D. Arnšek[1], F. Zupanič[1], B. Podgoršek[2], T. Bončina[1]
[1]Univerza v Mariboru, Fakulteta za strojništvo, Maribor, Slovenija / University of Maribor, Faculty of Mechanical Engineering, Maribor, Slovenia,
[2]Omco Metals Slovenia d. o. o., Žalec, Slovenija / Slovenia

Opredelitev mikrostrukturnih sestavin v naprednem aluminijevem bronu

Determination of Microstructural Constituents in an Advanced Aluminium Bronze

Izvleček

V tej raziskavi smo preučevali aluminijev bron, ki vsebuje 18 % Ni. Bron je bil lit v pesek. Analize so bile opravljene na prilitih napetih vzorcih. Vzorce smo pripravili z ustreznimi metalografskimi metodami. Preučevali smo jih s pomočjo svetlobne mikroskopije, vrstične elektronske mikroskopije, mikrokemične analize, rentgenske difrakcije in diferenčnih toplotnih analiz.

Tipična mikrostruktura v litem stanju je sestavljena iz globulitne β-faze, ki je pravzaprav intermetalna faza AlNi. Obstajata dve vrsti dvofaznih sestavnih delov v interdentritnih prostorih; obe zajemata α-Cu (trdna raztopina, obogatena s Cu) in β. Pri prvi sestavini (α-Cu + β-AlNi) so grobe β-palice v fazi α, pri drugi sestavini (α-Cu + β-AlNi) pa je prisotna lamelna struktura (α-Cu + β-AlNi)). V mikrostrukturi so prisotni tudi železno-kromovi boridi. Mikrostruktura vzorca DTA je po kakovosti enaka, vendar pa so mikrostrukturni sestavni deli precej bolj grob.

Ključne besede: aluminijev bron, nikelj, mikrostruktura, strjevanje

Abstract

In this investigation, we investigated a sand-cast aluminium bronze containing 18 % Ni. The bronze was sand cast. The analyses were carried out on cast-on tensile specimens. The samples were prepared by appropriate metallographic methods. They were investigated using light microscopy, scanning electron microscopy, microchemical analysis, X-ray diffraction and differential thermal analysis.

A typical as-cast microstructure consists of globular β-phase, which is fact the intermetallic phase AlNi. There are two types of two-phase constituents in the interdendritic spaces; both comprising of α-Cu (Cu-rich solid solution) and β. In the first (α-Cu + β-AlNi) constituent, there are coarse β-rods in phase α, while the second (α-Cu + β-AlNi) constituent exhibits a lamellar (α-Cu + β-AlNi structure. Also, iron-chromium borides are present in the microstructure. The microstructure of the DTA-sample is qualitatively the same, but the microstructural constituents are considerably coarser.

Keywords: aluminium bronze, nickel, microstructure, solidification

1 Uvod

Napredni aluminijevi broni, ki vsebujejo cink in imajo večjo vsebnost niklja, se uporabljajo v steklarski industriji, zlasti za kalupe za steklenice [1]. Ti kalupi so dolgotrajno izpostavljeni visokim temperaturam in mehanskim obremenitvam, njihova površina pa je v stiku s staljenim steklom. Zlitina za kalupe mora omogočati izjemno toplotno prevodnost, imeti pa mora tudi odlične mehanske lastnosti tako pri sobni kot pri visokih temperaturah [2]. Dodatki niklja k tem aluminijevim bronom so pomembni za zagotavljanje želenih lastnosti. V razpoložljivi literaturi ni nobenih objav v zvezi z mikrostrukturo teh naprednih bronov. Vendar pa brez znanja o tem racionalna optimizacije sestave teh zlitin ni mogoča.

1 Introduction

Advanced aluminium bronzes, containing zinc and higher contents of nickel, have been used in the glass industry, predominantly for the bottle moulds [1]. These moulds are exposed to high heat and mechanical loads for extended periods, and their surface is exposed to the molten glass. The mould alloy should have excellent heat conductivity and superb mechanical properties at room and elevated temperatures [2]. The additions of nickel to these aluminium bronzes are significant for attaining the desired properties. There are no publications regarding the microstructure of these advanced bronzes in the available literature. However, without this knowledge, any rational optimisation of the composition of these alloys is not viable.

2 Poskusni postopek

V tej raziskavi je poseben poudarek na aluminijevem bronu, ki vsebuje 18 % Ni. Bron je bil lit v pesek. Analize so bile opravljene na prilitih napetih vzorcih. Vzorce smo pripravili z ustreznimi metalografskimi metodami. Preučevali smo jih s pomočjo svetlobne mikroskopije (LM), vrstične elektronske mikroskopije (SEM), mikrokemične analize (EDS), rentgenske difrakcije (XRD) in diferenčnih toplotnih analiz (DTA). Uporabili smo svetlobni mikroskop Nikon 300, Sirion 400 NC, FEI, vrstični elektronski mikroskop, ki je bil opremljen z EDS-analizatorjem, INCA 350. Rentgenska difrakcija (XRD) je bila posneta v sinhotronu Elettra v Trstu, Italija, s pomočjo sevanja z valovno dolžino 0,099996 nm v načinu prenosa. Diferenčna toplotna analiza (DTA) je bila opravljena na temperaturi med sobno temperaturo in 1.200 °C s hitrostjo segrevanja in ohlajanja 10 °C/min.

2 Experimental

In this investigation, we give special attention to the aluminium bronze containing 18 % Ni. The bronze was sand cast. The analyses were carried out on cast-on tensile specimens. The samples were prepared by appropriate metallographic methods. They were investigated using light microscopy (LM), scanning electron microscopy (SEM), microchemical analysis (EDS), X-ray diffraction (XRD) and differential thermal analysis (DTA). We used a Nikon 300 light microscope, a Sirion 400 NC, FEI, scanning electron microscope, which was equipped with an EDS-Analyser, INCA 350. XRD was carried at synchrotron Elettra, Trieste, Italy, using radiation with a wavelength of 0.099996 nm in the transmission mode. DTA was carried out between room temperature and 1200 °C with the heating and cooling rates of 10 °C/min.

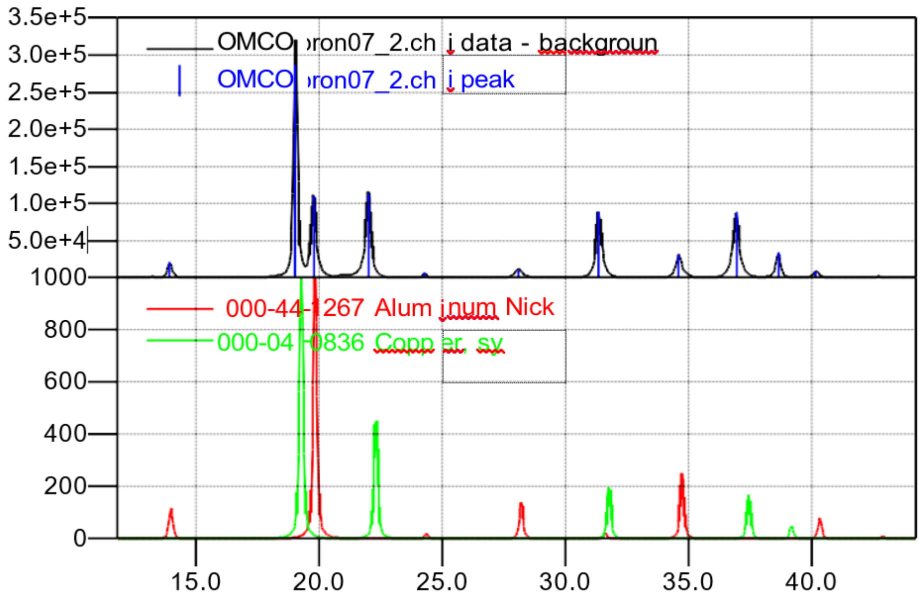
3 Rezultati in razprava

Slika 1 kaže vzorec rentgenske difrakcije raziskovane zlitine v litem stanju. Jasno je razvidno, da zlitina pretežno zajema dve fazi. V prvi fazi ima strukturo FCC, ki je enaka kot pri čistem bakru. Zato je ta faza trdna raztopina, bogata s Cu, α-Cu. Vendar pa je parameter mreže α-Cu zaradi raztopljenih elementov večji kot pri istem bakru. Dodatni vrhovi spadajo k AlNi-fazi [3]. Ta faza je intermetalna sestavina, ki izhaja iz diagrama binarne faze Al-Ni. Ima urejeno strukturo tipa CsCl. Ta struktura je enaka urejeni β-fazi iz sistema Cu-Zn. Faza β je sestavina valentnega elektrona s koncentracijo valentnega elektrona 3/2, ozna ena pa bo kot β-AlNi.

Na Sl. 2 je prikazan svetlobni mikroposnetek s tipično mikrostrukturo v litem stanju. Zajema globule faze β, ki je

3 Results and discussion

Fig. 1 shows an X-ray diffraction pattern of the investigated alloy in the as-cast condition. It can be clearly seen that the alloy mainly consists of two phases. The first phase has an FCC-structure, which is the same as in the pure copper. Therefore, this phase is a Cu-rich solid solution α-Cu. However, the lattice parameter of α-Cu is larger than that of pure copper due to the dissolved elements. Additional peaks belong to the AlNi-phase [3]. This phase is an intermetallic compound arising from the Al-Ni binary phase diagram. It has an ordered structure, type CsCl. This structure is equal to the ordered β'-phase from the system Cu-Zn. The phase β is an electron-valence compound with a valence electron concentration of 3/2, and will be denoted as β-AlNi.

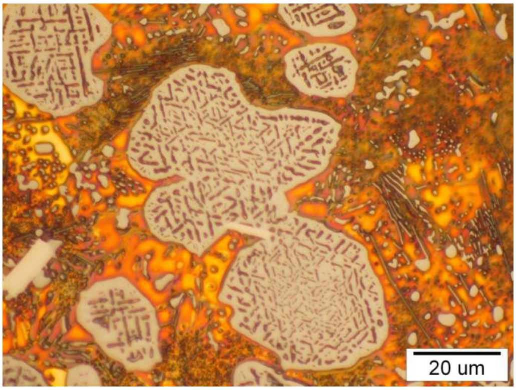


Slika 1. Difraktogram aluminijevega brona, posnet v sinhrotronu Elettra

Figure 1. X-ray diffraction pattern, taken at synchrotron Elettra.

intermetalna faza AlNi. Precipitate faze α-Cu je mogoče videti v globulah β-AlNi.

Obstajata dve vrsti dvofaznih sestavnih delov v interdentritnih prostorih. Obe zajemata α-Cu in β-AlNi. Pri prvi sestavini (α-Cu + β-AlNi) so grobe β-palice v fazi α, pri drugi sestavini (α-Cu + β-AlNi) pa je prisotna lamelna struktura (α-Cu + β-AlNi)). Podobno mikrostrukturo so odkrili Dobrašek in sod. [4]. V mikrostrukturi so prisotni tudi železno-kromovi boridi. Bor in krom vstopita v bron prek reciklažnega materiala.



Slika 2. Mikrostruktura aluminijevega brona v litem stanju (svetlobna mikroskopija)

Figure 2. A microstructure of the investigated aluminium bronze in the as-cast state (light microscopy)

Mikrostrukturne raziskave vzorca DTA so pokazale, da so bile mikrostrukturne sestavine v smislu kakovosti enake kot pri vzorcu v litem stanju, a bistveno bolj grobe (Slika 3). Primarna b-AlNi je nastala v obliki enakoosnih dendritov (Sl. 3a). Znotraj dendritov so bili tudi precipitati α-Cu in občasno boridov. Bolj groba interdentritna struktura je zajemala β-palice v α-Cu. b-palice na sliki imajo obliko ločenih okroglih delcev. Raziskava SEM je pokazala, da je večina β-palic imela v notranjosti precipitate

Fig. 2 shows a light micrograph with a typical as-cast microstructure. It consists of globules of the phase β, which is the intermetallic phase AlNi. Precipitates of the phase α-Cu can be seen within the globules of β-AlNi.

There are two types of two-phase constituents in the interdendritic spaces. Both consist of α-Cu and β-AlNi. In the first, (α-Cu + β-AlNi) constituent, the coarse β-rods are in phase α-Cu, while the second (α-Cu + β-AlNi) constituent exhibits a lamellar (α-Cu + β-AlNi) structure. Similar microstructure was found by Dobršek et al. [4]. In the microstructure are also iron-chromium borides. Boron and chromium come to the bronze through the recycle material.

The microstructural investigations of the DTA-sample showed that the microstructural constituents are qualitatively the same as were found in the as-cast sample, but were considerably coarser (Figure 3). The primary β-AlNi grew in the shape of equiaxed dendrites (Fig. 3a). Inside the dendrites, there were also precipitates of α-Cu and occasionally borides. The coarser interdendritic structure consisted of β-rods in α-Cu. The β-rods possessed in the images the shapes of separate rounded particles. SEM investigation revealed that most of the β-rods possessed precipitates α-Cu within them. The finer two-phase structure consisted of fine lamellas of α-Cu and β-AlNi.

EDS has shown that in the primary dendrites of β-AlNi the atomic ratio Al:Ni was approximately 1:1, which is consistent with the stoichiometry of AlNi, but it was also found that the phase β-AlNi dissolves almost 30 at.% of Cu, and few atomic percentages of Si, Fe and Zn.

The compositions of both two-phase areas (α-Cu + β-AlNi) were similar. The regions contained around 10 at.% Al, 13−15 α-Cu. Finejai fazi sta zajemali fine lamele α-Cu in β-AlNi.

EDS je pokazala, da je v primarnih dentritih b-AlNi atomsko razmerje Al:Ni znašalo pribl. 1:1, kar je skladno s stehiometrijo AlNi, izkazalo pa se je tudi, da faza β-AlNi raztopi skoraj 30 at.% Cu in nekaj atomskih odstotkov Si, Fe in Zn.

Sestava je bila podobna pri obeh dvofaznih območjih (α-Cu + β-AlNi). Območja so vsebovala pribl. 10 at.% Al, 13−15 at.% Ni, pribl. 70 at.% Cu, 5 at.% Zn in 1 at.% Si v Fe. Sestava najtemnejše faze (β-AlNi) je ustrezala $Al_4Ni_4Cu_2$ (atomski odstotki Ni in Al so bili skorajda identični, vsebnost Cu je bila 18 at.%, dodatno pa je vsebovala 3 5 at.% Si in okrog 2 at.% Fe).

Ti rezultati so jasno pokazali, da faza b-AlNi, ki izhaja iz diagrama binarne faze Al-Ni, lahko raztopi veliko količino bakra in drugih legirnih elementov. Ta faza je stabilna od sobne temperature do temperature likvidusa. Pri evtektoidni reakciji se ne razgradi, kot β-faza v sistemu Cu-Zn, ali b-faze v aluminijevem bronu vsebuje 5 mas.% Al in 5 mas.% Ni. Pri nižjih temperaturah se faza α loči od prenasičene trdne raztopine β-AlNi. Precipitacija je obilna pri dendritih β-AlNi, pojavi se pa tudi pri palicam podobni evtektični β-AlNi, pri tem pa nismo opazili precipitacije α od lamelnih b-AlNi. Mikrostruktura aluminijevega brona z visoko vsebnostjo niklja je precej drugačna kot pri aluminijevem bronu, ki vsebuje pribl. 5 wt.% Ni in 5 wt.% Fe [5].

Ta zlitina je vsebovala tudi boride (Sl. 4). Približna sestava boridov je bila B_2(Cr, Fe). Vsota kroma in železa je bila 30−35 at.%, pri čemer je vsebnost kroma nihala. Vsebnost kroma je nihala od 3 at.% (skoraj čisti B_2Fe) do 30 at.% (praktično čisti B_2Cr).

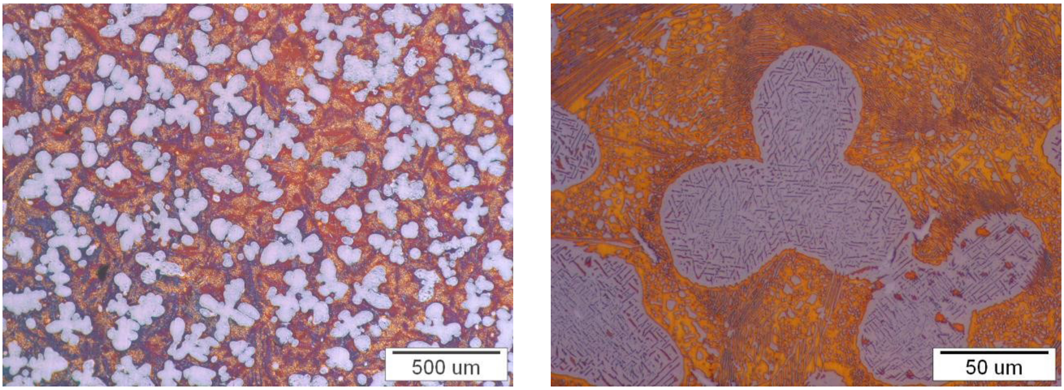
Krivulja ohlajanja DTA je pokazala, da so se med postopkom strjevanja tvorili trije vrhovi (Sl. 5). Prvi vrh je nastal pri temperaturi 1.170–1.180 °C in je vezan na

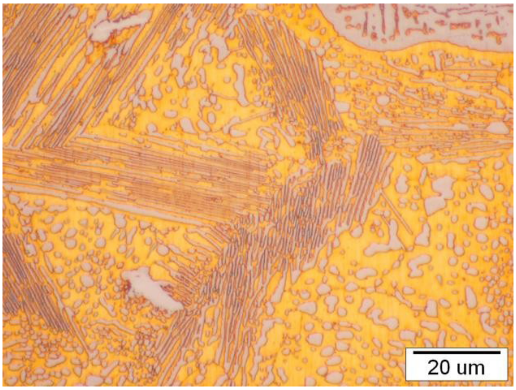
at.% Ni, around 70 at.% Cu, 5 at.% Zn and 1 at.% of Si in Fe. The composition of the darkest phase (β-AlNi) corresponded to $Al_4Ni_4Cu_2$ (the atomic percentages of Ni and Al were almost the same, the content of Cu was 18 at.%, and it additionally contained 3−5 at.% Si and around 2 at.% Fe).

These results clearly showed that the phase β-AlNi, which arises from the binary phase diagram Al-Ni, can dissolve a lot of copper and other alloying elements. This phase is stable from the room temperature up to the liquidus temperature. It does not decompose through the eutectoid reaction as β-phase in the Cu-Zn system, or β-phase in the aluminium bronzes containing 5 mas.% Al and 5 mas.% Ni. At lower temperatures, phase α precipitates from the supersaturated solid solution β-AlNi. The precipitation is abundant from the β-AlNi dendrites, it also appears from the rodlike eutectic β-AlNi, whereas we were not able to observe the precipitation of α from the lamellar β-AlNi. The microstructure of aluminium bronze, containing high nickel content is considerably different than in aluminium bronze containing around 5 wt.% Ni and 5 wt.% Fe [5].

This alloy also contained borides (Fig. 4). The approximate composition of borides was B_2(Cr, Fe). The sum of chromium and iron was 30−35 at.%, whereas the content of chromium varied. The content of chromium varied from 3 at.% (almost pure B_2Fe) to 30 at.% (practically pure B_2Cr).

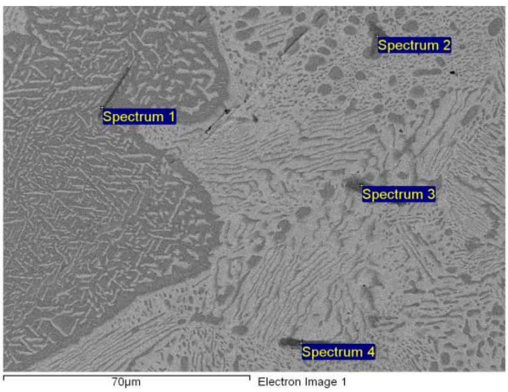
The cooling DTA-curve showed that during solidification three peaks appeared (Fig. 5). The first peak appeared at 1170−1180 °C. It is related to the formation and growth of β-AlNi. The second peak appeared at 1030−1050 °C. It is connected with the creation of the rodlike eutectic (α-Cu + β-AlNi). The third peak appeared when the lamellar two-phase constituent started to form (between 915 and 950 °C).





Slika 3. Mikrostrukture DTA-vzorca preiskanega aluminijevega brona po barvnem jedkanju (svetlobna mikroskopija)

Figure 3. Microstructures of the DTA-sample of the investigated Al-bronze after colour etching (light microscopy)



Slika 4. Elektronski mikroposnetek z odbitimi elektroni DTA-vzorca zlitine OMBX3

Figure 4. The backscattered electron micrograph of a DTA-sample showing all microstructural constituents, borides are specially indicated

tvorbo in rast b-AlNi. Drugi vrh je nastal pri temperaturi 1.030 1.050 °C in je povezan z nastankom palici podobne evtektike (α-

The greatest peculiarity of the microstructure of this aluminium bronze is the presence of the two two-phase

Cu + b-AlNi). Tretji vrh je nastal, ko se je začela tvoriti lamelna dvofazna sestavina (pri temperaturi 915 950 °C).

Največja posebnost mikrostrukture pri tem aluminijevem bronu je prisotnost dveh dvofaznih mikrostrukturnih sestavin (α-Cu + β-AlNi), ki se tvorita med strjevanjem. Mikrokemična analiza (EDS) je pokazala, da imata obe sestavini skorajda isto kemično sestavo. Zdi se tudi, da je bila sestava b-AlNi enaka tudi pri obeh sestavinah. Tukaj so na voljo številne možnosti. Prva možnost je prehod iz neurejenega b-AlNi na urejeni b'-AlNi. Druga možnost je razlika v sestavi faze α-Cu pri obeh sestavinah. Preliminarno modeliranje s programom Thermocalc kaže na to možnost, vendar pa EDS, izvedena s SEM, tega ni potrdila. Vendar pa je mikrostruktura precej fina in rešitev te težave morda leži v transmisijski elektronski mikroskopiji. Tretja možnost je izcejanje legirnih elementov v preostalo tekočino,, kar povečuje volumenski delež b-AlNi v evtektiki in povzroča pretvorbo iz paličaste evtektike v lamelno. Modeliranje s programom Thermocalc je prav tako pokazalo, da se lahko v aluminijevih bronih, ki vsebujejo manj kot 17 wt.%, tvori vrzel v likvidusu. To fazno ločevanje privede do tvorbe dveh tekočih faz; ena je bogata z nikljem, druga pa z bronom. Če je strjevanje obeh potekalo ločeno, to lahko privede do edinstvenih mikrostruktur [6].

Pri tej raziskavi smo opredelili mikrostrukturne sestavine tega aluminijevega brona z visoko vsebnostjo niklja. Vendar pa so potrebne nadaljnje študije za določitev razvoja mikrostrukture med strjevanjem in reakcijami v trdnem stanju.

microstructural constituents (α-Cu + β-AlNi) that form during solidification. EDS showed that both constituents possess almost the same chemical compositions. It also seems that the compositions of β-AlNi were the same in both constituents. There are several possibilities for these. The first possibility is the transition from disordered β-AlNi to ordered β'-AlNi. The second possibility is the difference in composition of the α-Cu phase in both constituents. The preliminary modelling using Thermocalc indicates this option, but EDS in SEM did not confirm this. However, the microstructure is rather fine and the solution of this problem may be solved by transmission electron microscopy. The third possibility is the segregation of the alloying elements into the remaining liquid, which increases the volume fraction of the β-AlNi in the eutectic, and causes the transformation from the rodlike eutectics to the lamellar one. The Thermocalc modelling also indicated that a liquidus gap can form in the aluminium bronzes containing more than 17 wt.%. This phase separation leads to the formation of two liquid phases; one is rich in nickel and the other in copper. If the solidification of both occurred separately, this could lead to unique microstructures [6].

With this investigation, we have identified microstructural constituent of this aluminium bronze with a high content of nickel. However, further studies are required to determine the microstructure evolution during solidification and solid-state reactions.

4 Conclusions

The results of this investigation revealed that in this type of aluminium bronzes two phases prevail. The first is a phase α, which is a Cu-rich solid solution with a face-centered cubic structure. The second phase is a phase

4 Sklepi

Rezultati te raziskave so pokazali, da pri tem tipu aluminijevih bronov prevladujeta dve fazi. Prva je faza α, ki je z bakrom bogata trdna raztopina s ploskovno centrirano strukturo kocke. Druga faza je faza β-Cu z urejeno strukturo CsCl in predstavlja vse intermetalne sestavine AlNi, ki izhajajo iz binarnega sistema Al-Ni, ki lahko pri visokih temperaturah raztaplja bron, silicij in železo. Preiskovani aluminijev bron je vseboval tudi boride, ki v zlitino vstopi prek recikliranih materialov.

Strjevanje te zlitine se začne s primarno kristalizacijo β-AlNi pri temperaturi 1.170 1.180 °C, ki lahko raste v obliki globul ali dentritov. Pri temperaturi 1.030-1.050 °C binarna evtektika za ne tvoriti L’ (α-Cu + β-AlNi), pri čemer b-AlNi raste v obliki palic. Proces strjevanja se zaključi s tvorbo lamelne evtektike (α-Cu + β-AlNi) pri temperaturi 915 950 °C.

V trdnem stanju se lahko odvija več procesov. Faza β-AlNi ne začne razpadati z evtektoidno reakcijo. Namesto tega se znotraj prenasičene b-AlNi tvorijo precipitati α. Nasprotno se znotraj faze α-Cu verjetno tvorijo precipitati faze β-AlNi.

Preliminarno modeliranje z uporabo programa Thermocalc kaže na možnost vrzeli v likvidusu pri aluminijevih bronih z več kot 17 wt.%. Ta fazno ločevanje lahko privede do tvorbe dveh tekočih faz; ena je bogatejša z nikljem kot druga. Strjevanje pri obeh lahko poteka ločeno, kar lahko povzroči nastanek edinstvenih mikrostruktur. Prav tako je mogoče, da iz različnih sestav evtektične faze α nastanejo različne morfologije evtektike α-Cu + β-AlNi.

β-Cu, with an ordered CsCl-structure, and represents an AlNi intermetallic compound arising from the binary Al-Ni system, which can dissolve copper, silicon and iron at elevated temperatures. The investigated aluminium bronze also contained borides, which comes to the alloy from the recycle material.

The solidification of this alloy starts with the primary crystallisation of β-AlNi at 1170−1180 °C, which can grow as globules or dendrites. At 1030-1050 °C, a binary eutectic starts to form L → (α-Cu + β-AlNi), in which β-AlNi grows in the form of rods. The solidification ends with the formation of a lamellar eutectic (α-Cu + β-AlNi) at 915−950 °C.

Several processes can take place in the solid state. The phase β-AlNi does not decompose by the eutectoid reaction. Instead, precipitates α form within the supersaturated β-AlNi. Conversely, within the phase α-Cu probably form precipitates of phase β-AlNi. Preliminary modelling using Thermocalc indicates the possibility of a liquidus gap in the aluminium bronzes containing more than 17 wt.%. This phase separation can lead to the formation of two liquid phases; one is richer in nickel than the other. The solidification of both can occur separately, which might cause the formation of unique microstructures. It is also possible that different morphologies of α-Cu + β-AlNi eutectic arise from different compositions of the eutectic α-phase.

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AKTUALNO / CURRENT

Koledar livarskih prireditev 2019 in 2020

Datum dogodka	Ime dogodka	Lokacija
26.-30.11. 2019	Online Kongress “Einkauf 4.0”http://einkauf4.lpages.co/kongres	Preko spleta
11.-12.12. 2019	Formstoffbedingte Gussfehler (Seminar)	Düsseldorf, Nemčija
12.-13.12. 2019	Digitale Lösungen für Gießereien (Seminar)	Mainz, Nemčija
18.-19.12. 2019	Werkstoffkunde der Gusseisenwerkstoffe (Seminar)	Düsseldorf, Nemčija
14.-16.01. 2020	Euroguss internationale Fachmesse für Druckguss	Nürnberg, Nemčija
27.01. 2020	OÖ Gießereiindustrie-Treffen	Vöcklabruck, Avstrija
15.-19.06. 2020	AMB-Internationale Ausstellung für Metallbearbeitung	Stuttgart, Nemčija
18.-22.10. 2020	75th World Foundry Congress	Busan, Južna Koreja