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Mikrostruktura mikrolegirane zlitine Al-Mn-Cu, ulite s hitrim asimetričnim dvovaljnim litjem

Microstructure of a Microalloyed Al-Mn-Cu alloy, Cast by Rapid Asymmetric Dual-Roll Casting

Povzetek

Dvovaljno litje se v industriji aluminija pogosto uporablja za izdelavo nekaj milimetrov debelih trakov. Hitrosti so le nekaj metrov na minuto. Dodatna pomanjkljivost je izcejanje v sredini trakov in bolj groba mikrostruktura zaradi razmeroma počasnega ohlajanja. Haga in njegovi sodelavci so razvili visokohitrostno litje z enim in dvema valjema, s katerim so dosegli vrednosti do 60 m min^{-1}. S tem postopkom je mogoče v laboratorijskih razmerah vlivati majhne količine livnih in gnetnih aluminijevih zlitin.

V tej raziskavi je bila z asimetričnim dvovaljnim postopkom litja ulita mikrolegirana zlitina Al-Mn-Cu, ki ima zaradi majhne vsebnosti skandija, cirkonija, kroma in vanadija veliko toplotno odpornost. Hitrosti litja so bile 25 m/min in 10 m/min, pri čemer so bili trakovi debeli približno 3 mm oziroma 6 mm. Uporabljeni hitrosti med strjevanjem nista ustvarili kvazikristalnega evtektika v meddendritnih prostorih, temveč heterogen zlog, sestavljen iz Q-Al_2Cu, $Al_2Cu(Sc)$ in faze, bogate z Mn (Al_4Mn). Mikrostruktura je bila sestavljena iz stebričastih dendritnih zrn v stiku z valji in enakoosnih dendritnih zrn na sredini. Na sredini traku je bilo prisotnih nekaj poroznosti zaradi krčenja in velikih intermetalnih delcev.

Ključne besede: aluminij, litje, trak, mikrostruktura, trdota

Abstract

Twin-roll casting is often used in the aluminium industry to produce strips a few millimetres thick. The speeds are only a few meters per minute. An additional disadvantage is the segregation in the middle of the strips and a rougher microstructure due to relatively slow cooling. Haga and his colleagues have developed high-speed single-roll and dual-roll casting, achieving up to 60 m min^{-1}. They can cast small amounts of both cast and wrought aluminium alloys in the laboratory.

In this research, a micro-alloyed Al-Mn-Cu alloy was cast using the asymmetric dual-roll casting process, which has high thermal resistance due to the small content of scandium, zirconium, chromium, and vanadium. The casting speeds were 25 m/min and 10 m/min, producing strip thicknesses around 3 mm and 6 mm, respectively. The applied speeds did not create a quasi-crystalline eutectic in the interdendritic areas during solidification, but a heterogeneous structure consisting of Θ-Al_2Cu, $Al_2Cu(Sc)$, and a Mn-rich phase (Al_4Mn). The microstructure consisted of columnar dendritic grains in contact with the rolls and equiaxed dendritic grains at the centre. Some shrinkage porosity and large intermetallic particles were present at the strip centre.

Keywords: aluminium, casting, strip, microstructure, hardness

1 Uvod

Pri konvencionalnem dvovaljnem litju lahko lijemo aluminijaste trakove neposredno iz tekočega stanja, kar omogoča manjšo porabo energije in s tem manjši ogljični odtis [1]. Strjevanje je sorazmerno hitro, kar vodi do udrobnitve kristalnih zrn in škodljivih intermetalnih faz [2]. Hitrost litja je sorazmerno nizka, navadno okoli 5 m/min, kar ima negativen vpliv na produktivnost. V Laboratoriju za procesiranje materialov na Inštitutu za tehnologijo v Osaki so raziskovali zvišanje hitrosti litja in produktivnosti z uporabo valjev z velikim premerom. Hitrost litja so povečevali tudi z izboljšanjem toplotne prevodnosti valjev in metalostatičnim tlakom tekoče kovine. Razvili so nova visokohitrostna enovaljna in dvovaljna ulivalnika [3], s katerima je mogoče doseči hitrosti litja vse do 60 m/min.

V Laboratoriju za materiale, UM FS, je bila razvita nova aluminijeva zlitina, ki vsebuje dvojne kvazikristalne izločke in izločke $L1_2$-Al_3X, ki bi lahko občutno vplivali na toplotno obstojnost zlitine [4]. Eksperimentalno smo vlili zlitino Al-Mn-Cu, mikrolegirano z zlitinskimi elementi Be, Sc, Zr, Cr in V. Vsak od dodanih elementov naj bi imel točno določen učinek. Mangan omogoča nastanek kvazikristalnih izločkov [5], medtem ko berilij izrazito poveča njihovo številčnost [6]. Skandij in cirkonij se dodata, da nastanejo izločki tipa $L1_2$ [7], medtem ko dodatka Cr in V povečata toplotno obstojnost.

Cilj raziskave je ugotoviti mikrostrukturo mikrolegirane zlitine Al-Mn-Cu s hitrim asimetričnim dvovaljnim litjem z dvema različnima hitrostma.

1 Introduction

The conventional twin-roll caster can cast aluminium strips directly from molten metal, considerably reducing energy consumption and carbon footprint [1]. The solidification is relatively rapid, leading to refinement of grain sizes and harmful intermetallic phases [2]. However, the roll speed is very low, typically lower than 5 m/min, implying low productivity. The Material Processing Laboratory at the Osaka Institute of Technology has investigated the increase in casting speed and productivity using a large-diameter roll, a thin gauge, and a lengthy setback. They have also increased casting speed by increasing the rolls' thermal conductivity and metallostatic pressure of the molten metal. They have developed new high-speed single-roll and twin-roll casters [3]. It is possible to achieve casting speeds up to 60 m/min.

A new alloy with quasicrystalline and $L1_2$-Al_3X precipitates was developed in the Laboratory of Materials, UM FS, having promising room and elevated temperature properties [4]. A microalloyed Al-Mn-Cu alloy was cast experimentally. It contains several elements with dedicated tasks, Be, Sc, Zr, Cr, and V. It contains Mn to form quasicrystalline precipitates [5] and Be to increase their number density enormously [6]. Sc and Zr are added to form $L1_2$-precipitates [7], and Cr and V increase the thermal stability.

This research aims to determine the microstructure of a microalloyed Al-Mn-Cu alloy by rapid asymmetric dual-roll casting with two speeds.

2 Experimental work

The composition of the investigated Al-alloy was 2.18% Mn, 2.73% Cu, 0.18% Sc,

2 Eksperimentalno delo

Sestava raziskane Al-zlitine je bila 2,18 % Mn, 2,73 % Cu, 0,18 % Sc, 0,08 % Be, 0,16 % Zr, 0,08 % Cr, 0,16 % Fe, 0,0003 % Mg, 0,009 % Ti and 0,07 % V (masni deleži v odstotkih; izmerjena je bila z masno spektroskopijo v induktivno sklopljeni plazmi ICP-MS). Zlitina je bila pripravljena iz tehnično čistega aluminija Al 99,8 ter legirana s predzlitinami AlBe5,5, AlCr20, AlV10, AlZr10, AlSc2, AlCu50 in AlMn10. Zlitina je bila staljena v vakuumski indukcijski talilni peči in ulita v ingote s premerom 20 mm. Zlitina je bila nato pretaljena in ulita z asimetričnim dvovaljnim litjem v Osaki (Slika 1). Livna temperatura je bila 700 °C, obremenitev valjev pa je bila 20 N/mm. Lili smo z dvema hitrostma litja: 10 m/min in 25 m/min. Pri prvi hitrosti je bila debelina trakov 5,5–6,5 mm, pri drugi pa 3,5–4 mm.

Odliti trakovi so bili razrezani na manjše vzorce ter metalografsko pripravljeni z brušenjem in poliranjem. Nekateri vzorci so bili analizirani v poliranem stanju, drugi so bili dodatno jedkani. Uporabljene so bile analizne metode svetlobne mikroskopije SM (svetlobni mikroskop NIKON Epiphot 300), visokoločljive elektronske mikroskopije (Jeol JSM IT-800SHL). Kemična sestava v mikroobmočju je bila izmerjena z energijskodisperzijsko spektroskopijo rentgenskih žarkov (EDS detektor Oxford AZtec Live AUTO UltimMax 100). Trdoto smo izmerili z metodo po Vickersu HV (DuraScan 50, EmcoTest).

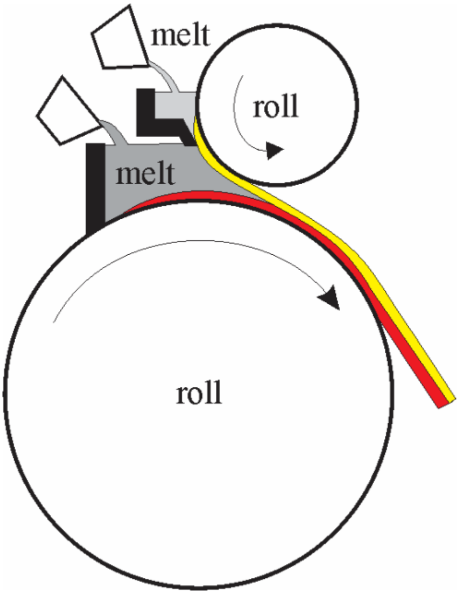
3 Rezultati in diskusija

3.1 Napake

Slika 2 prikazuje prerez tanjšega traku. Usmerjeno strjevanje kristalnih zrn aluminija

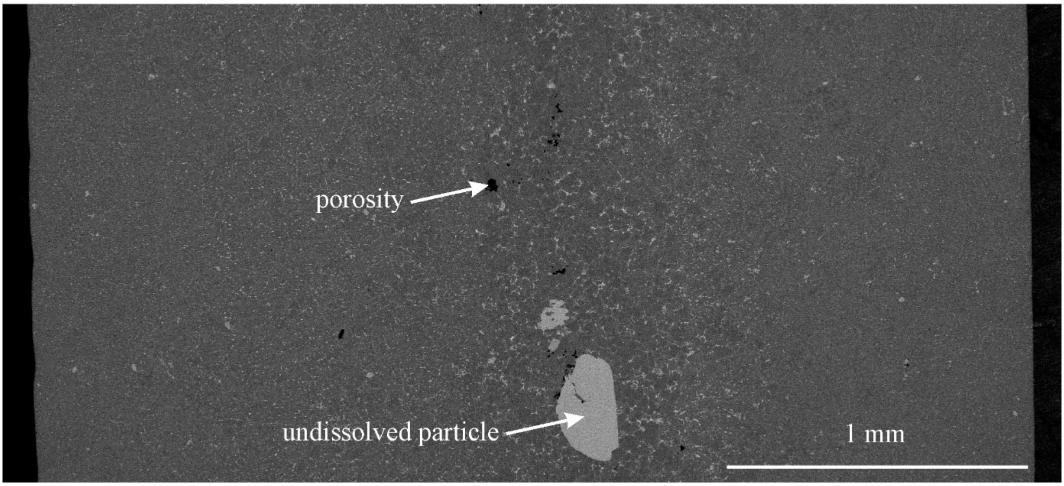
0.08% Be, 0.16% Zr, 0.08% Cr, 0.16% Fe, 0.0003% Mg, 0.009% Ti and 0.07% V (the content is in wt.% and was determined using Inductively Coupled Plasma, Mass Spectroscopy). The alloy was prepared using Al 99.8 alloyed by AlBe5.5, AlCr20, AlV10, AlZr10, AlSc2, AlCu50, and AlMn10 master alloys. The alloy was melted under vacuum in a vacuum induction furnace and cast into ingots with a diameter of 50 mm.

The alloy was cast by asymmetric dual-roll casting in Osaka (Figure 1). The casting temperature was 700 °C, and the roll load was 20 N/mm. Two casting speeds were selected: 10 m/min and 25 m/min. The first casting speed produced strips of a 5.5–6.5 mm thickness, and the second 3.5–4 mm.



Slika 1. Shematični prikaz asimetričnega dvovaljnega litja

Figure 1. Schematic presentation of an asymmetric dual-roll caster

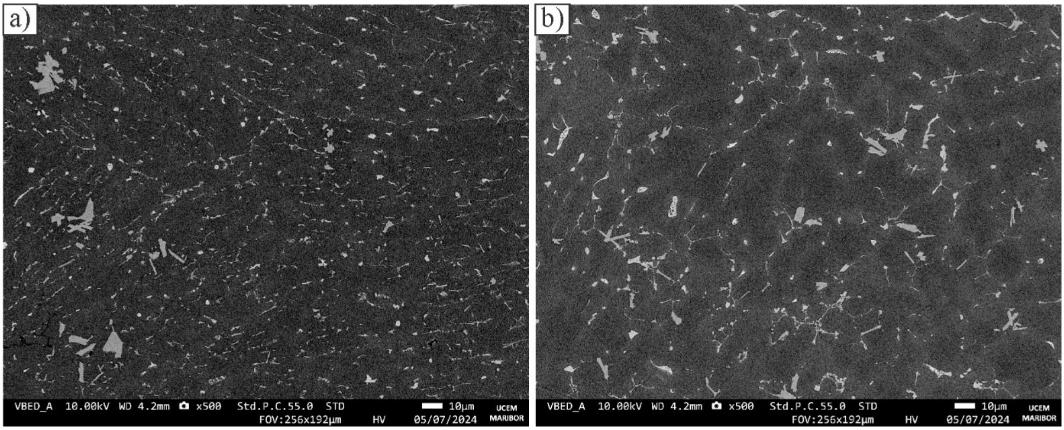


Slika 2. Prečni prerez tanjšega traku, hitrost litja 25 m/min (SEM, odbiti elektroni)

Figure 2. Cross-section of the thinner strip, casting speed 25 m/min (SEM, BSE (Back-scattered Electron micrograph))

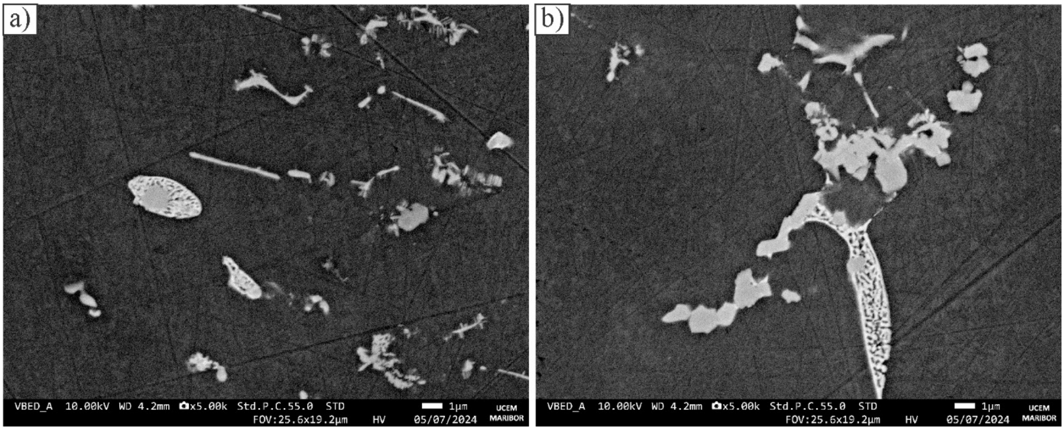
je potekalo od valjev proti sredini. Debelina te plasti je bila približno 1 mm. Na sredini traku so bila zrna enakoosna. Tam je bila pogosto opazna poroznost, ki je posledica krčenja pri strjevanju. Veliki neraztopljeni delci so nastali pri strjevanju ingota pri vakuumskem indukcijskem taljenju.

The cast strips were cut and prepared by metallography, ground, polished, and etched. Some samples were analysed after polishing, and others were additionally etched. They were investigated by light microscopy LM (light microscope NIKON Epiphot 300) and high-resolution scanning



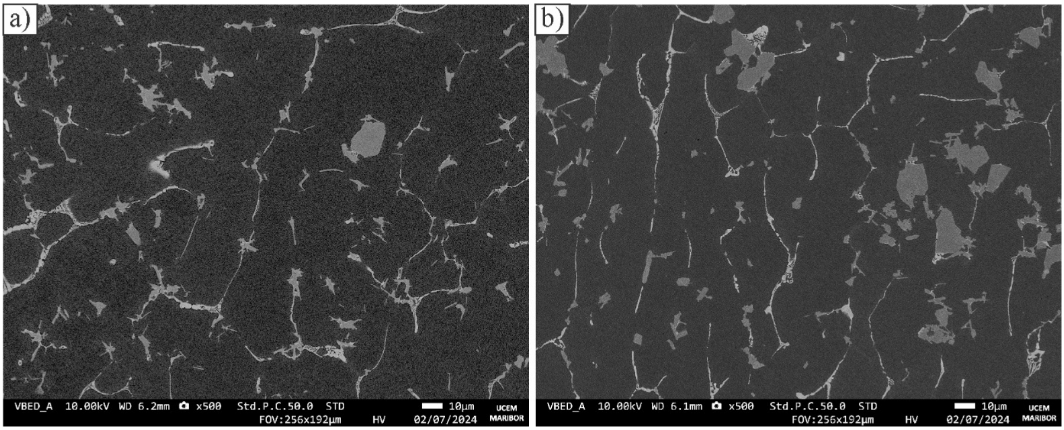
Slika 3. Mikrostruktura tanjšega traku pri manjši povečavi, hitrost litja 25 m/min. a) Blizu površine, b) v središču traku (SEM, odbiti elektroni)

Figure 3. Microstructure of the thinner strip at a smaller magnification. a) Close to the surface, b) at the strip centre (SEM, BSE)



Slika 4. Mikrostruktura tanjšega traku pri večji povečavi, hitrost litja 25 m/min. a) Blizu površine, b) v središču traku (SEM, odbiti elektroni)

Figure 4. Microstructure of the thinner strip at a higher magnification, casting speed 25 m/min. a) Close to the surface, b) at the strip centre (SEM, BSE)



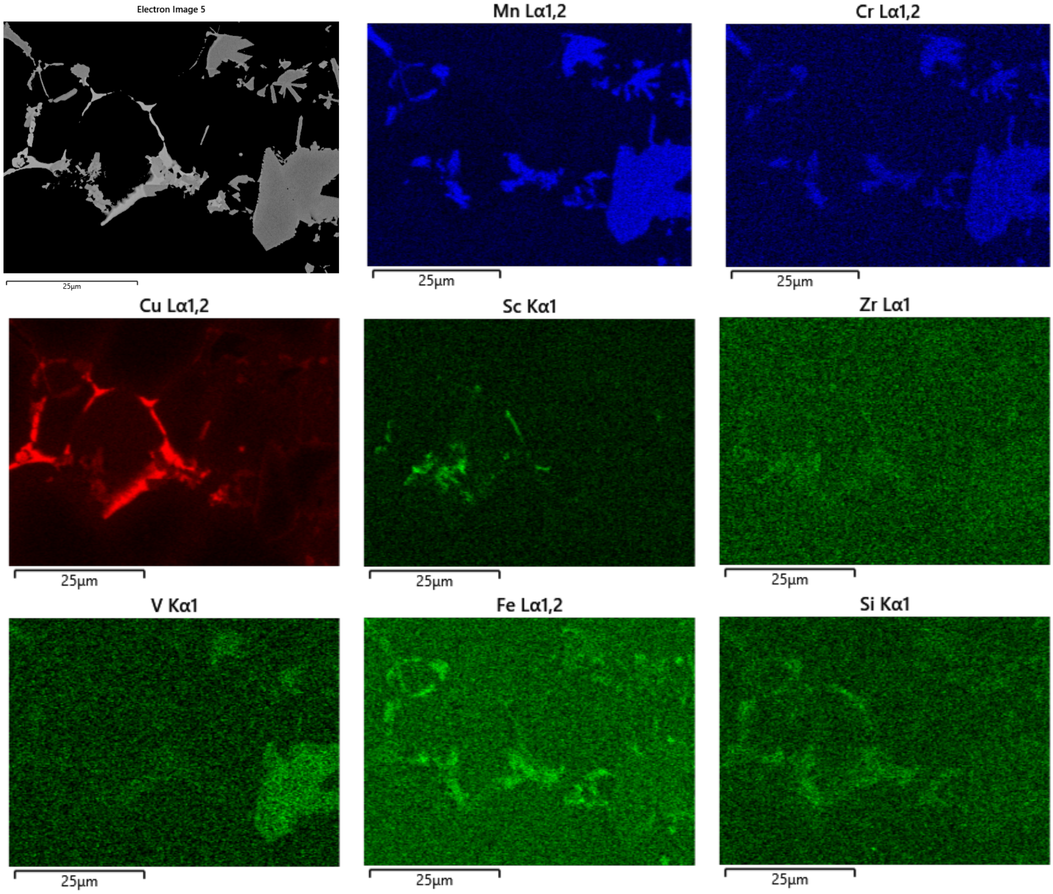
Slika 5. Mikrostruktura debelejšega traku, hitrost litja 10 m/min, a) blizu površine, b) v središču traku (SEM, odbiti elektroni)

Figure 5. Microstructure of the thicker strip, casting speed 10 m/min. a) Close to the surface, b) at the strip centre (SEM, BSE)

Sliki 3 in 4 prikazujeta mikrostrukture tanjšega traku pri dveh različnih povečavah. Slika 3 prikazuje drobnejša dendritna zrna blizu površine, medtem ko Slika 4 prikazuje območja z manjšimi meddendritnimi prostori blizu površine.

Makro- in mikrostruktura debelejšega traku je bila kvalitativno podobna kot pri

electron microscopy SEM (Jeol JSM IT-800SHL). Chemical compositions in the micro area were determined using energy dispersive spectroscopy (EDS detector Oxford AZtec Live AUTO UltimMax 100). Hardness was measured using the Vickers method HV (DuraScan 50, EmcoTest).



Slika 6. Porazdelitev elementov v tanjšem traku, hitrost litja 25 m/min. Na zgornji levi sliki je analizirano območje (SEM, odbiti elektroni). Druge slike prikazujejo porazdelitev označenih elementov.

Figure 6. Elemental mapping of an area in the thinner strip, casting speed 25 m/min. The top left is the SEM BSE micrograph; the other images show the distribution of the indicated elements.

tanjšem traku (Slika 5). Posledično je bila tudi trdota zelo podobna. Znašala je 110 ±7 HV 0,1 (112 meritev) pri tanjšem traku in 110 ±12 HV 0.1 (203 meritve) pri debelejšem traku.

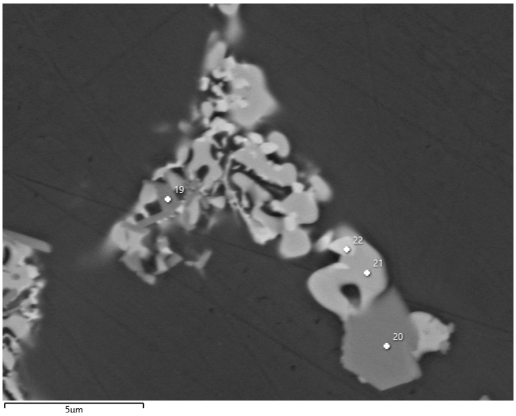
Slika 6 prikazuje porazdelitev elementov v meddendritnem prostoru. Analiza EDS je odkrila fazo, bogato z Mn, ki vsebuje tudi Cr. Večji delci te intermetalne faze so vsebovali tudi vanadij. Sestava te faze je bila približno Al_4Mn. Baker je bil pretežno v meddendritnih prostorih, kjer je tvoril

3 Results and discussion

3.1 Defects

Figure 2 shows the cross-section of the thinner strip. Directional solidification of aluminium crystal grains took place from the rolls toward the centre. The thickness of this layer was about 1 mm. There were equiaxed grains at the strip centre. Porosity, resulting from the solidification shrinkage, was often observed at the centre. Large značilen heterogen zlog (α-Al + Θ-$\mathrm{Al_2Cu}$). Slika 6 nakazuje, da je Sc bolj koncentriran Sc v meddendritnih prostorih.

Podrobnejši mikroposnetek meddendritnega prostora na Sliki 7 nakazuje, da so v njem vsaj tri faze. Najtemnejša faza (številka 20) je bogata z manganom, kar ustreza $\mathrm{Al_4Mn}$. Delec, označen z 21, ima sestavo, ki se ujema s Θ-$\mathrm{Al_2Cu}$. Najsvetlejša faza, označena z 22, ima sestavo podobno Θ-$\mathrm{Al_2Cu}$, vendar vsebuje še 3–4 at.% Sc.



Slika 7. Mikrostruktura na območju meddendritnega prostora v debelejšem traku, hitrost litja 10 m/min (SEM, odbiti elektroni). Točkovna analiza EDS je bila izvedena v točkah, označenih 19–22.

Figure 7. Microstructure in the interdendritic region in the thicker strip, casting speed 10 m/min (SEM, BSE). Point EDS was carried out at sites labelled 19–22.

V mikrostrukturi so bile ugotovljene mikroizceje. Dendritna središča so vsebovala več V, Zr in Mn, medtem ko so dendritni robovi (izognili smo se delcem v meddendritnem prostoru) vsebovali več Si, Sc, Fe in Cu. Vendar stopnja izcejanja ni bila zelo visoka zaradi majhne razdalje med dendritnimi vejami.

undissolved particles formed during the ingot solidification after vacuum induction melting.

Figures 3 and 4 show the microstructure of the thinner strip at two different magnifications. Figure 3 indicates finer dendritic grains near the surface, while Figure 4 shows smaller interdendritic spaces near the surface.

The macro- and microstructure of the thicker ribbon was qualitatively similar to that of the thinner strip (Figure 5). As a result, hardness was also very similar. It was 110 ± 7 HV 0.1 (112 measurements) for the thinner strip and 110 ± 12 HV 0.1 (203 measurements) for the thicker strip.

Figure 6 shows the typical elemental distribution in the interdendritic space. The EDS analysis revealed a Mn-rich phase containing Cr, while V was in larger particles. This phase has an approximate composition of $\mathrm{Al_4Mn}$. Copper was mainly present in the interdendritic areas, forming a typical heterogeneous structure (α-Al + Θ-$\mathrm{Al_2Cu}$). Figure 6 also shows the presence of Sc in the interdendritic regions.

A detailed image of an interdendritic region (Figure 7) showed the presence of at least three phases. The darkest phase (number 20) is Mn-rich ($\mathrm{Al_4Mn}$). The particle labelled by 21 has a composition close to Θ-$\mathrm{Al_2Cu}$. The brightest phase, labelled by 22, has a composition like Θ-$\mathrm{Al_2Cu}$ but contains 3–4 at.-% Sc.

There was microsegregation in the dendritic grains. Dendrite centres contained more V, Zr, and Mn, while dendrite rims (we avoided particles in the interdendritic region) contained more Si, Sc, Fe, and Cu. However, the segregation rate was not very high due to the small dendrite arm spacing.

4 Zaključki

Trakovi so bili sestavljeni iz stebričastih zrn, ki so rasla od površine proti sredini.

Na sredini je bilo območje z enakoosnimi zrni.

Na sredini traku je bilo opaziti tudi krčilno poroznost in neraztopljene delce.

V meddendritnih prostorih so bile tri faze: z manganom bogata faza s približno sestavo Al_4Mn, s Cu bogata faza Θ-Al_2Cu in s Cu bogata faza, ki vsebuje 3–4 at.-% Sc.

V mikrostrukturi tanjšega in debelejšega traku ni bilo veliko razlik. Zato je bila trdota statistično praktično enaka.

Raziskava navaja osnovno obnašanje mikrolegirane zlitine Al-Mn-Cu. Vendar so potrebne dodatne raziskave za optimizacijo postopka litja.

4 Conclusions

The strips consisted of columnar grains growing from the surface towards the centre.

There was a region with equiaxed grains at the centre.

There was also solidification shrinkage and undissolved particles at the strip centre.

There were three phases in the interdendritic regions: a manganese-rich phase with an approximate composition of Al_4Mn, a Cu-rich phase Θ-Al_2Cu and a Cu-rich phase containing 3–4 at.-% Sc.

There were not many differences in the microstructure of the thinner and the thicker ribbon. Thus, the hardness was statistically indistinguishable.

The research gives the basic behaviour of the microalloyed Al-Mn-Cu alloy. However, additional research is required to optimise the casting process.

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