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Izločki v aluminijevi kvazikristalni zlitini

Precipitates in an aluminium quasicrystalline alloy

Izvleček

Aluminijeve kvazikristalne zlitine predstavljajo nov razred visokotrdnostnih aluminijevih zlitin in imajo velik potencial za uporabo na različnih področjih. Raziskali smo aluminijevo kvazikristalno zlitino, ki vsebuje baker. Dodatek bakra omogoča utrjanje zlitine pri toplotni obdelavi. Zlitine smo izdelali z gravitacijskim litjem v bakreno kokilo. Pri strjevanju so nastali kvazikristali, ki so bili navzoči v obliki primarnih delcev ter kot sestavni del evtektika. Potem smo zlitine toplotno obdelali, da smo dosegli stanje T5. Po tej toplotni obdelavi se trdota vzorcev ni bistveno spremenila. Podrobna preiskava s presevno elektronsko mikroskopijo (TEM) je pokazala, da so v zlitini različni izločki. Na nastanek izločkov je imela največji vpliv temperatura toplotne obdelave. Pri nižjih temperaturah so nastali binarni izločki Al-Cu, v vmesnem temperaturnem območju so nastajali kvazikristalni izločki, medtem ko so se pri višjih temperaturah tvorili izločki faze Al₂₀Mn₂Cu₂.

Ključne besede: toplotna obdelava, aluminijeva zlitina, izločki, kvazikristal, TEM.

Abstract

Aluminium quasicrystalline alloys represent a novel class of high-strength alloys. They possess a great potential for practical applications in many fields. In this study, we used a quasicrystalline Al-alloy with a copper addition in order to achieve strengthening by heat treatment. The alloys were prepared by casting into a copper mould. Quasicrystals formed during solidification. They were present either as a primary phase or as a part of a quasicrystal-containing eutectic. Afterwards, the samples were subjected to T5 heat treatment. The hardness of melt-spun ribbons increased considerably, while the hardness of gravitationally cast samples did not change noticeably. Detailed investigation using transmission electron microscopy (TEM) showed that different types of precipitates can form, depending primarily on the heat treatment temperature. At lower temperatures binary Al-Cu precipitates formed, in the intermediate region prevailed the formation of quasicrystalline precipitates, while at higher temperatures formed Al₂₀Mn₂Cu₂ precipitates.

Keywords: heat treatment, aluminium alloy, precipitates, quasicrystal, TEM.

1 Uvod

S kvazikristali utrjene Al-zlitine imajo lahko izvrstno kombinacijo trdnosti in duktilnosti. Ikozaedrična kvazikristalna faza (IQC), ki je prisotna v teh zlitinah, je metastabilna.

1 Introduction

Quasicrystal-strengthened Al-alloys can possess excellent combinations of strength and ductility. An icosahedral quasicrystalline phase (IQC) present in these alloys is Zlitine se lahko izdelajo s hitrim strjevanjem in dodatnim stiskanjem [1]. Nekateri elementi (Be, Ce, Si) stabilizirajo zlitine s kvazikristalno fazo v sistemu Al-Mn in se lahko izdelajo z navadno tehnologijo ulivanja. Te zlitine lahko v ulitem stanju dosežejo veliko trdnost, ki se lahko v nekaterih primerih celo poveča s toplotno obdelavo. Tak primer predstavljajo trakovi iz zlitine Al-Mn-Be-Cu, brizgane na hlajeno podlago. Trdota teh trakov se poveča za 50 % po toplotni obdelavi pri 300 °C in 400 °C v primerjavi z ulitim stanjem [2]. Te zlitine lahko tudi ohranijo trdnost do mnogo višjih temperatur kot trgovske Al-zlitine. Vendar bo potrebnih še veliko raziskav, preden se bodo lahko industrijsko izdelovale.

Pri naši raziskavi smo uporabljali 1000krat manjše ohlajevalne hitrosti, kot se uporabljajo pri postopku brizganja taline na hlajeno podlago. Kljub temu so nastali primarni kvazikristali pri strjevanju zlitine Al-Mn-Be-Cu, osnova pa je bila prenasičena z zlitinskimi elementi. Cilj naše raziskave je bil ugotaviti učinek toplotne obdelave na izločevalne procese in vlogo primarne kvazikristalne faze.

2 Eksperimentalni del

Preiskovana zlitina je vsebovala 4,24 mas. % Mn, 0,68 mas. % Be, 4,44 mas. % Cu in 90,94 mas. % Al. Narejena je bila iz predzlitin Al-Mn, Al-Cu, Al-Be in aluminija 99,89, ki smo jih stalili v vakuumski indukcijski peči. Najprej smo talino ulili v valjaste kokile s premerom 50 mm, potem pa nastali ulitek pretalili v bakreni kokili z izmerami 100 mm x 10 mm x 1 mm, da bi dosegli ohlajevalne hitrosti v območju 1000 K/s. Vzorce smo umetno starali 24 h na zraku pri 200 °C, 300 °C, 400 °C in 500 °C. Nato smo jih preiskali z XRD s sinhrotronskimi rentgenskimi žarki z valovno dolžino 0,1 nm (Sincrotrone Elettra,

metastable. These alloys can be made using rapid solidification and then compacted [1] [3]. Some elements (Be, Ce, Si) stabilize quasicrystalline phase alloys based on Al-Mn system, and can be produced by common casting methods. These alloys can attain considerably strength in the ascast condition, and in some cases, heat treatment can further enhance the strength. The examples were Al-Mn-Be-Cu meltspun ribbons. In this ribbons, the hardness increased for 50% after heat treatment at 300 °C and 400 °C in comparison to the ascast state [2]. These alloys can also retain strength to much higher temperature than commercial Al-alloys. However, much work is still necessary for industrial production of these alloys.

In this work, about 1000-times smaller cooling rates were used as during melt spinning. Nevertheless, primary IQC formed upon solidification in an Al-Mn-Be-Cu alloy, and the matrix was supersaturated with alloying elements. The aim of this investigation was to study the effect of heat treatment on the precipitation processes and the role of the primary IQC-phase.

2 Experimental

The investigated alloy contained 4.24 % Mn, 0.68 % Be, 4.44 % Cu and 90.64 % Al mass fraction. It was vacuum induction melted using Al-Mn, Al-Cu and Al-Be master alloys, and Al 99.89. It was first cast into cylindrical moulds with 50 mm in diameter, and after remelting into a copper mould with the dimensions: $100 \times 10 \times 1$ mm to obtain cooling rates in the range of 1000 K/s. The samples were heat-treated by artificial aging at 200 °C, 300 °C, 400 °C and 500 °C for 24 hours in the air. Afterwards, they were investigated by XRD using synchrotron X-rays with a wavelength

Trieste, Italy). Folije iz vzorcev v ulitem in toplotno obdelanem staniu za preiskavo s TEM, okoli 50 nm debele, smo izdelali s fokusiranim elektronskim snopom (FIB) (FEI, Helios). Večje, podolgovate izločke iz vzorcev, toplotno obdelanih pri 500 °C, smo ekstrahirali iz osnove s kemičnim postopkom. Z elektronskim presevnim mikroskopom z veliko ločljivostjo (HRTEM), elektronskim presevnim mikroskopom s filtrirano energijo (EFTEM), vrstičnim presevnim elektronskim mikroskopom (STEM), s spektroskopijo z izgubo energije elektronov (EELS) in energijskodisperzijsko rentgensko spektroskopijo (EDXS) smo preiskovali vzorce v napravi FEI Titan 60-300.

3 Rezultati in razprava

Z rentgensko difrakcijo (XRD) smo odkrili, da je zlitina sestavljena iz trdne raztopine, bogate z α -Al, kvazikristalov in Al₂Cu. of 0.1 nm (Sincrotrone Elettra, Trieste, Italy). TEM foils with thicknesses around 50 nm were prepared using FIB (FEI, Helios) for samples in the as-cast and heat-treated states. Larger, elongated precipitates present in a sample heat-treated at 500 °C were extracted from the matrix using a chemical method. HRTEM, EFTEM, STEM, ELLS and EDXS were carried out using FEI Titan 60-300.

3 Results and Discussion

XRD revealed that the alloy consisted of an Al-rich solid solution α -Al, IQC and Al₂Cu. The grain sizes of α -Al were between 5 to 10 μ m. The primary IQC- and Al₂Cu particles were allocated predominantly in the intergranular regions. XRD showed no changes in the phase composition after heat treatments at 200 °C, 300 °C and 400 °C. A profound alteration occurred at 500 °C. Al₂Cu and IQC disappeared,



Slika 1. EFTEM-slike Cu in Mn v zlitinah Al-Mn-Be-Cu. a, b) 24 h pri 200 °C, in c,d) 24 h pri 400 °C

Figure 1. EFTEM maps for Cu and Mn in the Al-Mn-Be-Cu alloys. a, b) 24 h at 200 °C, and c,d) 24 h at 400 °C

Velikost zrn faze α -Al je bila 5–10 µm. Primarni kvazikristali in delci Al₂Cu so se nahajali predvsem v območjih med zrni. XRD ni pokazala sprememb po toplotnih obdelavah pri 200 °C, 300 °C in 400 °C. Velika sprememba se je pojavila pri 500 °C. Faza Al₂Cu in kvazikristali so izginili, nadomestili sta jih fazi Be₄Al(Mn,Cu) and T-Al₂₀Mn₃Cu₂.

Preiskava s presevnim elektronskim mikroskopom je odkrila spremembe celo pri toplotni obdelavi pod 500 °C. Pri 200 °C so nastali ploščičasti, z bakrom bogati izločki v območjih med zrni (slika 1a,b) zaradi velike prenasičenosti z zlitinskimi elementi. Ploščice so bile dolge okoli 200 nm in debele okoli 5 nm. Razdalje med središči delcev so bile 70-100 nm. HRTEM je pokazal, da delci tvorijo semikoherentno mejo z osnovo α -Al po ravninah {1 0 0} faze α -Al. Izločki so identični izločkom 0' v binarnih zlitinah Al-Cu [3]. Difuzijske razdalje, izračunane po metodi najmanjših kvadratov, za Cu in Mn pri 200 °C so bile 150 mn oz. 1 nm. Zato lahko nastajajo le izločki, bogati z bakrom, ker njihova tvorba zahteva difuzijo dolgega reda.

Pri 400 °C so bili prisotni paličasti izločki okroglega ali kvadratastega prereza (slika 1c,d). Bili so dolgi do 2 μ m in debeli 50–100 nm.

Po toplotni obdelavi 24 h pri 500 °C so v α -Al prevladovali paličasti izločki s kvadratastim prerezom. Bili so dolgi okoli 2 ¼m in debeli 200 400 nm (slika 2a). EDS je odkrila, da vsebujejo Al, Cu in Mn, medtem ko Be s to metodo nismo zaznali. Njihove povprečne atomske koncentracije so bile 81,4±0,64%, 12,0±0,61% oz. 6,6±0.63%. Slika 2b kaže HRTEM-posnetek in slika 2c ustrezni sliki hitre Fourierove transformacije (FFT). Slika 2d kaže drugo sliko hitre Fourierove transformacije z veliko simetrijo. Obratna vektorja **g**(1) and **g**(3) sta bila pravokotna drug na drugega. Podroben being replaced by $Be_4Al(Mn,Cu)$ and T-Al₂₀Mn₃Cu₂.

Examinations using TEM revealed microstructural changes even during heat treatments at temperatures below 500 °C. At 200 °C, plate-like copper-rich precipitates formed predominantly in the intergranular regions (Fig. 1a,b) because of higher supersaturation with alloying elements there. The plates were about 200 nm long and only about 5 nm thick. The centre-to-centre interparticle distances were between 70 and 100 nm. HRTEM showed that the particles formed a semicoherent interface with α -Al matrix on {1 0 0} planes of α -Al. The precipitates were identical to θ'-precipitates in binary Al-Cu alloys [3]. The root-mean-square (RMS) diffusion distances for Cu and Mn at 200 °C are 150 nm and 1 nm, respectively [4]. Thus only Cu-rich precipitates can form because their formation requires long-range diffusion.

At 400 °C, the rodlike precipitates were present with circular and square-like cross-sections (Fig. 1c,d). They were up to 2 μ m long, and 50–100 nm thick. The manganese RMS-diffusion distance was about 460 nm, thus Mn-mobility was sufficient to enable formation and growth of particles.

After heat treatment for 24 h at 500 °C, rodlike precipitates with the square-like cross-sections prevailed within α -Al. They were about 2 μ m long, and between 200 nm and 400 nm thick. (Fig. 2a). EDS revealed that they contain Al, Cu and Mn, while Be could not detected using this method. The average atomic concentration for Al, Mn and Cu is 81.4 \pm 0.64%, 12.0 \pm 0.61% and 6.6 \pm 0.63%, respectively. Fig. 2b shows a HRTEM-image, and Fig. 2c is the corresponding FFT. Fig. 2d shows another highly symmetrical FFT. The reciprocal lattice vectors **g**(1) and **g**(3) were also perpendicular to each other. Detailed



Slika. 2. Toplotna obdelava 24 h 500 °C

a) ekstrahiran paličast izloček,
b) HRTEM-posnetek , posnet v
smeri dvoštevne osi
c) FFT HRTEM-posnetka,
posnetega v smeri dvoštevne osi,
d) FFT HRTEM-mikroposnetka v
smeri druge dvoštevne osi.

Figure 2: Heat treatment at 500 °C for 24 h.

a) An extracted rodlike precipitate,b) HRTEM-micrograph taken along a twofold axis,

c) FFT of the HRTEM-micrograph taken along twofold axis,d) FFT of a HRTEM-micrograph taken along another twofold axis

pregled literature je odkril, da so ti izločki znani kot T-faze [5].

Vzorec smo segrevali na 300 °C. Čas segrevanja s sobne temperature na 300 °C je bil 2 minuti. V tem obdobju nismo opazili razlik v mikrostrukturi. Potem smo study of the literature revealed that these precipitates are known as T-phase [5].

The in-situ heating was carried out at 300 °C. The heating time from room temperature to 300 °C was 2 minutes. No differences in microstructure were observed



Slika 3. Obdelava 150 minut na licu mesta pri 300 °C. a) TEM-posnetek v svetlem polju, b) EFTEM porazdelitev Cu, in c) EFTEM porazdelitev Mn

Figure 3. In-situ treatment at 300 °C for 150 min a) TEM bright-field image, b) EFTEM distribution of Cu, and c) EFTEM distribution of Mn

opazili tri značilne procese: (1) nastanek in rast kroglastih izločkov v osnovi α-Al, (2) nastanek in rast delcev, ki so se nukleirali na prisotnih delcih v ulitem stanju, (3) nastanek, rast in raztapljanje 300 ^{oo}Al₂Cu. Elektronski mikroposnetek in EFTEM-slike mangana in bakra iz TEM vzorca po segrevanju 150 minut pri 300 °C prikazuje slika 3.

Znotraj zrn α-Al smo odkrili kroglaste izločke, ko je njihov premer dosegel okoli 10 nm. Hitrost nukleacije se je izračunala iz števila delcev po različnih časih zadrževanja pri določeni temperaturi. Inkubacijski čas pri teh razmerah je bil okoli 10 min. Po 15 min se je hitrost nukleacije začela občutno povečevati in po 20 min je dosegla največjo vrednost okoli 10¹⁸ m⁻³ s⁻¹. Po 30 min se je nukleacijska hitrost zmanjšala skoraj za dva velikostna reda in zdi se, da so po 60 min prenehali nastajati novi delci. Potem so rasli samo obstoječi izločki. Nekateri izločki so se enakomerno debelili, medtem ko so drugi postajali podolgovati. Med opazovanjem nismo zasledili zorenja. Slika 3a kaže preiskovano območje po 150 min zadrževanja pri 300 °C. Ustrezne EFTEMslike Cu in Mn kažejo, da izločki vsebujejo tako Cu kot Mn, medtem ko z uporabljeno metodo nismo zasledili Be. A je bil delež mangana mnogo večji kot delež bakra. Koncentracija Cu v izločkih je bila skoraj enaka kot v primarnih kvazikristalnih delcih, tj. v območju nekaj atomskih odstotkov. Velikosti delcev in razdalje med delci so primerljive z razdaljami za Mn, ki so izračunane po metodi najmanjših kvadratov, kar pomeni, da difuzija mangana krmili nastajanje in rast izločkov.

Po ohladitvi vzorca na sobno temperaturo smo naredili HRTEM-posnetke, da bi analizirali zgradbo nastalih izločkov. Slika 4a kaže HRTEM-posnetek fazne meje med α -Al in izločkom. Odboj α -Al na FFTsliki kaže, da leži aluminijska osnova na osi cone [111]. Izloček kaže troatevno simetrijo. during this period. Afterwards, three distinct processes were observed: (1) the formation and growth of spherical precipitates within the α -Al matrix; (2) the formation and growth of particles nucleated on the particles present in the as-cast condition, and (3) the formation, growth and dissolution of Al₂Cu. An electron micrograph and EFTEM maps for Mn and Cu from the TEM specimen after 150 minutes heating to 300 °C are shown in Fig. 3.

The spherical precipitates formed within α -Al grains were detected when their diameter reached about 10 nm. The nucleation rates were calculated from the particle count after different holding times. incubation period under The these circumstances was about 10 min. The nucleation rate started to increase considerably after 15 min, and attained the maximum value of around 10¹⁸ m⁻³ s⁻¹ after 20 min holding. Nucleation rate dropped nearly by two orders of magnitude after 30 min, and the formation of new precipitates apparently stopped after 60 min. Thereafter, only growth of the existing precipitates took place. Some precipitates thickened uniformly while others became elongated. No precipitate ripening was observed during observation. Fig. 3a shows the investigated area after 150 min of holding the temperature at 300°C. Corresponding EFTEM-maps for Cu and Mn showed that precipitates contained both Cu and Mn, while Be was not detected using this method. However, the content of Mn was much higher than that of Cu. The Cu concentration in precipitates was almost the same as in the primary IQC-particles; in the range of few atomic percents. The precipitate sizes and the interparticle distances are comparable with RMS distances for Mn, indicating that Mn-diffusion limits the formation and growth of precipitates.



Slika 4. HRTEM-posnetek meje med α-Al in kvazikristalnim izločkom (a), in njegova FFTslika (b). HRTEM-posnetek na kvazikristalni fazi (c), in njegova FFT-slika (d)

Figure 4. HRTEM of an interface between α -Al and IQC-precipitate (a), and its corresponding FFT (b). HRTEM of a precipitate formed on IQC (c), and its corresponding FFT (d)

Njegova FFT ni periodična. Razdalja med konicami se veča s srednjo vrednostjo

zlatega razmerja τ $((1+\sqrt{5})/2 \approx 1.62)$ v glavnih smereh. To močno kaže, da imajo izločki ikozaedrično kvazikristalno zgradbo [6]. Mrežne ravnine se epitaksialno raztezajo od kvazikristalne faze do ploskovno centrirane kubične mreže [7]. To omogoča nastanek nizkoenergijske meje med kvazikristalno fazo in α-Al in prednost za nastajanje kvazikristalnih izločkov pred drugimi izločki. Več podrobnosti se lahko dobi v [8].

Primarni kvazikristalni izločki predstavljajo mesta heterogene nukleacije za izločke T-faze. Vsak kvazikristalni delec omogoča nastanek več izločkov T-faze. Začnejo nastajati po zadrževanju 15 min pri temperaturi. Njihove začetne velikosti so bile dolžina okoli 15 nm (vzporedno z mejo med kvazikristalno fazo in α-Al) in debelina 10 nm (navpično na mejo med kvazikristalno

After cooling the sample down back to room temperature, HRTEM images were acquired to study the structure of formed precipitates. Fig. 4a shows a HRTEMimage of the interface between α -Al and one precipitate. The α -Al reflections in FFT indicated that the Al matrix is on [111] zone axis. The precipitate exhibited a threefold symmetry. Its FFT was not periodic. The distances between peak positions increased

with the golden mean τ ((1+ $\sqrt{5}$)/2 \approx 1.62) in the main directions. This strongly indicates precipitates had an icosahedral that Closer quasicrystalline structure [6]. inspection showed an excellent matching between the matrix and the precipitate in the HRTEM-image, and coincidence of some diffraction spots in the corresponding FFT. The lattice planes extended epitaxially from the quasicrystalline phase to the FCC-matrix [7]. This allows the creation of a low energy interface between IQC and α -AI, and the fazo in α -Al). Hitrost rasti je bila pribli~no linearna s časom v smeri vzporedni z mejo in sorazmerna $t^{0.5}$ v smeri, navpični nanjo. Na koncu je bil izloček dolg 135 nm in debel 45 nm. HRTEM je pokazal dobro ujemanje med kvazibinarno fazo in α -Al. FFT-slika izločka je kazala periodičnost.

Difuzijski koeficient Cu pri 300 °C je sorazmerno velik. Temperatura je bila nad metastabilnimi temperaturami solvus za GPcone θ " in θ '. Tako se je lahko prenasičenost z bakrom izrazila v nastanku faze Al₂Cu. Al, Cu je začel nastajati heterogeno na kvazikristalnih delcih približno po istem času, kot so prvi kvazikristalni delci nastali v osnovi. Razdalje med delci Al, Cu, izračunane po metodi najmanjših kvadratov, so bile okoli 100 nm, kar ustreza razdaljam difuzije Cu. Delci Al_aCu so rasli prvih 30 min in potem so se začeli raztapljati. Po 60 min so se skoraj vsi delci Al₂Cu raztopili, samo na določenih mestih so delci Al₂Cu vztrajali v mikrostrukturi po 150 min (delec označen s puščico na sliki 3b).

Mikrotrdotna analiza z obremenitvijo 100 mN je pokazala, da se je trdota vzorcev, toplotno obdelanih pri 200 °C, povišala z 120 HV na 130 HV po 24-urnem zadrževanju pri tej temperaturi, medtem ko se je trdota vzorcev pri 400 °C zmanjšala z 120 HV na 110 HV. Na drugi strani so imeli vzorci, toplotno obdelani pri 300 °C, največjo trdoto po približno 60 min, kar ustreza zmanjševanju nastajanja izločkov pri preiskavi s TEM na licu mesta. To kaže, da hitrost difuzije v foliji ni bila mnogo večja od difuzije v masivnem vzorcu kljub mnogo večji površini.

Rezultati te preiskave so pokazali, da lahko nastajajo različne vrste izločkov po toplotni obdelavi zlitine Al-Mn-Be-Cu. Pri 200 °C lahko zaradi omejene difuzije Mn nastajajo le z bakrom bogati izločki. Pri višjih temperaturah (550 °C) nastajajo ternarni izločki, katerih sestava je blizu ravnotežni preference for formation of IQC precipitates before other possible precipitates. More details can be found in [8].

Primary **IQC**-particles represented heterogeneous nucleation sites for T-phase precipitates. Each IQC-particle enabled formation of several T-phase precipitates. They started to form after holding for around 15 min. Their initial sizes were about 15 nm in length (parallel to the IQC/ α -Al interface) and 10 nm in thickness (perpendicular to the IQC/ α -Al interface). The growth rate was approximately linear with time in the direction parallel to the interface, and proportional to t^{0.5} in the direction perpendicular to it. At the end, the precipitate was 135 nm in length and 45 nm in thickness. HRTEM showed nice matching between both IQC and α -AI. FFT of the precipitate exhibited periodicity.

Diffusivity of Cu at 300 °C was rather fast. The temperature was above the metastable solvus temperatures for GP-zones, θ " and θ '. Thus the Cu-supersaturation could only be released by formation of Al₂Cu. Al₂Cu started to form heterogeneously on primary IQC-particles approximately after the same time as first quasicrystalline precipitates formed in the matrix. The Al₂Cu interparticle distances were about 100 nm, which corresponded to Cu RMS diffusion distances. Al₂Cu particles increased in size for the first 30 min, and then started to dissolve. After 60 min almost all Al,Cu dissolved, only occasionally remains of Al₂Cu particles persisted in microstructure after 150 min (e.g. the particle indicated by the arrow in Fig. 3b).

The microindentation study using a load of 100 mN revealed that the hardness of samples heat-treated at 200 °C slowly increased up to 24 h holding from 120 HV to 130 HV, whereas hardness of samples exposed to 400 °C decreased from 120 HV to 110 HV. On the other hand, the samples heat-treated at 300 °C exhibited

fazi τ₁ (Al₂₉Mn₆Cu₄). Pri vmesni temperaturi (300 °C) nastajajo v trdnoraztopinski osnovi, bogati z Al, kvazikristalni delci. Primarne kvazikristalne faze predstavljajo primerno mesto za nukleacijo drugih kristalnih izločkov. Tako kvazikristalni izločki kot heretogeno nastali izločki imajo z osnovo določena kristalografska razmerja.

4 Sklepi

Rezultati te preiskave so pokazali, da lahko nastajajo različne vrste izločkov po toplotni obdelavi zlitine Al-Mn-Be-Cu. Pri 200 °C lahko zaradi omejene difuzije Mn nastajajo le z bakrom bogati izločki. Pri višjih temperaturah (550 °C) nastajajo ternarni izločki, katerih sestava je blizu ravnotežni fazi T, (Al₂₀Mn_eCu₄). Pri vmesni temperaturi (300 °C) nastajajo v trdnoraztopinski osnovi, bogati z Al, kvazikristalni delci. Primarne kvazikristalne faze predstavljajo primerno nukleacijo drugih kristalnih mesto za izločkov. Tako kvazikristalni izločki kot heretogeno nastali izločki imajo z osnovo določena kristalografska razmerja.

5 Zahvale

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The results of this study showed that several types of precipitates can form upon heat treatment of an Al-Mn-Be-Cu alloy. At 200 °C only Cu-rich precipitates can form, due to limited diffusion of Mn. At higher temperatures (550 °C) ternary precipitates formed, closely related to the equilibrium τ_{1} (Al₂₀Mn_eCu₄). In the intermediate temperature range (300 °C) IQC-particles formed in the Al-rich solid solution matrix, which primary IQC presented a convenient nucleation site for other crystalline precipitates. Both IQC-precipitates and heterogeneously formed crystalline precipitates had definite crystallographic relationships with the matrix.

4 Conclusions

The results of this study showed that several types of precipitates can form upon heat treatment of an Al-Mn-Be-Cu alloy. At 200 °C only Cu-rich precipitates can form, due to limited diffusion of Mn. At higher temperatures (550 °C) ternary precipitates formed, closely related to the equilibrium τ_{1} (Al₂₀Mn_eCu₄). In the intermediate temperature range (300 °C) IQC-particles formed in the Al-rich solid solution matrix, which primary IQC presented a convenient nucleation site for other crystalline precipitates. Both IQC-precipitates and heterogeneously formed crystalline precipitates had definite crystallographic relationships with the matrix.

so bile financirane iz 7. okvirnega programa evropske unije po pogodbi 312483 – ESTEEM2 (Integrirana infrastrukturna iniciativa–I3)

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Viri / References

- 1 T. Oz, E. Karakose and M. Keskin, *Mater. Des.* **50**, 399-412 (2013).
- 2 F. Zupanic, G. Lojen, L. Barba and T. Boncina, Mater. Charact. 70, 48-54 (2012).
- 3 A. Biswas, D. J. Siegel, C. Wolverton and D. N. Seidman, *Acta Mater.* **59**, 6187-6204 (2011).
- 4 Y. Du, Y. A. Chang, B. Huang, W. Gong, Z. Jin, H. Xu, Z. Yuan, Y. Liu, Y. He and F. Y. Xie, *Materials Science and Engineering: A* **363**, 140-151 (2003).
- 5 Z. Shen, C. Liu, Q. Ding, S. Wang, X. Wei, L. Chen, J. Li and Z. Zhang, *Journal of Alloys and Compounds* **601**, 25-30 (2014).
- 6 V. Elser, *Phys. Rev. B* **32**, 4892-4898 (1985).
- 7 A. Singh and A. P. Tsai, J. Phys.-Condes. Matter 202008).
- 8 F. Zupanič, D. Wang, C. Gspan and T. Bončina, Mater. Charact. 106, 93-99 (2015).