu - bearing particles in the extracted powder (Fig. 11). The EDS analysis results proof that analyzed particles extracted from the AlSi5Cu1Mg alloy were: Si, AlMnFeSi, Al₅FeSi, Al₅Mg₈Cu₂Si₆ phases.

DSC curves obtained by heating (Fig. 12a) and cooling (Fig. 12b) of as-cast specimens of AlCu₄Ni₂Mg₂ alloy are shown in Fig. 12. DSC curves demonstrate reactions which occurred during heating and solidification process of the alloy. The obtained results were similar to the peaks observed during cooling of the samples of AlSi5Cu1Mg alloy - the recorded peaks were shifted to the lower values (Fig. 12b).

The solidification sequence of this alloy can be quite complex and dependent upon the cooling rate (Fig. 12). Possible reactions which occurred during solidification of AlCu₄Ni₂Mg₂ alloy are presented in Tab. 5. Aluminum rich (α -Al) dendrites are formed at the beginning of solidification process. Additional alloying elements into the alloys (Ni, Cu, Mg) as well as impurities (eg. Fe) change the solidification path and reaction products. Therefore, as-cast microstructure of the tested alloy exhibit the appearance of mixture of intermetallic phases (Fig. 13a). The solidification reactions (the exact value of temperature) obtained during DSC investigation presented in Tab. 5.

Possible reactions	Temperature, °C
$L \rightarrow (Al) + Al_6Fe$	612
$L \rightarrow (Al) + Al_4CuMg$	584
$L \rightarrow (Al) + Al_2Cu + Al_2CuMg$	558
$L \rightarrow (Al) + Al_2Cu + Al_7Cu_4Ni$	542
$L \rightarrow (Al) + Al_2Cu + Al_2CuMg + Al_3(CuFeNi)_2$	493
Solidus	480

Table 5. Possible solidification reactions during nonequilibrium conditions in investigated AlCu₄Ni₂Mg₂ alloy, at a heating rate 5°C/min

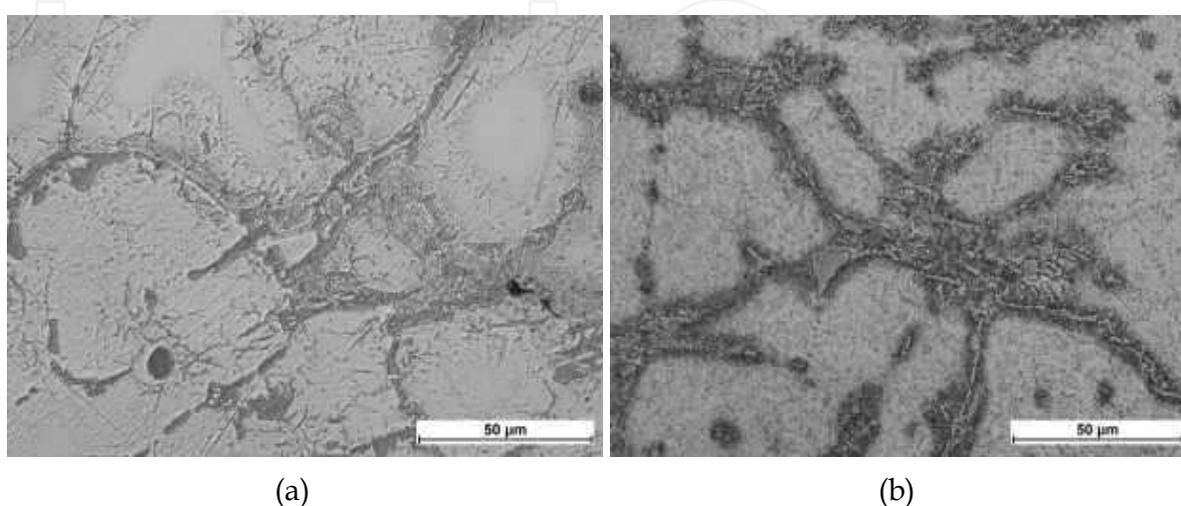


Fig. 13. The microstructure of AlCu₄Ni₂Mg₂ alloy in as-cast state (a) and the T6 condition (b)

The analyzed microstructure in as-cast state (Fig. 13a) contains of primary aluminium dendrites and substantial amount of different intermetallic phases constituents varied in shape, size and distribution. They are located at the grain boundaries of α -Al and form dendrites network structure (Fig. 13a).

The analyzed microstructure of investigated AlCu₄Ni₂Mg₂ alloy in T6 condition (Fig. 13b) consists different precipitates varied in shape, i.e.: fine sphere-like, complex rod-like and ellipse-like distributed within interdendritic areas of the α -Al alloy. Large number of fine sphere-like strengthening phase are located in the boundary zone. However, small volume of this phase is also present homogenously throughout the sample (Fig. 13b). In order to identify the intermetallic phases in the examined alloy, series of distribution maps were performed for the elements line Mg-K, Al-K, Fe-K, Ni-K, Cu-K (Fig. 14). The maximum pixel spectrum clearly shows the presence of Ni and Cu in the scanned microstructure. In order to identify the presence of the elements in the observed phases, two regions of the mapped phase with high nickel and copper concentration were marked and their spectra evaluated.

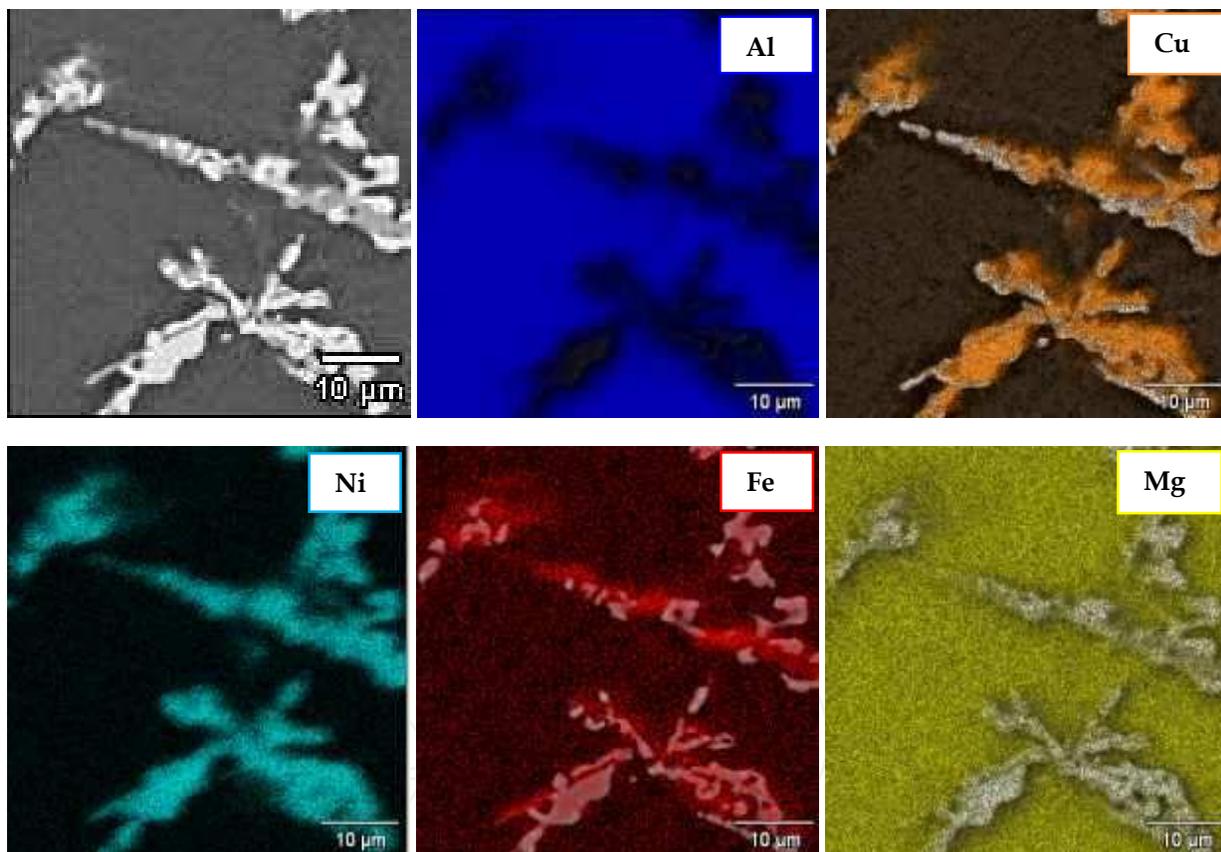


Fig. 14. SEM image of the AlCu₄Ni₂Mg₂ alloy and corresponding elemental maps of: Al, Mg, Fe, Ni and Cu

As seen in the elemental maps in Fig. 14, the regions enriched in Ni and Cu correspond to the formation of type precipitates (complex rod-like) and ellipse-like precipitates observed in Fig. 13. Fig. 15 shows the scanning electron micrographs and EDS analysis of particles in the AlCu₄Ni₂Mg₂ alloy.

The EDS analysis performed on the phases present in microstructure of the alloy revealed, that complex rod-like phase is the Al₇Cu₄Ni one, whereas the ellipse-like is Al₃(CuFeNi)₂ (Fig. 15 and Tab. 6)

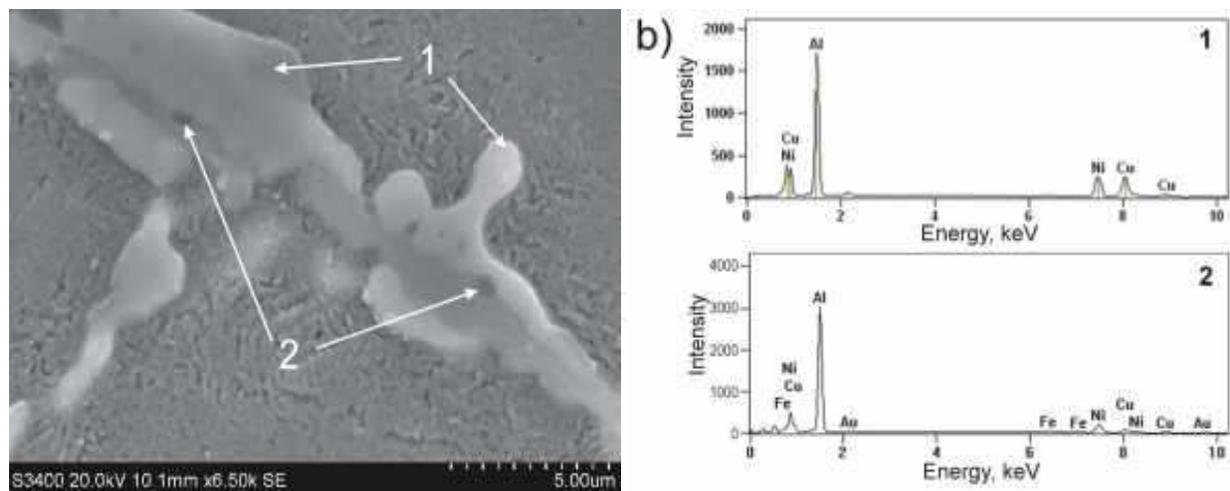


Fig. 15. a) SEM micrographs of the AlCu4Ni2Mg2 alloy in the T6 condition; b) The corresponding EDS-spectra acquired in positions indicated by the number 1 and 2

No. of analyzed particles	Suggested type of phases	Chemical composition of determined intermetallic phases, (%at)			Reference
		Ni	Cu	Fe	
20	Al ₇ Cu ₄ Ni	11.8÷22.2	38.7÷50.7		Belov, 2005 Chen, 2010 This work
		18.08	34.33		
		14.2÷22.6	29.7÷45.2		
25	Al ₃ (CuFeNi) ₂	18÷22	9÷15	8÷10	Belov, 2002 This work
		17.1÷20.5	10.5÷19.3	7.2÷9.5	
12	Al ₂ Cu		52.5		Belov, 2005 This work
			47.7÷51.9		

Table 6. The chemical composition and volume fraction of the intermetallic phases in the AlCu4Ni2Mg2 alloy

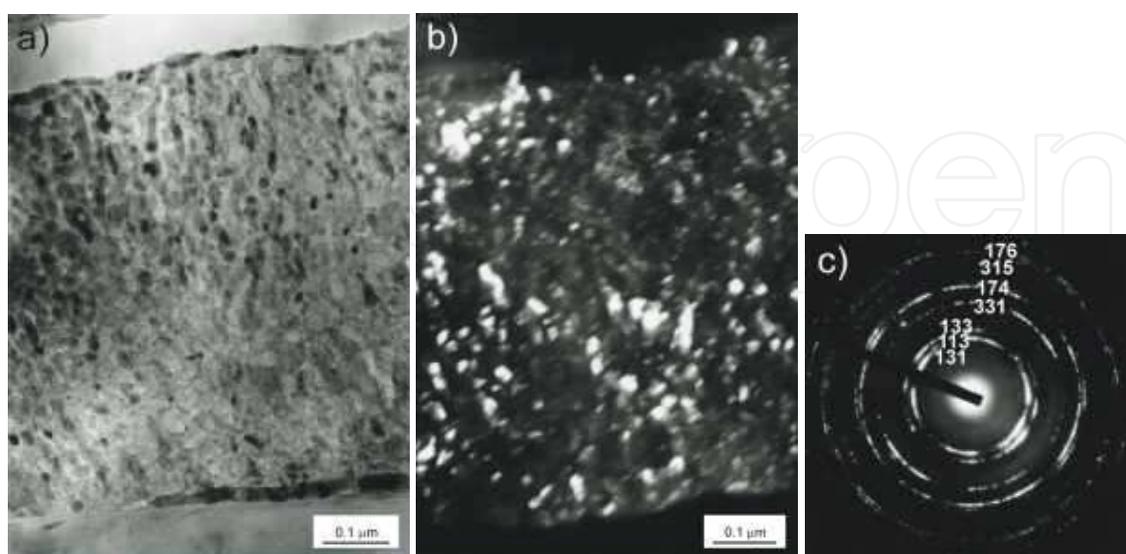


Fig. 16. TEM micrograph of AlCu4Ni2Mg2 alloy in T6 conditions showing the precipitate of the S-Al₂CuMg phase (a,b), and corresponding electron diffraction pattern (c)

The microstructure of the examined alloy AlCu₄Ni₂Mg₂ in T6 state consists of the primary precipitates of intermetallic phases combined with the highly dispersed particles of hardening phases. The TEM micrographs and the selected area electron diffraction patterns analysis proved that the dispersed precipitates shown in Fig. 13b are the intermetallic phases S-Al₂CuMg (Fig. 16) and Al₆Fe (Fig. 17) besides the precipitates of hardening phase θ' -Al₂Cu were present in AlCu₄Ni₂Mg₂ alloy (Fig. 18). The approximate size of the S phase was 0,5 μ m.

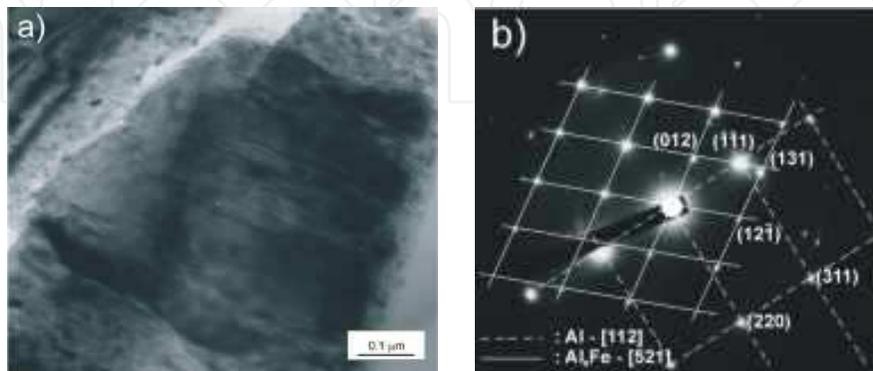


Fig. 17. TEM micrograph of AlCu₄Ni₂Mg alloy in T6 condition showing the precipitate of the Al₆Fe phase (a), and corresponding electron diffraction pattern (b)

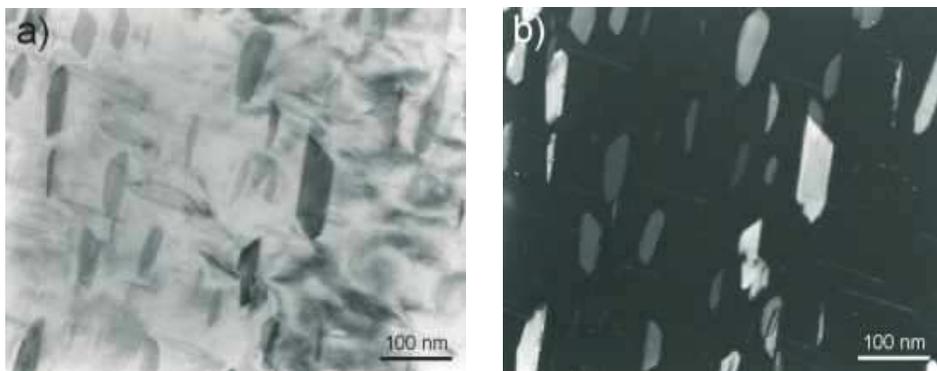


Fig. 18. TEM micrograph of AlCu₄Ni₂Mg alloy in T6 condition showing the precipitates of hardening phase θ' -Al₂Cu, bright field (a) and dark field (b)

The results of the SEM/EDS analysis of the particles extracted with boiling phenol from AlCu₄Ni₂Mg₂ alloy (Fig. 19) were compared with X-ray diffraction pattern (Fig. 20). The observed peaks confirmed SEM and TEM results. The majority of the peaks were from Al₇Cu₄Ni, Al₆Fe, S-Al₂CuMg, and Al₃(CuFeNi)₂.

On the other hand, it is nearly impossible to make unambiguous identification of the all intermetallics present in an aluminium alloy which are rather complex, even applying all well-known experimental techniques. X-ray diffraction analysis is one of the most powerful and appropriate technique giving the possibility to determine most of verified intermetallics based on their crystallographic parameters. Our analysis shows that the difficulties of having reliable results of all the possible existing phases in a microstructure of the alloy is related to the procedure of phase isolation. The residue is separated by centrifuging and since some of the particles are very fine and available sieves are having too big outlet holes there is no chance prevents them from being flowing out from a solution.

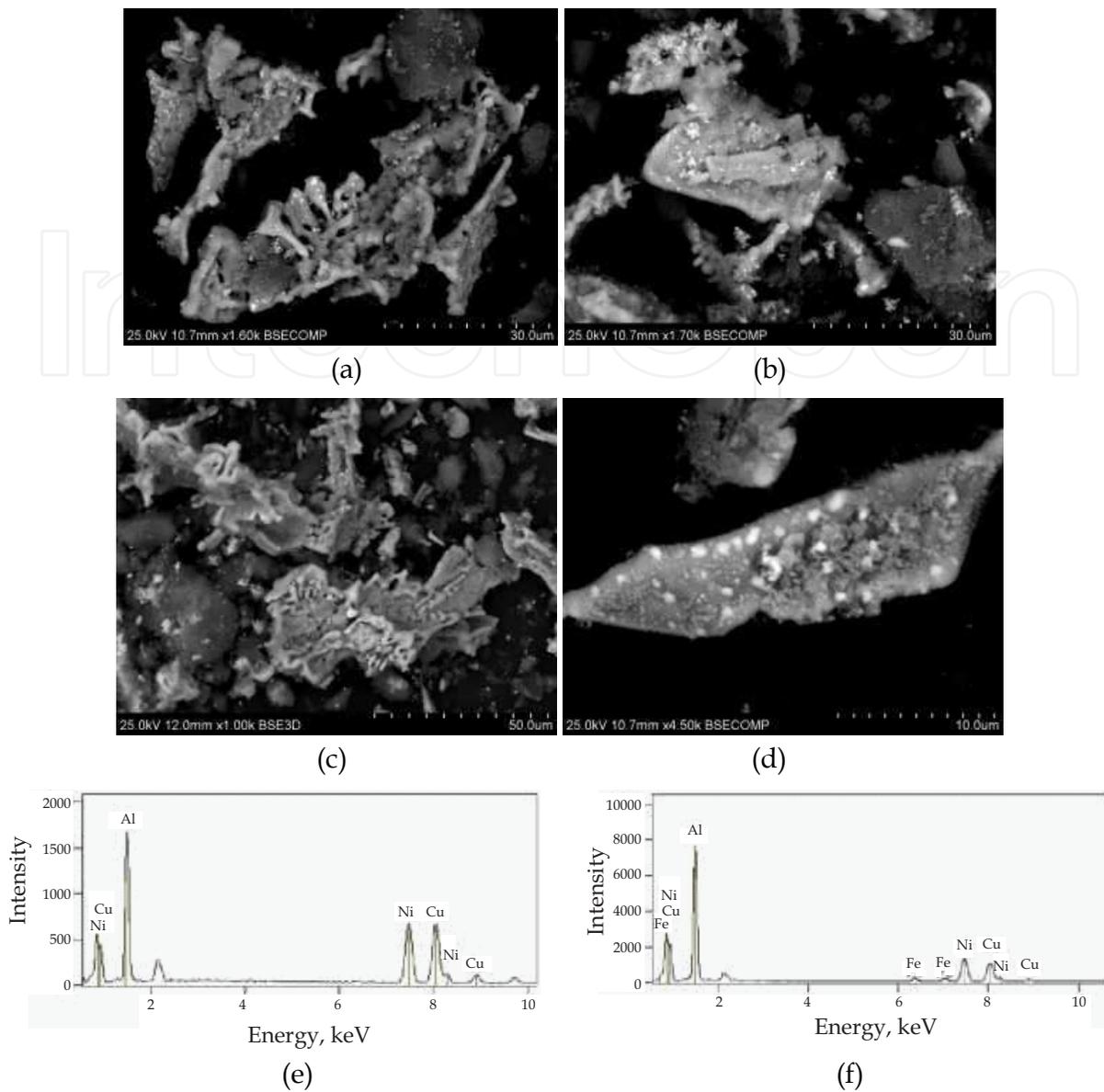


Fig. 19. SEM micrographs of the particles Al₇Cu₄Ni (a,c) and Al₃(CuFeNi)₂ (b,d) extracted from the AlCu₄Ni₂Mg₂ alloy along with EDS spectra (e,f)

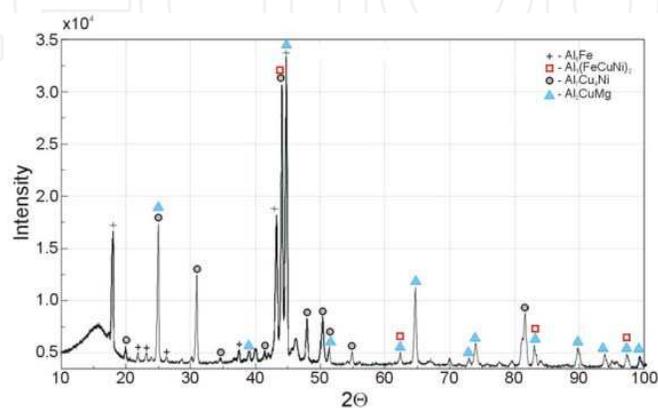


Fig. 20. The X-ray diffraction from the particles extracted from AlCu₄Ni₂Mg₂ alloy

4. Conclusion

Currently, efforts are being directed towards the development of analytical techniques which rapidly achieve an accurate determination of phase components in an alloy. According to the obtained results, the applicability of the proposed methods provides a practical alternative to other techniques. The phenol extraction procedure was also successfully applied to the examined aluminium alloys. The main advantages of dissolution techniques are its reliability – when used properly you will always get pure residue – and its low price. The major disadvantages of phenol extraction method are the possible contamination of the residue and the time needed.

The examined alloys AlSi5Cu1Mg and AlCu4Ni2Mg2 possessed a complex microstructure. By using various instruments and techniques (LM, SEM-EDS, TEM and XRD) a wide range of intermetallic phases were identified. The microstructure of investigated AlSi5Cu1Mg alloy included: β -Al₅FeSi, α -Al₁₂(FeMn)₃Si, Al₂Cu, Q-Al₅Cu₂Mg₈Si₆, Si and Mg₂Si phases. The microstructure of AlCu4Ni2Mg2 alloy included five phases, namely: Al₇Cu₄Ni, θ' -Al₂Cu, Al₆Fe, S-Al₂CuMg, and Al₃(CuFeNi)₂. A size and distribution of these various dispersoids depend on the time and temperature of the homogenization and/or annealing processes. Fine intermetallic particles (<1 μ m) are formed during artificial aging of heat-treatable alloys and are more uniformly distributed than constituent particles or dispersoids. Dimensions, shape and distribution of these particles may have important effects on the ductility of alloys and more needs to be known regarding their formation, structure and composition. For example, the coarse particles can influence the recrystallization, fracture, surface, and corrosion behavior, while the dispersoids control grain size and provide stability to the metallurgical structure. The dispersoids can also affect the fracture performance and may limit strain localization during deformation. The formation of particles drains solute from the matrix and, consequently, changes the strength properties of the material. This is specially relevant in the heat-treatable alloys, where depletion in Cu, Mg, and Si can significantly change the metastable precipitation processes and age hardenability of a material. Therefore, particle characterization is essential not only for choosing the best processing routes, but also for designing optimized alloy composition. 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. A variety of microscopic techniques are well appropriate to characterize intermetallics but only from a small section of an analyzed sample. From commercial point of view it is extremely advantageous to provide use quick, reliable and economical examination technique capable of providing data of particles from different locations of a full scale-sized ingot. One of these methods is dissolving the matrix of an aluminium alloy chemically or electrochemically.

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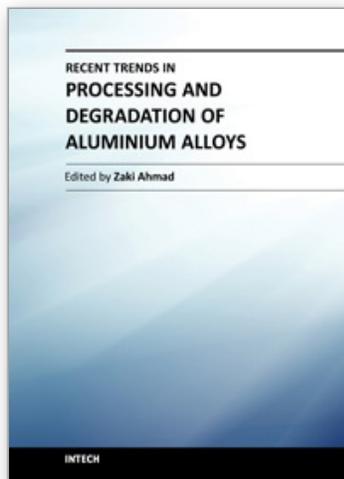
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