

Vpliv toplotne obdelave na razvoj lastnosti pri zlitini AlSi7Mg(Cu)

Influence of Heat Treatment on AlSi7Mg(Cu) Alloy Properties Development

Izvleček

Zasnova materialov skozi izbor kemijske sestave, termodinamično modeliranje, obdelava s cepljenjem in modificiranjem, pravičen razvoj tehnologije za litje, ki mu sledi ustrezna toplotna obdelava, bi lahko izboljšali lastnosti ulitkov. Zaradi zapletenih geometrij ulitkov in značilnosti strank so proizvajalci ulitkov prisiljeni k uvajanju proizvodnih postopkov, ki vključujejo dodatne korake, npr. toplotno obdelavo. Zahteven postopek, ki poteka pri razmeroma visoki temperaturi in je poleg tega še dolgotrajen, učinkuje na mikrostrukturne spremembe materiala ter tako neposredno vpliva na njegove mehanske lastnosti. Pomemben vidik za nove izboljšave in uporabe je poglobitev razumevanja spreminjanja mikrostrukturnih sestavin kot posledice toplotne obdelave in posledično izboljšanih mehanskih lastnosti. Toplotno obdelavo spremljajo povišani stroški in daljši proizvodni časi, ki vodijo do slabše konkurenčnosti proizvajalcev ulitkov. Primerjava pridobljenih lastnosti navadnih ulitkov ter toplotno obdelanih ulitkov razkriva, da so uvedeni proizvodni postopki tesno povezani z denarnimi in časovnimi naložbami v odnosu z doseženimi izboljšavami lastnosti.

Običajne zlitine AlSi7Mg so pogosta izbira za proizvodnjo ulitkov zapletenih geometrij. Snovanje novih kemijskih sestav zlitine AlSi7Mg(Cu) z dodatkom Cu (do 1,435 wt.%) predstavlja izziv na poti k doseganju naprednih mehanskih lastnosti že pri običajnih, torej neobdelanih ulitkih. Širok razpon zapletenih reakcij in intermetalnih faz izhajata iz številnih interakcij legirnih elementov (Si, Mg, Cu) in elementov v sledovih (Fe, Mn). Toplotna obdelava učinkuje na spremembo morfologije železnih faz in izboljšavo mikrostrukturne sestavine ter izboljša kovinsko matriko sekundarnih elementov za legiranje, kot sta Cu in Mg. Natančna določitev vedenja zlitine je bila izvedena z modeliranjem faznega diagrama ravnovesja, sočasne toplotne analize in metalografskih raziskav obeh stanj, torej tako običajnih kot toplotno obdelanih ulitkov. Toplotno obdelane mikrostrukture se spremenijo, npr. prečistijo se, v njih pa poteče enakomerna porazdelitev mikrosestavin, izboljša se kovinska matrika magnezija in bakra ter posledično fragmentacije faz, ki se strdijo nazadnje.

Inovativna kemijska sestava in znano zaporedje strjevanja določenih faz ter njihova morfologija in porazdelitev pa pomenijo znatno izboljšanje nateznih mehanskih lastnosti že pri običajnih ulitkih.

Toplotna obdelava ne vpliva na bistveno izboljšanje mehanskih lastnosti.

Ključne besede: zlitina AlSi7Mg(Cu), baker, mikrostruktura, toplotna obdelava, mehanske lastnosti

Abstract

The design of materials through the selection of the chemical composition, thermodynamic modelling, melt treatment by inoculation and modification, correctly developed casting

technology followed by adequate heat treatment could improve casting properties. Due to castings complex geometry and customer properties, casting manufacturers are forced to adopt production process with additional steps such as heat treatment. Rigorous regime with relatively high temperature and long holding time indicates the microstructural changes in the material, thus indirectly affects the mechanical properties of the material. An important aspect for further improvement and application is to develop a better understanding of the microstructural constituent changes due to performed heat treatment and consequently improved mechanical properties. Heat treatment is accompanied with increased costs and longer time for total production, which cause lower competitiveness of casting producer per produced casting. Comparison of obtained properties in as-cast and heat-treated state reveals an evaluation of applied production procedures, closely connected with investments in money and time vs. obtained properties improvement.

Conventional AlSi7Mg alloy represents a frequent selection for complex geometry castings production. Designing of new chemical composition of AlSi7Mg(Cu) alloy with extra addition of Cu (up to 1.435 wt.%) represents a challenge in order to achieve advanced mechanical properties already in as-cast state. A wide range of complex reactions and intermetallic phases occurs due to numerous alloying (Si, Mg, Cu) and trace elements (Fe, Mn) interaction. Performed heat treatment influences on iron bearing phases' morphology change, refining of microstructural constituents and enrichment of metal matrix on secondary alloying elements such as Cu and Mg. Exact determination of alloy behaviour was performed by modelling of equilibrium phase diagram, simultaneous thermal analysis and metallographic investigations in both states, as-cast and heat-treated, respectively. Heat-treated microstructure experiences the change like refining and uniform distribution of microconstituents, metal matrix enrichment on magnesium and copper, and therefore fragmentation of last solidifying phases'.

Innovated chemical composition and determined solidification sequence of particular phases as well as their morphology and distribution comprehend to significant increase of tensile mechanical properties already in as-cast state. Heat treatment did not influenced on significant improve of mechanical properties.

Keywords: AlSi7Mg(Cu) alloy, copper, microstructure, heat treatment, mechanical properties

1 Uvod

Varnostni in obremenjeni ulitki iz aluminijeve zlitine so izpostavljeni visokim zahtevam na tržišču glede kakovosti materiala in lastnosti. Kakovost aluminijevih ulitkov je v veliki meri odvisna od procesa litja in parametrov, pa tudi kemijske sestave, ki pomembno vpliva na njihove mehanske lastnosti [1]. Zato postaja inovativen razvoj zelo trdnih aluminijevih zlitin obvezna sestavina strukturnih komponent v avtomobilski industriji.

1 Introduction

Safety loaded aluminium alloy castings have been exposed to high market demands related to material and properties quality as a whole. The quality of aluminium components mainly depends from casting process and parameters, as well as from chemical composition, which significantly affects the mechanical properties [1]. Therefore, the innovative development of high strength aluminium alloys becomes

Zaradi odlične livnosti in dobrega razmerja med trdnostjo in težo, zlasti v primeru toplotno obdelanih ulitkov, je tradicionalna zlitina AlSi7Mg pogosta izbira pri izdelavi ulitkov zapletenih geometrij z visokimi varnostnimi zahtevami. Te zlitine so primerne za široko paleto aplikacij, kot so hladilni ventilatorji motorjev, ohišja motorjev, dele, ki se vrtijo z visoko hitrostjo, strukturne letalske komponente, črpalke za gorivo, ohišja kompresorjev itd. [2, 3]. Strjevalno zaporedje se pri hipoevtektičnih zlitinah AlSi7Mg začne z razvojem primarnih aluminijevih dendritov α_{Al} in nastankom dendritne mreže, sledi pa evtektična reakcija ($\alpha_{Al} + \beta_{Si}$) na primarnih zrnih α_{Al} ali samostojno na prisotnem nosilcu železa in/ali drugih nečistočah nukleantov z drugačno kristalografsko usmerjenostjo [4]. Način nastanka evtektika določa obseg in morfologijo evtektične faze, pa tudi delež poroznosti v mikrostrukturi. Glavni in najpomembnejši legirni element pri klasični zlitini AlSi7Mg je Si, za katerega sta značilni visoka pretočnost in zmanjšanje krčenja, sledi pa mu Mg, ki je odgovoren za večjo trdnost zlasti v primeru toplotne obdelave [5–7]. Vsebnost sekundarnih legirnih elementov (Mg, Cu) pomembno vpliva na postopek strjevanja zlitine Al-Si in izboljšanje njenih lastnosti, zlasti trdnosti pri sobni in zvišani temperaturi [8–17].

Dodani magnezij do 0,7 wt.% krepi precipitacijo evtektične faze α_{Al} -Mg₂Si in/ali intermetalnih spojin, bogatih z Mg [17, 18]. Pomembno zvečanje trdnosti pri visoki temperaturi smo dosegli po obdelavi raztopine zaradi aktivacije strjevanja pri precipitaciji z razvojem magnezijevih faz. Večjo trdnost zlitin, ki vsebujejo Mg, pri visoki temperaturi je mogoče pripisati samo precipitaciji sekundarne faze Mg₂Si [17]. Glede na vsebnost Mg se lahko trdnost teženja, natezna trdnost in elongacija zlitin Al-Si-Mg v litem stanju spreminjajo. Trdnost

an imperative for structural components in automotive industry.

Excellent castability and favourable relation between strength and weight, especially in the heat-treated state, indicate conventional AlSi7Mg alloy as a frequent choice for complex geometry castings with high properties demand. These alloys are suitable for a wide range of applications, such as engine cooling fans, crank cases, high speed rotating parts, structural aerospace components, fuel pumps, compressor cases, etc. [2,3]. The solidification sequence of hypoeutectic AlSi7Mg alloys begins with development of primary aluminum dendrites α_{Al} and formation of dendritic network, followed by eutectic reaction ($\alpha_{Al} + \beta_{Si}$) on the primary grains α_{Al} or independently on present iron bearing and/or other impurities nucleants with different crystallographic orientation [4]. The way of eutectic occurs determines the amount and morphology of eutectic phase, and also the porosity ratio in the microstructure. The primary and most important alloying element in conventional AlSi7Mg alloy is Si, which is characterized by high fluidity and reduction in shrinkage, followed by Mg responsible for strength increase especially in heat-treated state [5-7]. Secondary alloying elements content (Mg, Cu) significantly influences on solidification manner of an Al-Si alloy and their properties improvement, especially in strength at room and elevated temperatures [8-17].

Magnesium addition up to 0.7 wt. % has a strengthening effect by precipitation of α_{Al} -Mg₂Si eutectic phase and/or Mg-rich intermetallics [17, 18]. The significant increase in strength at high temperature was achieved after solution treatment due to activating precipitation hardening through evolution of Mg bearing phases. The increase in high temperature strength of the Mg containing alloys can only be

teženja se povečuje ob večji vsebnosti Mg. Nasprotno pa večje fazno razmerje Mg_2Si nima bistvenega vpliva na pridobljene vrednosti z vidika trdnosti. Obratno pa se elongacija z zvečanjem razmerja Mg in Mg_2Si zmanjša [11].

Tudi Cu se pogosto uporablja kot legirni element za večjo trdnost litih zlitih, zlasti pri toplotni obdelavi. Pri zlitinah Al-Si je Cu dodan v razmerju med 1,5–3,5 wt.% in zato ustvarja intermetalno fazo Al_2Cu in/ali druge bakrove intermetalne faze, kot je Al_2CuMg [12–14]. Baker povečuje obseg strjevanja zlitine in omogoča boljše pogoje za nastanek poroznosti [19, 20].

Literatura navaja številne raziskave vpliva legirnih elementov na lastnosti zlitine AlSi7Mg [21–23]. Soodvisnost modeliranja faznega diagrama ravnovesja, rezultatov toplotne analize in prepoznavanje mikrosestavin omogočajo določitev termodinamične stabilnosti zlitine in njenega vedenja v litem stanju ter po toplotni obdelavi [24]. Klasična zlitina AlSi7Mg, ki je skladna s številnimi standardi (EN 1706, IDM 4234) [25, 26], je bila predhodno predmet raziskave [27, 28]. Kemijska sestava, ki jo zahteva standard, je prikazana v Preglednici 1.

Standard avtomobilskih proizvajalcev [26] omejuje dovoljeno vsebnost določenih elementov, kot je magnezij, povečuje pa dovoljeno vsebnost železa, faze katerega pri ustreznih morfologijah pomenijo večjo trdnost aluminijevih zlitin. Zahtevane mehanske lastnosti vzorcev iz zlitine

atributed to the precipitation of secondary phase Mg_2Si [17]. The yield strength, tensile strength and elongation of the as-cast Al-Si-Mg alloys can vary with the Mg content. The yield strength increases with increasing of Mg content. In contrary, an increase of Mg_2Si phase ratio does not affect significantly obtained strength values. Conversely, the elongation was decreased with an increase of Mg and Mg_2Si ratio [11].

Also, Cu is commonly used as an alloying element to increase the strength of cast alloys, especially when heat treatment is applied. In Al-Si alloys, Cu is usually added in levels between 1.5 – 3.5 wt.% thus forming the intermetallic phase Al_2Cu and/or other Cu-bearing intermetallics like Al_2CuMg [12–14]. Copper increases the solidification range of an alloy, and facilitates the condition of porosity formation [19, 20].

Literature survey reveals a number of investigations related to the influence of alloying elements on the AlSi7Mg alloy properties [21–23]. Correlation of equilibrium phase diagram modelling, thermal analysis results and microconstituents identification enables determination of thermodynamic stability of an alloy and its behaviour in as-cast and heat-treated state [24]. The conventional AlSi7Mg alloy, corresponded to the numerous standards (EN 1706, IDM 4234) [25, 26], has been investigated previously [27, 28]. Chemical composition requested by standard is presented in Table 1.

Preglednica 1. Kemijska sestava zlitine AlSi7Mg [25]

Table 1. Chemical composition of AlSi7Mg alloy [25]

Standardna oznaka / Standard mark	wt. %	Kemijski element / Chemical element									
		Si	Fe	Cu	Mn	Mg	Zn	Ti	Ni	Pb	Bal.
EN AC 42000	min.	6,5				0,20		0,05			
	maks.	7,5	0,55	0,20	0,35	0,65	0,15	0,25	0,15	0,15	0,15

AlSi7Mg v litem stanju so prikazane v Preglednici 2 [25].

Preglednica 2. Mehanske lastnosti kokilno litih vzorcev iz zlitine AlSi7Mg v litem stanju [25]

Table 2. Mechanical properties of AlSi7Mg alloy of die cast samples in as-cast state [25]

	$R_{p0.2}$ [MPa]	R_m [MPa]	A50 [%]
EN AC 42000	90	170	2,5

Namen te raziskave je bil določiti vpliv toplotne obdelave na spreminjajoče lastnosti zlitine AlSi7Mg(Cu). Vedenje zlitine in mehanske lastnosti so bili določeni s predhodno določitvijo strjevalnega zaporedja s pomočjo termodinamičnega modeliranja in sočasne toplotne analize. Učinek toplotne obdelave je bil ocenjen na podlagi razvoja mikrostrukture in preiskav mehanskih lastnosti ter njihovi primerjavi s podatki, pridobljenimi v litem stanju.

2 Poskusni postopek

Lastnosti na novo zasnovane zlitine AlSi7Mg(Cu) so bile opredeljene v litem stanju in po toplotni obdelavi. Raziskava zajema razvoj faznega diagrama ravnovesja, sočasno toplotno analizo ter analizo mikrostrukturnih in mehanskih lastnosti.

Modeliranje faznega diagrama ravnovesja smo opravili s programom ThermoCalc (TCW 5.0). Talino AlSi7Mg(Cu) smo pripravili v indukcijski peči ABB IMTK 2000, obdelava taline pa zajema cepljenje s predzlitino AlTi5B in modifikacijo s predzlitino AlSr10. Analizo kemijske sestave smo opravili z optičnim spektrometrom ARL-3460.

Talino novo zasnovane zlitine AlSi7Mg(Cu) smo pripravili v indukcijski peči ABB IMTK 2000 z ingoti in povratnim razmerjem dovajanega materiala 1:1. Po

Automotive producers standard [26] naravnost dovoljeno vsebnost za posamezne elemente, kot so Mg, pri čemer se povečuje dovoljena vsebnost Fe, katerih faze v ustreznih morfologijah prispevajo k povečanju trdnosti aluminijevih zlitin. Zahtevane mehanske lastnosti vzorcev AlSi7Mg zlitine v litem stanju so prikazane v Preglednici 2 [25].

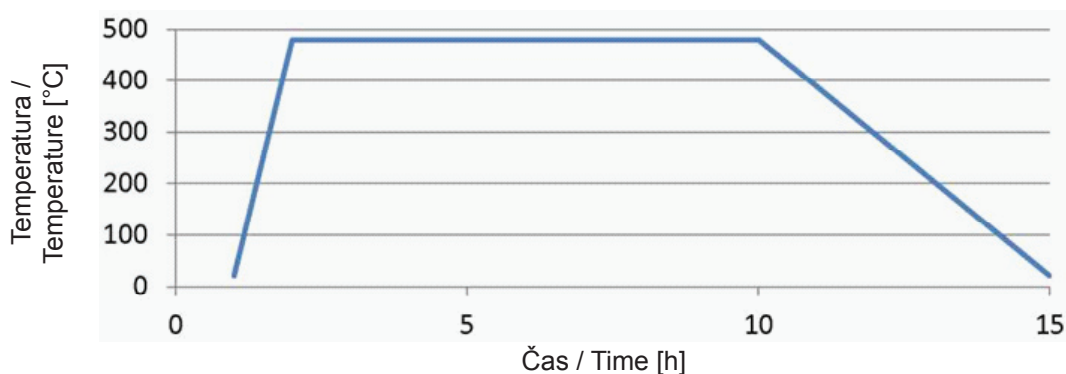
Cilj tega raziskovanja je bil določiti vpliv toplotne obdelave na inovativno AlSi7Mg(Cu) zlitino. Lastnosti zlitine in mehanske lastnosti so bile določene s predhodno določitvijo strjevalnega zaporedja s pomočjo termodinamičnega modeliranja in sočasne toplotne analize. Učinek toplotne obdelave je bil ocenjen na podlagi razvoja mikrostrukture in preiskav mehanskih lastnosti ter njihovi primerjavi s podatki, pridobljenimi v litem stanju.

2 Experimental

Characterization of newly designed AlSi7Mg(Cu) alloy has been performed in as-cast and heat-treated state. Investigation comprehends the development of an equilibrium phase diagram, simultaneous thermal analysis, microstructural, and mechanical properties analysis.

The modelling of an equilibrium phase diagram has been performed using ThermoCalc (TCW 5.0) program. The melt of AlSi7Mg(Cu) was prepared in an induction furnace ABB IMTK 2000 Melt treatment comprehends inoculation with AlTi5B and modification with AlSr10 master alloy. Chemical composition analysis was obtained on an optical spectrometer ARL-3460.

Newly designed AlSi7Mg(Cu) alloy melt was prepared in an induction furnace ABB IMTK 2000 with the ingot and return ratio



Slika 1. Režim toplotne obdelave

Figure 1. Heat treatment regime

topljenju pri temperaturi $770 \pm 5^\circ\text{C}$ smo talino razplinili z nitrogenom (N_2) s pomočjo opreme MTS 1500 – Foseco. Talino smo obdelali s cepljenjem s predzlitino AlTi5B in modifikacijo s predzlitino AlSr10 . Analizo kemijske sestave smo opravili z optičnim spektrometrom ARL-3460.

Toplotna obdelava je potekala po naslednjem postopku: segrevanje od sobne temperature do temperature žarjenja $480^\circ\text{C} - 2$ uri, ohranjanje končne temperature žarjenja – 8 ur, sledilo je zračno hlajenje, kot je prikazano na Sliki 1.

Vzorci za metalografsko raziskavo smo pripravili s standardnim metalografskim postopkom za pripravo z mletjem in poliranjem, sledilo pa je jedkanje v 0,5-odstotni fluorovodikovi kislini. Metalografske raziskave zajemajo metalografske raziskave (Olympus GX 51) in mikrostrukturne raziskave (vrstični elektronski mikroskop Tescan Vega TS 5136 MM).

Sočasno toplotno analizo z metodo diferencialne toplotne analize smo opravili z izvajanjem segrevanja in hlajenja pri hitrosti 10 K/min z opremo Netzsch STA 409 C/CD, da bi določili pomembne temperaturne vrednosti pri faznih transformacijah in precipitaciji.

in charge material 1: 1. After melting at a temperature of $770 \pm 5^\circ\text{C}$, the degassing of the melt was performed with the nitrogen (N_2) using a MTS 1500 - Foseco equipment. Melt treatment was performed through inoculation with AlTi5B master alloys and modification with AlSr10 master alloy. Chemical composition analysis was performed on an optical emission spectrometer ARL-3460.

Heat treatment was performed following the regime: heating, starting from room temperature to the annealing temperature of 480°C for 2 hours, and the retention of the final annealing temperature during 8h, followed by air cooling, as shown in Figure 1.

Samples for metallographic investigation were prepared by standard metallographic preparation procedure by grinding and polishing, followed by etching in 0.5% HF. Metallographic investigations comprehends metallographic investigations (Olympus GX 51) and microstructural investigations (scanning electron microscopes Tescan Vega TS 5136 MM).

A simultaneous thermal analysis by differential thermal analysis method was performed by heating and cooling rates

Raziskave mehanskih nateznih lastnosti smo opravili na preizkusni napravi MTS 810 pri sobni temperaturi $T = 20\text{ }^{\circ}\text{C}$ skladno s standardom EN 10002-1: 1998 [29].

3 Rezultati in razprava

Novo kemijsko spojino zlitine AlSi7Mg(Cu) z dodatkom bakra smo zasnovali in primerjali s predhodno raziskano klasično zlitino AlSi7Mg [28], kot je prikazano v Preglednici 3.

Novo zasnovana zlitina je skladna s standardom EN 42000 AC za zlitino AlSi7Mg v povezavi z vsebnostjo osnovnih legirnih elementov (Si in Mg) ter elementov v sledih, kot sta Fe in Mn. Odklon je bil uveden z bistvenim zvečanjem vsebnosti Cu.

Modeliranje novo zasnovane zlitine AlSi7Mg(Cu) s programom ThermoCalc (TCW 5.0) je potekalo skladno s predhodno izračunanim faznim diagramom ravnovesja [29]. Interakcija legirnih elementov in elementov v sledovih odkriva širok nabor intermetalnih faz, ki jim sledi izračun strjevalnega zaporedja zlitine AlSi7Mg(Cu). Izračunano strjevalno zaporedje ravnovesja zlitine AlSi7Mg(Cu) je prikazano v Preglednici 4.

Mikrostrukturo zlitine AlSi7Mg(Cu) v litem stanju (F) in po toplotni obdelavi (T) smo raziskali s pomočjo svetlobne mikroskopije, kot je prikazano na Sliki 2.

Manjša povečava (100-kratna) kaže enakomerno porazdeljenost primarne dendritne mreže z enakomerno

of 10 K/min at Netzsch STA 409 C / CD equipment in order to reveal significant temperature of phase transformations and precipitation.

Mechanical tensile properties investigations were performed on testing machine MTS 810, at room temperature $T = 20\text{ }^{\circ}\text{C}$ in accordance to EN 10002-1: 1998 [29].

3 Results and Discussion

New chemical composition of AlSi7Mg(Cu) alloy with extra addition of copper has been designed and compared with previously investigated conventional AlSi7Mg alloy [28], as shown in Table 3.

The newly designed alloy complies with the EN 42000 AC standard for AlSi7Mg alloy in relation to the content of the base alloying elements (Si and Mg), and trace elements such as Fe and Mn. Deviation has been implemented with significant increase in Cu content.

Modelling of newly designed AlSi7Mg(Cu) alloy by ThermoCalc (TCW 5.0) program resulted with previously calculated equilibrium phase diagram [29]. The interaction of alloying and trace elements reveals a wide range of intermetallic phases, followed with calculation of solidification sequence of AlSi7Mg(Cu). Calculated equilibrium solidification sequence of AlSi7Mg(Cu) alloy is shown in Table 4.

Microstructure of AlSi7Mg(Cu) alloy in as-cast (F) and heat-treated (T) state was

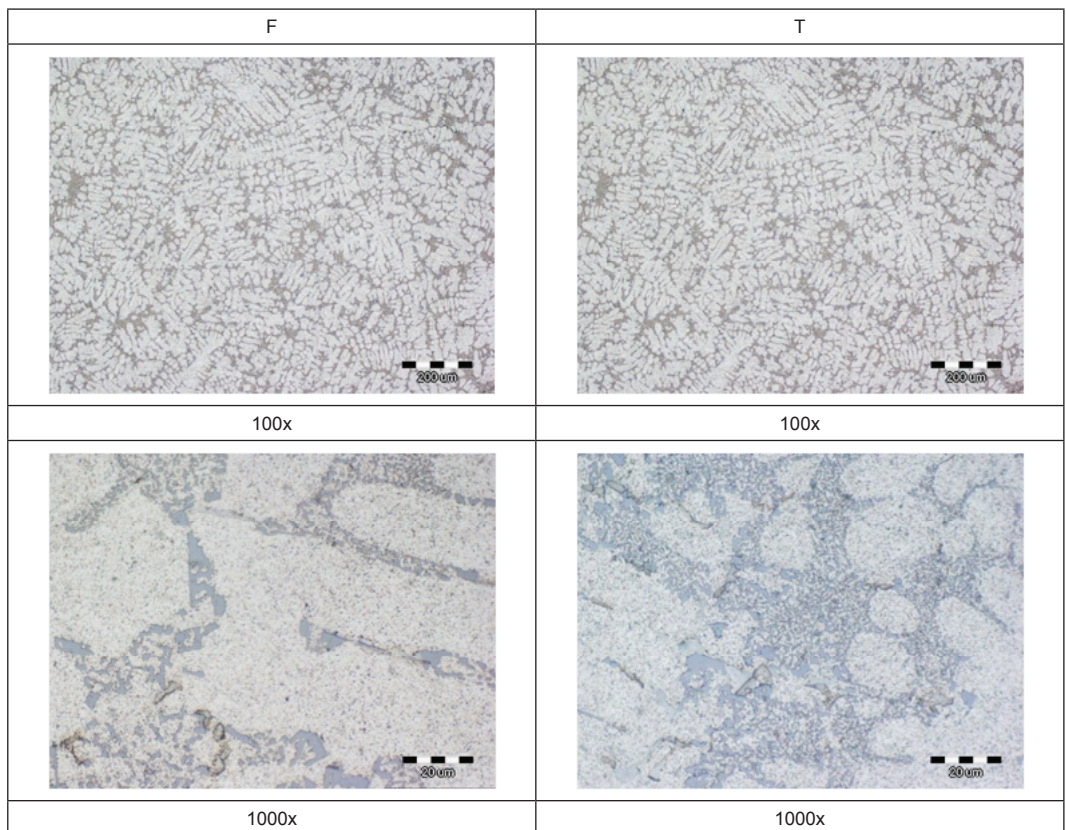
Preglednica 3. Kemijska sestava zlitin AlSi7Mg/AlSi7Mg(Cu)

Table 3. The chemical compositions of AlSi7Mg / AlSi7Mg(Cu) alloys

Element, wt. %	Si	Fe	Cu	Mn	Mg	Ti	Sr
AlSi7Mg	7,008	0,101	0,130	0,010	0,320	0,139	0,0121
AlSi7Mg(Cu)	7,527	0,235	1,435	0,076	0,348	0,147	0,0223

Preglednica 4. Izračunano strjevalno zaporedje ravnovesja zlitine AlSi7Mg(Cu)**Table 4.** Calculated equilibrium solidification sequence of AlSi7Mg(Cu) alloy

Opis reakcije	Reaction description	Reakcija
Temperatura likvidusa, T_l	Liquidus temperature, T_l	$L \rightarrow L_1 + \alpha\text{Al}$
Evteksična temperatura, T_e	Eutectic temperature, T_e	$L_1 + \alpha_{\text{Al}} \rightarrow L_2 + \alpha_{\text{Al}} + (\alpha_{\text{Al}} + \beta_{\text{Si}})$
Temperatura pri precipitaciji sekundarnih intermetalnih faz, T_1	Precipitation of secondary intermetallic phases temperature, T_1	$L_2 + (\alpha_{\text{Al}} + \beta_{\text{Si}}) \rightarrow L_3 + (\alpha_{\text{Al}} + \beta_{\text{Si}}) + \text{Al}_{15}(\text{FeMn})_3\text{Si}_2$
Temperatura pri precipitaciji sekundarnih intermetalnih faz, T_2	Precipitation of secondary intermetallic phases temperature, T_2	$L_3 \rightarrow L_4 + \text{Al}_5\text{Cu}_2\text{Mg}_8\text{Si}_6$
Temperatura pri precipitaciji sekundarnih intermetalnih faz, T_3	Precipitation of secondary intermetallic phases temperature, T_3	$L_4 + \text{Al}_5\text{Cu}_2\text{Mg}_8\text{Si}_6 \rightarrow L_5 + \text{Al}_8\text{FeMg}_3\text{Si}_6$
Temperatura pri precipitaciji sekundarnih intermetalnih faz, T_4	Precipitation of secondary intermetallic phases temperature, T_4	$L_5 \rightarrow L_6 + \text{Al}_7\text{Cu}_2\text{M}$
Temperatura pri precipitaciji sekundarnih intermetalnih faz, Temperatura solidusa, T_s	Precipitation of secondary intermetallic phases temperature, Solidus temperature, T_s	$L_6 \rightarrow \text{Al}_2\text{Cu}$

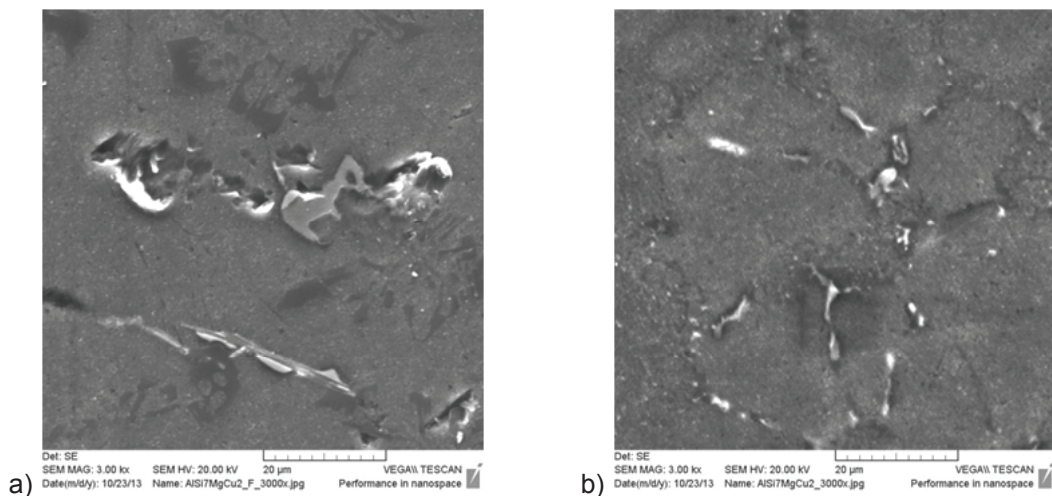
Slika 2. Mikrografi zlitine AlSi7Mg(Cu), pridobljeni s svetlobno mikroskopijo**Figure 2.** Micrographs of AlSi7MG(Cu) alloy obtained by light microscopy

porazdeljenimi interdendritnimi območji v obeh stanjih, litem (F) in po toplotni obdelavi (F). Večja povečava kaže bolj grobe in prekinjene veje dendrita, posejane z intermetalnimi fazami nosilcev železa iglaste oblike (Al_5SiFe) in hrapave sekundarne intermetalne faze na mejah zrn v litem stanju (F). Največja povečava (1.000-kratna) kaže prisotnost nespremenjenih evtektov (mešana vlakna in lamelarna oblika). Po toplotni obdelavi (T) se kaže enakomerna razporejenost vlaknastih oblik glavnega evtekt ($\alpha_{\text{Al}} + \beta_{\text{Si}}$). Zadnje faze strjevanja so bile opažene na mejah zrn. Njihova oblika in barva kaže sekundarne evtektične faze $\alpha_{\text{Al}} - \text{Al}_2\text{Cu}$ (delci in klastri ploščic) ter $\alpha_{\text{Al}} - \text{Mg}_2\text{Si}$ (tanki razvejani črni delci) v litem stanju (F). Stanje po toplotni obdelavi (F) kaže dobro razdelane sekundarne intermetalne faze na mejah zrn.

Primerjavaporazdelitvemikrostrukturnih sestavnih delov in velikosti, zaznane z vrstičnimi elektronskimi mikroskopi, pri največji povečavi je prikazana na Sliki 3.

investigated using light microscopy, as shown in Figure 2.

A smaller magnification (100x) reveals uniform distribution of primary dendritic network with evenly distributed interdendritic areas in both states, as-cast (F) and heat-treated (F), respectively. Higher magnification indicates rougher and broken dendritic branches dotted with iron-bearing intermetallic phases with needlelike morphology, (Al_5SiFe), and coarse secondary intermetallic phases at grain boundaries in as-cast state (F). The highest magnification (1000x) indicates the presence of undermodified eutectic (mixed fiber and lamella morphology). Heat-treated state (T) indicated uniformly distributed fiber morphology of main eutectic ($\alpha_{\text{Al}} + \beta_{\text{Si}}$). Last solidifying phases have been noticed at grain boundaries. Their morphology and colour reveals secondary eutectic phases $\alpha_{\text{Al}} - \text{Al}_2\text{Cu}$ (platelets particles and clusters) and $\alpha_{\text{Al}} - \text{Mg}_2\text{Si}$ (thin ramified black particles) in as-cast (F) state. Heat-



Slika 3. Vrstični elektronski posnetki zlitine AlSi7Mg(Cu) v a) litem stanju in b) po toplotni obdelavi (F)

Figure 3. Scanning electron images of AlSi7Mg(Cu) alloy in a) as-cast and b) heat-treated state (F)

Toplotna obdelava pozitivno vpliva na enakomerno porazdelitev in izboljšavo intermetalnih faz. Morfološko škodljive intermetalne faze, obogatene v železu in znane kot faza P (Al_5SiFe), pri tovrstni obliki niso več prisotne. Po toplotni obdelavi so odpravljeni tudi klastri Al_2Cu . Opazili smo obogateno kovinsko matriko (α_{Al}) pri Cu in Mg, kot je prikazano v Preglednici 5.

Preglednica 5. Kemijska sestava kovinske matrike zlitine AlSi7Mg(Cu)

Table 5. Chemical composition of AlSi7Mg(Cu) alloy metal matrix

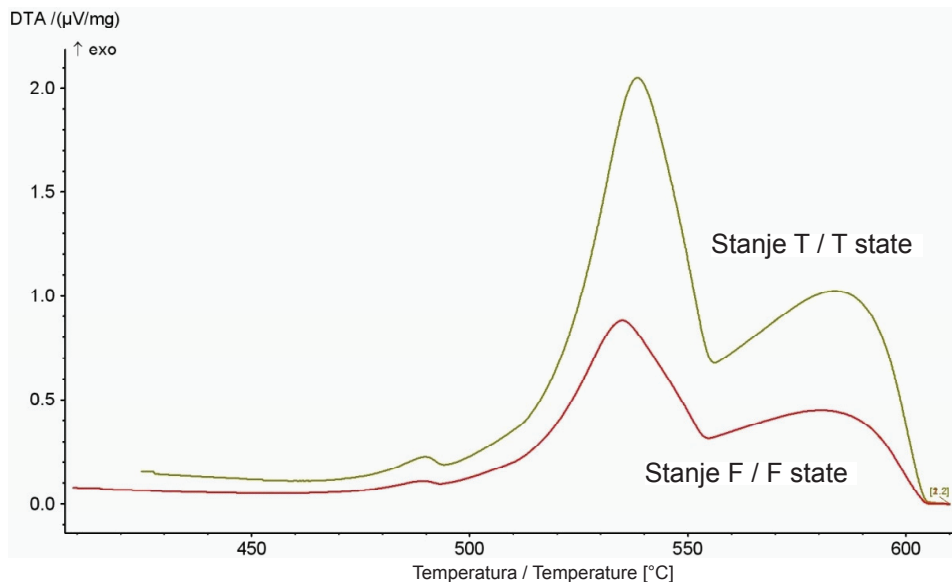
Kemijska sestava / Chemical composition, wt. %	Al	Si	Cu	Mg
AlSi7Mg(Cu)-F	96,02	0,52	1,49	1,93
AlSi7Mg(Cu)-T	96,06	-	2,55	1,40

treated state (F) reveals fine fragmented secondary intermetallic phases' at the grain boundaries.

Comparison of microstructural constituents' distribution and size revealed with scanning electron images at higher magnification is given in Figure 3.

Heat treatment has a positive influence on uniform distribution and refining of intermetallic phases'. Morphological detrimental intermetallic phases enriched in iron known as β phase (Al_5SiFe) are no longer present in that particular morphology. Also, Al_2Cu clusters have been resolved in heat-treated state. Enrichment of metal matrix (α_{Al}) in Cu and Mg has been noticed as shown in table 5.

Simultaneous thermal analysis enabled identification of significant thermodynamic changes in AlSi7Mg(Cu) alloy during solidification process. Comparison of the



Slika 4. Primerjava hladilnih krivulj zlitine AlSi7Mg(Cu) pri sočasni toplotni analizi v litem stanju in po toplotni obdelavi

Figure 4. Comparison of simultaneous thermal analysis cooling curves of AlSi7Mg(Cu) alloy in as-cast and heat-treated state

Sočasna toplotna analiza je omogočila prepoznavanje pomembnih toplotnih sprememb pri zlitini AlSi7Mg(Cu) med procesom strjevanja. Primerjava oblike krivulje hlajenja v litem stanju in po toplotni obdelavi je pokazala podobno vedenje, kot je prikazano na Sliki 4.

Krivulji segrevanja in hlajenja zlitine AlSi7Mg(Cu) v obeh stanjih sta pokazali doseganje istih temperatur pri faznih transformacijah in precipitaciji, kot je prikazano v Preglednici 6. Primerjava modeliranja faznega diagrama ravnovesja se kaže v sočasni toplotni analizi, mikrostruktura raziskava pa kaže strjevalno zaporedje zlitine AlSi7Mg(Cu).

Poleg dendritske mreže je treba najprej oceniti železove iglaste formacije Al_5SiFe in/ali kompleksno formacijo, podobno kitajskim pismenkam, $Al_{15}(Fe, Mn, Cu)_3Si_2$. Ustrezno razmerje precipitata Cu in Mg v kovinski matrici, medtem ko masa kohezijsko tvori kompleksne evtektične klastre $Al_8Mg_3(Fe, Mn)Si_6$ in faze $Al_5Mg_8Si_6Cu_2$. Strjevanje se zaključuje s precipitacijo

cooling curves shape for as-cast and heat-treated state revealed similar behaviour as shown in Figure 4.

The heating and cooling curves of AlSi7Mg(Cu) alloy in both states resulted in establishing the exact temperatures of phase transformations and precipitation, as shown in Table 6. Comparison of equilibrium phase diagram modelling results with simultaneous thermal analysis and microstructural investigations indicates the solidification sequence of AlSi7Mg(Cu) alloy.

Beside dendrite network, first to evaluate is iron-based needlelike Al_5SiFe and/or complex Chinese script formation $Al_{15}(Fe, Mn, Cu)_3Si_2$. Appropriate ratio of Cu and Mg precipitate in a metal matrix, while the bulk cohesively forms complex eutectic clusters of $Al_8Mg_3(Fe, Mn)Si_6$ and $Al_5Mg_8Si_6Cu_2$ phase. Solidification ends with secondary eutectic phase precipitations $\alpha_{Al} + Mg_2Si$ and $\alpha_{Al} + Al_2Cu$. Heat-treated state reveals higher significant temperatures of phase transformation and precipitation.

Preglednica 6. Pomembne temperaturne vrednosti pri fazni transformaciji in precipitaciji zlitine AlSi7Mg(Cu)

Table 6. Significant temperatures of phase transformation and precipitation of AlSi7Mg(Cu) alloy

Opis reakcije / Reaction description	Reakcija / Reaction	Stanje T/F T / F state [°C]	Stanje T/T, T / T state [°C]
Temperatura likvidusa, Tl / Liquidus temperature, Tl	$L \rightarrow L_1 + \alpha_{Al} + Al_5SiFe + Al_{15}(Fe, Mn, Cu)_3Si_2$	603,7	603,7
Evtektična temperatura, Te / Eutectic temperature, Te	$L_1 + \alpha_{Al} \rightarrow L_2 + \alpha_{Al} + (\alpha_{Al} + \beta_{Si})$	550,5	554,6
Temperatura pri precipitaciji sekundarnih intermetalnih faz, T1 / Precipitation of secondary intermetallic phases temperature, T1	$L_2 \rightarrow L_3 + Al_5Cu_2Mg_8Si_6 + Al_8Mg_3(Fe, Mn)Si_6$	527,8	531,6
Temperatura pri precipitaciji sekundarnih intermetalnih faz, T2 / Precipitation of secondary intermetallic phases temperature, T2	$L_3 \rightarrow L_4 + \alpha_{Al} + Mg_2Si$	494,0	493,6
Temperatura pri precipitaciji sekundarnih intermetalnih faz, temperatura solidusa, Ts / Precipitation of secondary intermetallic phases temperature, Solidus temperature, Ts	$L_4 \rightarrow L_5 + \alpha_{Al} + Al_2Cu$	467,9	534,1

sekundarne evtektične faze $\alpha_{Al} + Mg_2Si$ in $\alpha_{Al} + Al_2Cu$. Toplotna obdelava razkriva pomembnejše temperaturne vrednosti pri fazni transformaciji in precipitaciji.

Raziskali smo tudi natezne mehanske lastnosti zlitine AlSi7Mg(Cu). Primerjava lastnosti nove zlitine AlSi7Mg(Cu) tako v litem stanju (F) [28, 30] in po toplotni obdelavi (T) ter predhodno raziskane klasične zlitine AlSi7Mg [27] je prikazana v Preglednici 6.

Opazili smo pomembno zvečanje meje teženja in natezne trdnosti nove zlitine AlSi7Mg(Cu) v litem stanju, kar je mogoče pripisati kompleksnim interakcijam faze $Al_{15}(Fe,Mn,Cu)_3Si_2$, spremenjene z dodatkom Cu, pa tudi formaciji gostih faz $Al_5Cu_2Mg_8Si_6$ in $Al_8Mg_3(Fe,Mn)Si_6$. Elongacija v litem stanju se je bistveno zmanjšala. Opazili smo slabšo trdnost teženja in natezno trdnost po toplotni obdelavi. Edina prednost toplotne obdelave je bistveno večja elongacija A_{50} .

4 Sklepi

Zasnova inovativne zlitine AlSi7Mg(Cu) odkriva širok razpon zapletenih reakcij in možnih intermetalnih faz zaradi interakcije legirnih elementov in elementov v sledovih. Razvoj mikrostrukture in določitev strjevalnega zaporedja omogoča opredelitev nove zlitine in njenih lastnosti tako v litem stanju kot po toplotni obdelavi. Njihove interakcije pri procesu strjevanja smo določili z modeliranjem faznega diagrama ravnovesja, sočasno toplotno analizo ter raziskavo mikrostrukturnih in mehanskih lastnosti. Dodatek Cu (do 1,435 wt.%) kot sekundarni legirni element sproža dodatno interakcijo s Fe, Mn in Mg. Določitev strjevalnega zaporedja zlitine AlSi7Mg(Cu) z natančno določenimi pomembnimi temperaturnimi vrednostmi fazne transformacije in precipitacije ter korelacija z zaznano obliko in velikostjo

Tensile mechanical properties AlSi7Mg(Cu) alloy has been also investigated. Comparison of those obtained for new one AlSi7Mg(Cu) in both as-cast (F) [28, 30] and heat-treated (T) state and previously investigated conventional AlSi7Mg [27] is shown in Table 6.

Preglednica 6. Natezne mehanske lastnosti zlitine AlSi7Mg(Cu) v litem stanju

Table 6. Tensile mechanical properties of AlSi7Mg(Cu) alloy in as-cast state

Zlitina / Alloy	$R_{p0.2}$ [MPa]	R_m [MPa]	A_{50} [%]
AlSi7Mg	120	165	6,3
AlSi7Mg(Cu)-F	143	234	3,1
AlSi7Mg(Cu)-T	100	221	6,5

Significant increase in yield strength and tensile strength of innovative AlSi7Mg(Cu) alloy in as-cast state has been noticed, which can be attributed to the complex interactions of $Al_{15}(Fe,Mn,Cu)_3Si_2$ phase modified with Cu addition as well as to the formation of compacted $Al_5Cu_2Mg_8Si_6$ and $Al_8Mg_3(Fe,Mn)Si_6$ phases'. Elongation in as-cast state has been significantly decreased. Lowering the yield and tensile strength in heat-treated state was noticed. The only benefit of heat treatment was indicated in significant increase of elongation A_{50} .

4 Conclusion

Designing of innovative AlSi7Mg(Cu) alloy reveals a wide range of complex reactions and possible intermetallic phases due to the interaction of alloying and trace elements. Evolution of microstructure and determination of solidification sequence enables the characterization of new alloy and its properties in both states as-cast and heat-treated. Determination of their

mikrostrukturnih sestavin omogoča predvidevanje njenega vedenja.

Širok nabor ugodnih intermetalnih faz omogoča pregled možnih povezav, vključno z razvojem nateznih mehanskih lastnosti že v litem stanju. Primerjava trdnosti teženja in natezne trdnosti pri pogosto uporabljeni zlitini AlSi7Mg kaže bistveno zvečanje preiskovanih lastnosti za inovativno kemijsko sestavo zlitine AlSi7Mg(Cu) v litem stanju, medtem ko toplotna obdelava ni bistveno vplivala na izboljšave.

5 Zahvala

Raziskave so bile opravljene v sodelovanju z družbami CIMOS P.P.C. Buzet, d.o.o., Buzet, Hrvaška. Raziskave smo opravili v okviru preiskovane teme »Design and Characterization of Innovative Engineering Alloys« (Zasnova in opredelitev inovativnih tehničnih litin), oznaka: TP167, ki jo je finančno podprla Univerza v Zagrebu v okviru zagotavljanja finančne podpore raziskavam.

interactions in solidification process was performed by modelling of equilibrium phase diagram, simultaneous thermal analysis, microstructural and mechanical investigations. An extra addition of Cu (up to 1,435 wt.%) as a secondary alloying element initiates additional interaction with Fe, Mn and Mg. Determination of AlSi7Mg(Cu) alloy solidification sequence with exact significant temperatures of phase transformation and precipitation and correlation with revealed morphology and size of microstructure constituents enables prediction of its behaviour.

Wide spectra of favourable intermetallic phases enable an overview of strong connections comprehending to the tensile mechanical properties development already in as-cast state. The comparison of yield and tensile strength with commonly used AlSi7Mg alloy indicates a significant increase of investigated properties for innovative chemistry of AlSi7Mg(Cu) alloy in as-cast state, while performed heat treatment did not influence on significant improvement.

5 Acknowledgement

Investigations have been performed in collaboration with companies CIMOS-P.P.C. Buzet, d.o.o., Buzet, Croatia. Investigations were performed within the research topic "Design and Characterization of Innovative Engineering Alloys", Code: TP167 funded by the University of Zagreb within the Framework of Financial Support of Research.

Viri / References

- [1] S. Seifeddine, Effect of cooling rate and Fe and Mn content on the tensile and fatigue properties of the Al-10%Si-2%Cu casting alloy, PhD Thesis, Jonkoping University, Jonkoping, 2008
- [2] F. Stadler, H. Antrekowitsch, W. Fragner, H. Kaufmann, E. Pinatel, P. Uggowitzer, The effect of main alloying elements on the physical properties of Al-Si foundry alloys, *Materials Science and Engineering A*, 560 (2013), str. 481–491
- [3] J. Olofsson, I.L. Svensson, P. Lava, D. Debruyne, Characterisation and investigation of local variations in mechanical behaviour in cast aluminium using gradient solidification, Digital Image Correlation and finite element simulation, *Materials & Design*, 56 (2014), str. 755–762
- [4] A. K. Dahle, J. Hjelen, L. Arnberg, Formation of hypoeutectic Al-Si alloys, *Proceedings of the 4th International Conference on Solidification Processing*, Sheffield, 1997, 527530
- [5] ASM Specialty Handbook: Aluminum and Aluminum Alloys. Ohio: ASM International, Materials Park 1993.
- [6] L. Backerund, G. Chai, J. Tamminen, *Solidification Characteristics of Aluminium Alloys: Foundry Alloys Vol. 2*, Stockholm: AFS/Skanaluminium; 1999.
- [7] D. Dispinar, J. Campbell, Metal quality studies in secondary remelting of aluminium, *J. Inst. Cast Met. Eng.*, 178 (2004), str. 78–86.
- [8] K. Dahle, L. Arnberg, Development of strength in solidifying aluminium alloys, *Acta Materialia*, 45 (2) (1997) 547-559, doi: 10.1016/S1359-6454(96)00203-0
- [9] N. A. Belov, D. G. Eskin, A. A. Aksenov, *Multicomponent phase diagrams, Applications for commercial aluminum alloys*, Elsevier, London, 2005, str. 47–52,
- [10] Z. Zovko Brodarac, F. Unkić, J. Medved, P. Mrvar, Determination of solidification sequence of the AlMg9 alloy, *Kovove Mater.* 50 (1) (2012), str. 59–67.
- [11] S. Ji, F. Yan, Z. Fan, *A High Strength Aluminium Alloy for High Pressure Die Casting*, Light Metals 2016, Aluminum Alloys, Processing and Characterization, Alloy Development and Applications (E. Williams), The Minerals, Metals & Materials Society, John Wiley & Sons, Inc., Hoboken, New Jersey, p. 207-212
- [12] J. Backman, *Processing aspects for improving mechanical properties in aluminium castings* (PhD Thesis, Linköping: Linköping University, Jonkoping: Jonkoping University), 1999, urn:nbn:se:liu:diva-30074
- [13] E. R. Wang, X. D. Hui, S. S. Wang, Y. F. Zhao, G. L. Chen, Improved mechanical properties in cast Al-Si alloys by combined alloying of Fe and Cu, *Materials Science and Engineering A*, 527 (29-30) (2010) 7878-7884, doi: 10.1016/j.msea.2010.08.058
- [14] Z. Zovko Brodarac, N. Dolić, F. Unkić, Influence of copper content on microstructure development of AlSi9Cu3 alloy, *J. Min. Metall. Sect. B-Metall.* 50 (1) B (2014), str. 53–60, doi:10.2298/JMMB130125009B
- [15] F. H. Samuel, A. M. Samuel, H. W. Doty, Factors controlling the type and morphology of Cu-containing phases in 319 Al alloy, *AFS Trans.*, 104 (1996), str. 893–901.
- [16] T. Pabel, S. Bozorgi, C. Kneibl, K. Faerber, P. Drivetrain, P. Schumacher, Einfluss der Legierungselemente auf die HeiBrissneigung bei AlSi7MgCu-Gusslegierungen, *Giesserei*, 99, 2012, 9, str. 30–37

- [17] F. Stadler, H. Antrekowitsch, W. Fragner, H. Kaufmann, P. Uggowitzner Effect of main alloying elements on strength of Al-Si foundry alloys at elevated temperatures, *International Journal of Cast Metals Research*, 25 (2012), str. 215–224, doi: 10.1179/1743133612Y.0000000004
- [18] M. Zamani, Al-Si Alloys - Microstructure and Mechanical Properties at Ambient and Elevated temperature, PhD Thesis, Jonkoping University, School of Engineering, Department of Materials and Manufacturing, 2015
- [19] C. H. Cacers, M. B. Djurdjević, T. J. Stockwell, J. H. Sokolowski, The effect of Cu content on the level of microporosity in Al-Si-Cu-Mg casting alloys, *Scripta Mater.* 40 (5) (1999), str. 631–637
- [20] S. G. Shabestari, H. Moemeni, Effect of copper and solidification conditions on the microstructure and mechanical properties of Al-Si-Mg alloys, *Mater Process Technol.* 153–154 (2004), str. 193–198, doi: 10.1016/j.jmatprotec.2004.04.302
- [21] I. Dugić, F. Henriksson, C. Strebel, O. Kosmaz, S. Seifeddine, On the Effect of Alloying Element Range on the Mechanical Properties of Recycled Aluminium Alloy EN AB-46000, *Light Metals 2016, Aluminum Alloys, Processing and Characterization, Alloy Development and Applications* (E. Williams), The Minerals, Metals & Materials Society, John Wiley & Sons, Inc., Hoboken, New Jersey, str. 115–120
- [22] M. Zamani, S. Seifeddine, E. Ghassemali, Effect of cooling rate and eutectic modification on texture and grain structure, *La Metallurgia Italiana*, 108 (6) (2016) 2932
- [23] M. Tocci, A. Pola, L. Raza, L. Armellin, U. Afeltra Optimization of heat treatment parameters for a nonconventional Al-Si-Mg alloy with Cr addition by DOE method, *La Metallurgia Italiana*, 108 (6) (2016) 141-144
- [24] M. B. Djurdjević, G. Huber, Z. Odanović, Synergy between thermal analysis and simulation *J Therm Anal Calorim.* 111 (2) (2013) 1365-1373, doi: 10.1007/s10973-012-2389-0
- [25] EN 1706:2010 Aluminij in aluminijeve zlitine – Ulitki – Kemična sestava in mehanske lastnosti
- [26] IDM 4234, Honeywell – Garret, Industrial Division specification, Aluminium alloy castings 356-F, revision K, 2008
- [27] D. Stanić, Z. Zovko Brodarac, F. Unkić, Mikrostrukturna i mehanicka svojstva kokilno lijevanih uzoraka AlSi7Mg legure (Microstructure and mechanical properties of AlSi7Mg alloy samples cast in permanent mould, in Croatian), *Proceedings book of 9* International Foundrymen Conference* (ed. F. Unkić), Sisak, Sveučilište u Zagrebu, Metalurški fakultet, 2009, CD_ROM 17-2009
- [28] Z. Zovko Brodarac, D. Stanić, Study of innovative AlSi7MgCu alloy with improved properties, 56. IFC – Mednarodno livarsko posvetovanje Portorož 2016, *Zborniki konferenc* (ed. A. Križman, P. Mrvar, J. Medved, P. Schumacher, R. Deike, M. Jan-Blažič, M. Debelek), Ljubljana, Slovenija, 2016., 66-67, CD_ROM (prispevek in extenso)
- [29] EN 10002-1:1998 Kovinski materiali – preverjanje natezne trdnosti – 1. del: preskusna metoda (pri sobni temperaturi)
- [30] Z. Zovko Brodarac, D. Stanić, T. Holjevac Grgurić, Solidification sequence of innovative AlSi7Mg(Cu) alloy, 48th International October Conference on Mining and Metallurgy (N. Strbac, D. Zivkovic), University of Belgrade Technical Faculty in Bor, Zajecar, 2016, str. 375–378