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Razvoj zlitine AlSi7Mg z dodatki Li

Development of AlSi7Mg Alloy with Additions of Li

Povzetek

Aluminijeve zlitine z dodatkom litija so poznane že več desetletij. Glavno prednosti dodatka Li je zmanjšanje gostote zlitine in izboljšanje mehanskih lastnosti, še posebej modula elastičnosti. Izboljšanje mehanskih lastnosti v glavnem povzroča strjevanje precipitata metastabilne faze δ’ (Al_3Li). Glavna pomanjkljivost zlitin Al-Li je reaktivnost Li, Saj je močno nagnjen k tvorbi oksidov in lahko reducira različne klovinske okside in vodo. Med temi reakcijami lahko nastajajo plini (npr. Vodik), ki lahko povzročijo poroznost. Podobne težave nastajajo med reakcijami z materiali za forme, pri čemer lahko produkti reakcij vplivajo na kakovost ulitkov.

V tem delu smo preučili dodatek Li zlitini AlSi7Mg. Dodatek Li je enak 1 mas.%. Različne materiale form, kot so croning peščena mešanica, peščena mešanica za postopek CO2, silikatne opeke ter grafitne in jeklene forme, smo preizkusili z namenom določevanja površinske kakovosti ulitka in količino reakcijskih produktov. Najboljši rezultati brez reakcijskih produktov so bili doseženi z grafitnimi in jeklenimi formami. Sočasno je bila izvedena enostavna termična analiza z namenom primerjave poteka strjevanja nove zlitine v povezavi z izračuni termodinamskih ravnotežij. Optično mikroskopijo in vrstično elektronsko mikroskopijo z analizami EDS in XRD smo uporabili za opredelitev mikrostrukturnih elementov v fazah AlLiSi, ki vsebujejo Li. Mehanski preizkusi so pokazali, da pride do naravnega staranja z izločanjem faze Al3Li, s čemer se poveča trdnost materiala.

Ključne besede: aluminijeve zlitine, mikrostruktura, litij, mehanske lastnosti

Summary

Aluminium alloys with lithium additions have been known to the world for several decades. The main advantage of the Li addition is the reduced alloy density and increased mechanical properties, especially the modulus of elasticity. Increased mechanical properties are mainly caused by precipitation hardening of the metastable δ’ phase (Al3Li). The main difficulty of Al- Li alloys is Li reactivity where its tendency to form oxide is very high and can reduce different metal oxides and water. At these reactions it can form gases especially hydrogen which can cause gas porosity. Similar problems are met at reactions with moulding materials where reaction products can influence casting quality.

In the current work the investigation of Li additions to the cast AlSi7Mg alloy is treated. The addition of Li amounts to 1 wt.%. Different moulding materials such as Cronning sand mixture, CO2 sand mixture, silicate brick, graphite and steel mould were tested to determinate the surface quality of a casting and the amount of reaction products. The best results without reaction products were given by graphite and steel mould. At the same time a simple thermal analysis was performed to explain the solidification course of the new alloy in correlation with thermodynamic equilibrium calculations. Optic, scanning electron microscopy with EDS analysis and XRD analysis were used to identify microstructural constituents of which the AlLiSi phase contained Li. Mechanical tests showed that natural ageing occurs, and hardness is increased, indicating that the Al3Li phase precipitates.

Key words: aluminium alloys, microstructure, lithium, mechanical properties.

1 Uvod

Osnovne zlitine Al-Li imajo nizko gostoto, visok modul elastičnosti in visoko trdnost, zaradi česar so te zlitine primerne za vesoljsko industrijo. Visoka trdnost je posledica postopka Izločevalnega utrjevanja. Dodatki elementov lahko tvorijo nekoherentne disperzoide ali semikoherentne precipitate ter spremenijo mikrostrukturo in mehanske lastnosti zlitin [1–5]. Zlitine Al-Li se uporabljajo v letalski industriji, saj zmanjšanje teže ob uporabi teh zlitin nizke gostote močno zmanjša stroške goriva in izboljša učinkovitost delov, kot so: letalski deli, npr. prednji in zadnji robovi, pokrovi loput, vodila za sedeže; vojaški deli in deli za vesoljsko industrijo, kot so osrednje krilne strukture, trup, kontrolne površine – vsi so izdelani iz zlitin Al-Li. Zlitine Al-Li se uporabljajo za zamenjavo konvencionalnih aluminijevih zlitin v helikopterjih, raketah in satelitskih sistemih [5–8].

Izločki zlitin Al-Li so v več metastabilnih in stabilnih fazah, ki so lahko prisotne v zlitinah Al-Li, odvisne pa so od uporabljenih legirnih elementov. Metastabilni izločki v tej študiji so označeni s δ' (Al3Li), medtem ko je stabilna faza označena s δ (AlLi) [8]. Izločki so odvisni od kemije, strukture zrn in celotne termomehanske zgodovine. Pri zlitinah Al-Li je utrjevanje zaradi dodatka Li posledica tako utrjevanja trdne raztopine kot tudi izločevalnega utrjevanja [1–20]. izločevalno utrjevanje je primarno posledica metastabilne utrjevalne faze δ' (Al3Li), ki tvori kroglaste, celovite in urejene delce izločkov, ki so v aluminijevi matrici orientirani v smeri ploskev proti ploskvi [20–25]. Pri

1 Introduction

Al-Li base alloys have low density, high elastic modulus and high strength properties which make these alloys attractive for aerospace applications. The high strength is produced by the process of precipitation hardening. The addition of elements can form incoherent dispersoid or semicoherent precipitates and change the microstructure and mechanical properties of alloys [1-5]. Al-Li alloys use in aircraft applications, where the weight savings effected by using these low- density alloys greatly reduce the vehicle fuel costs and increase performance of parts such as: Aircraft parts such as leading and trailing edges, access covers, seat tracks; military and space applications such as main wing box, centre fuselage, control surfaces are made by Al-Li alloys. Al-Li alloys are used as substitute for conventional aluminium alloys in helicopters, rockets and satellite systems [5-8].

The precipitates of Al-Li alloys are in several metastable and stable phases which can be present in Al-Li alloys, depending on the alloying elements. The metastable precipitates in this section are δ' (Al3Li), while the stable phase discussed is δ (AlLi) [8]. The precipitation, in turn, depends upon chemistry, grain structure, and total thermomechanical history. In Al-Li alloys, the strengthening from Li additions is due to both solid solution strengthening and precipitation hardening [1-20]. The precipitation hardening is primarily due to the metastable strengthening phase, δ' (Al3Li), which forms spherical, coherent ravnotežnem strjevanju in v najenostavnejši obliki binarnih zlitin Al-Li sta v mikrostrukturi prisotni samo z aluminijem bogata trdna raztopina in faza δ (AlLi) [25–30].

Nova aluminijeva zlitina je bila pripravljena in analizirana z dodatki Li na podlagi zlitine AlSi7Mg z izboljšanimi mehanskimi lastnostmi, potek strjevanja pa je bil karakteriziran ob upoštevanju vseh mikrostrukturnih elementov. Za razvoj nove zlitine z dodatkom Li smo uporabili optično mikroskopijo in vrstično elektronsko mikroskopijo (SEM). Toplotno analizo in Diferenčno vrstično kalorimetrijo smo skladno z izračuni termodinamičnega ravnotežja faz uporabili za določitev poteka strjevanja. Prav tako smo izvedli tudi analize XRD in mehansko testiranje (preskušanje trdnosti).

2 Eksperimentalni del

Novo aluminijevo zlitino AlSi7MgLi smo preiskali z različnimi metodami. Termodinamične izračune smo izvedli s programsko opremo ThermoCalc in kemijske sestave zlitin v Pregl. 1 smo uporabiti za izračun faznih diagramov zlitin. Vzorce smo stalili v indukcijski peči v grafitnem talilnem lončku in jih ulili v jeklene forme ter izvedli enostavno termično analizo. Po zajemu numeričnih podatkov smo narisali krivulje hlajenja in njihove izpeljave ter določili značilne temperature. Izvedena je bila diferenčna vrstična kalorimetrija in vzorci so bili pripravljeni za mikrostrukturne preiskave z optičnim mikroskopiranjem in vrstičnim elektronskim mikroskopiranjem z EDS, da bi določili faze, prisotne v zlitini AlSi7Mg. Preizkus trdote po Vickersu smo uporabili za določitev mehanskih lastnosti v 13-dnevnem obdobju po litju.

and ordered precipitate particles having a cube-on-cube orientation relationship with the aluminium matrix [20-25]. At equilibrium, and at its simplest in binary Al-Li alloys, the only phases present are the aluminium-rich solid solution and the δ (AlLi) phase [25-30].

New aluminium cast alloy was produced and analysed with Li additions based on AlSi7Mg alloy with improved mechanical properties where characterisation of solidification path was determined with all microstructural constituents. In order to develop new alloys with Li addition an optic microscope and Scanning electron microscopy (SEM) Thermal analysis and differential scanning calorimetry in accordance with thermodynamic equilibrium calculations were used to determine the solidification course. Also, XRD analyses and mechanical testing (hardness testing,) were performed.

2 Experimental Work

New aluminium cast alloy AlSi7MgLi was investigated experimentally. The thermodynamic calculations were performed with ThermoCalc software and the chemical compositions of alloys given in tab.1 were used in order to calculate phase diagrams of alloys. Samples were melted in an induction furnace in a graphite crucible and cast in a steel mould where simple thermal analysis was performed. After data acquisition their numerical data, cooling curves and their derivatives were plotted and characteristic temperatures determined. Differential scanning calorimetry was performed, and sample prepared for microstructural investigation with optic microscope and scanning electron microscope with EDS in order to determine the phases present in alloy AlSi7Mg. Vickers Hardness test was

Preglednica 1. Kemijska sestava zlitine v wt. %

Table 1. Chemical composition of alloy in wt. %

Zlitina / Alloy	Al	Si	Fe	Cu	Mg	Zn	Ti	Li
AlSi7Mg	Preostanek / Rest	6,7	0,44	0,01	0,35	0,01	0,01	-
AlSi7MgLi	Preostanek / Rest	7,05	0,10	0,05	0,36	0,02	0,09	0,80

3 Rezultati in razprava

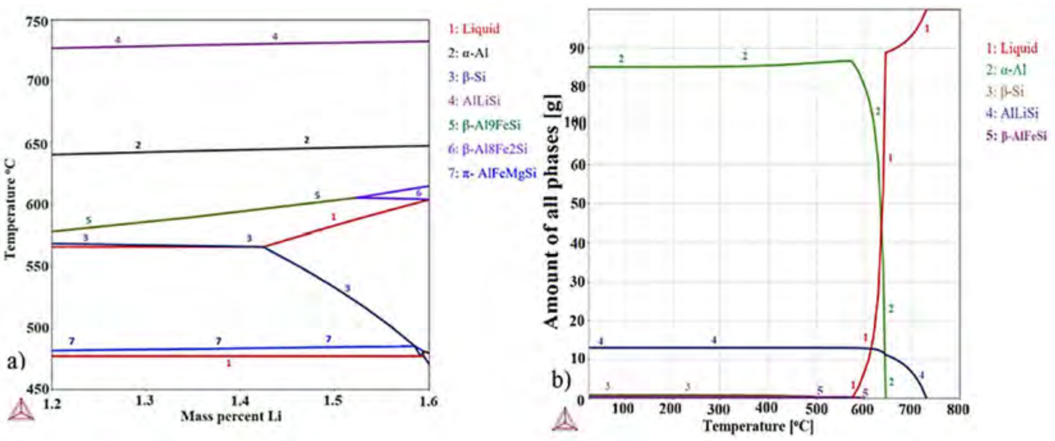
Termodinamični opis sistema zlitin AlSi7MgLi je bil izdelan s programsko opremo ThermoCalc. Na podlagi kemične sestave v Pregl. 1 smo izračunali strjevanje in fazno ravnovesje preskusnih zlitin. Glede na izračune termodinamičnega ravnovesja smo napovedali strjevanje primarne faze α-Al, faze β-Si, nove faze (AlLiSi), Mg2Si, železne faze π-AlMgFeSi in β-AlFeSi.

used to determine mechanical properties in period of 13 days after casting.

Alloy Al Si Fe Cu Mg Zn Ti Li AlSi7Mg Rest 6.7 0.44 0.01 0.35 0.01 0.01 - AlSi7MgLi Rest 7.05 0.10 0.05 0.36 0.02 0.09 0.80

3 Results and Discussion

Thermodynamic description of system AlSi7MgLi cast alloys was constructed using Thermocalc software. From the chemical composition in tab.1 the solidification and equilibrium phases were calculated for experimental alloy. According to the thermodynamic equilibrium calculation we



Slika 1. a) fazni diagram preskusne zlitine AlSi7MgLi in b) količina faz zlitine med strjevanjem

Figure 1. a) phase diagram of experimental alloy AlSi7MgLi and b) amount of phases of alloy during solidification

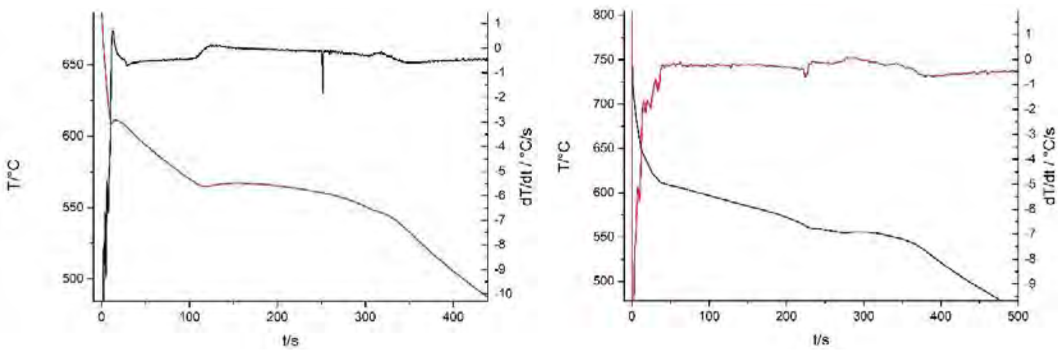
4 Enostavna termična analiza

Proces strjevanja je bil analiziran s termično analizo vzorcev, litih v jeklene forme in celico Croning, vsak vzorec se je ohlajal na zraku. Po pridobitvi podatkov smo izvedli grafično obdelavo z označenimi temperaturami faznih transformacij. Krivulje ohlajanja in odvodi krivulj zlitine AlSi7Mg z dodatkom Li od temperature litja 740 °C v jeklene forme so prikazane na Sl. 2. Z diagrama s krivuljami ohlajanja je razvidno znatno odstopanje od vrednosti značilnih temperatur ohlajanja; glede na izračune in diagram ohlajevalnih krivulj pri temperaturi litja 740°C se strjevanje začne pri pribl. 650 °C. Glede na rezultate izračunov s programsko opremo ThermoCalc bi se morala strjevati najprej faza AlLiSi, nato faza α-Al, železne faze, faza β-Si v evtektični obliki in na koncu faza Mg2Si. Interval strjevanja se zaključi pri temperaturi 536 °C. Po drugi strani je temperatura likvidusa zlitine AlSi7Mg precej nižja, in sicer pribl. 610 °C, ob koncu strjevanja pa meri pribl. 533 °C, kar je podobno zlitini z Li.

have predicted solidification of the primary α-Al, β-Si, new phases (AlLiSi), Mg2Si, iron bearing phase π- AlMgFeSi and β-AlFeSi.

4 Simple Thermal Analysis

Solidification process was analysed by thermal analysis on the samples cast in the steel mould and Croning cell, each sample subjected to the solidification by cooling in the air. After the data acquisition their numerical and graphical processing with the marked temperatures of the phase transformations was performed. The cooling and differentiated curves of AlSi7Mg alloy with Li addition from casting temperature 740 °C in steel mould are shown in fig. 2. The diagram with cooling curves indicates significant deviation in values of the characteristic temperatures of the solidification, according to calculation and diagram of cooling curves at 740 °C with solidification start at around 650°C. according to ThermoCalc results the AlLiSi phase should precipitate, followed by α-Al, iron bearing phases, β-Si in form of eutectic and Mg2Si at the end. The solidification interval ends at 536 °C. On the other hand,



(a) (b)

Slika 2. Ohlajevalna krivulja in njen odvod za zlitino a) AlSi7Mg in b) AlSi7MgLi

Figure 2. The cooling curve and differentiated curves of a) AlSi7Mg and b) AlSi7MgLi alloy

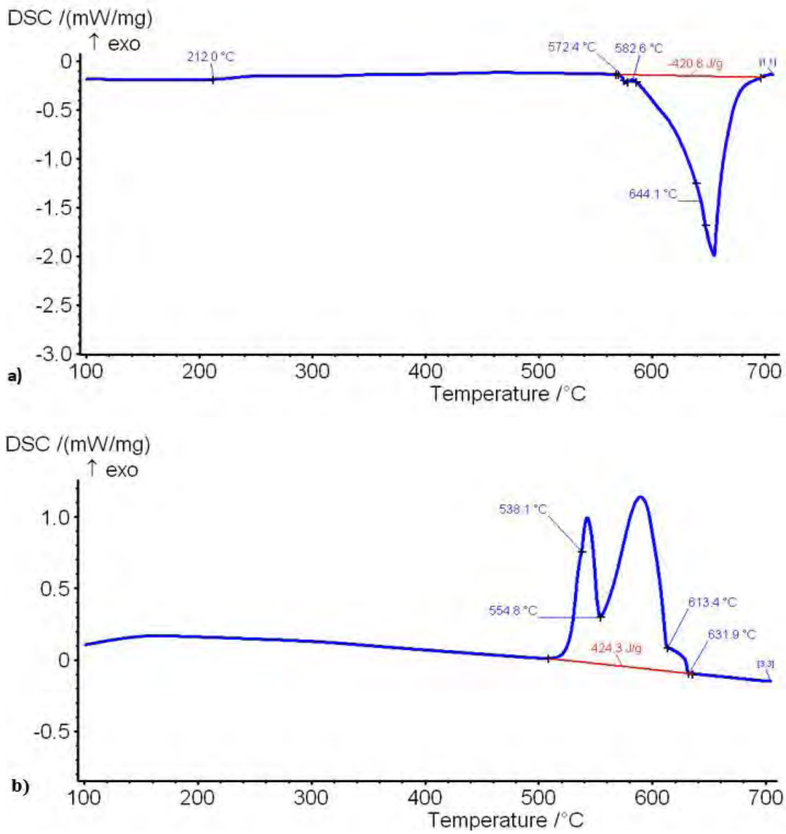
5 Diferenčna vrstična kalorimetrija

Krivulja segrevanja zlitine z dodatkom Li nakazuje, da se taljenje začne pri temperaturi 572 °C, in naklon krivulje se spremeni pri temperaturi 582,6 °C. Pri temperaturi 644,1 °C ven smo nadaljevali z oceno faz; majhen vrh pri temperaturi 212 °C ven nakazuje precipitacijo faze Al3Li. simultana termična analita je bila izvedena na vzorcu litem v jekleno kokilo (formo). Na podlagi diferenčne vrstične kalorimetrije (DSC) smo izdelali diagrame krivulj segrevanja in ohlajanja, ki so prikazane

the liquidus temperature of AlSi7Mg alloy is much lower at around 610 °C and the end of solidification is at around 533 °C which is similar to the alloy with Li.

5 Differential Scanning Calorimetry

The heating curve of alloy with Li addition indicates the melting start at 572 °C and the curve changes after 582.6 °C and continued with evaluation of phase at 644.1 °C, furthermore small peaks at 212 °C indicates precipitation process of Al3Li

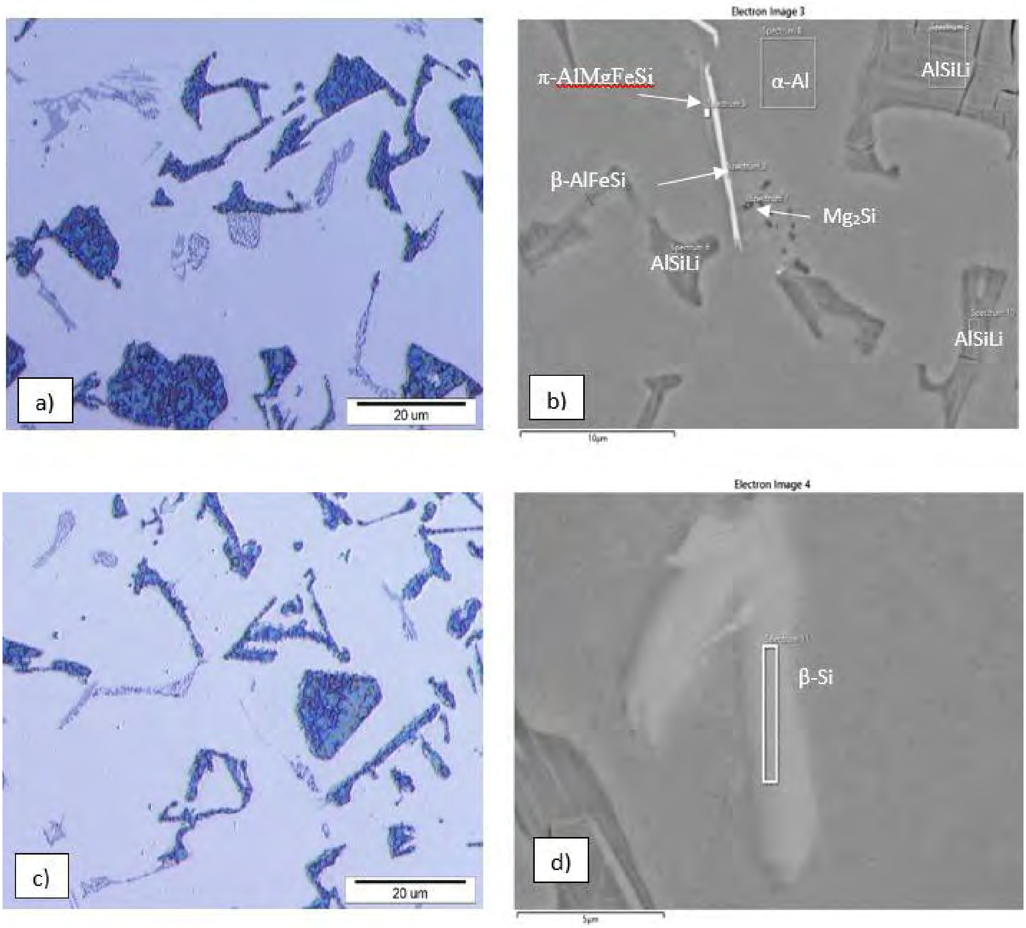


Slika 3. Krivulja segrevanja DSC (a) in krivulja ohlajanja DSC(b) zlitine AlSi7MgLi

Figure 3. Heating DSC curve (a) and cooling DSC curve (b) of AlSi7MgLi alloy

na Slikah 3-a in 3-b. Diagrami na Sl. 3 prikazujejo pomembne temperature faznih transformacij. Temperatura na začetku strjevanja (temperatura likvidusa) je merila 631,9 °C pri fazi AlLiSi, pri temperaturi 613 °C bi se morala strjevati faza α-Al+AlLiSi ven, glede na izračune s programsko opremo ThermoCalc pa je ta temperatura 554,8 °C, sledi železna faza β-AlFeSi pri temperaturi 538 °C ter fazi β-Si in Mg2Si pri temperaturi 554 °C.

phase. Simultaneous thermal analysis was performed on the sample part from the sample poured in the steel mould, differential scanning calorimetry (DSC) resulted in diagrams of the heating and cooling curves shown in the figure 3-a and 3-b. The diagrams in fig. 3 resulted in values of significant temperatures of the phase transformations. The temperature of the solidification starts (liquidus temperature) at 631.9 °C with AlLiSi phase, at 613°C



Slika 4. Optična mikroskopija (a, c) in mikroskopija SEM (b, d) zlitine AlSi7MgLi

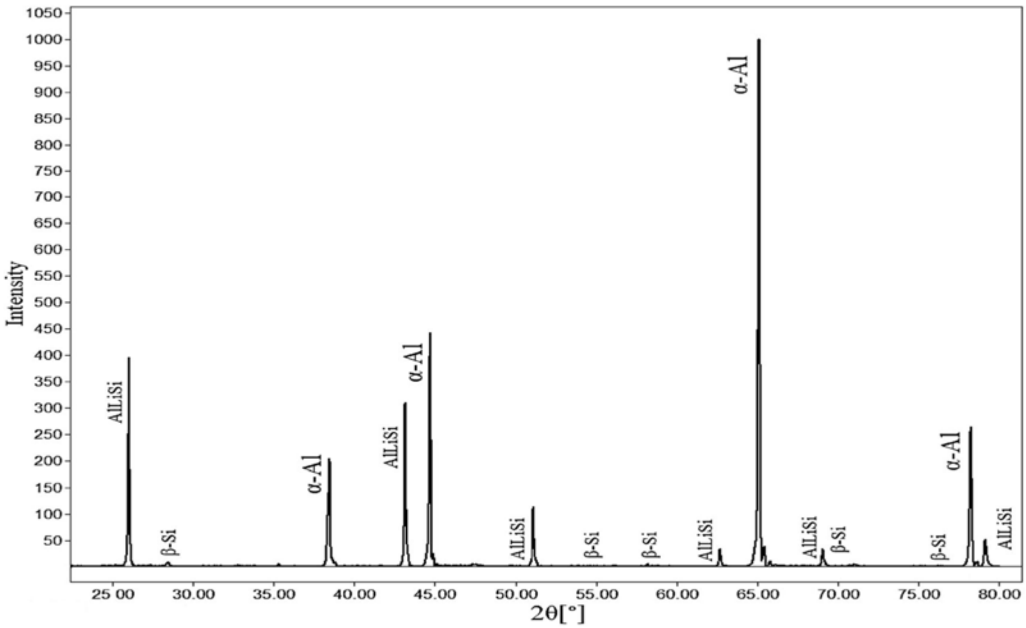
Figure 4. Optical (a, c) and SEM micrographs (b, d) of AlSi7MgLi

6 Mikrostrukturna analiza

Po analizi strjevanja z metodo termične analite smo vzorce pripravili za metalografske raziskave. Glede na metalografijo je mikrostruktura sestavljena iz faze α-Al, faze AlLiSi, faze π-AlMgFeSi ter faze Mg_2Si, kar potrjujeta analizi EDS in XRD, ki sta prikazani na Sl. 4b, 4d, 4f in Sl. 5. Z vrstično elektronsko mikroskopijo smo opredelili faze, ki nastajajo pri dodatku Li. Na podlagi Sl. 4 je mogoče zaključiti, da se pojavi nova faza AlLiSi. Glede na izračune s programsko opremo ThermoCalc meri sestava elementov faze, ki je prisotna v zlitini v litem stanju, 33 mas. % vsakega elementa faze AlLiSi, z analizama SEM in EDS pa smo dokazali, da AlSi vsebuje litij, in sicer na podlagi termodinamičnih izračunov. Mikrostruktura zlitine z dodatkom Li je sestavljena iz faz α-Al, β-AlFeSi, the α-Al+AlLiSi should solidify according to thermocalc at 554.8 °C, followed by iron bearing phase β-AlFeSi at 538°C, β-Si and Mg2Si at 554 °C.

6 Microstructure Analysis

After analysis of solidification by thermal analysis technique samples were prepared for metallographic investigation. According to micrographs the microstructure consists of α-Al phase, AlLiSi phase, π- AlMgFeSi phase, Mg2Si which they are confirmed by EDS and XRD analysis and they presented in fig.4b, 4d, 4f and fig.5. With SEM we observed phases which they are formed with Li addition. From fig.4. it can be concluded that new phase AlLiSi appears. According to ThermoCalc calculation the elemental composition of phase present as-cast state



Slika 5. Vzorec XRD zlitine AlSi7MgLi

Figure 5. XRD pattern of AlSi7MgLi alloy

π-AlMgFeSi in Mg_2Si. Z analizo XRD smo opredelili faze, ki nastajajo pri dodatku Li. Na podlagi Sl. 5 lahko potrdimo nastanek alfa faze α-Al ter faz AlLiSi in β-Si.

7 Mehanske lastnosti

Vzorci so se s staranjem utrjevali 13 dni, meritve mikro trdote so bile izvedene pri sobni temperaturi in izračunane so bile povprečne vrednosti.

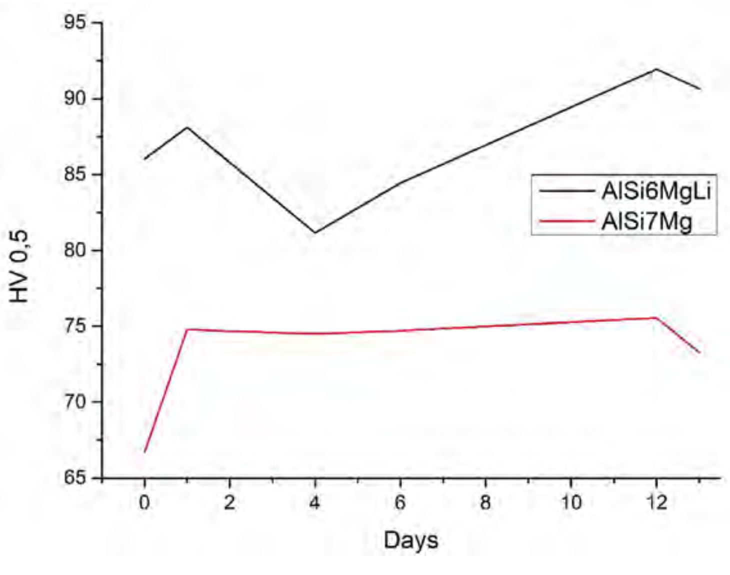
Analiza ven trdnosti vzorca AlSi7Mg za prvo meritev je dala rezultat 66 HV. Med 2.–4. meritvijo so se vrednosti trdote linearno višale od 66 HV do 74,5 HV. Zaključimo lahko, da je bila najvišja trdota dosežena po 12 dneh. Analiza preizkusa trdote vzorca AlSi7Mg za prvo meritev je dala rezultat 86 HV. Po štirih dneh staranja je trdota vzorca padla do 81 HV. Po 6 do 13 dneh se je trdota zlitine AlSi7MgLi postopoma višala s 84 HV do 90 HV, vrh pa je bil dosežen pri vrednosti 91 HV. Zaključimo lahko, da je bila najvišja trdota dosežena po 12 dneh, in sicer je merila 102 HV. Naše raziskave so pokazale, da se je trdota v 13 dneh staranja

alloy the percentage of each element in the phase of AlLiSi is 33 wt %, after analysis by SEM- EDS it was proved that AlSi contains lithium according to the thermodynamic calculation. Furthermore, microstructure in the alloy with Li addition consists from α-Al, β-AlFeSi, π-AlMgFeSi and Mg_2Si. With XRD analysis we observed phases which were formed with Li addition. From fig. 5 we confirm the formations of alpha α-Al, AlLiSi phases and β-Si.

7 Mechanical Properties

The samples were age-hardened for 13 days where micro hardness measurements were performed at room temperature and average values were calculated.

Hardness test analysis for sample AlSi7Mg for first measurement was 66 HV. During 2-4 measurements, values showed a linear increase of hardness from 66 HV-74.5 HV. It can be concluded that peak hardness was achieved after 12 days. Hardness test analysis for sample AlSi7MgLi showed



Slika 6. Graf trdnosti naravnega staranja zlitin AlSi7Mg in AlSi7MgLi

Figure 6. Graph of hardness for natural ageing of AlSi7Mg and AlSi7MgLi.

povečala, in sicer iz 66 na 75 za zlitino AlSi7Mg, pri zlitini AlSi7Mg z dodatkom Li pa se je povečala iz 86 na 91 HV. Glede na rezultate obeh zlitin ima dodatek Li zlitini AlSi7Mg pozitiven vpliv na izboljšanje trdote v primerjavi z zlitino AlSi7Mg.

8 Sklepi

Preučil smo novo zlitino AlSi7Mg z dodatkom Li. V ta namen sta bili zasnovana zlitina AlSi7MgLi, ki vsebuje tudi 0,36 wt. % Mg in 7,05 wt. % Si, dodanega 0,80 wt. % Li. Odkrili smo, da pri zlitini AlSi7Mg z dodatkom Li poteče drugačno strjevanje, prav tako pride do nastanka novih mikrostruktur kot tudi nove faze AlLiSi. Li vpliva na izboljšanje mehanskih lastnosti zlitine AlSi7Mg v litem stanju. Analize SEM/EDS in XRD so razkrile, da dodatek Li spodbuja nastanek nove faze AlLiSi.

the hardness for 0 day was 86 HV in as cast state. After four days of ageing, the hardness of sample dropped until 81 HV. After 6 to 13 days hardness of AlSi7MgLi alloy increased gradually from 84 HV to 90 HV where plateau is reached at 91 HV. It can be concluded that peak of hardness was achieved after 12 days at 102 HV. Our research showed that value of hardness increased during 13 days of ageing time from 66-75 at AlSi7Mg whereas at AlSi7Mg with Li addition from 86-91 HV. According to results of both alloys, the Li addition to AlSi7Mg has great influence on increasing of hardness compared to AlSi7Mg.

8 Conclusion

New alloy with Li addition to AlSi7Mg alloy was studied. For this purpose, 0.80 wt. % Li was added to designed AlSi7MgLi alloy containing 0.36 wt. % Mg and 7.05 wt. % Si. It was found that Li combination with AlSi7Mg resulted in different solidification, development of new microstructure and creation of new phase AlLiSi. Li has the influence to increase the mechanical properties in as cast state to AlSi7Mg. SEM/EDS and XRD analysis revealed that the addition of Li promotes the formation of new phase AlLiSi.

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