

Z. Zovko Brodarac<sup>1</sup>, F. Kozina<sup>1</sup>, J. Burja<sup>2,3</sup>, B. Šetina Batič<sup>3</sup>, D. Stanić<sup>4</sup>, M. Folta<sup>5</sup>

<sup>1</sup>Univerza v Zagrebu, Fakulteta za metalurgijo, Sisak (HR) / University of Zagreb Faculty of Metallurgy, Sisak (HR), <sup>2</sup>Univerza v Ljubljani, Naravoslovnotehniška fakulteta, Ljubljana (SI) / University of Ljubljana Faculty of Natural Sciences and Engineering, Ljubljana (SI), <sup>3</sup>Inštitut za kovinske materiale in tehnologije, Ljubljana (SI) / Institute of Metals and Technology, Ljubljana (SI), <sup>4</sup>Istrska univerza uporabnih znanosti, Pula (HR) / Istrian University of Applied Sciences, Pula (HR), <sup>5</sup>Škoda Auto Vysoká Škola o.p.s., Mladá Boleslav (CZ) / Škoda Auto Vysoká Škola o.p.s., Mladá Boleslav (CZ)

## Vpliv toplotne obdelave na mikrostrukturo reciklirane zlitine AlSi9Cu3(Fe)

### Influence of Heat Treatment on Microstructure of Recycled AlSi9Cu3(Fe) Alloy

#### Izvleček

Surovine veljajo za najpomembnejši korak pri določanju ogljičnega odtisa proizvodnje. Proizvajalci ulitkov se zaradi pravnih, gospodarskih in političnih razmer v Evropski uniji ter pomanjkanja kakovostnih surovin soočajo s številnimi težavami. Večina vsakodnevno proizvedenih komponent, kot so aluminij in njegove zlitine, spada med kritične surovine (CRM) in strateške surovine (SRM). Te surovine so bistvenega pomena za različne strateške sektorje, kot so obnovljivi viri energije, digitalna industrija, letalski in obrambni sektor ter zdravstveni sektor, ki so vsi tesno povezani s kovinsko industrijo. Standardna aluminijeva zlitina AlSi9Cu3(Fe) (EN AC 46000) se pogosto uporablja v avtomobilski industriji in sektorju prevoza. Zaradi visokih zahtev proizvajalcev avtomobilov glede varnosti in mehanskih lastnosti, kot so trdnost, trdota in raztezek, ter nizke mase in odpornosti proti koroziji je zlitina AlSi<sub>9</sub>Cu<sub>3</sub>(Fe) prednostna surovina za proizvodnjo varnostno pomembnih komponent. Večina teh lastnosti je posledica razvoja mikrostrukture. Funkcionalne in uporabne lastnosti aluminijevih zlitin so odvisne od kemijske sestave, načina taljenja, hitrosti strjevanja, postopka litja in morebitne toplotne obdelave. Zaradi krize surovin se posebna pozornost namenja uporabi sekundarnih, tj. recikliranih surovin ter njihovi funkcionalnosti in trajnosti ob koncu življenjske dobe.

Za nadaljnje izboljšanje kakovosti popolnoma reciklirane zlitine AlSi9Cu3(Fe) je bila izvedena toplotna obdelava. Termodinamični izračuni in metalografske analize recikliranih zlitin AlSi9Cu3(Fe) kažejo naslednje zaporedje strjevanja: primarni aluminij  $\alpha_{Al}$ , eutektik ( $\alpha_{Al} + \beta_{Si}$ ), intermetalni fazi na osnovi železa v igličasti morfologiji Al<sub>5</sub>FeSi in Al<sub>15</sub>Si<sub>2</sub>M<sub>4</sub> »kitajske pisave«, intermetalni fazi na osnovi magnezija in bakra, kot sta Mg<sub>2</sub>Si in Al<sub>2</sub>Cu, ter kompleksni intermetalni fazi, kot sta Al<sub>8</sub>Mg<sub>3</sub>FeSi<sub>2</sub> in Al<sub>5</sub>Mg<sub>8</sub>Si<sub>2</sub>Cu<sub>2</sub>. Toplotna obdelava je pomembno vplivala na izboljšanje mikrostrukture in spremembo morfologije nekaterih mikrostrukturnih komponent. Nov vpogled v razvoj mikrostrukture smo pridobili s konfokalno lasersko mikroskopijo, ki omogoča kartiranje mikrostrukturne topografije in določanje višinskih odstopanj mikrostrukturnih komponent.

**Ključne besede:** CRM, recikliranje, zlitina AlSi9Cu3(Fe), mikrostruktura, toplotna obdelava

#### Abstract

Raw materials are considered the most important step in determining the carbon footprint of production. Manufacturers of castings face a number of difficulties due to the legal, economic, and political situation in the European Union and the shortage of high-quality

raw materials. Most of the components produced on a daily basis, such as aluminium and its alloys, belong to the critical raw materials (CRMs) and strategic raw materials (SRMs). These raw materials are essential for a variety of strategic sectors such as renewable energy, the digital industry, the aerospace and defence sector, and the healthcare sector, all of which are closely linked to the metals industry. The standard aluminium alloy AISi9Cu3(Fe) (EN AC 46000) is widely used in the automotive and transport industries. The high requirements of automotive manufacturers in terms of safety and mechanical properties, such as strength, hardness, and elongation, as well as low mass and corrosion resistance, make the AISi9Cu3(Fe) alloy the preferred raw material to produce safety-critical components. Most of these properties are due to the development of the microstructure. The functional and useful properties of cast aluminium alloys depend on the chemical composition, the melting treatment, the solidification rate, the casting process, and the possible heat treatment. Given the raw materials crisis, particular attention is being paid to the use of secondary, i.e., recycled, raw materials and their functionality and sustainability at the end of their service life.

To further improve the quality of the fully recycled AISi9Cu3(Fe) alloy, a heat treatment was carried out. Thermodynamic calculations and metallographic analyses of recycled AISi9Cu3(Fe) alloys show the following solidification sequence: primary aluminium  $\alpha_{Al}$ , eutectic phase ( $\alpha_{Al} + \beta_{Si}$ ), iron-based intermetallic phase in needle-like  $Al_5FeSi$  and  $Al_{15}Si_2M_4$  "Chinese script" morphology, magnesium- and copper-based intermetallic phase such as  $Mg_2Si$  and  $Al_2Cu$ , and complex intermetallic such as  $Al_8Mg_3FeSi_2$  and  $Al_5Mg_8Si_2Cu_2$  phases. The heat treatment had a significant influence on the refinement of the microstructure and the change in morphology of certain microstructural components. New insights into the evolution of the microstructure were gained using confocal laser microscopy, which allows mapping of the microstructural topography and determination of the height deviation of the microstructural components.

**Keywords:** CRM, recycling, AISi9Cu3(Fe) alloy, microstructure, heat treatment

## 1 Uvod

Surovine so ključnega pomena za vsak proizvodni proces, ko gre za določanje ogljičnega odtisa proizvodnje, zlasti če veljajo za kritične surovine (CRM) in/ali strateške surovine (SRM) [1–4]. Kartiranje potenciala za recikliranje aluminijevih zlitin razkriva težave, probleme, pa tudi rešitve za boljšo kakovost izdelkov [5,6].

Funkcionalne in uporabne lastnosti zlitine Al-Si za litje so močno odvisne od kakovosti surovine, zlasti če gre za recikliran material. V avtomobilski industriji so zlitine Al-Si-Cu prepoznane kot kakovostni izdelki za različne varnostno pomembne

## 1 Introduction

Raw materials are crucial for any production process when it comes to defining the carbon footprint of production, especially when they are considered as critical (CRM) and/or strategic raw materials (SRM) [1-4]. Mapping the recycling potential of aluminium alloys reveals difficulties, problems, but also solutions for better quality products [5,6].

The functional and useful properties of a casting Al-Si alloy depend strongly on the quality of the raw material, especially if it is a recycled material. The automotive industry has recognised Al-Si-Cu alloys as quality products for various safety-critical parts,

dele, za katere je značilna visoka trdnost pri sobnih in povišanih temperaturah [7–9]. Izhodiščna točka vsake industrijske proizvodne verige je kemijska sestava, ki zaradi vrste interakcij med elementi in uporabljenih tehnoloških proizvodnih parametrov ne omogoča vedno podvajanje mikrostrukture in s tem razvoja mehanskih lastnosti [10,11]. Uporabljena tehnologija, tj. hitrost hlajenja in s tem strjevanja, vpliva na številne interakcije med postopkom strjevanja. Medsebojno delovanje silicija, bakra, železa in magnezija je bilo raziskano drugje [12–16]. Ocena kemijske sestave se začne z modeliranjem ravnotežnega faznega diagrama in napovedjo poteka strjevanja, kar zaradi uporabe sekundarnih surovin postaja vse bolj zapleteno. Sinergija modeliranja in izvedene termodinamične in mikrostrukturne analize zlitine AlSi9Cu3(Fe) kaže na prisotnost širokega spektra legirnih elementov zlitin AlSi9Cu3(Fe) in razvoj  $\alpha$ -Al<sub>15</sub>Si<sub>2</sub>M<sub>4</sub> (M= Cr, Fe, Mn, Mo),  $\beta$ -Al<sub>5</sub>FeSi, Al<sub>2</sub>Cu in še bolj kompleksnih, kot je Al<sub>3</sub>Cu<sub>2</sub>Mg<sub>9</sub>Si<sub>7</sub>, z uporabo teoretičnega modeliranja. Kompleksna pot strjevanja kaže primarni aluminij  $\alpha_{Al}$ , evtektik ( $\alpha_{Al} + \beta_{Si}$ ), intermetalno fazo na osnovi železa v morfologiji Al<sub>5</sub>FeSi in »kitajske pisave«, intermetalni fazi na osnovi magnezija in bakra, kot sta Mg<sub>2</sub>Si in Al<sub>2</sub>Cu, ter kompleksni intermetalni fazo, kot sta Al<sub>8</sub>Mg<sub>3</sub>FeSi<sub>2</sub> in Al<sub>5</sub>Mg<sub>8</sub>Si<sub>2</sub>Cu<sub>2</sub>. Termodinamični učinki medsebojnega delovanja elementov med postopkom strjevanja pomembno vplivajo na potek in način strjevanja [17]. Druge študije so se osredotočile na uporabo popolnoma sekundarnih surovin [18–20]. Pri ponovnem taljenju vložka se kemijska sestava nekoliko poslabša, kar se odraža v termodinamičnih učinkih in razvoju mikrostrukture, visoka natezna trdnost in raztezek pa se ohranita. Ugotovili smo, da vpliv različnih postopkov toplotne obdelave

characterised by high strength at room and elevated temperatures [7-9]. The starting point of any industrial production chain is the chemical composition, which, due to a series of element interactions and applied technological production parameters, does not always allow replication of the microstructure and thus the development of the mechanical properties [10,11]. The applied technology, i.e., the cooling and thus the solidification rate, has an influence on numerous interactions during the solidification process. The interaction of silicon, copper, iron, and magnesium has been investigated elsewhere [12-16]. The evaluation of the chemical composition begins with the modelling of the equilibrium phase diagram and the prediction of the solidification sequence, which is becoming increasingly complex due to the use of secondary raw materials. The synergy of modelling and performed thermodynamic and microstructural analysis of AlSi9Cu3(Fe) alloy shows the presence of a wide range of alloying elements. AlSi9Cu3(Fe) alloys show the evolution of  $\alpha$ -Al<sub>15</sub>Si<sub>2</sub>M<sub>4</sub> (M= Cr, Fe, Mn, Mo),  $\beta$ -Al<sub>5</sub>FeSi, Al<sub>2</sub>Cu and even more complex ones such as Al<sub>3</sub>Cu<sub>2</sub>Mg<sub>9</sub>Si<sub>7</sub> using theoretical modelling. The complex solidification pathway shows primary aluminium  $\alpha_{Al}$ , eutectic ( $\alpha_{Al} + \beta_{Si}$ ), iron-based intermetallic phase in Al<sub>5</sub>FeSi, and “Chinese script” morphology, magnesium, and copper-based intermetallic phase such as Mg<sub>2</sub>Si and Al<sub>2</sub>Cu, and complex intermetallic such as Al<sub>8</sub>Mg<sub>3</sub>FeSi<sub>2</sub> and Al<sub>5</sub>Mg<sub>8</sub>Si<sub>2</sub>Cu<sub>2</sub> phases. The thermodynamic effects of the interaction of the elements during the solidification sequence have a significant influence on the solidification pathway and the mode of solidification [17]. Other studies have focused on the use of fully secondary raw material [18-20]. The remelting of charge material shows a slight deterioration of the chemical composition, which is

pozitivno vpliva na mikrostrukturo in mehanske lastnosti [7, 9, 21–26].

Za nadaljnje izboljšanje kakovosti popolnoma reciklirane zlitine AlSi9Cu3(Fe) je bila izvedena toplotna obdelava. Pri proizvodnji visokotlačnih ulitkov (HPDC) je treba skrbno določiti režim toplotne obdelave, da bi preprečili nastanek mehurjev zaradi ujetja zraka med postopkom litja [23]. V literaturi se priporočajo zelo nizke temperature taljenja < 450 °C in poznejše staranje pri temperaturi 160 °C, da bi zagotovili izvedljivost industrijske uporabe [23]. Na splošno toplotna obdelava vpliva na izboljšanje mikrostrukture z razdrobljenostjo in sferoidizacijo eutektskega Si [24–26]. Drugi učinki vključujejo razpad zadnjih strjenih faz na osnovi Cu in/ali Mg in njihovo delno raztapljanje v trdni raztopini  $\alpha_{Al}$  ter interakcijo z drugimi elementi, kot je Fe [24–26].

Namen te študije je uporabiti optimalni režim toplotne obdelave (vrsta, temperatura in čas) za zlitino za tlačno litje AlSi9Cu3(Fe) iz popolnoma sekundarne surovine in določiti vpliv na možne mikrostrukturne spremembe.

## 2 Materiali in metode

V tem delu je bil vložek za preučitev taljenja in naknadne toplotne obdelave vzorcev pripravljen iz 100-odstotnega, tj. popolnoma ponovno pridobljenega materiala.

Kemijska sestava je bila analizirana z optičnim emisijskim spektrometrom ARL-3460 in spektrometrom LECO GDS 900 s tlilno razelektrivijo. Termodinamični izračuni ravnotežnega in neravnotežnega faznega diagrama po postopku Scheil-Gulliver za zlitino AlSi9Cu3(Fe) so bili opravljeni s programsko opremo Thermo-Calc TCW 5.0 s podatkovno bazo TTAL7 [17, 19-20]. Opravljena je bila primerjava

reflected in the thermodynamic effects and the development of the microstructure, while the high tensile strength and elongation are maintained. It has been found that the influence of different heat treatment processes has a positive effect on the microstructure and mechanical properties [7, 9, 21-26].

To further improve the quality of the fully recycled AlSi9Cu3(Fe) alloy, a heat treatment was carried out. In the production of high-pressure die castings (HPDC), the heat treatment regime should be carefully defined to avoid possible blistering occurrence due to air entrapment during the casting process [23]. Very low solution temperatures < 450 °C and subsequent ageing at 160 °C are recommended in the literature to ensure the feasibility of industrial application [23]. In general, heat treatment influences the refinement of the microstructure by fragmentation and spheroidisation of the eutectic Si [24-26]. Other effects include the decomposition of the last solidifying stage phases based on Cu and/or Mg and its partial dissolution in the  $\alpha_{Al}$  solid solution, as well as the interaction with other elements such as Fe [24-26].

The aim of this study is to apply the optimal heat treatment regime (type, temperature, and time) for the HPDC AlSi9Cu3(Fe) alloy from completely secondary raw material and to determine the influence on possible microstructural changes.

## 2 Materials and Methods

In this work, the charge material for melting and afterward heat treatment samples investigation was prepared from 100%, i.e., completely returned material.

The chemical composition was analysed using the ARL-3460 optical emission

dobljenih rezultatov z rezultati simultane termične analize, opravljene na analizatorju STA 449 Jupiter. Simultana termična analiza omogoča določanje značilnih temperatur in toplotnih učinkov določenih dogodkov med taljenjem in/ali strjevanjem [18,19].

Režim toplotne obdelave je sestavljen iz raztopnega žarjenja pri temperaturi 430 °C, ki mu sledi staranje pri 160 °C, kot je prikazano na sliki 1.

Mikrostrukturo smo pregledali z optičnim mikroskopom (OM) Olympus BX51 in konfokalnim laserskim mikroskopom (CLM) Olympus Lext OLS5100 ter vrstičnim elektronskim mikroskopom, opremljenim z energijsko disperzijskim spektrometrom JEOL5610 in Zeiss CrossBeam 550.

### 3 Rezultati in razprava

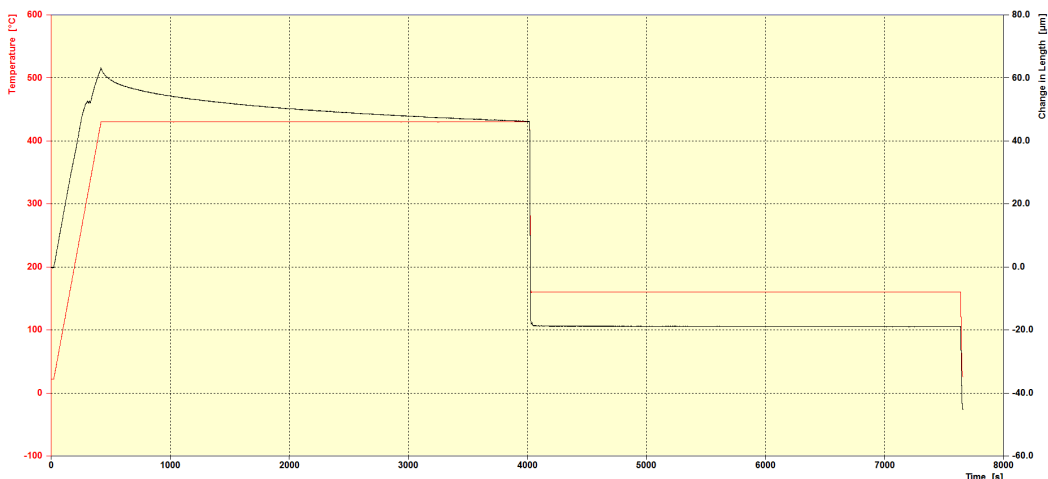
#### 3.1 Rezultati kemijske sestave

Kemijska sestava zlitine AlSi9Cu3(Fe) je navedena v preglednici 1. Primerjava kemijske sestave preskusnih vzorcev s

spektrometer and the LECO GDS 900 glow discharge spectrometer. Thermodynamic calculations of the equilibrium and Scheil–Gulliver non-equilibrium phase diagrams of the AlSi9Cu3(Fe) alloy were carried out using the Thermo-Calc software TCW 5.0 with the TTAL7 database [17, 19-20]. The results obtained were compared with those of the simultaneous thermal analysis performed with STA 449 Jupiter. STA allows the determination of the characteristic temperatures and the thermal effect of certain events during melting and/or solidification [18,19].

The heat treatment regime consists of solution heat treatment at 430 °C followed by ageing at 160 °C, as shown in Figure 1.

The microstructure was examined using optical microscopes (OM) Olympus BX51 and confocal laser microscope (CLM) Olympus Lext OLS5100, and scanning electron microscopes (SEM) equipped with energy dispersive spectrometer JEOL5610 and Zeiss CrossBeam 550.



**Slika 1.** Režim toplotne obdelave vzorcev sekundarne zlitine AlSi9Cu3(Fe), proizvedenih s tlačnim litjem

**Figure 1.** Heat treatment regime for secondary AlSi9Cu3(Fe) alloy samples produced using HPDC

certificiranimi podatki ni pokazala nobenih odstopanj od vrednosti, ki jih zahteva standard EN 1706:2010.

Primerjava zahtev in preskušanih vzorcev iz običajnega vložka (CCM 50 %) ter ponovno pridobljenega vložka (RCM 100 %) je pokazala, da so vse dobljene vrednosti pomembnih elementov skladne s standardi, vendar nižje pri ponovno pridobljenem vložku. Zaradi visokih vrednosti bakra in nizke vsebnosti magnezija se pričakuje nastanek intermetalnih faz  $Al_2Cu$  in  $Al_5Mg_8Si_2Cu_2$ . Glede na ustrezno vsebnost železa in mangana v preskušani zlitini RCM in zaradi uporabljene tehnologije HPDC se pričakuje nastanek  $Al_{15}(Mn,Fe)_3Si_2$  in  $Al_5FeSi$  zaradi neugodnega razmerja med Fe in Mn. Poleg tega lahko interakcija z magnezijem povzroči nastanek faze  $Al_8Mg_3FeSi_2$  [17]. Vsebnost drugih nečistoč, kot so svinec, krom in kositer so višje, vendar še zmeraj v mejah, ki jih predpisuje norma.

### 3.2 Rezultati preučitve mikrostrukture

Razvoj mikrostrukture pred toplotno obdelavo ni bistveno odstopal od zaporedja strjevanja, ki smo ga raziskali že prej [17–19, 21]. Ploskovna porazdelitev kemijskih elementov v značilnem interesnem območju v kovinski matrici, opravljena z vrstičnim

## 3 Results and Discussion

### 3.1 Chemical Composition Results

The chemical composition of the  $AlSi9Cu3(Fe)$  alloy is given in Table 1. The comparison of the chemical composition of the tested samples with certified data did not show any deviation from the values required by the EN 1706:2010 standard.

The comparison between the requirement and the tested samples in common charge material (CCM 50%) and recycled – return charge material (RCM 100%) showed that all obtained values for important elements are consistent with the norm, but lower in return charge material. Due to the high copper values and low magnesium content, the formation of  $Al_2Cu$  and  $Al_5Mg_8Si_2Cu_2$  intermetallic phases is expected. Concerning the corresponding content of iron and manganese in the tested RCM alloy, and due to the applied HPDC technology, the formation of  $Al_{15}(Mn,Fe)_3Si_2$  and  $Al_5FeSi$  is expected regarding unfavourable Fe/Mn ratio. In addition, interaction with magnesium can lead to the formation of the  $Al_8Mg_3FeSi_2$  phase [17]. Other impurities, such as lead, chromium and tin are higher, but still within the limits prescribed by norm.

**Preglednica 1.** Primerjava kemijske sestave vložka EN AB  $AlSi9Cu3$  po standardu EN 1706:2010, običajni vložek (CCM) je sestavljen iz 50 %, sekundarni vložek pa iz 100 % povratnega vložka (RCM)

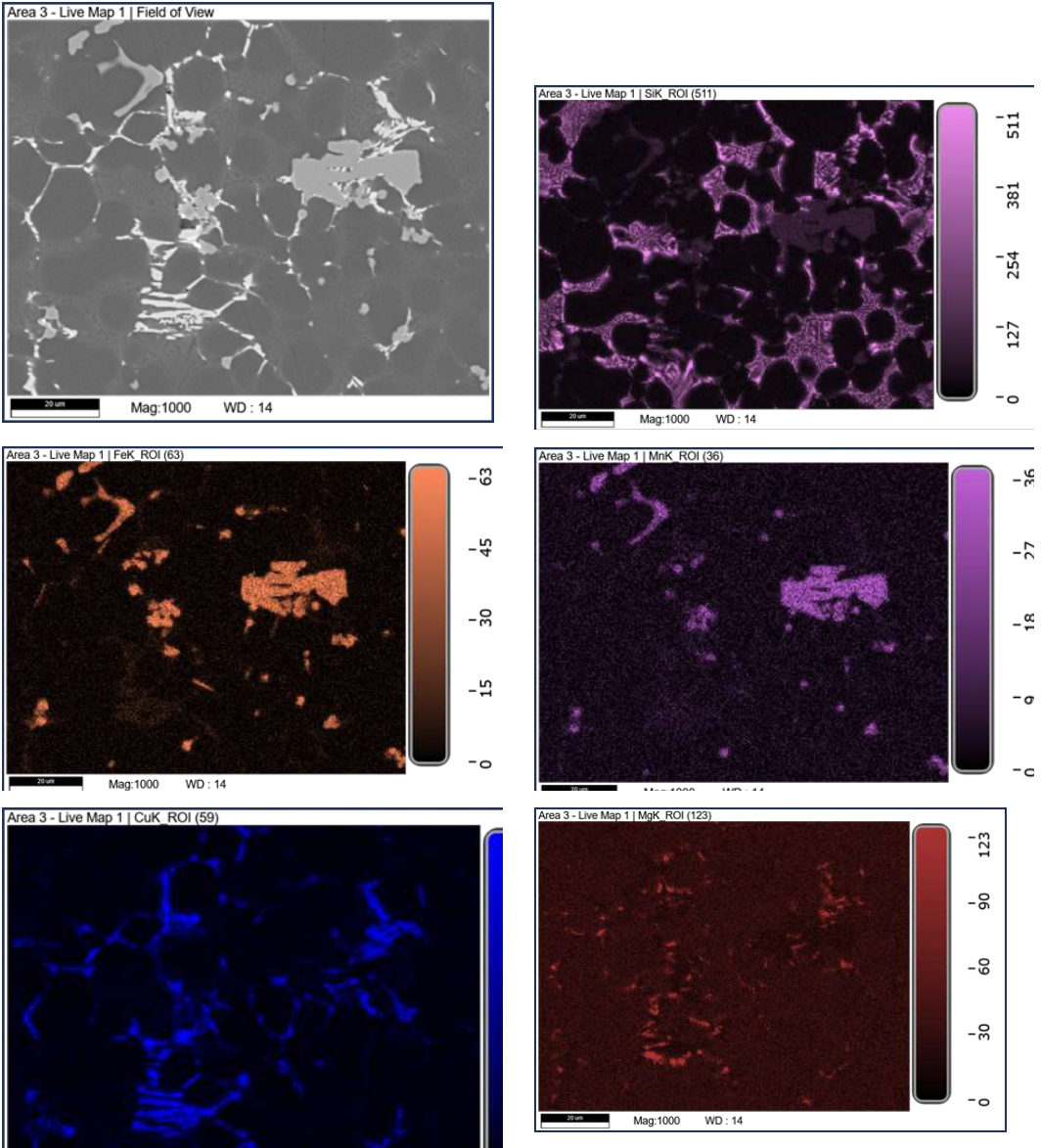
**Table 1.** Comparison of chemical composition of charge material EN AB  $AlSi9Cu3$  by norm EN 1706:2010, conventional charge material (CCM) consisted of 50%, and secondary charge material consisted form 100% of return charge material (RCM)

ELEMENT w/wt %	Si	Fe	Cu	Mn	Mg	Cr	Zn	Pb	Sn
EN 1706:2010	8,0–11,0	1,0	2,0–4,0	0,55	0,05–0,55	0,15	1,2	0,35	0,25
CCM (50 %)	9,75	0,89	3,26	0,24	0,12	0,04	0,99	0,06	0,012
RCM (100 %) – ARL	8,75	0,66	2,91	0,20	0,17	0,04	0,82	0,04	0,004
RCM (100 %) – LECO	9,36	0,80	2,65	0,21	0,17	0,03	0,78	0,05	0,024

elektronskim mikroskopom (SEM), opremljenim z energijsko disperzijskim spektrometrom (EDS), je prikazana na sliki 2.

### 3.2 Microstructure Investigation Results

The microstructure development prior heat treatment showed no significant deviation

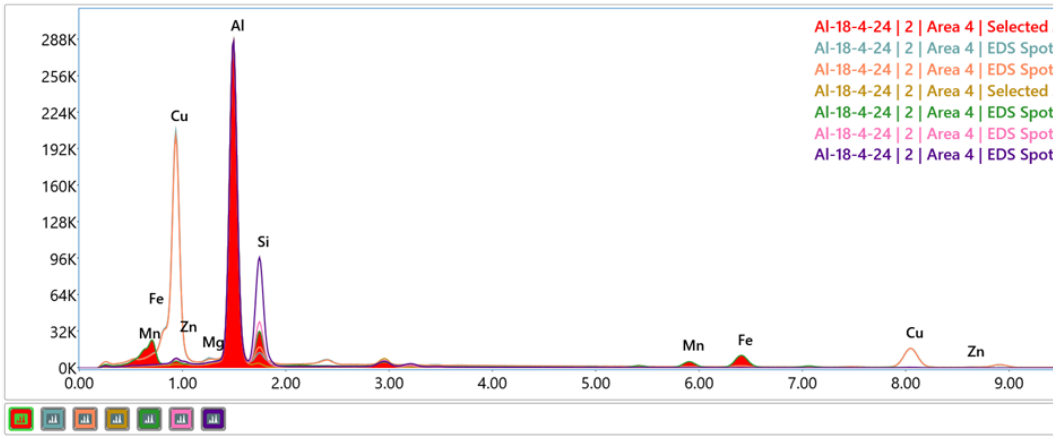


Slika 2. Ploskovna porazdelitev elementov značilnega interesnega območja zlitine AlSi9Cu3(Fe) pred toplotno obdelavo.

Figure 2. Mapping of the characteristic area of interest of AlSi9Cu3(Fe) alloy prior to heat treatment.

Identifikacija posameznih komponent mikrostrukture v interesnem območju v kovinski matrici, pridobljena z vrstičnim elektronskim mikroskopom in energijsko

from the solidification sequence investigated previously [17-19, 21]. Distribution of chemical elements in the characteristic area of interest in metal matrix, performed



Položaj / območje / Position / Area	Element	Teža / Weight %	Napaka / Error %	Položaj / Mesto / Position / Spot	Element	Teža / Weight %	Napaka / Error %
Area 1	Al K	65,20	4,1	Spot 1	Al K	55,0	5,7
	Si K	10,50	6,3		Si K	2,10	7,4
	Fe K	16,30	3,0		Fe K	1,31	9,9
	Mn K	6,80	3,3		Cu K	42,3	3,5
	Cu K	1,20	13,6		Al K	53,7	5,7
Area 2	Al K	95,60	2,6	Spot 2	Si K	3,20	7,0
	Si K	2,80	7,2		Fe K	0,50	9,9
					Cu K	41,60	3,5
Area 4				Spot 3	Al K	65,40	4,1
					Si K	10,40	6,3
					Fe K	16,50	3,0
					Mn K	6,30	3,3
					Cu K	1,50	10,9
Area 4				Spot 4	Al K	78,60	2,7
					Si K	19,60	6,2
					Cu K	1,80	7,9
Area 4				Spot 5	Al K	63,20	2,9
					Si K	34,10	5,7
					Cu K	1,30	10,5
					Mg K	0,90	4,2

**Slika 3.** Preučitev kemijske sestave posameznih mikrostrukturnih komponent pred toplotno obdelavo  
**Figure 3.** Investigation of the chemical composition of particular microstructural components prior to heat treatment

disperzijskim spektrometrom, je prikazana na sliki 3.

**Pred toplotno obdelavo:** Rezultati optične mikroskopije (OM) in vrstične elektronske mikroskopije (SEM) kažejo značilno mikrostrukturo zlitine AlSi9Cu3(Fe), izdelane s HPDC. Mikrostruktura je sestavljena iz matrice iz primarnega aluminija  $\alpha_{Al}$ , evtektika ( $\alpha_{Al} + \beta_{Si}$ ), intermetalne faze na osnovi železa v pretežno igličasti morfologiji –  $Al_5FeSi$  in tudi v morfologiji »kitajske pisave«  $Al_{15}(Fe,Mn,Cu)_3Si_2$ , ponekod črnega razvejanega  $Mg_2Si$ , intermetalne faze  $Al_8Mg_3FeSi_2$  ter faz  $Al_2Cu$  in  $Al_5Mg_8Si_2Cu_2$  na osnovi bakra. Bakrovi skupki se strjujejo na mejah zrn, medtem ko se razvejana bakrova faza nahaja v interdendritnih prostorih.

Primerjava mikrostrukture treh značilnih vzorcev je prikazana na sliki 4.

**Po toplotni obdelavi:** Rezultati optične mikroskopije (OM) in konfokalne laserske mikroskopije (CLM) kažejo na splošno izboljšano mikrostrukturo zlitine AlSi9Cu3(Fe) zaradi toplotne obdelave, sestavljene iz raztopnega žarjenja pri temperaturi 430 °C in naknadnega staranja pri temperaturi 160 °C. Mikrostruktura je pokazala razdrobljenost in sferoidizacijo finega evtektskega  $\beta_{Si}$ . Večja povečava je pokazala železovo fazo bistveno finejšo in bolj razdrobljeno v igličasti morfologiji –  $Al_5FeSi$  in morfologije kitajske pisave v obliki ploščic ali razdrobljeni obliki –  $Al_{15}(Mn,Fe,Cu)_3Si_2$ , ki so trše od matrice in zasedajo višje mesto v topografiji mikrostrukture. Bakrova in magnezijeva faza se med raztopnim žarjenjem večinoma raztopita v trdno raztopino  $\alpha_{Al}$ . Nekaj razdrobljenih ostankov pa je bilo mogoče zaznati na mejah zrn.

Merjenje topografije s konfokalnim laserskim mikroskopom omogoča oceno razvoja komponent mikrostrukture v osi Z in s tem primerjavo trdote posameznih

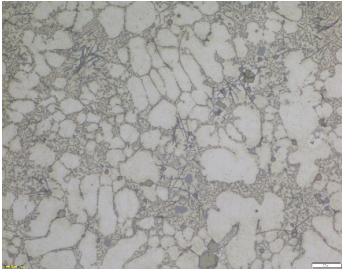
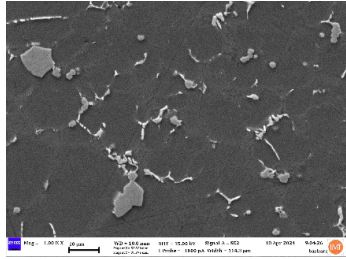
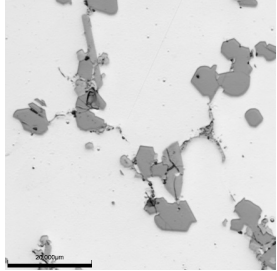
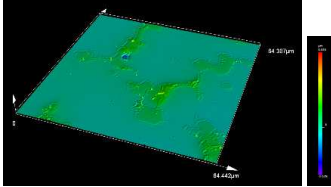
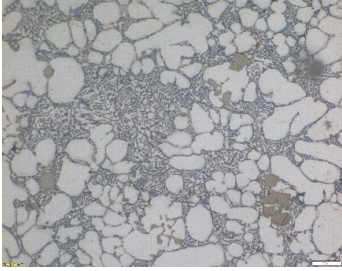
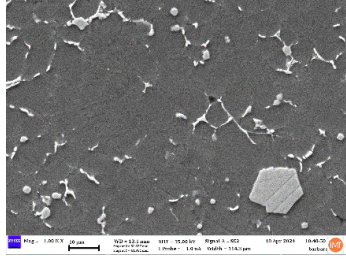
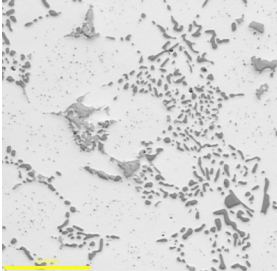
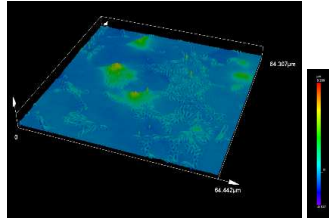
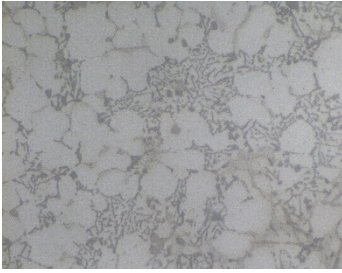
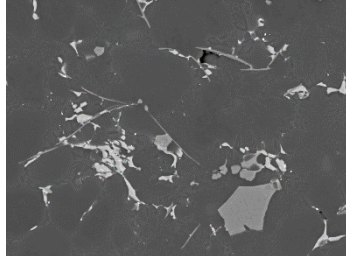
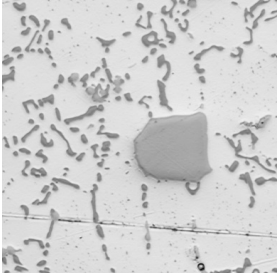
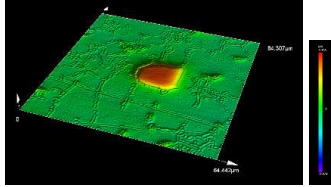
by mapping using SEM equipped with EDS, is shown in Figure 2.

Identification of particular microstructure components at the area of interest in metal matrix, obtained by SEM and EDS is shown in Figure 3.

**Prior heat treatment:** Results of optical microscopy (OM) and scanning electron microscopy (SEM) show the typical microstructure of AlSi9Cu3(Fe) alloy produced by HPDC. Microstructure investigation consists of a matrix of primary aluminium  $\alpha_{Al}$ , a eutectic ( $\alpha_{Al} + \beta_{Si}$ ), an intermetallic phase on the iron base in predominantly needle-like morphology -  $Al_5FeSi$  and also in  $Al_{15}(Fe,Mn,Cu)_3Si_2$  "Chinese script" morphology, somewhere black branched  $Mg_2Si$ , an intermetallic  $Al_8Mg_3FeSi_2$  phase, and  $Al_2Cu$  and  $Al_5Mg_8Si_2Cu_2$  phases on the copper base. Copper clusters are precipitated on the grain boundaries, while the ramified copper phase is located in interdendritic spaces.

Comparison of the microstructure for three characteristic samples is presented in Figure 4.

**After heat treatment:** The results of optical microscopy (OM) and confocal laser microscopy (CLM) show generally refined microstructure of the AlSi9Cu3(Fe) alloy due to heat treatment consisting of solution heat treatment at 430 °C and subsequent ageing at 160 °C. Microstructure revealed fragmentation and spheroidisation of fine eutectic  $\beta_{Si}$ . A higher magnification revealed significantly finer and fragmented iron base phase in small needle-like -  $Al_5FeSi$  and platelet or fragmented Chinese script morphology -  $Al_{15}(Mn,Fe,Cu)_3Si_2$ , which are harder than the matrix and occupy a higher position in microstructure topography. The copper and magnesium phases largely dissolve in the  $\alpha_{Al}$  solid solution during solution heat treatment. However, some of the fragmented residues could be detected at the grain boundaries.

PRED TOPLOTNO OBDELAVO / PRIOR HEAT TREATMENT		PO TOPLOTNI OBDELAVI / AFTER HEAT TREATMENT	
OM – Olympus BX51	SEM – JEOL5610, Zeiss CrossBeam 550	CLM – Olympus Lext OLS5100 – 2D	CLM – Olympus Lext OLS5100 – 3D
			
			
			

**Slika 4.** Razvoj mikrostrukture zlitine AlSi9Cu3(Fe) pred toplotno obdelavo in po njej

**Figure 4.** Microstructure development of AlSi9Cu3(Fe) alloy prior and after heat treatment

komponent na podlagi njihove višine po metalografskipripravi. Z laserskimi meritvami smo ugotovili, da je faza  $\text{Al}_{15}(\text{Mn,Fe,Cu})_3\text{Si}_2$  v obliki ploščic in razdrobljena (zeleno-rumeno-rdeča) najvišja in najtrša faza v mikrostrukturi. Ničelna raven je večinoma namenjena aluminijasti kovinski matrici. Najnižje intermetalne faze (zeleno-modre) so verjetno najmehkejše faze na osnovi bakra in/ali magnezija, ki po toplotni obdelavi ostanejo na mejah zrn.

#### 4 Sklepne ugotovitve

Kot varnostno pomembni material se standardna aluminijeva zlitina  $\text{AlSi9Cu3(Fe)}$  (EN AC 46000) pogosto uporablja v avtomobilski in transportni industriji ter zanjo veljajo visoke zahteve glede funkcionalnih in mehanskih lastnosti. Nanje vplivajo predvsem lastnosti surovin, obdelava taline in tehnologija litja. Zaradi velike uporabe sekundarnih, tj. recikliranih surovin in tudi kritičnih surovin (CRM), je treba posebno pozornost nameniti kemijski sestavi zaradi možnega poslabšanja zaradi večkratnega ponovnega taljenja, ki lahko povzroči poslabšanje mehanskih in drugih lastnosti uporabe. Ustrezna toplotna obdelava je vedno veljala za režim homogenizacije mikrostrukture in s tem izboljšanja mehanskih lastnosti. V tej preučitvi smo z različnimi mikroskopi analizirali mikrostrukturo ponovno taljene zlitine za tlačno litje  $\text{AlSi9Cu3(Fe)}$  v različnih stanjih pred toplotno obdelavo in po njej.

Pred toplotno obdelavo zlitino  $\text{AlSi9Cu3(Fe)}$  sestavljajo  $\alpha_{\text{Al}}$ , evtektk ( $\alpha_{\text{Al}} + \beta_{\text{Si}}$ ), intermetalne faze  $\text{Al}_5\text{FeSi}$ ,  $\text{Al}_{15}(\text{Fe,Mn,Cu})_3\text{Si}_2$ ,  $\text{Mg}_2\text{Si}$ ,  $\text{Al}_8\text{Mg}_3\text{FeSi}_2$ ,  $\text{Al}_2\text{Cu}$ ,  $\text{Al}_5\text{Mg}_8\text{Si}_2\text{Cu}_2$ . Po natančno določenem režimu toplotne obdelave (raztopno žarjenje pri temperaturi 430 °C, ki mu sledi staranje pri temperaturi 160 °C), da bi preprečili nastanek mehurjev, je bila

Measurement of the topography using a confocal laser microscope enables evaluation of microstructure components development in Z axes and thus to compare the hardness of particular components based on their height after metallographic preparation. The laser measurement reveals that the platelet-shaped and fragmented  $\text{Al}_{15}(\text{Mn,Fe,Cu})_3\text{Si}_2$  phase (green-yellow-red) is the highest and hardest phase in the microstructure. Zero level is mostly dedicated to the aluminium metal matrix. The lowest intermetallic phases (green-blue) are probably the softest phases, on the base of copper and/or magnesium, remaining at the grain boundaries after heat treatment.

#### 4 Conclusions

As a safety-critical material, the standard aluminium alloy  $\text{AlSi9Cu3(Fe)}$  (EN AC 46000) is widely used in the automotive and transport industries and exposed to high requirements in terms of its functional and mechanical properties. These are mainly influenced by the raw material properties, melt treatment, and casting technology. The significant use of secondary, i.e., recycled raw material and also CRM, requires special attention to the chemical composition due to potential degradation as a result of repeated remelting, which can lead to deterioration in mechanical and other usage properties. Appropriate heat treatment has always been considered a regime for homogenisation of microstructure and thus improvement of mechanical properties. In this investigation, the microstructure of the remelted HPDC  $\text{AlSi9Cu3(Fe)}$  alloy was analysed using different microscopes, in different states prior and after the heat treatment.

Prior heat treatment HPDC  $\text{AlSi9Cu3(Fe)}$  alloy microstructure consists

mikrostruktura bistveno finejša. Opazovanje mikrostrukture kaže:

- homogeno in popolnoma izboljšano mikrostrukturo;
- razdrobljen in fino sferičen evtektski  $\beta_{Si}$ ;
- razdrobljeno igličasto morfologijo  $Al_5FeSi$  in morfologijo kitajske pisave v obliki ploščic ali razdrobljeni obliki –  $Al_{15}(Mn,Fe,Cu)_3Si_2$ , ki je na podlagi laserske meritve topografije bistveno trša od vseh drugih sestavin;
- na mejah zrn je bilo mogoče zaznati nekaj razdrobljenih ostankov bakrove in magnezijeve faze.

of  $\alpha_{Al}$ , eutectic ( $\alpha_{Al}+\beta_{Si}$ ), intermetallic phases'  $Al_5FeSi$ ,  $Al_{15}(Fe,Mn,Cu)_3Si_2$ ,  $Mg_2Si$ ,  $Al_8Mg_3FeSi_2$ ,  $Al_2Cu$ ,  $Al_5Mg_8Si_2Cu_2$ . After a carefully defined heat treatment regime (solution heat treatment at 430 °C followed by ageing at 160 °C) to avoid blistering, the microstructure was significantly finer. Observation of microstructure shows:

- homogeneous and completely refined microstructure;
- fragmented and spherical fine eutectic  $\beta_{Si}$ ;
- fragmented needle-like  $Al_5FeSi$  and platelet-shaped or fragmented Chinese script morphology -  $Al_{15}(Mn,Fe,Cu)_3Si_2$ , which is significantly harder than all other components, based on laser topography measurement;
- Some of the fragmented residues of copper and magnesium phases could be detected at the grain boundaries.

## Viri / References

1. European Aluminium, <https://european-aluminium.eu/projets/a-low-carbon-footprint/>
2. European Commission, Directorate-General for Internal Market, Industry, Entrepreneurship and SMEs, Grohol, M., Veeh, C., Study on the critical raw materials for the EU 2023: final report, Publications Office of the European Union, 2023, <https://data.europa.eu/doi/10.2873/725585>
3. European Commission, EU principles for sustainable raw materials, Publications Office of the European Union, 2021, <https://ec.europa.eu/docsroom/documents/47815>
4. Z. Zovko Brodarac, F. Kozina, J. Medved, J. Burja, Investigation of recycled  $AlSi9Cu3(Fe)$  alloy, 63rd International Foundry Conference, Ljubljana: Slovenian Foundrymen Society, Portorož, 2023, 10, 1-9
5. Metallurgical Innovation and Materials for Industrial Technology Improvement, Mapping Study on Aluminium Melt Purification from Consumer Scrap, 2023, file:///C:/Users/Admin/Downloads/Mapping-Study\_Full-Report\_Final.pdf
6. IRT M2P, Technical report: Market Development for secondary Casting Alloys beyond Motor Blocks - Study on casting alloy market and recycling, 2023, file:///C:/Users/Admin/Downloads/IRT\_M2P\_Market-Development-for-2ary-Casting-Alloys-beyond-Motor-Blocks\_final.pdf
7. A. Fabrizi, S. Capuzzi, A. De Mori, G. Timelli, Effect of T6 Heat Treatment on the Microstructure and Hardness of Secondary  $AlSi9Cu3(Fe)$  Alloys Produced by Semi-Solid SEED Process, *Metals*, 8 (2018)10, 750, doi:10.3390/met8100750
8. J. Kasińska, D. Bolibruchová, M. Matejka, The Influence of Remelting on the Properties

- of AlSi9Cu3 Alloy with Higher Iron Content, *Materials* 13 (2020)3, 575, doi:10.3390/ma13030575
9. Z. Zovko Brodarac, D. Stanić, M. Folta, F. Kozina, Influence of remelting on mechanical properties of AlSi9Cu3(Fe) alloy, *74th\_WFC\_E-Abstract\_Book*, (ed. Park, Eun Soo), Busan: The World Foundry Organization, Korea Foundry Society, Busan, South Korea, 2022, 1-0437
  10. A. Bjurenstedt, On the influence of imperfections on microstructure and properties of recycled Al-Si casting alloys, *Jönköping University School of Engineering*, 2017, Dissertation Series No. 28
  11. L. Hurtalová, E. Tillová, M. Chalupová, The structure analysis of secondary (recycled) AlSi9Cu3 cast alloy with and without heat treatment, *Engng. Trans.* 61 (2013)3, 197–218
  12. Z. Zovko Brodarac, T. Holjevac Grgurić, M. Vončina, D. Stanić, Thermodynamic and microstructural characterization of AlSi8Cu3 alloy, *Livarski vestnik*, 61 (2014)4, 193-209
  13. Z. Zovko Brodarac, N. Dolić, F. Unkić, Influence of chemical composition on microstructure development of EN AB AlSi9Cu3 alloy, *IOC 2011 Proceedings* (ed. D. Marković, D. Živković, S. Nestorović), Bor: Tehnički fakultet u Boru Univerziteta u Beogradu, Bor, Srbija, 2011, 683-686
  14. Z. Zovko Brodarac, D. Stanić, F. Unkić, Microstructural evaluation of AlSi9Cu3 alloy, *Conference proceedings of 51st International Foundry Conference Portorož 2011*, (ed. M. Debelak), Ljubljana: Društvo livarjev Slovenije, Portorož, Slovenija, 2011, 74-75
  15. Z. Zovko Brodarac, M. Trbižan, F. Unkić, Study of shrinkage cavity occurrence in AlSi9Cu3 high pressure die casting, *Livarski vestnik* 50 (2003)3
  16. C.H. Cacers, M.B. Djurdjevic, T.J. Stockwell, J.H. Sokolowski, The Effect of Cu Content on the Level of Microporosity in Al–Si–Cu–Mg Casting Alloys, *Scr. Mater.*, 40 (1999)5, 631–637
  17. Z. Zovko Brodarac, F. Kozina, D. Stanić, M. Folta, Quality assessment of the AlSi9Cu3(Fe) alloy with regard to recycling ratio, *Congress Proceedings - MME SEE 2023 5th Metallurgical & Materials Engineering Congress of South-East Europe* (ed. M. Sokić, B. Marković, V. Manojlović). Beograd: Association of Metallurgical Engineers of Serbia (AMES), Trebinje, Bosnia and Herzegovina, 2023, 251-256
  18. Z. Zovko Brodarac, F. Kozina, J. Medved, J. Burja, Investigation of recycled AlSi9Cu3(Fe) alloy, *Conference Proceedings 63rd International Foundry Conference (IFC)*, (ed. A. Križman, P. Mrvar, J. Medved, Jožef et al.), Ljubljana: Slovenian Foundrymen Society, Portorož, Slovenia, 2023, 10, 1-9
  19. Z. Zovko Brodarac, F. Kozina, D. Stanić, M. Folta, Solidification of remelted AlSi9Cu3(Fe) alloy, *Book of abstracts of the 7th Central and Eastern European Conference on Thermal Analysis and Calorimetry (CEEC-TAC7)*, Brno: SITECH, Brno Czech Republic, 2023, 134-134
  20. Z. Zovko Brodarac, D. Stanić, M. Folta, F. Kozina, Recycling potential of AlSi9Cu3(Fe) alloy, *Conference proceedings 61st International Foundry Conference (IFC)*, (ed. A. Križman, P. Mrvar, J. Medved, Jožef et al.), Ljubljana: Slovenian Foundrymen Society, Ljubljana: Slovenian Foundrymen Society, Portorož, Slovenia, 2021, 1-8
  21. Z. Zovko Brodarac, D. Stanić, K. Terzić, Utjecaj toplinske obrade žarenjem na svojstva mikrostrukturnih konstituenata AlSi8Cu3 legure, *Conference Proceedings MATRIB*

- 2013 (ed. Ž. Alar, S. Jakovljević, S. Šolić), Zagreb: Hrvatsko društvo za materijale i tribologiju (HDMT), Vela Luka, Croatia, 2013, 76-83
22. D. Stanić, Z. Zovko Brodarac, K. Terzić, , Utjecaj žarenja radi odstranjivanja jezgri na mehanička i mikrostrukturna svojstva AlSi8Cu3 legure, Proceedings book of 13th International Foundrymen Conference – IFC (ed. Z. Glavaš, Z. Zovko Brodarac, N. Dolić), Sisak: Metalurški fakultet Sveučilišta u Zagrebu, Opatija, Croatia, 2013, 376-385
23. S. Cecchel, A. Panvini, G. Cornacchia, Low Solution Temperature Heat Treatment of AlSi9Cu3(Fe) High-Pressure Die-Casting Actual Automotive Components, JMEPEG 27(2018), 3791–3802, doi:10.1007/s11665-018-3478-4
24. E. Sjölander, S. Seifeddine, Optimisation of solution treatment of cast Al-Si-Cu alloys. Mater. Des. 31(2010), 44-49
25. D.L. Zhang, L.H. Zheng, D.H. StJohn, Effect of a short solution treatment time on microstructure and mechanical properties of modified Al-7wt.%Si-0.3wt.%Mg alloy, J. Light Met. 2(2002), 27–36.
26. Y. Han, A.M. Samuel, H.W. Doty, S. Valtierra, F.H. Samuel, Optimizing the tensile properties of Al-Si-Cu-Mg 319-type alloys: Role of solution heat treatment, Mater. Des. 58(2014), 426–438.

## AKTUALNO / CURRENT

### Livarske prireditve 2025/26

Datum dogodka	Ime dogodka	Mesto in država
16. - 18. 07. 2025	China Diecasting & China Nonferrous Expo	Shanghai, Kitajska
17. - 19. 09. 2025	65. IFC Portorož 2025	Portorož, Slovenija
2. - 3. 10. 2025	World Foundry Summit	Pariz, Francija
24. - 25. 10. 2025	Ledebur-Kolloquium	Freiberg, Nemčija
25. - 28. 10. 2025	The 17th Asian Foundry Congress	Xi'an, Kitajska
12. - 13. 11. 2025	Parts Finishing	Karlsruhe, Nemčija
12. 12. 2025	Polish Foundry Day	Krakov, Poljska
13. - 15. 01. 2026	Euroguss 2026	Nürnberg, Nemčija
09. - 11. 06. 2026	CastForge 2026	Stuttgart, Nemčija