



Microstructure and mechanical properties of C355.0 cast aluminium alloy

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ABSTRACT

Purpose: The main task of this work was to study the solidification process through analysis of the DSC curves that were obtained at solidification rate of 5 K/min. During C355.0 alloy solidification an amount of different intermetallic phases may form. Their volume fraction, chemical composition and morphology exert significant influence on a technological and mechanical properties of the aluminium alloys. Therefore the examination and identification of intermetallic phases in examined alloy is very important part of complex investigation. In this research the effect of precipitation hardening process on the microstructure and mechanical properties of C355.0 alloy has also been investigated.

Design/methodology/approach: To study the solidification process differential scanning calorimetry (DSC) was used. Hardness measurements have been utilized to examine the effect of a precipitation hardening (T6) on the mechanical properties. The plastic and mechanical properties were evaluated by uniaxial tensile test at room temperature. To identify intermetallics in C355.0 alloy optical light microscopy (LM), X-ray diffraction (XRD), scanning (SEM) and transmission (TEM) electron microscope were used.

Findings: The results show that the as-cast microstructure of C355.0 alloy after slow solidification at a cooling rate 5K/min, consisted a wide range of intermetallics phases. The microstructure of investigated C355.0 alloy included: β -Al₅FeSi, α -Al₁₂(FeMn)₃Si, Al₂Cu, Q-Al₅Cu₂Mg₈Si₆, Si and Mg₂Si phases. Significant changes in as-cast microstructure and mechanical properties followed after artificial aging due to a precipitation strengthening process were observed.

Practical implications: The aim of this work was to analyze the solidification process and how T6 heat treatment influenced the microstructure and mechanical properties of C355.0 alloy. Additionally this paper proposes the best experimental techniques for analysis of the intermetallic phases occurring in the cast and T6 condition.

Originality/value: The paper has provided essential data about influence of solidification process and aging parameters on the microstructure and mechanical properties of C355.0 alloys

Keywords: Metallic alloys, Microstructure, Intermetallic phases, Heat treatment, Mechanical properties

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MATERIALS

1. Introduction

The excellent castability, lightness and mechanical properties of cast Al-Si-Cu-Mg aluminum alloys - include C355.0 alloy, make them very important group of materials for industrial application. They are widely used especially in aerospace, automotive and household industries [1-7]. These group of alloys have become increasingly important in recent years, mainly in the aircraft and automotive industry in the form of aircraft supercharger covers, fuel-pump bodies, air-compressor pistons, liquid-cooled aircraft engine crankcases, motor mounts, cylinder heads, heat exchangers, air conditioners, transmissions housings, wheels, fenders, loads floor and suspension components due to their high strength at room and high temperature [1-10]. 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. Their relative volume fraction, chemical composition and morphology exert significant influence on a technological properties of the aluminium alloys [2-6,11-30]. Additional alloying elements like as: Cu and Mg, as well as impurities like as: Fe, Mn lead to more complex solidification reaction [1-7,12,13,31,32]. Therefore, the C355.0 as-cast microstructure presents many intermetallic phases. During solidification of alloy C355.2 Bäckerud [31] identified five reactions (Table 1).

Table 1.
Solidification reactions under nonequilibrium conditions in a C355.2 alloy [31,32]

No	Reactions	Temp., °C
1.	$L \rightarrow (Al)$ dendrite network	621-557
2.	$L \rightarrow (Al)+Si+Al_5FeSi$, $L \rightarrow (Al)+Si+Al(FeMnSi)$	570-551
3.	$L+Al_5FeSi \rightarrow (Al)+Si+ Al_8FeMg_3Si_6$	551-535
4.	$L \rightarrow (Al)+Si+ Al_8FeMg_3Si_6+Mg_2Si$	535-501
5.	$L \rightarrow (Al)+Al_2Cu+Al_5FeSi$, $L \rightarrow (Al)+Al_2Cu+Si+Al_5Mg_8Cu_2Si_6$	501-489

The solidification reactions experimentally observed by Bäckerud [31] in C355.2, alloy solidified under nonequilibrium conditions. A primary α -aluminum dendrites network forms between 621-557°C. The exact temperature depends mainly on the amount of Si and Cu concentration in the alloy [1,32]. About range of temperature 570-551°C the eutectic mixture of α -aluminum and Si forms, leading to a further localized increase in Cu content of the remaining liquid. The Fe rich phases Al_5FeSi and $Al(FeMnSi)$ can also precipitate in this temperature range. At approximately 535°C the $Al_8FeMg_3Si_6$ and Mg_2Si phases begin to precipitate. A decrease in the temperature allows for precipitation of Al_2Cu and $Al_5Mg_8Cu_2Si_6$ phases between 501-489°C.

In order to improve the mechanical properties and ductility of Al-Si-Cu-Mg cast alloys they are usually heat-treated [1-11,20-24]. The heat treatment consists of solution treatment, quenching and artificial aging. After solution treatment and quenching, the magnesium and copper-rich intermetallic compounds dissolve into Al matrix. For Al-Si-Cu-Mg alloys, the age hardening is

caused by the precipitation of metastable strengthening phases θ'' , β'' and/or θ' , β' precursors of the equilibrium θ (Al_2Cu) and β (Mg_2Si) phases. For Al-Si-Mg-Cu alloys, the precipitation behaviors are rather complicated and others several phases such as S (Al_2CuMg) or Q ($Cu_2Mg_8Si_6Al_5$) in metastable conditions may exist [1,4-6,31,32].

The main objective of this work was to examine a solidification process of C355.0 alloy and analyze morphology and composition of the complex microstructure of intermetallic phases in as-cast and T6 conditions. Hardness measurements have been utilized to study the effect of the precipitation hardening (150°C and 220°C for 42h) on the mechanical properties in aluminium alloy C355.0. The mechanical and plastic properties of the examined alloy were evaluated by uniaxial tensile test at room temperature.

2. Material and experimental

The investigation was carried out on the C355.0 casting aluminium alloy. The chemical composition of the alloy is indicated in Table 2.

Table 2.
Chemical composition of the C355.0 alloy, %wt

Si	Cu	Mg	Fe	Mn	Ti	Others	Al
5.2	1.33	0.53	0.2	0.14	0.1	0.15	bal

DSC measurements were performed using a SETARAM Setsys Evolution 1200 with a sample weight of approximately 80-90 mg. Temperature scans were made from room temperature $\sim 25^\circ C$ to $800^\circ C$ with constants heating rates of $5^\circ C$ 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.

Microstructure analysis was carried out on the as-cast and in T6 condition C355.0 aluminium alloy. The alloy were subjected to T6 heat treatment: solution heat treated at $520^\circ C$ for 5 h followed by water cooling. Subsequently the specimens were subjected to artificial aging at temperature $150^\circ C$ and $220^\circ C$ for 48 h followed by air cooling. To monitor the hardness changes during aging treatments Brinell hardness was measured with the tester under 250 N load for 10 sec. After artificial aging, a set of specimens were prepared for tensile testing to study the effect of T6 heat treatments on mechanical properties of the examined alloys. The specimens were strained by tensile deformation on Instron TTF-1115 servohydraulic universal tester at a constant rate at room temperature, in according to standard PN-EN 10002-1:2004 [33]. Tensile properties (tensile and yield strength, elongation) were evaluated using round test specimens of 10 mm diameter and 100 mm gauge length.

The microstructure of examined alloy was observed using an optical microscope - Nikon 300 on the polished sections etched in Keller solution (0.5% HF in 50 ml H_2O). The observation of specimens morphology was performed on the scanning electron microscope (SEM) HITACHI S-3400, operating at 6-10 kV in a conventional back-scattered electron mode and the transmission electron microscopes (TEM) Tesla BS-540 and Jeol-2100

operated at 120, 180 and 200 kV. The thin foils were prepared by the electrochemical polishing in: 260 ml CH₃OH + 35 ml glycerol + 5 ml HClO₄ using Tenupol-3. The chemical composition of the intermetallics was made by energy dispersive spectroscopy (EDS) attached to the SEM manufactured by Thermo Noran. The intermetallic particles from investigated C355.0 alloy 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 in as-cast alloy were identified by using X-ray diffraction analysis. The X-ray diffraction analysis was performed using ARL-XTR's diffractometer - Cu K α radiation at 40 kV.

3. Results and discussion

DSC curves obtained by heating (Fig. 1a) and cooling (Fig. 1b) as-cast specimens of the examined C355.0 alloy are shown in Fig. 1. DSC curves demonstrate precisely each reactions during heating and solidification process.

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 alloy (Fig. 1a). In this material alloying elements such as: Mg, Cu, as well as impurities: Mn, Fe, leads to more complex solidification reaction. Therefore, as-cast microstructure of C355.0 alloy presents a mixture of intermetallic phases (Fig. 2). The solidification reactions (the exact value of temperature) obtained during DSC investigation are presented in Table 3. Results obtained in this work very well corresponding to the [1-3,32]. Solidification process of this alloy starts from formation of aluminum rich (α -Al) dendrites at 610°C. The eutectic mixture of α -aluminum, Si and Al₃FeSi forms at temperature of 564°C. The Al(FeMnSi) phase can precipitate in temperature 532°C. A decrease in the temperature allows for precipitation of Al₂Cu (510°C, 499°C) and Al₅Mg₈Cu₂Si₆ phases (499°C).

Table 3.

Solidification reactions during nonequilibrium conditions in C355.0 alloy, heating rate was 5°C/min

Reactions	Temperature, °C
L→(Al) dendrite network	610
L→(Al)+Si+Al ₃ FeSi	564
L→(Al)+Si+AlMnFeSi	532
L→(Al)+Al ₂ Cu+Al ₃ FeSi	510
L→(Al)+Al ₂ Cu+Si+Al ₅ Mg ₈ Cu ₂ Si ₆	499

Fig. 2 shows as-cast microstructure of C355.0 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). 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). 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. 4).

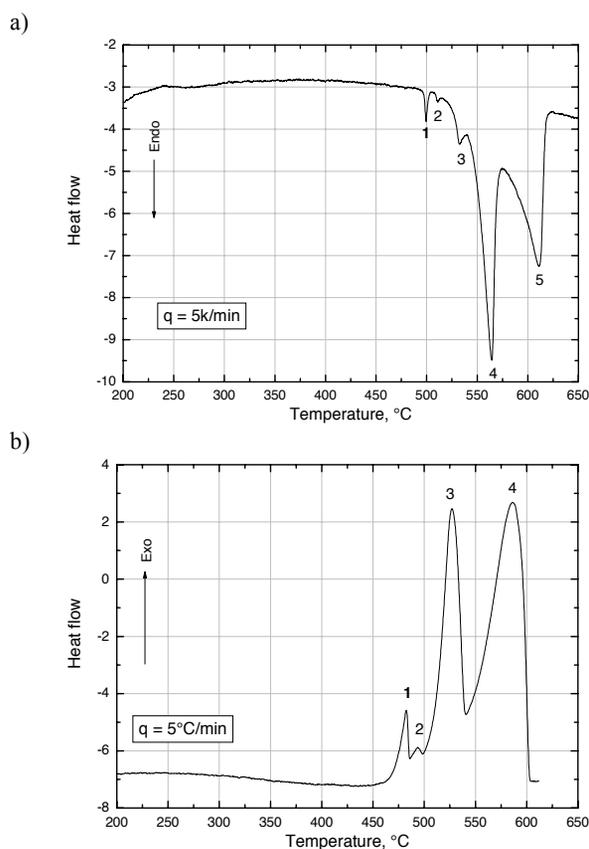


Fig. 1. DSC thermograms of as-cast specimens of C355.0 alloy, obtained during a) heating and b) cooling at rate of 5°C

Fig. 4 shows the SEM micrographs with corresponding EDS-spectra (Table 4) of intermetallics observed in the as-cast C355.0 alloy. The EDS analysis indicate that the oval particles are Al₂Cu (Fig. 4a). Besides Al₂Cu phase, another Cu containing phase Al₅Mg₈Cu₂Si₆ was observed (Fig. 5b). 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) [7], and most of Fe in aluminium alloys

form a wide variety of Fe-containing intermetallics depending on the alloy composition and its solidification conditions [1-7]. In the investigated as-cast C355.0 alloy Fe-containing intermetallics such as light grey needle like β -Al₅FeSi (Fig. 4b) and blockly phase consisting of Al, Si, Mn and Fe (Fig. 4a) were observed. On the basis of literature data [6-9,16,30,32] and EDS results (Fig. 4 and Table 4) these particles were identified as α -Al(FeMn)Si phase.

Since it is rather difficult to produce detailed identification of intermetallic using only one method (e.g. microscopic examination) therefore XRD technique was utilized to provide confidence in the results of phase classification based on metallographic study. The X-ray diffraction spectra from as-cast specimen are shown in Fig. 5.

The analysis of results obtained from the X-ray diffraction spectra were consistent with microscopic examination (Figs. 2-4). Microstructure of C355.0 alloy in T6 condition is presented in Fig. 6. Analyzing the micrographs of the alloy after heat treatment at 520°C for 5 h 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 [16,19,20] as shown in Figures 6 and 7.

The EDS analysis performed on the phases presented in microstructure of the C355.0 alloy in T6 condition (Fig. 7) revealed, that spherical in shape inclusions are the eutectic silicon ones, whereas the rod-like and "Chinese script" shaped, are inclusions of the phase consisting of Al, Si, Mn and Fe (Fig. 2 and Table 3).

During solution heat treatment primary particles Si- and Cu-containing phases: β -Mg₂Si, Al₂Cu and Al₅Cu₂Mg₈Si₆ dissolve in the α -Al matrix. The subsequent aging heat treatment at 150 and 220°C leads to formation from the supersaturated solid solution fine intermetallic strengthening particles of β -Mg₂Si and Al₂Cu (Fig. 6). Analysis of the particles extracted from the C355.0 alloy using phenolic dissolution technique (Fig. 8) confirmed metallographic observation of specimens in T6-condition (Figs. 6 and 7).

The EDS spectra revealed the presence of Al, Mg, Mn, Si, Fe and Cu - bearing particles in the extracted powder (Fig. 8). The EDS analysis results prove that analyzed particles extracted from the C355.0 alloy were: Si, AlMnFeSi, Al₅FeSi, Al₅Mg₈Cu₂Si₆ phases.

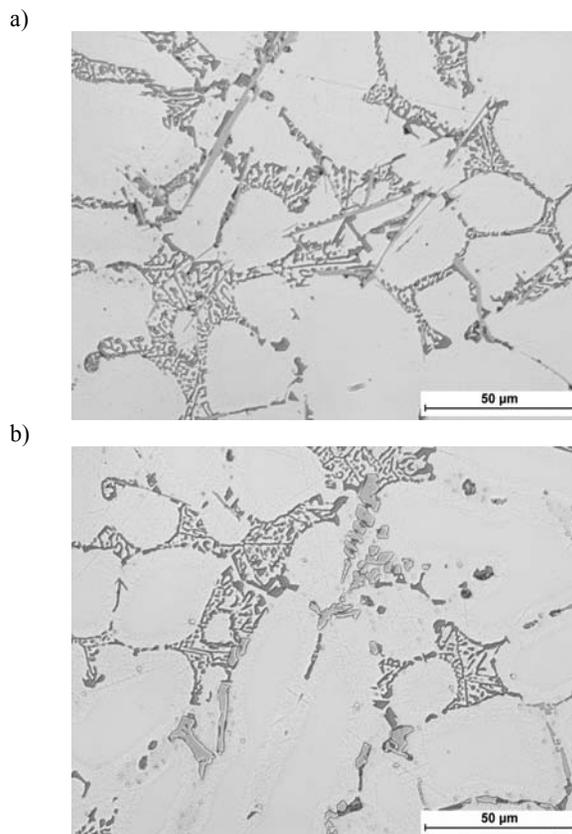


Fig. 2. Morphology of C355.0 alloy in the as-cast state: (a) unetched and (b) etched.

Table 4.

The chemical composition of the intermetallic phases in C355.0 alloy in the as-cast state

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	31.1	33			Ji [34]
		15.2	26.9	29.22			Lodgaard [5]
		17.97	27.48	28.49			This work
25	β -Al ₅ FeSi	12-15			25-30		Mondolfo [7]
		12.2			25		Warmuzek [9]
		14.59			27.75		Liu [16]
		13-16			23-26		This work
12	α -Al ₁₂ (FeMn) ₃ Si	10-12			10-15	15-20	Mondolfo [7]
		5.5-6.5			5.1-28	14-24	Warmuzek [9]
		5-7			10-13	19-23	Liu [16]
		8-12			11-13	14-20	This work
10	Al ₂ Cu		52.5				Belov [32]
			49.51				This work
25	Si	85-95					This work

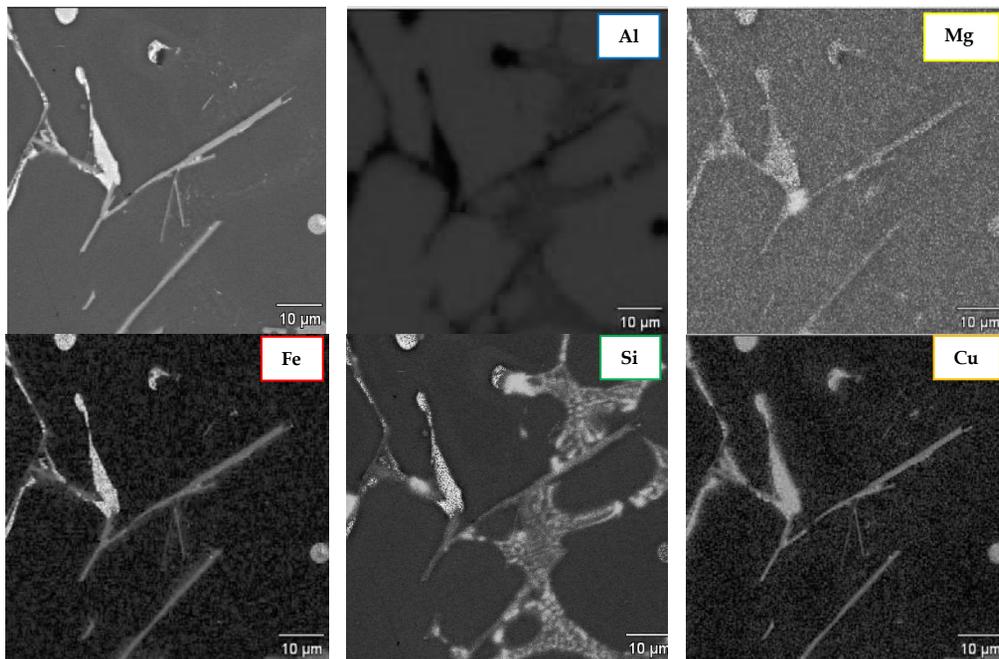


Fig. 3. SEM image of the C355.0 alloy and corresponding elemental maps of: Al, Mg, Fe, Si and Cu

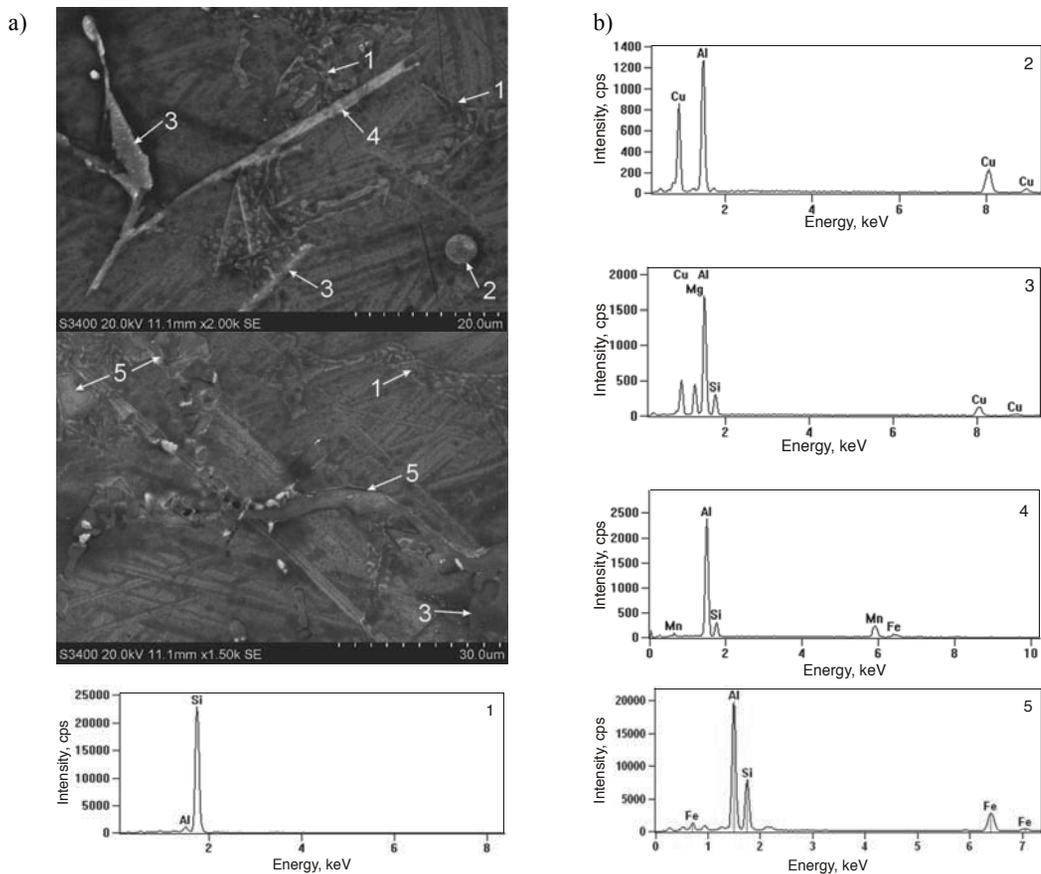


Fig.4. a) SEM micrographs of the C355.0 alloy in the as-cast state; b) the corresponding EDS-spectra acquired in positions indicated by the number 1 and 2.

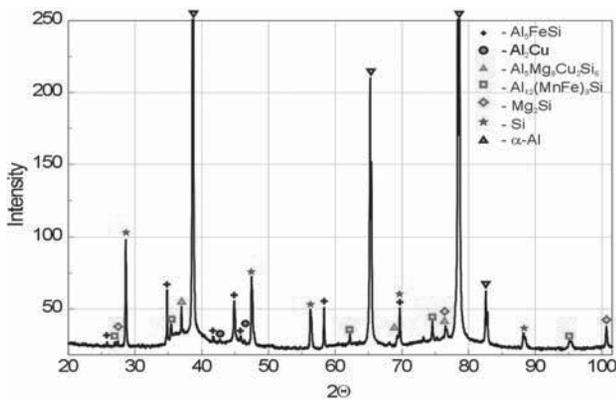


Fig. 5. X-diffraction pattern of C355.0 alloy in as-cast state

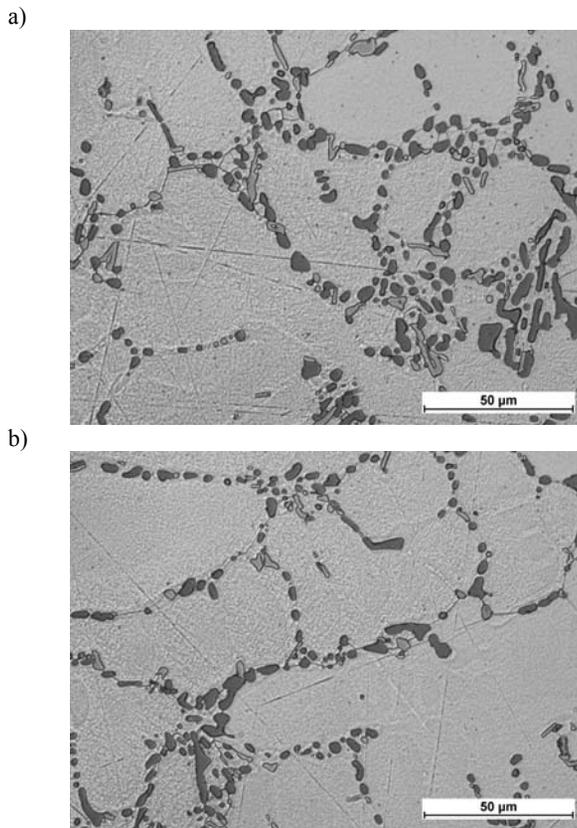


Fig. 6. The microstructure of C355.0 alloy in the T6 condition (a,b)

Previous works [9,17,23,25,30] and literature data [1-7] reveals that the presence of β (Mg_2Si) and θ - Al_2Cu phases and precipitates of intermetallic phases containing Si, Fe and Mn plays significant role in mechanical properties increasing. Fig. 9 shows the effect of artificial aging time on hardness of C355.0 alloy. It can clearly be seen that the hardness of examined alloy depends on the temperature and time of aging. The hardness of samples aged at 150°C increases with the prolongation of aging time (Fig. 9). The hardness of samples aged at 220°C initially

increased rapidly with increasing the aging time reaching the peak value, after which hardness decreased. Maximum hardness was received after aging at 150°C - 116HB for 48 h and 220°C - 102HB for 6 h (Fig. 9).

The strength (yield and tensile strength) and plastic (elongation) properties of C355.0 alloy after solutionizing and artificial aging at various temperatures were determined from static tensile test. The results of these tests are presented in summary Table 5. It can be observed that as the aging time increases, a continuous increase in tensile strength, with approximately no elongation changes is noticed. An intensive hardening increment of the alloy in a relatively short time of aging not exceeding the value of 10 h - 220°C and 15 h - 150°C, followed by further almost uniform increase in the mechanical properties within the range of longer aging times can be observed.

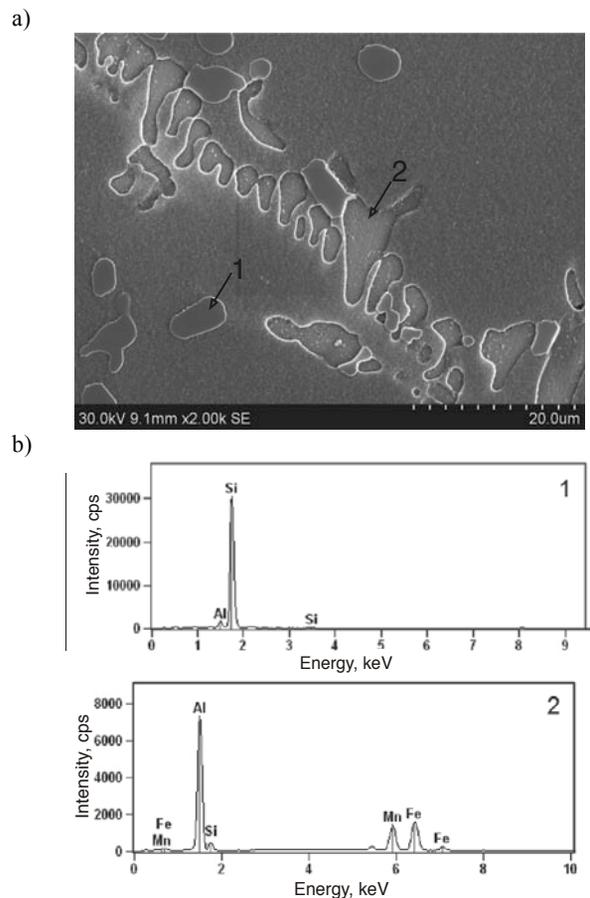


Fig. 7. a) SEM micrographs of the C355.0 alloy in the T6 condition; b) the corresponding EDS-spectra acquired in the positions indicated by the number 1 and 2.

Changes in mechanical properties of the alloy treated at aging temperature of 150°C and 220°C are shown in Fig. 10. These results show that tensile properties of the C355.0 alloy aged at higher temperature - 220°C were almost the same as the properties obtained after aging at 150°C, and the trend of both

yield strength and tensile strength variation due to aging time in principle did not change (Fig. 10a,b).

Strength of the alloy continues to increase up to 10 hours - 220°C and 15 hours - 150°C of aging, after which there is no visible increase in mechanical properties. In samples aged at 220°C longer than 20 hours decrease of tensile strength were observed (Fig. 10a). Additionally slightly decrease of yield strength of the alloy aged in both temperature was observed (Fig. 10b). After first 10 hours of aging, a simultaneous

deterioration of plastic properties with the increase in tensile and yield strength, was observed (Fig. 10c). However with elongation of aging time, the plastic properties increases with increasing time.

The initial increase in the tensile strength and yield strength is due to vacancies assisted diffusion mechanism and formation of high volume fraction of (GP) zones followed by formation precipitation of metastable strengthening phases θ' , β'' and/or θ'' , β' precursors of the equilibrium β -Mg₂Si and θ -Al₂Cu phases [2-7,32].

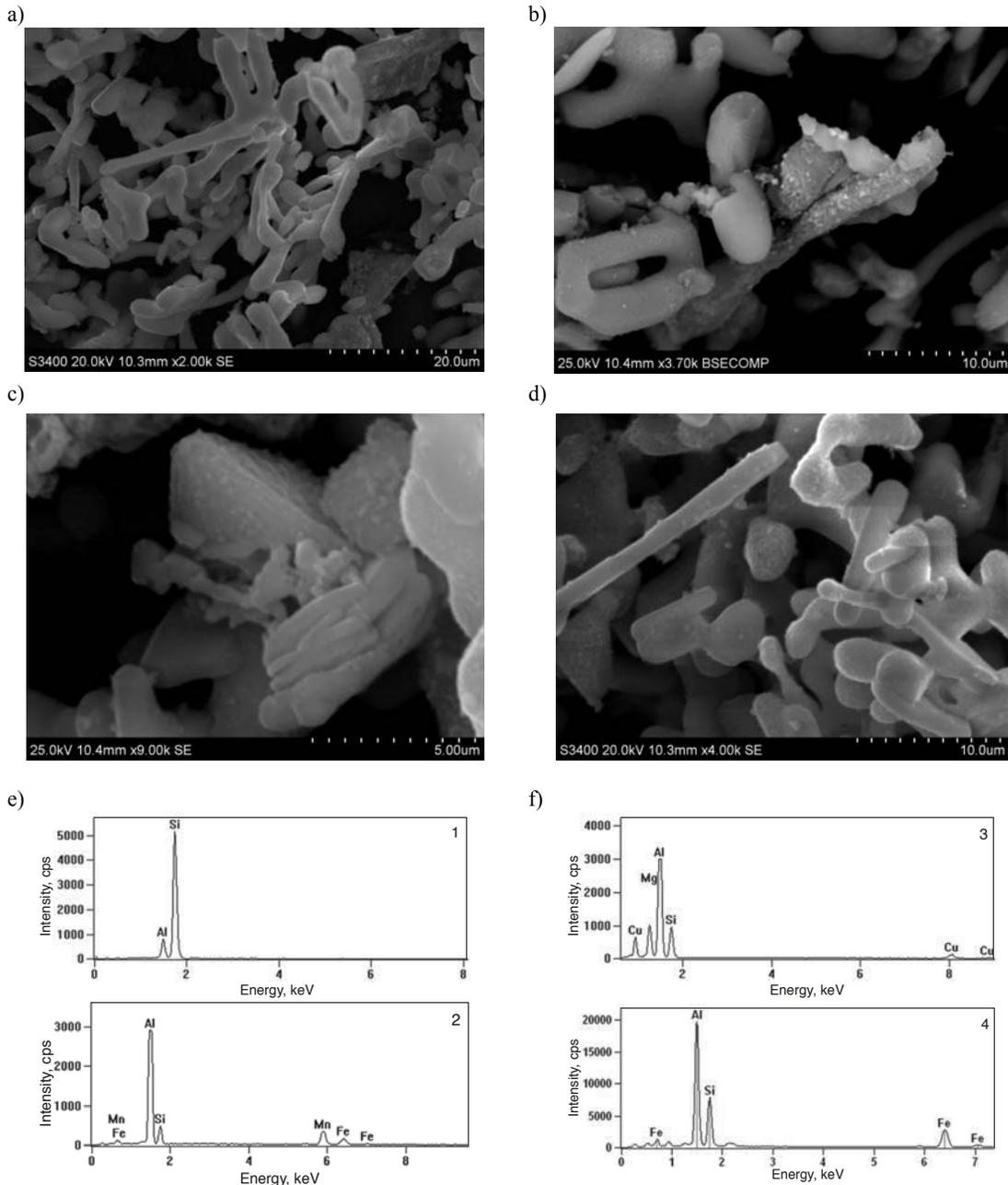


Fig. 8. SEM micrographs (a-d) of the particles extracted from the C355.0 alloy and EDS spectra (e,f)

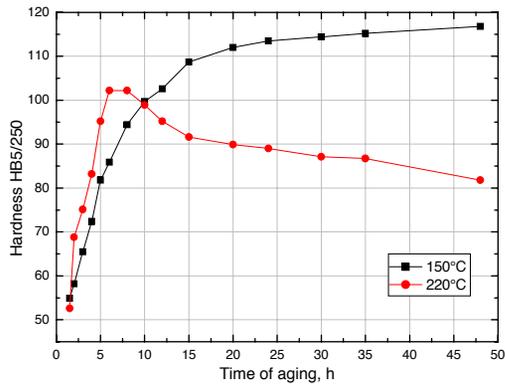


Fig. 9. Variation of hardness with time of aging of investigated alloy at temperature of 150 and 220°C

TEM observation of the microstructure of C355.0 alloy in T6 state and the selected area electron diffraction patterns analysis proved that the dispersed precipitates shown in Fig. 6 were the precipitates of hardening phase β -Mg₂Si (Fig. 11) and θ' -Al₂Cu (Fig. 12).

Table 5. The effect of the temperature and time of aging on the mechanical properties of C355.0 alloy

Aging temperature, °C	Aging time, h	Mechanical properties		
		Yield Strength, MPa	Tensile Strength, MPa	Elongation, %
150	3	148.2	153.2	9.0
	5	159.7	185.7	8.1
	7	172.8	230.0	7.8
	10	190.0	269.2	7.6
	15	202.3	397.0	8.0
	21	205.1	307.3	8.1
	32	204.7	306.9	8.3
	42	202.8	300.6	8.2
220	3	165.1	187.1	8.3
	5	178.6	242.6	7.6
	7	188.4	282.8	7.5
	10	208.0	294.9	7.7
	15	211.9	294.9	8.2
	21	211.9	290.7	8.4
	32	211.9	281.3	8.6
	42	211.6	272.0	8.9

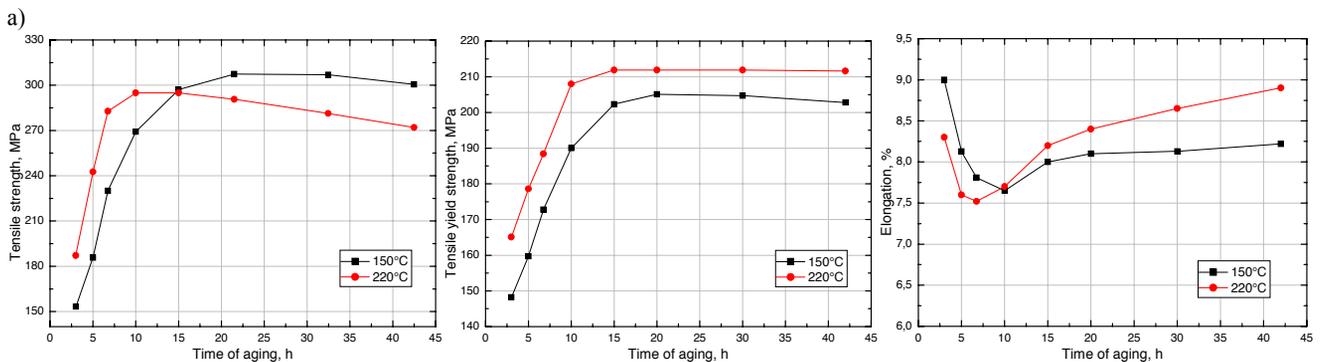


Fig. 10. Effect of aging time on: a) tensile strength, b) tensile yield strength and c) elongation of C355.0 aluminium alloy aged at 150°C and 220°C



Fig. 11. TEM micrograph of C355.0 alloy in T6 conditions showing the precipitate of the β -Mg₂Si phase (a,b), and corresponding electron diffraction pattern (c)

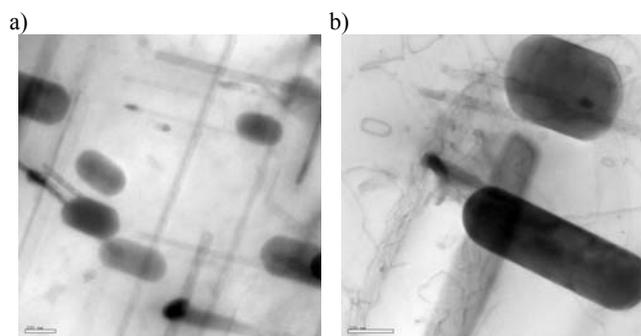


Fig. 12. Precipitation of strengthening β -Mg₂Si i θ' -Al₂Cu phases in C355.0 (a,b) - TEM

4. Conclusions

- The present study allowed to identified in the as-cast C355.0 alloy five intermetallic phases: Si, Al₅FeSi, α -AlMnFeSi, Al₂Cu, Al₅Cu₂Mg₈Si₆. These phases are the products of following reactions:
 $L \rightarrow (Al)$ dendrite network
 $L \rightarrow (Al) + Si + Al_5FeSi$
 $L \rightarrow (Al) + Si + AlMnFeSi$
 $L \rightarrow (Al) + Al_2Cu + Al_5FeSi$
 $L \rightarrow (Al) + Al_2Cu + Si + Al_5Cu_2Mg_8Si_6$
- Microscopic examination (LM, SEM, TEM) shows that during solution heat treatment a primary particles Si- and Cu-containing phases: Al₂Cu and Al₅Cu₂Mg₈Si₆ dissolve in the α -Al matrix. During subsequent aging heat treatment at 150 and 220°C the formation of fine intermetallic strengthening particles of β -Mg₂Si and Al₂Cu from the supersaturated solid solution were observed.
- The highest mechanical properties (hardness, yield and tensile strength) connected with a good plastic properties was achieved for C355.0 alloy during ageing at 150°C. Maximum value of hardness was obtained after aging at 150°C - 116HB for 48 h and 220°C - 102HB for 6 h. However the tensile properties of the C355.0 alloy aged at higher temperature - 220°C were almost the same (max. R_{0.2} - 211.9 MPa, R_m - 295 MPa) as those of the alloy after aging at 150°C (max. R_{0.2} - 205 MPa, R_m - 307 MPa). Strength of the alloy continues to increase up to 10 hours - 220°C and 15 hours - 150°C of aging, after which there is no visible increase in mechanical properties. In samples aged at 220°C longer than 20 hours decrease of tensile strength were observed.

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