LIVARSKI VESTNIK

Izdajatelj / Publisher:
Društvo livarjev Slovenije
Lepi pot 6, P.P. 424, SI-1001 Ljubljana
Tel.: ++ 386 1 25 22 488
Fax: ++386 1 426 99 34
E-mail: drustvo.livarjev@siol.net
www.drustvo-livarjev.si

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Angleški jezik / English:
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Slovenski jezik / Slovene:
prof. Janina Šifrer

Tisk / Print:
Fleks d.o.o.

Naklada / Circulation:
4 številke na leto / issues per year
800 izvodov / copies

Letna naročnina: 35 EUR z DDV
Year subscription: 35 EUR (included PP)

Dano v tisk: september 2016

SIAPRO d.o.o.
Postaja 9
SI-5216 MOST NA SOČI

Direktor: Franci Jezeršek
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AKTUALNO / ACTUAL

Izdajanje Livarskega vestnika sofinancira Javna agencija za raziskovalno dejavnost Republike Slovenije
Publishing supported by Slovenian Research Agency
Livarski vestnik je vpisan v razvid medijev Ministrstva za kulturo pod zaporedno številko 588

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Izdelava jekla 3Cr-3W s precizijskim litjem

Production of 3Cr-3W Steel by Investment Casting

Izvleček

V prispevku je prikazana izdelava jekla 3CrW z litjem. Jeklo je bilo legirano s Co in Ta. Preiskovali smo vpliv toplotne obdelave na mikrostrukturo in trdoto vzorcev. Ugotovili smo, da je toplotna obdelava pri različnih temperaturah spreminjala mikrostrukturo in trdoto materiala.

Abstract

In this study, 3CrW steel was produced by casting. 3CrW steel alloyed with Co and Ta. The effect of heat treatment on microstructure and hardness of samples was examined. It was observed that the heat treatments performed at different temperatures change the microstructure and hardness of the materials.

1 Uvod

Rafinerije nafte uporabljajo krom-molibdenova jekla pri procesih kemične industrije in v elektrarnah. Ta jekla imajo majhen razteznostni koeficient in so namenjena obratovanju, kjer je potrebna velika odpornost proti lezenju ter koroziji pri visokih temperaturah in tlakih. Lastnosti teh jekel nadzorujejo veliki dodatki karbidotvornih elementov, kot so Cr, Mo, W, V, Ti, in Nb. Kot pri vseh kovinskih materialih tudi pri jeklih Co-Mo prihaja do slabšanja mehanskih lastnosti, kadar so izpostavljena napetostim pri dolgotrajnem obratovanju pri visokih temperaturah. Glavni vzrok za poslabšanje lastnosti teh jekel pri visokotemperaturni uporabi je povezana z deformacijami zaradi lezenja (Fu in sodel., 2007, Fu in sodel., 2009, Sawada in sodel. 2009).

1 Introduction

Petroleum refineries utilize chromium-molybdenum (Cr-Mo) alloy steel in the manufacturing of chemical industry and power plants. These steels having low thermal expansion coefficient are designed for a higher creep and corrosion resistance to operate at high temperatures and pressure. The properties of these steels are controlled with the addition of powerful carbide forming elements such as Cr, Mo, W, V, Ti, and Nb. As is in all metallic materials, Cr-Mo steels display degradation for its mechanical properties by being exposed to stress under long term service temperature. The main reason for the degradation in the properties of these steels in high temperature applications is related with the creep deformation.(Fu et al., 2007, Fu et al., 2009, Sawada et al., 2009)

Za visokotemperaturno obratovanje elektrarn so potrebna jekla, ki so zelo učinkovita pri teh temperaturah, njihovi stroški izdelave so majhni in imajo dobre mehanske lastnosti, ker morajo obratovati pri visokih temperaturah ter velikih tlakih vodne pare. Te zahteve izpolnjujejo feritna jekla. Obratovanje pri višjih zahtevah je možno s povečanjem debeline sten. Vendar pa je možno obratovanje pri veliki učinkovitosti tudi pri manjši debelini sten, ker to omogoča legiranje in toplotna obdelava materialov. V zadnjem času je bilo objavljeno manjše število raziskav s Cr-W jekli, pri katerih se da doseči zelo dobre mehanske lastnosti. V literaturi so objavljene številne raziskave Cr-Mo jekel v razvoju (Abe in sodel., 1988, Bendick in sodel., 2007, Fu in sodel., 2009).

2 Eksperimentalno delo

Sestavo jekla, ki smo ga uporabili pri poskusih in je bilo izdelano s precizijskim litjem v keramične modele, prikazuje razpredelnica 1. Uliti vzorci so imeli približne dimenzije 270 mm x 25 mm x 15 mm. S svetlobno mikroskopijo in SEM smo preiskali ulite in toplotno obdelane vzorce ter izmerili njihovo trdoto. Vsi vzorci so bili avstenitno žarjeni 6 ur pri 1200 ºC in toplotno obdelani pri različnih časih ter temperaturah 1100 ºC, 710 ºC in 660 ºC.

V naši preiskavi smo lito jeklo 3CrW različno toplotno obdelali in ugotavljali učinek teh toplotnih obdelav z merjenjem

For the power plants operating at high temperature, the production of the steel operating with high efficiency at these temperatures, having low cost and exhibiting good mechanical properties is required. Low cost and high efficiency is possible to operate under high steam pressures and at high temperatures. These conditions have made the production of ferritic steels to operate under such requirements an obligation. Operating under higher conditions is possible with higher wall thickness. However, it is also possible to operate with high efficiency at low wall thickness thanks to the materials developed with alloying and heat treatments. That is why there are a limited number of researches on CrW steels being recently produced and having potential to exhibit high mechanical properties and, even though there are numerous studies in the literature on CrMo steels starting to be developed (Abe et al., 1988, Bendick et al., 2007, Fu et al., 2009).

2 Experimental Studies

The steel from the compound used in the experiment and stated in Table 1 was produced by using investment casting method in ceramic mould after its compound was set. The cast samples are approximately in dimensions of 270 mm x 25 mm x 15 mm. The optical microscope and SEM examinations of cast and heat treated samples were performed and their hardness measurements were taken. After all the materials were austenitized for 6 h at

Razpredelnica 1: Kemična sestava jekla, izdelanega s precizijskim litjem, v mas. %

Table 1: The Chemical composition of produced steel by investment casting (mass fraction, %)

C	Cr	W	V	Si	Mn	Co	P	S
0,023	2,916	3,504	0,191	0,049	0,194	3,182	0,014	0,009

trdote ter preiskavo mikrostrukture. Polirane obruse smo 30 s jedkali z jedkalom Viella.

3 Simulacije

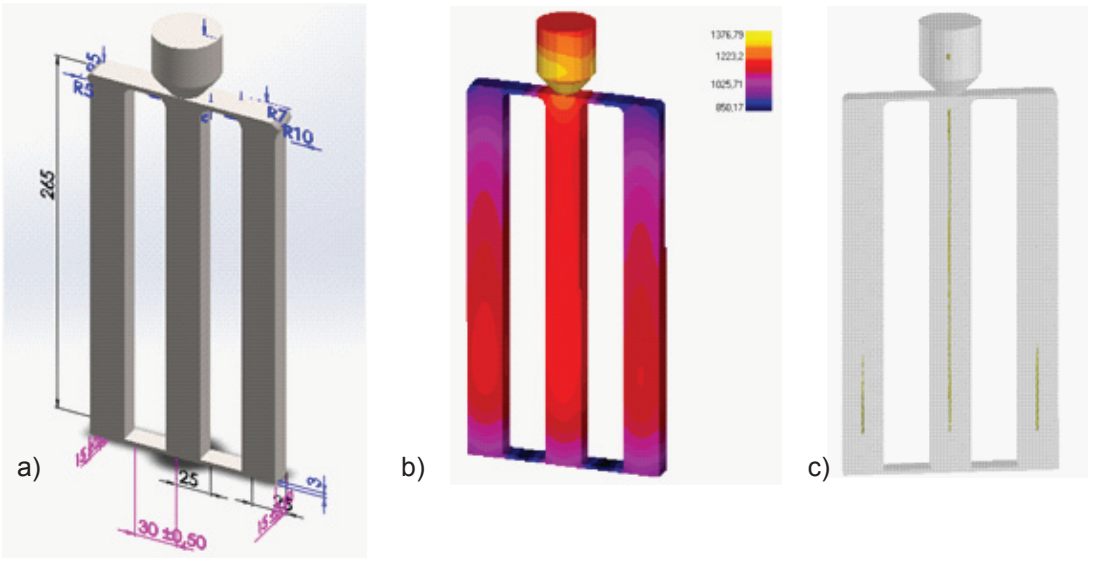
Narejena je bila simulacija litja na osnovi kemične sestave in dimenzij, opisanih v eksperimentalnem delu. 3D-risbo ulitega sestavnega dela smo pripravili s programsko opremo SolidWorks glede na dimenzije in konstrukcijo (slika 1a). Risbe so bile shranjene v STL-formatu, potem pa smo s programom SolidCast izdelali temperaturno porazdelitev in analizirali mikroporoznost. Sliko temperaturne porazdelitve ulite komponente prikazuje slika 1b. Kot se vidi na sliki 1b, se tanki prerezi med komponentami strdijo v kratkem času, kar lahko pripelje do napak predvsem v stranskih komponentah. Mikroporoznost ulitih delov, analizirana po kriteriju FCC

1200 ºC, heat treatments were performed in different duration and temperatures as 1100 ºC, 710 ºC, and 660 ºC'.

In this study, different heat treatments were performed on the 3CrW steel produced by using casting method and the effects of these heat treatments on hardness and microstructure were examined. The polished materials were etched by using Viella etching agent for 30 seconds.

3 Simulation Studies

The casting simulation was carried out for casting process that was performed in chemical composition and dimensions given in Experimental Procedure section. The component drawing was drawn on SolidWorks software as 3-D according to this dimensions and design (Figure 1a). Drawn components were saved as STL



Slika 1: a) 3D posnetek vzorca iz jekla 3Cr3W, b) analiza porazdelitve temperature med litjem, c) mikroporoznost ulitih delov, narejena po kriteriju FCC (FranCi Chiesa)

Figure 1: a) 3D figure of 3Cr3W steel b) temperature analyze during casting and c) The microporosity of cast parts, which was made according to FCC (FranCo Chiesa) criterion

(FranCo Chiesa) [Chiesa in Mammen], je prikazana na sliki 1c. Kot se vidi na tej sliki, je zelo majhna nevarnost nastanka mikroporoznosti v srednji komponenti, ker se le-ta strdi zadnja. Stranski komponenti se strdita prej.

4 Rezultati in razprava

Na osnovi faznih diagramov in CCT-krivulj iz literature smo pričakovali, da bo osnova iz feritne ali bainitne mikrostrukture. Slika 1a prikazuje lito mikrostrukturo ulitega materiala. Iz posnetkov svetlobne mikroskopije se vidi, da je lita mikrostruktura po litju nehomogena. Velika verjetnost je, da bo osnova imela feritno in bainitno mikrostrukturo, ker je bila talina ulita v keramično formo in je bilo zato njeno strjevanje počasnejše.

Ugotovili smo, da je postala prvotna nehomogena mikrostruktura po toplotni obdelavi bolj homogena in iz drobnejših zrn (slika 2 a-f). Pri ohlajanju na zraku s temperature avstenitnega žarjenja se je pričakovalo, da bo bainitna mikrostruktura odvisna od ohlajevalne hitrosti. Kot kažejo mikroposnetki, je bila mikrostruktura v glavnem sestavljena iz večjih bainitnih območij, čeprav ni bila 100 % bainitna. Pričakovalo se je, da bodo toplotne obdelave najprej zmanjšale, nato pa povečale delež bainita (Chen in sodel., 2004). Vidi se, da so mikrostrukture iz večjih zrn, čeprav imajo nekatere tudi drobnejša zrna. Drobnozrnate mikrostrukture smo zasledili v normaliziranih jeklih. Vendar pa so se velika zrna pojavila pri toplotnih obdelavah po normalizacijskem žarjenju. Vidi se, da so bila zrna bolj groba pri toplotni obdelavi pri 710 ºC v primerjavi s popuščanjem pri 660 ºC. Trdota po Brinellu se je merila po litju in po toplotni obdelavi. Vpliv toplotnih obdelav na trdoto jasno kaže slika 4. Vidi se, da se je trdota na zraku ohlajenih vzorcev

format and then temperature distribution and microporosity analysis was performed on SolidCast programme. The image of temperature distribution of cast component is shown in Figure 1 b. As seen in Figure 1b, thin connection sections between components solidify in a short time, which can cause to occur a failure especially in side components. The microporosity of cast parts, which was made according to FCC (FranCo Chiesa) criterion [Chiesa and Mammen] is given in Figure 1 c . As seen in Figure 1 c, because of the later solidification of the middle component than side components, there is very little risk of microporosity in middle component.

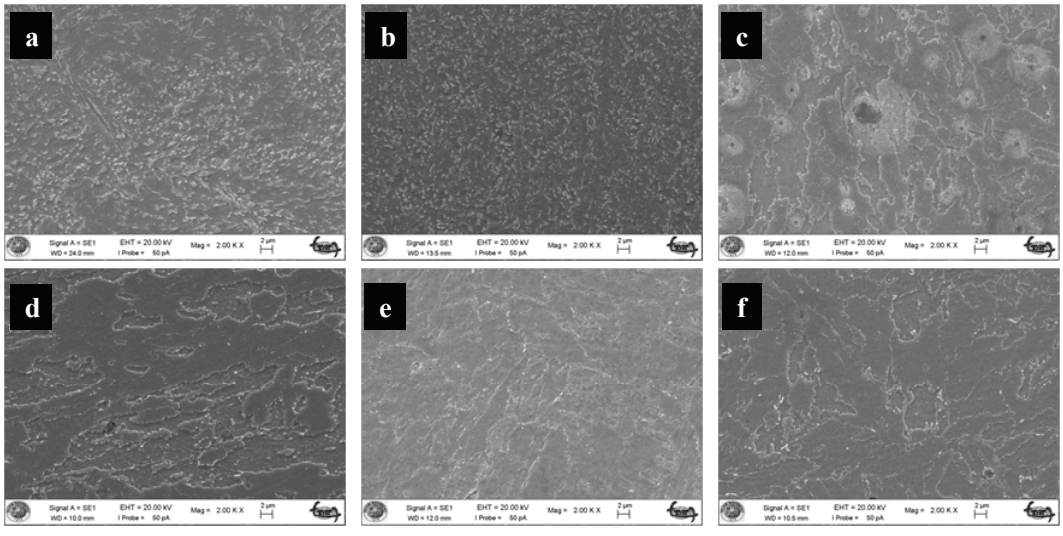
4 Experimental Results and Discussion

It is expected that the matrix is in the ferritic or bainitic structure as a result of examination of the phase diagrams and CCT curves upon the literature review as per the compound. Figure 1a illustrates the casting microstructure of the cast material. It can be understood that the casting structure had a non-homogenous microstructure from the optical microscope images taken after casting. There was a higher possibility that the matrix phase in the casting structure had a ferrite and bainite structure as a result of the fact that the casting was made into a ceramic mould and consequently the cooling rate was slower.

It was observed that after the heat treatments, the non-homogeneous casting microstructure became more homogeneous and had finer particles (Figure 2 a-f). In the air cooling processes carried out from austenite temperatures, bainitic microstructure is expected depending on cooling rate. As seen in the following microstructures, despite the fact that microstructures are not 100%,

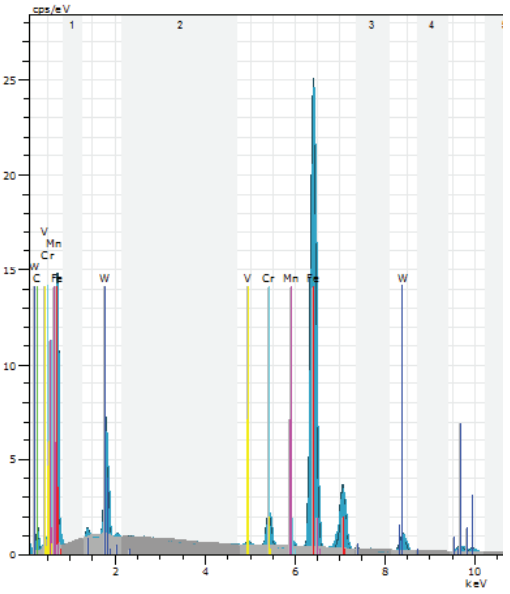
po avstenitizaciji (pri 1200 °C in 1100 °C) zmanjšala po popuščanju (pri 710 °C in 660 °C). Dejstvo, da je bila trdota po popuščanju pri 710 °C manjša kot pri popuščanju pri 660 °C, je bilo pričakovano. Znano je, da višje temperature popuščanja zmanjšujejo trdoto in povečujejo žilavost. Trdota po avstenitizacijski toplotni obdelavi je bila tudi višja kot po drugih toplotnih obdelavah. Različne izmerjene trdote so povezane z deležem bainitnih območij v mikrostrukturi materiala. Povečanje bainitnih območij vpliva na trdoto. EDS-analize smo naredili na vzorcu, ki je bil 6 ur homogeniziran pri 1200 °C in 1 uro normaliziran pri 1100 °C, potem pa 4 ure popuščan pri 710 °C.

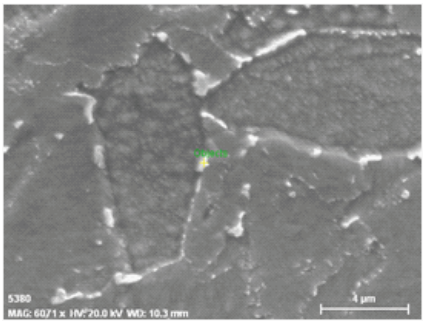
they were mostly comprised of a majority of bainite clusters. It is expected that the heat treatments will decrease and then increase the amount of bainite.(Chen et al., 2004) As is seen from the microstructures, while some microstructures had finer grains, some had larger grains. Fine grained bainitic structures were observed in the normalized steels. However, coarser bainite grains took place of finer bainite grains in the heat treatments after the normalization. It can be seen that the grains were coarser in the tempering process made at 710 °C compared to the tempering process made at 660 °C. Brinell hardness of the materials was taken after the casting structure and



Slika 2: SEM-mikroposnetki jekel a) lita mikrostruktura, b) homogenizirano 6 ur pri 1200 °C, nato popuščano 4 ure pri 660 °C, c) homogenizirano 6 ur pri 1200 °C, nato popuščano 4 ure pri 710 °C, d) homogenizirano 6 ur pri 1200 °C, nato normalizirano 1 uro pri 1100 °C, e) homogenizirano 6 ur pri 1200 °C, nato normalizirano 1 uro pri 1100 oC in popuščano 4 ure pri 660 °C, f) homogenizirano 6 ur pri 1200 °C, nato normalizirano 1 uro pri 1100 oC in popuščano 4 ure pri 710 °C

Figure 2: SEM microstructures of steels, a) Casting microstructure b) Homogenized at 1200 °C for 6 h and then tempering at 660 ° C for 4 h c) Homogenized at 1200 °C for 6 h and then tempering at 710 ° C for 4 h d) Homogenized at 1200 °C for 6 h and normalizing at 1100 °C for 1 h e) Homogenized at 1200 °C for 6 h and normalizing at 1100 °C for 1 h and then tempering at 660 ° C for 4 h f) Homogenized at 1200 °C for 6 h and normalizing at 1100 °C for 1 h and then tempering at 710 ° C for 4 h





Element	C norm. [m.f.,%]	C Atom. [at.%]
Iron	77.43	82.62
Carbon	1.40	6.96
Tungsten	16.83	5.45
Chromium	3.47	3.97
Manganese	0.39	0.43
Vanadium	0.48	0.56
Total: 99.57	100.00	100.00

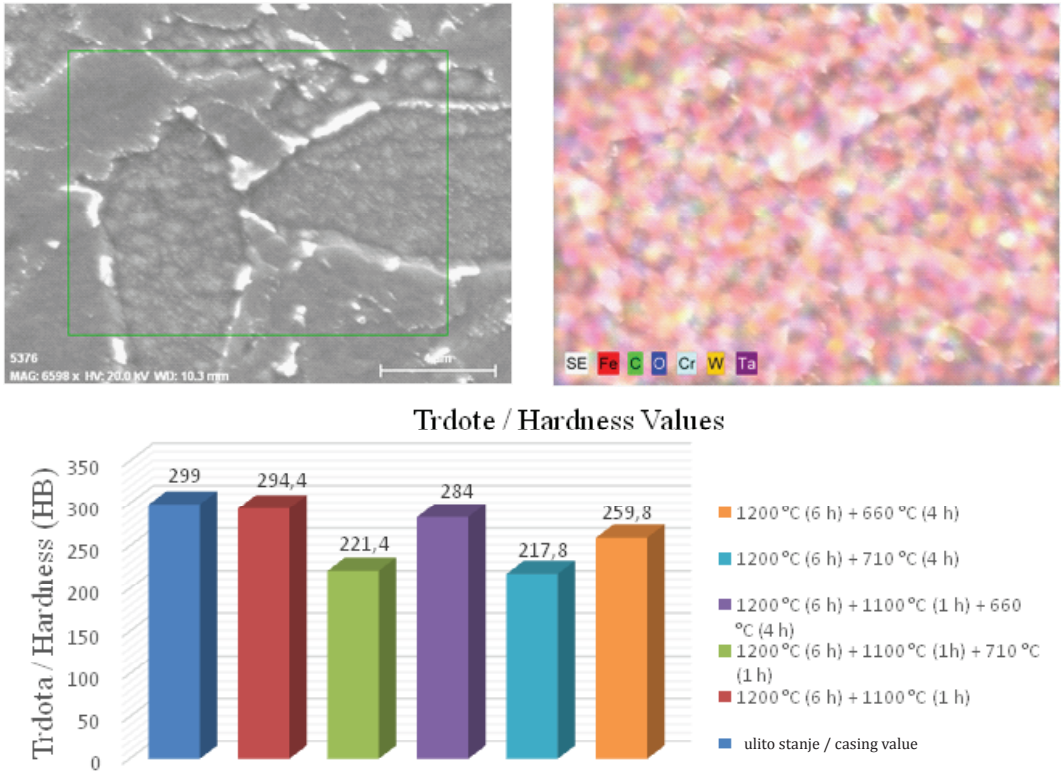
Slika 3: EDS-analiza delca v vzorcu, homogeniziranem 6 ur pri 1200 °C in normaliziranem 1 uro pri 1100 °C, nato popuščanem 4 ure pri 710 °C

Figure 3: EDS result of a particle in the sample homogenized at 1200 °C for 6 h and normalized at 1100 °C for 1 h and then tempered at 710 ° C for 4 h

heat treatments. The effects of the heat treatments on the hardness can be seen clearly in Figure 4. It was observed that the hardness values obtained with air cooling after the austenitization (at 1200 °C and 1100 °C) decreased after the tempering (at 710 °C and 660 °C). The fact that the hardness value after the tempering process made at 710 °C is lower than the hardness value obtained following the tempering process made at 660 °C was an expected result. As it is known, high tempering temperature signifies low hardness and high toughness. The hardness value after the austenitizing heat treatment was also higher than the hardness value after the other heat treatments. Obtaining different hardness values can be related to the amount of bainite structure occurring in the structure of material. The increase in the bainitic structure affected the hardness EDS analysis was taken from the sample, which homogenized at 1200 °C for 6 h and normalized at 1100 °C for 1 h and then tempered at 710 ° C for 4 h. Also mapping analyses were performed in the same sample. As seen from the Figure 3 and 4, carbides, which were rich in Cr and Fe, were found in the sample homogenized at 1200 °C for 6 hours and normalized at 1100 °C for 1 hour and then tempered at 710 ° C for 4 hours.

5 Conclusion

As it can be understood from the microstructure images and hardness, the heat treatments performed at different temperatures change the microstructure and hardness of the material. Finer grains were obtained in the tempering process made at 660 °C for 4 hours compared to the tempering process made at 710 °C for



Slika 4: Trdote jekel

Figure 4: Hardness values of steels

5 Sklepi

Mikroposnetki in meritve trdot kažejo, da toplotne obdelave pri različnih temperaturah različno vplivajo na mikrostrukturo in trdoto materiala. 4-urno popuščanje pri 660 °C je dalo drobnejša zrna kot enako popuščanje pri 710 °C. Zato je bila trdota večja.

Iz mikroposnetkov se lahko sklepa, da ima material feritno-bainitno mikrostrukturo. Delež bainita se spreminja v odvisnosti od toplotne obdelave. Povečanje količine bainita povečuje trdoto. Trdota jekla z manjšimi bainitnimi območji je bila večja kot pri jeklih z bolj grobimi zrni.

4 hours, and consequently higher level of hardness was obtained.

It could be observed from the obtained microstructures that the material had a ferrite-bainite microstructure. The rate of bainite varied depending on the heat treatment. The fact that amount of bainite increased also increased the hardness. The hardness of the steel having finer bainite clusters was higher than the steel having coarse grains.

6 Zahvala

Ta prispevek je bil pripravljen na osnovi doktorske dizertacije Gökhana Arıcıja na Podiplomski šoli za naravoslovje in uporabne vede Univerze Sekcuk, Konya, Turčija. Delo je financirala Univerza Selcuk v okviru raziskovalnih projektov pod številko projekta 14401084.

6 Acknowledgment

This study was carried out as a PhD thesis by Gökhan Arıcı in the Graduate School of Natural and Applied Science at Selcuk University, Konya, Turkey. This work was also supported by Selcuk University Scientific Research Projects under Grant Numbers 14401084.

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Centrifugalna atomizacija Nd-Fe-B lusk za izdelavo trajnih magnetov

Centrifugal atomization of Nd–Fe–B flakes used for production of permanent magnets

Povzetek

Nd-Fe-B trajni magneti se veliko uporabljajo v aplikacijah, kjer je zahtevan visok energijski produkt magnetov s ciljem zmanjšanja mase izdelka. Njihovo uporabo lahko najpogosteje zasledimo na področju avtomobilske industrije, v računalništvu (trdi diski) ali za izdelavo generatorjev v vetrnih turbin. Konvencionalne tehnologije litja so zelo neugodne za izdelavo trajnih magnetov, saj povzročajo tvorbo nehomogene mikrostrukture, ki vključuje segregate ?-Fe in področja bogata z RE elementi. Na drugi strani, sodobne tehnologije hitrega strjevanja (litje trakov, hitro strjevanje trakov na vrtečem se bobnu, centrifugalna atomizacija,...) omogočajo dosego homogene in drobnozrnate mikrostrukture.

Ta prispevek opisuje uporabo hitrega strjevanja z metodo centrifugalne atomizacije za izdelavo Nd-Fe-B lusk. V raziskavi so predstavljeni rezultati študije vpliva sestave zlitine in procesnih parametrov centrifugalne atomizacije na mikrostrukturo Nd-Fe-B zlitin. Metalografska analiza mikrostrukture je bila raziskana z optičnim in vrstičnim elektronskim mikroskopom. V prispevku so prav tako raziskani vplivi procesnih parametrov na razvoj mikrostrukture hitro strjenih lusk ter posledično vpliv le-teh na magnetne lastnosti izdelanih magnetov.

Abstract

Nd–Fe–B-type permanent magnets are widely used in applications that require a high magnetic energy product in order to reduce weight. Automotive industry, hard drives, or wind turbines are examples of applications where their use can be found. Conventional casting techniques cause the formation of ?-Fe and large RE-rich regions. On the other hand, techniques like strip casting, melt-spinning, and centrifugal atomization, produce homogeneous and fine scaled microstructures.

This paper discusses the application of rapid solidification by the centrifugal atomization method for preparation of Nd–Fe–B flakes. The effect of alloy composition and various process parameters of centrifugal atomization on the microstructure of rapidly solidified Nd–Fe–B alloy were investigated. The microstructures and the phase composition were examined by metallographic techniques, namely optical and scanning electron microscopy. Additionally, the influence of the processing methods on the microstructures of as-cast flakes and subsequent magnetic properties of the prepared magnets will be discussed.

1 Uvod

Tehnike hitrega strjevanja omogočajo zaradi velikih ohlajevalnih hitrosti izdelavo zlitin, katerih mikrostruktura se zelo razlikujejo od tistih, ki so značilne za konvencionalne postopke litja. V primeru kristalnih materialov je mogoči doseči tvorbo drobnih kristalov primarne faze, prenasičenost trdne raztopine ter mikrostrukturo z visoko stopnjo kemijske homogenosti [1,2]. Morfologija mikrostrukture prav tako močno vpliva na magnetne lastnosti NdFeB magnetov [3,4]. Pri postopkih konvencionalnega litja vsebuje mikrostruktura NdFeB zlitin ob trdo magnetni fazi prav tako velike deleže α-Fe faze ter groba zrna Nd bogate faze. Na drugi strani, omogočajo metode hitrega strjevanja doseganje višjih ohlajevalnih hitrosti, ki vodijo do nastanka drobnozrnate homogene mikrostrukture, kar posledično omogoča izdelavo magnetov z nižjo vsebnostjo redko zemeljskih elementov (Nd+Dy ~ 14at%) [4-7]. S postopkom centrifugalne atomizacije dosežemo velike ohlajevalne hitrosti s brizganjem taline na hitro se vrteč disk [1], pri čemer se talina strdi v obliki tankih lusk debeline ca. 100 μm, katerih mikrostruktura je drobnozrnata.

Ta članek obravnava uporabo hitrega strjevanja z metodo centrifugalne atomizacije za pripravo Nd-Fe-B lusk. V raziskavi so predstavljeni rezultati študije vpliva sestave zlitine in procesnih parametrov centrifugalne atomizacije na razvoj mikrostrukture Nd-Fe-B zlitin. Metalografska analiza mikrostrukture je bila raziskana z optičnim in vrstičnim elektronskim mikroskopom. V prispevku so prav tako raziskani vplivi procesnih parametrov na mikrostrukturo hitro strjenih lusk ter posledično vpliv le-teh na magnetne lastnosti izdelanih magnetov.

1 Introduction

The rapid solidification technique with high cooling rates allows preparation of alloys with microstructures which are very different from those obtained by conventional casting procedures. In the case crystalline materials, crystals of fine primary phase, supersaturated solid solution and improved chemical homogeneity have been reported [1,2]. The magnetic properties of the NdFeB magnets are also strongly influenced by morphology of its microstructure [3, 4]. Conventional casting results in the formation of a substantial quantity of α-Fe and coarse Nd-rich regions, while rapid solidification techniques enable higher cooling rates that lead to formation of fine scale microstructures with higher homogeneity which requires a lower rare-earth content (Nd+Dy ~ 14 at%) [4-7]. For the centrifugal atomization are also characteristic higher cooling rates, which are achieved by pouring the melt on a rapidly rotating disc [1]. Whereby the melt solidified forming thin flakes with a thickness about 100 μm with fine scale microstructure.

This paper discusses the application of rapid solidification by means of the centrifugal atomization method for preparation of Nd–Fe–B flakes. The effect of alloy composition and various process parameters of centrifugal atomization on the microstructure of rapidly solidified Nd–Fe–B alloy were investigated. The microstructures and the phase composition were examined by metallographic techniques, namely optical and scanning electron microscopy. Additionally the influence of the processing parameters on the microstructures of as-cast flakes and subsequent magnetic properties of the prepared magnets will be discussed.

2 Eksperimentalni del

Da bi zadostili zahtevam doseganja visoke čistosti Nd-Fe-B zlitin, ki je nujno potrebna za dosego dobrih magnetnih lastnosti, je bila centrifugalna atomizacija izvedena v indukcijski vakuumski peči (slika 1a). Pri izdelavi zlitine smo posebno pozornost posvetili izogibanju kontaminacije taline s kisikom in ogljikom. Za izdelavo zlitin smo uporabili predzlitine (Nd-Pr, FeB, FeDy, GaDy) in čiste kovine (Fe, al, Cu, Co) z visoko čistostjo vsaj 99,9 wt%. Nominalna sestava zlitine je podana v tabeli 1 in je bila enaka za vse vzorce.

V izogib oksidaciji je bila vakuumska peč evakuirana do 10^{-3} mbar-a in trikrat prepihana z argonom visoke čistosti Ar (5.0). V začetni fazi prepihovanja peči je bila nadzorovana

2 Experimental Procedure

In order to satisfy the demand for high purity of Nd-Fe-B alloy, which is essential for obtaining good magnetic properties, the centrifugal atomization was performed in a inductively heated vacuum furnace (figure 1a). Special care was taken to avoid the contamination of the melt with oxygen and carbon. High purity master alloys (Nd-Pr, FeB, FeDy, GaDy) and pure components (Fe, Al, Cu, Co) were used, with a purity of no less then 99,9 %. The nominal composition, which is presented in table 1, was the same for all samples.

In order to avoid oxidation, the vacuum furnace was operated at 10^{-3} mbar and then refilled with high purity Ar (5.0) three times. As the initial stage of purging the furnace

a)

b)

Slika 1: Vakuumska peč za centrifugalno atomizacijo ter prikaz vmesne livne posode z izlivno odprtino na dnu

Figure 1: Vacuum furnace for centrifugal atomization (a) and tundish with an orifice at the bottom (b)

Tabela 1: Nominalna kemijska sestava zlitine s tolerancami

Table 1: Nominal chemical composition mass fraction, % with tolerances

Dy	Fe	Nd	Pr	B	Ga	Al	Cu	Co
3,6	64	27,4	0,6	0,93	0,2	0,15	0,15	3
± 0,2	± 0,5	± 0,5	± 0,1	± 0,05	± 0,05	± 0,05	± 0,05	± 0,2

vsebnost kisika v peči, z analizatorjem kisika, ki je bil nameščen na izpušni cevi Ar iz peči. Izmerjena vsebnost kisika je bila manjša od 0,1 ut%. Pretaljevanje in litje je bilo izvedeno v zaščitni atmosferi argona. Posebna pozornost je bila posvečena zagotavljanju kemijske stabilnosti kokile zaradi velike reaktivnosti redko zemeljskih elementov v tekočem stanju. Kontaminacija taline s kokilo je nedopustna, saj so za dosego dobrih magnetnih lastnosti dovoljena le minimalna odstopanja od želene kemijske sestave zlitine. V izogib temu sta bili kolika in vmesna livna posoda zaščiteni s keramičnim premazom iz Al_2O_3, ki ga odlikuje dobra kemijske stabilnosti in adhezivnosti. V dno livne posode je bila izvrtana izlivna odprtina s premerom 10 mm, ki je bila prav tako premazana s tanko plastjo Al_2O_3 (slika 1b).

Pred litjem sta bila tako kokila kot livna posoda predgreti. Kokila je bila predgreta induktino na 300 °C pred zalaganjem s surovinami. Livna posoda pa je bila uporovno segreta z grelcem, ki je bil vertikalno vstavljena v livno posodo. Da bi preprečili strjevanje taline v trenutku, ko le-ta pride v stik z livno posodo, smo vertikalni uporovni grelec izvlekli iz livne posode tik pred začetkom litja. Dejanska temperatura v livni posodi je bila izmerjena s termoelementom, ki je bil nameščen v uporovnem grelcu. V osrednjem delu livne posode je bila izmerjena temperature približno 300 °C, medtem ko je bila na dnu, na mestu izlivne odprtine ta le 100°C. Hitrost vrtenja ohlajevalnega diska je bila regulirana s potenciometrom. Eksperimenti so bili izvedeni pri naslednjih vrtilnih hitrostih diska 210 RPM, 240 RPM in 280 RPM. Razdalja med ohlajevalnim diskom in izlivno površino je bila določena eksperimentalno na 10-15 mm. Za dosego optimalne homogenosti centrifugalno atomiziranih lusk je bil med taljenjem prav tako variiran čas taljenja

was reached, oxygen analyzer, which is mounted to the Ar exhaust pipe showed less then 0,1 % of oxygen. The melting and casting itself was performed under protective Ar atmosphere. Special caution was given to the chemical stability of the crucible due to the aggressive behavior of the rare earth elements in the molten state. A narrow chemical composition range is required in order to obtain good magnetic properties. Therefore no contamination of the melt in contact with the crucible can be expended. Al_2O_3 was chosen as a ceramic coating for the crucible and the tundish for its good chemical stability and proper adhesiveness to the crucible. An orifice of 10 mm was drilled in the bottom of the tundish and carefully painted with a thin layer of Al_2O_3 (figure 1b).

Both the crucible and the tundish were pre-heated. The crucible was inductively heated to approx. 300 °C prior to loading of the raw material. The tundish was resistance heated with a heater which was planted vertically into the tundish. Just moments prior to the casting stage, the vertical heater was lifted, thus preventing the instant solidification of the melt when it comes in contact with the tundish. The temperature of the tundish is measured by a thermocouple installed inside the heater and gives an approximation of the actual temperature of the tundish. The latter is approximated to 300 °C in the body of the tundish and 100 °C at the bottom, where the orifice is located. The cooling wheel frequency rate is regulated by a potentiometer. Frequencies of 210 RPM, 240 RPM and 280 RPM were selected for the experiment. The orifice to cooling wheel distance was set experimentally to 10-15 mm. Time of the melting stage was also varied in order to achieve optimum homogeneity of the centrifugally atomized flakes. By extending the time of the melting stage we assured

Tabela 2: Prikaz pogojev centrifugalne atomizacije

Table 2: Various melting conditions

	Vzorec / Sample 1	Vzorec / Sample 2	Vzorec / Sample 3
RPM	210	240	280
Čas taljenja / Melting time [min]	6	10	10

taline. S podaljšanjem časa taljenja smo zagotovili, da so bile vse vhodne komponente, še posebej Fe, ki ima visoko temperaturo tališča 1452°C, pretaljene.

V tabeli 2 so prikazani različni pogoji taljenja, pri katerih so bili izvedeni poskusi centrifugalne atomizacije. Po vsakem poskusu smo kemijsko sestavo vzorcev preverili z ICP-OES analizo (Perkin Elmar Optima 5300 DV). V vzorcih smo prav tako preverili vsebnost kisika z ELTRA ON9000 analizatorjem ter vsebnost ogljika z ELTRA CS 800 napravo. Sledila je priprava magnetov po principu prašne metalurgije.

Za proizvodnjo sintranih Nd-Fe-B magnetov so bile centrifugalno atomizirane luske najprej hidrirane in nato dehidrirane (HDD), pri čemer postanejo krhke ter razpadejo v prah velikosti približno 100 mikrometrov. Po HDD procesu sledi mletje prahu v JET mlinih za dosego drobnozrnatih prahov z ozko porazdelitvijo velikosti delcev ca. 50 mikronov (D50). Porazdelitev velikosti delcev prahov smo izmerili z napravo za analizo velikosti delcev Bettersize – BT-2001(dry). Parametri mletja so bili enaki za vse tri vzorce centrifugalno atomiziranih lusk. Po mletju so bili magneti stisnjeni v magnetnem polju in sintrani v vakuumski peči v zaščitni atmosferi Ar (5.0). Sledile so meritve zelene in dejanske gostote vzorcev po sintranju. Pri vseh vzorcih je bila dosežena zelena gostot 4,3 ± 0,1 g/cm^{3,} medtem ko je bila gostota po sintranju 7,5 ± 0,05 g/cm^3, kar je zelo blizu teoretični gostoti Nd-Fe-B zlitin (7,65 g/cm^3) in nakazuje na uspešen proces sintranja. Po sintranju smo

that all of the raw material, especially Fe, with a high liquid temperature of 1452 °C was melted.

Various samples and melting variables are presented in table 2. After each melting stage, the composition was measured by the ICP-OES method, Perkin Elmer Optima 5300 DV. Carbon and oxygen content was measured by ELTRA ON900 analyzer, and the carbon content was measured by the Carbon Sulphur Determinator ELTRA CS 800. Magnets were prepared according to powder metallurgy principles.

For production process of sintered Nd-Fe-B magnets the centrifugally atomized flakes are first hydrated, and then dehydrated (HDD) by which the material becomes brittle and turns to powder of approx. 100 microns in size. After the HDD process, the material is JET-milled to obtain fine powders with a narrow particle size distribution (PSD), with a D50 of 5 microns. PSD was measured using a Bettersize - BT-2001(dry) particle size analyzer. The milling parameters were fixed for all the samples. After JET-milling the magnets were pressed in a magnetic field and sintered in a vacuum furnace in protective Ar (5.0) atmosphere. Green density and sintered density were carefully measured. A green density of 4,3 ± 0,1 g/cm^3 and a sintered density of 7,6 ± 0,05 g/cm^3 were obtained for all the measured samples, which is close to the theoretical density of Nd-Fe-B (7,65 g/cm^3) and gives evidence of a successful sintering stage. After the magnets were prepared the composition was again measured and

ponovno preverili sestavo magnetov s VRF metodo (PANalytical AXIO MAX). Magnetne lastnosti smo izmerili z permagrafom (Magnet Phisic Steingroever).

Mikrostruktura in debelina Nd-Fe-B lusk je bila raziskana po različnih procesnih pogojih v prečnem prerezu na metalografskih obrusih z optičnim mikroskopom, Nikon Epiphot 300 opremljenim s sistemom za digitalno kvantitativno analizo slike (Olympus DB12 in programski paket Analysis). Pred metalografsko pripravo so bili vzorci sintranih magnetov vpeti v kovinske sponke, za zagotavljanje želene lege med vročim vlaganjem vzorcev v termoplastično maso. Za ta tip vzorcev, ki vsebujejo veliko majhnih por, je priporočljivo vroče vlaganje, saj pri hladnem vlaganju ne pride do zapolnitve vseh por z duroplastično maso. Sledilo je brušenje z uporabo SiC brusnega papirja z različno granulacijo od P320, P500, P1000, P2500 do P4000. Poliranje metalografskih vzorcev je bilo izvedeno z 1 mikronsko diamantno suspenzijo, ki mu je v zadnji stopnji še sledilo poliranje s 0,05 mikronsko koloidno raztopino korunda. V obeh primerih smo za poliranje uporabili polirno podlago iz klobučevine, ki je bila navlažena pred nanosom polirnega sredstva. Zaradi majhne debeline vzorcev so bili izbrani krajši časi poliranja s koloidnim korundom, največ 2 minuti. Metalografski vzorci so bili pripravljeni na avtomatski napravi za brušenje in poliranje, pri čemer se je polirna/brusna podlaga vrtela v smeri urinega kazalca s hitrostjo 250/40 rpm. Vzorci so bili med brušenjem obremenjeni z 10 N, v zadnji stopnji poliranja pa samo z 5 N. Dodatno je bila morfologija in mikrokemijska sestava NdFeB lusk raziskana z vrstičnim elektronskim mikroskopom FEI Srion NC opremljenim z energijsko disperzijskim spektrometrom (EDX).

verified by the XRF PANalytical AXIO MAX. The resulting magnetic properties were measured by a permeagraph (Magnet Physik Steingroever).

The microstructure and the thicknesses of Nd-Fe-B flakes after different process conditions were examined on the transversal cross-section on the metallographic samples with an optical microscope, Nikon Epiphot 300, equipped with a system for digital quantitative image analysis (Olympus DB12 and software programme Analysis). Before metallographic preparation the samples were positioned using metal clamps and carefully hot mounted in thermoset resin. Hot mounting is preferable since the small samples contain many cavities, which are not easily filled using cold mounting substances. Grinding was performed using SiC papers P320, P500, P1000, P2500, P4000. Followed by polishing using 1 micron diamond suspension and the final step was polishing using 0,05 micron colloidal alumina. In both polishing steps a micro-cloth was used which was wetted prior to applying the polishing agent for additional lubrication. Due to the small thickness of the samples, polishing times with colloidal alumina should be kept short, at a maximum of about 2 min. Samples were prepared on an automated grinder//polisher, using clockwise rotation 250/40 rpm, and a force of 10N, expect at the final step where the force was reduced to 5N. Additionally, the morphology and chemical microanalysis of NdFeB flakes was examined with the scanning electron microscope FEI Siron NC equipped with an energy-dispersive X-ray (EDX) detector.

3 Results and Discussion

As can be seen on table 3 the composition measurements show that in case of short

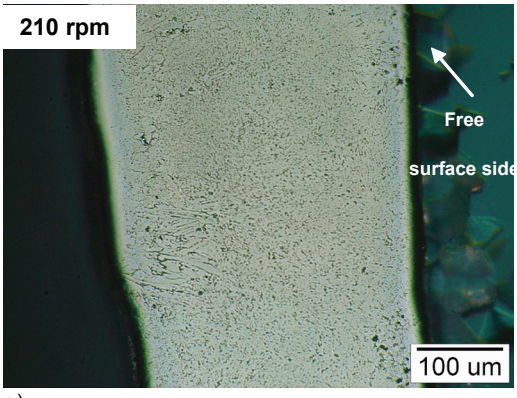
3 Rezultati in diskusija

Iz rezultatov izmerjene kemijske sestave vzorcev po taljenju, ki so prikazani v tabeli 3, je razvidno, da pri kratkem času taljenja, 6 minut, ostane še določena količina železa neraztopljena. Kot posledica le-tega je delež izmerjenega železa v vzorcu 1 z ICP analizo nekoliko manjši. S podaljšanjem časa taljenja na 10 minut se vsa količina železa raztopi v talini, kar je tudi potrjeno z meritvijo deležev železa z ICP analizo v vzorcu 2 in 3 (tabela 3). Iz tabele 3, ki prikazuje rezultate meritev ICP analize je razvidno, da v vseh vzorcih obstajajo majhna odstopanja od želene kemijske sestave. Vendarle, zaradi merilne nenatančnosti ter nenatančnosti priprave vzorcev lahko sklepamo, da je kemijska sestava vzorcev v mejah dovoljenih toleranc. Ustreznost rezultatov ICP-OES analize je bila prav tako potrjena s meritvijo kemijskih sestav sintranih vzorcev z XRF metodo (tabela 4). Zaradi omejitev merilnih metod vsebnosti bora v vzorcih ni bilo mogoče določiti. Vsebnost bora naj bi bila v vzorcih približno 1%, zato je bila sestava le-teh normalizirana na 99%. Odstopanja med ICP-OES in XRF metodama je mogoče pripisati merilni negotovosti posamezne metode ter negotovosti priprave vzorcev. Tako lahko zaključimo, da so razlike izmerjenih vrednosti še v okviru sprejemljivih toleranc. V vseh vzorcih je melting time, 6 min, some of the iron remains undissolved. As a consequence there is lack of iron content in the ICP measurement of sample 1. When the melting time was extended to 10 min, all of the iron has dissolved into the melt, proven by the ICP measurement shown in table 3 as sample 2 and 3. As can be seen from the table of ICP measurements, there is small deviation from the expected chemical composition. Yet, due to the measuring uncertainty and sample preparation uncertainty, we accepted the chemical compositions, to be within acceptable tolerances. The XRF method introduced to the sintered parts verifies the ICP-OES measurements (table 4). Due to the limitation of the method boron content was not measured. The value of the boron content was approximated to 1% and the results were normalized to 99%. The discrepancy between the ICP-OES and XRF measurements was accounted to the measuring uncertainty and sample preparation uncertainty. We deducted that the differences are within the acceptable tolerances. The measured fraction of impurities values is low for all the measured samples. The Argon chosen for the experiment was of high grade and successfully prevented the oxidation of the rare-earth (RE) elements. The RE's are extremely susceptible to oxidation, thus high vacuum and high purity protective

Tabela 3: Rezultati ICP-OES meritev

Table 3: ICP-OES measurements results (mass fraction, %)

	Dy (ut%)	Fe (ut%)	Nd (ut%)	Pr (ut%)	B (ut%)	Ga (ut%)	Al (ut%)	Cu (ut%)	Co (ut%)
Želena sestava / Target composition	3,60	64,00	27,40	0,60	0,93	0,20	0,15	0,15	3,00
Vzorec 1	3,67	61,56	29,18	0,61	0,99	0,22	0,25	0,26	3,25
Vzorec 2	3,38	63,46	27,90	0,66	1,03	0,18	0,20	0,19	2,99
Vzorec 3	3,38	63,36	27,97	0,65	1,06	0,19	0,22	0,21	2,96



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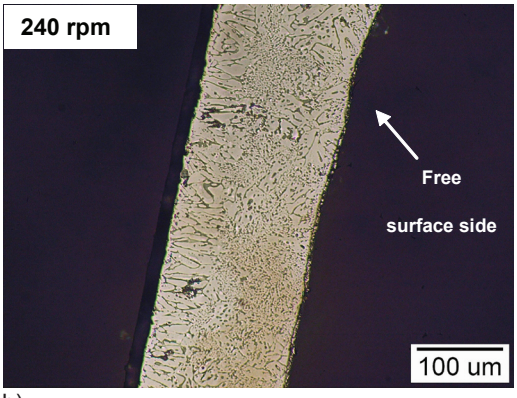

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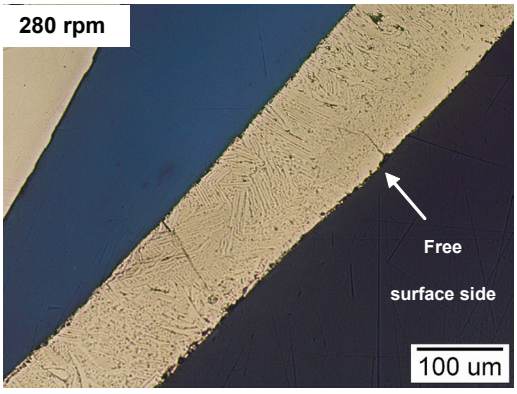


c)

Slika 2: Optični mikroposnetki prikazujejo mikrostrukturo NdFeB lusk v poliranem stanju v odvisnosti od hitrosti vrtenja diska

Figure 2: Optical micrographs of the polished cross section showing the microstructure of the NdFeB flakes at different wheel speeds

izmerjen delež nečistoč zelo majhen. Argon, ki je bil izbran za izvedbo eksperimentov, je visoke čistosti in je uspešno preprečil oksidacijo redko zemeljskih (RE) elementov. Visokokakovostne magnete lahko iz zlitin, ki vsebujejo redkozemeljske elemente, izdelamo le v visokem vakuumu oz. v atmosferi zaščitnega plina visoke čistost, saj so redko zemeljski elementi zelo dovzetni za oksidacijo (tabela 5).

Z metodo centrifugalne atomizacije je mogoče s spreminjanjem procesnih parametrov izdelati luske z različno debelino. Ugotovljeno je bilo, da z naraščanjem vrtilne hitrosti diska s 210 rpm na 280 rpm debelina lusk zmanjšuje od ~320 do ~120 µm. Z naraščanjem vrtilne hitrosti diska se zmanjšanje debelina lusk, kar je posledica

gases are a necessity for obtaining alloys of high quality (table 5).

With the centrifugal atomization technique by varying the parameters of the device, the flakes can be produced with different thicknesses. It was observed that the increase in the wheel speed from 210 rpm to 280 rpm results in a decrease in the flakes thickness from ~320 to ~120 µm. As the increase in the wheel speed leads to a reduced flakes thickness, the cooling rate increases, and therefore the finer microstructure was obtained. Moreover, with increasing wheel speed changes also the mode of solidification from equiaxed to directional (fig. 2 a,b,c and fig. 3a and fig. 4a). This leads to formation of columnar grains of hard magnetic $Nd_2Fe_{14}B$ phase,

Tabela 4: Rezultati XRX meritev

Table 4: XRF measurements results (mass fraction, %)

	Al (ut%)	Co (ut%)	Cu (ut%)	Dy (ut%)	Fe (ut%)	Ga (ut%)	Nd (ut%)	Pr (ut%)	SUM (ut%)
Vzorec / Sample 1	0,22	3,2	0,21	3,88	61,36	0,18	29,1	0,85	99
Vzorec / Sample 2	0,29	3,08	0,29	3,82	63,54	0,23	27,02	0,73	99
Vzorec / Sample 3	0,18	3,02	0,23	3,64	63,78	0,23	27,21	0,71	99

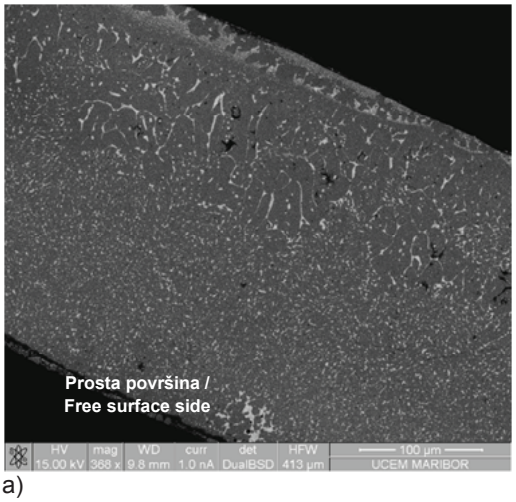
naraščanja ohlajevalne hitrosti, ki pripomore k tvorbi drobnozrnate mikrostruktura. Nadalje, z naraščanjem vrtilne hitrosti diska se prav tako spremeni način strjevanja zlitine od enakoosnega k usmerjenemu (slika 2a, b, c in slika 3a, b, c). Kar vodi do nastanka stebričastih zrn trdo magnetne faze $Nd_2Fe_{14}B$, obdanih z Nd- bogato fazo ter zatre nastanek mehko magnetne α-Fe faze (slika 3 in slika 4).

Slika 5 prikazuje magnetne lastnosti vzorcev, ki nakazujejo nedvoumno odvisnost med časom taljenja (povezano s raztopitvijo železa v talini), kot tudi pojavom nastanka α-Fe faze (povezano surrounded by Nd-reach phase and suppresses the formation of soft magnetic α-Fe phase (fig. 3 and fig. 4).

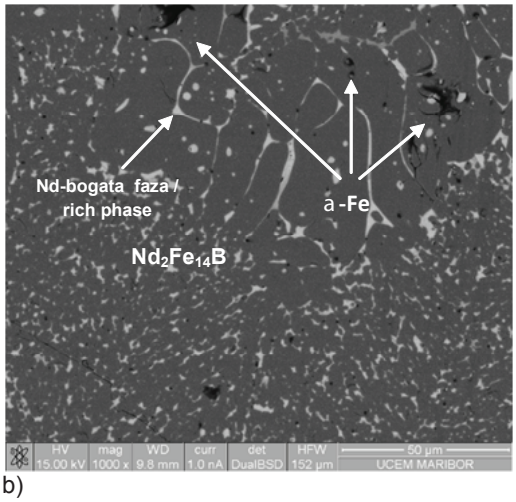
Tabela 5. Meritev nečistoč v luskah v litem stanju

Table 5. Impurities measurements on as-cast flakes (mass fraction, %)

	Kisik (ut%) / Oxygen	Ogljik (ut%) / Carbon
Vzorec / Sample 1	0,04	0,08
Vzorec / Sample 2	0,029	0,06
Vzorec / Sample 3	0,024	0,11



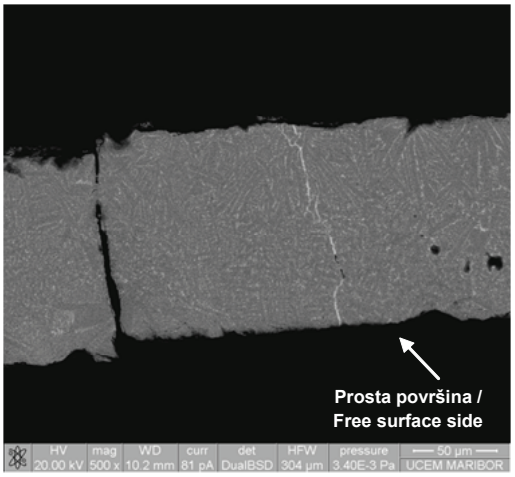
a)



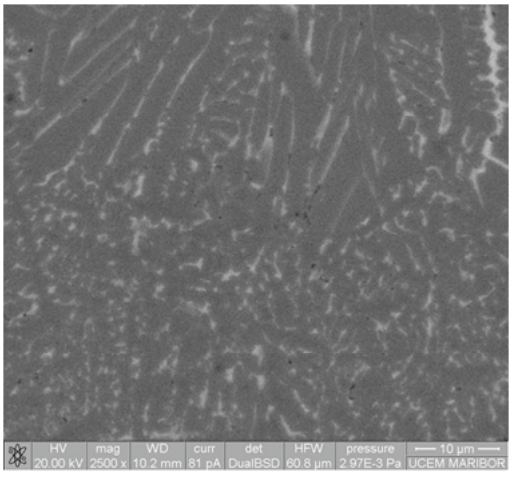
b)

Slika 3: Morfologija centrifugalno atomiziranih NdFeB lusk pri hitrosti diska 210 rpm; SEM mikroposnetek, polirano stanje v prečnem prerezu

Figure 3: The morphology of the centrifugally atomized NdFeB flake at wheel speed of 210 rpm; SEM micrograph, polished cross section



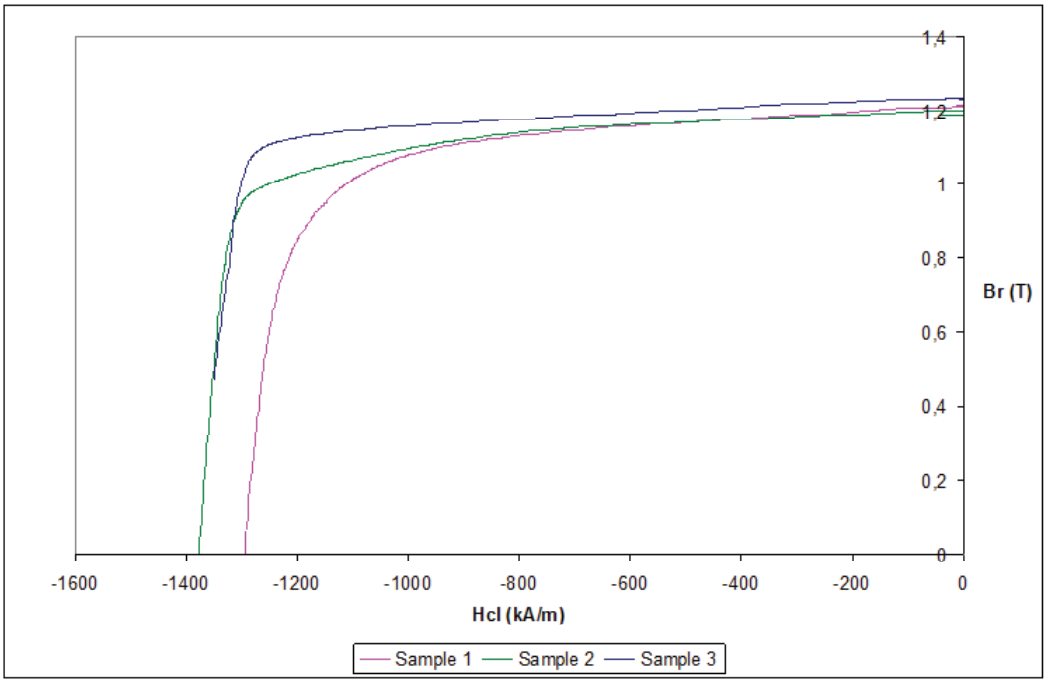
a)



b)

Slika 4: Morfologija centrifugalno atomiziranih NdFeB lusk pri hitrosti diska 280 rpm; SEM mikroposnetek, polirano stanje v prečnem prerezu

Figure 4: The morphology of the centrifugally atomized NdFeB flake at wheel speed of 280 rpm; SEM micrograph, polished cross section



Slika 5: B-H krivulja vzorcev magnetov od 1 do 3

Figure 5: B-H curves for samples 1 to 3

z ohlajevalno hitrostjo in hitrostjo vrtenja hladilnega diska). Iz diagram na sliki 5, kjer x os prikazuje remanenco (Br), je razvidno, da pomanjkanje železa v zlitini zmanjšuje Br (vzorec 1). Na drugi strani, z naraščanjem hitrosti strjevanja se zatre nastanek α-Fe faze, kar posledično pripomore k naraščanju koercitivnosti (HcI) magnetov.

4 Zaključki

Metoda centrifugalne atomizacija omogoča ob pravilno izbranih procesnih parametrih (hitrost vrtenja diska, čas taljenja) izdelavo NdFeB lusk z želeno mikrostrukturo, ki je sestavljena iz drobnih stebričastih zrn glavne $Nd_2Fe_{14}B$ faze, ki so ločena s tankim filmom Nd- bogate faze. Takšna morfologija mikrostrukture je zelo zaželene za izdelavo visoko zmogljivih magnetov z visoko koercitivnostjo.

Magnetic properties can be seen in figure 5. They show unambiguous dependency with the melting stage time (tied to dissolving of the iron in liquid phase), as well as the formation of the α-Fe (tied to the cooling rate and the cooling wheel speed). We can see on axis x, representing the remanence (Br), that the lack of iron in the alloy reduces the Br (sample 1). On the other hand, we can see that with increasing solidification rate, which suppresses the formation of the α-Fe the coercivity (HcI) of the magnets increases.

4 Conclusions

Centrifugal atomization technique enables at optimal processing parameters (wheel speed, melting time) the production of NdFeB flakes with desired microstructures consisting of fine columnar grains of main $Nd_2Fe_{14}B$ phase separated by thin films of Nd-rich phase. Such microstructure morphology is strongly desirable for preparation of high performance magnets with high coercively.

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Inovativni pristop k izboljšanju disperzije kovinskega oksida in strukturno odvisnih lastnosti električnih kontaktnih materialov iz srebrovega/kovinskega oksida brez Cd

Innovative Approach on Improvement of Metal Oxide Dispersion and Structure Dependent Properties of Cd Free Silver/Metal Oxide Electrical Contact Materials

Izvleček

V tem prispevku je opisan poskus inovativnega pristopa za izboljšanje disperzija kovinskih oksidov (MeO) v električnih kontaktnih materialih iz Ag-MeO z vnosom nanodelcev kovinskih oksidov (SnO_2, ZnO In_2O_3, Bi_2O_3 in WO_3) v srebrovo osnovo in pripravo nanokompozitnih prahov z metodo šablone iz topnega škroba. Morfologijo in mikrostrukturo pripravljenih sestavljenih prahov Ag-MeO smo analizirali s SEM in FESEM. Dobljeni rezultati kažejo, da imajo izdelani sestavljeni prahovi veliko stopnjo in enakomernost disperzije nanodelcev kovinskega oksida. Poleg tega smo ugotovili, da so zelo primerni za izdelavo električnih kontaktnih materialov na osnovi Ag-MeO in da so njihove prednosti v izboljšanju homogenosti mikrostrukture ter izboljšanju strukturno odvisnih lastnosti. Pripravljeni in preiskani kontaktni materiali iz Ag-SnO_2 in Ag-ZnO kažejo fino disperzijo oksidov, večjo trdoto in gostoto ter manjšo poroznost in rahlo manjšo električno prevodnost kot ustrezni standardno pripravljeni materiali vendar še vedno v zahtevanem območju za trgovske kontaktne materiale te vrste.

Ključne besede: električni kontakti brez Cd, metoda s šablono, Ag-MeO sestavljeni prahovi, morfologija, mikrostruktura, fizikalne lastnosti

Abstract

In the present study an innovative attempt has been made to improve dispersion of metal oxides (MeO) in Ag-MeO electrical contact materials by introducing metal oxide nanoparticles (SnO_2, ZnO, In_2O_3, Bi_2O_3, and WO_3), into silver matrix and preparation of nanocomposite powders by template method using soluble starch as template material. The morphology and microstructure of the prepared Ag-MeO composite powders were studied using SEM and FESEM. The obtained results show that the prepared composite powders exhibit high degree of dispersion of metal oxide nanoparticles and high uniformity. Furthermore, it was found that they are very suitable for further production of Ag-MeO electrical contact materials and that the benefits are primarily illustrated as an improvement of homogeneity of microstructure and structure dependent properties. The prepared and

studied final Ag-SnO2 and Ag-ZnO contact materials exhibit finer dispersion of oxides, higher hardness, higher density, lower porosity and slightly lower electrical conductivity compared to corresponding conventionally prepared materials, however still within the required range for commercial contact material of this type.

Keywords: Cd free electrical contacts, template method, Ag-MeO composite powders, morphology, microstructure, physical properties

1 Uvod

Električni kontakti na osnovi srebra predstavljajo pomembno in široko uporabljano skupino funkcionalnih materialov [1]. Ker strožja okoljska zakonodaja EU za materiale, ki vsebujejo Cd (RoHS, WEEE), omejuje uporabo splošno zelo dobrih kontaktov iz Ag-CdO, se vlagajo veliki napori v razvoj alternativnih električnih kontaktov brez Cd. Čeprav so se materiali iz Ag-SnO_2 in Ag-ZnO zdeli kot zelo obetavna, okolju bolj prijazna zamenjava, se pri povišanih temperaturah sorazmerno slabo obnašajo in imajo slabo preoblikovalnost, če so izdelani po klasičnih postopkih. Dosedanje raziskovalne izkušnje kažejo, da je najpomembnejši parameter za doseganje homogene mikrostrukture zelo fina disperzija kovinskega oksida v srebrovi osnovi in da je splošno sprejeto dejstvo, da se delovanje kontaktnih materialov iz Ag-MeO lahko občutno izboljša z boljšo disperzijo sestavljenih prahov, kar se navadno doseže z uporabo ali sintezo drobnejših delcev kovinskega oksida [2]. Drobnejše mikrostrukture so boljše, še posebej, kadar se uporabljajo različni kovinski oksidi, ki spreminjajo lastnosti materialov s spreminjanjem interakcije med srebrovo osnovo in glavnim kovinskim oksidom [2,3,4]. Poleg tega je sprejeto dejstvo, da drobnejši kovinski oksidni delci izboljšuje odpornost proti zvarjanju kontaktov in v določenih razmerah zmanjšujejo stopno erozije [5]. Če upoštevamo, da je disperzija oksida neposredno povezana

1 Introduction

Silver based electrical contacts represent a significant and widely used group of functional materials [1]. As stricter EU environmental legislations concerning materials containing Cd (RoHS, WEEE) limit the use of generally superior Ag-CdO contacts, significant efforts are undertaken to develop alternative Cd-free electrical contacts. Although, Ag-SnO_2 and Ag-ZnO have emerged as promising, more environmentally friendly replacements, when they are produced by common processing routes they are characterized by rather poor over-temperature behavior and poor workability. So far research experience shows that the most important factor for obtaining homogenous microstructure is very fine metal oxide dispersion in silver matrix and as it is generally accepted that the performance of Ag-MeO contact materials can be enhanced by increase in the composites dispersion, which is usually achieved by use or synthesis of finer metal oxide particles [2]. Finer microstructures are advantageous especially when different metal oxides are used that alter the materials properties by changing the interaction between silver matrix and the main metal oxide [2,3,4]. Furthermore, it is accepted that smaller metal oxide particles promote anti-welding characteristics an under certain conditions decrease erosion rate [5]. Considering that the oxide dispersion is directly related to an applied processing technique, a variety of production routes has

z uporabljenim načinom izdelave, je bilo razvitih več teh načinov. Vendar dosežejo standardne mokre ali suhe metode mešanja prahov svoje tehnične meje pri velikosti delcev 1–2 mikrometra zaradi bolj ali manj izrazitega skepljanja [6]. Poleg tega je izboljšana disperzija kovinskega oksida in s tem homogenost mikrostrukture kontaktnih materialov iz Ag-MeO možna le z alternativnimi pristopi priprave začetnih sestavljenih prahov. Zato so raziskovalci preiskali različne negalvanske kemične metode izločanja delcev. Dodatno moderne metode s polimeri podprte tvorbe anorganskih nanokompozitov in metod bioulivanja, ki uporabljajo različne organske šablone, nudijo nove možnosti za pripravo zelo enakomernih nanokompozitnih delcev [5-8].

Glede na povedano smo v tem prispevku opisali inovativni poskus, kako povečati disperzijo Ag-MeO v kontaktnih materialih z uvajanjem kovinskih oksidnih nanodelcev v srebrovo osnovo in pripravo nanokompozitnih prahov z metodo škrobove šablone. Prikazali bomo vpliv uporabe kovinskih oksidnih nanodelcev in njihovega uvajanja v srebrovo osnovo na izboljšanje disperzije in enakomernosti mikrostrukture pripravljenih sestavljenih prahov ter o tem razpravljali. Poleg tega bomo prikazali mikrostrukturno odvisne lastnosti, kot so gostota, poroznost, trdota in električna prevodnost, na primerih izdelanih kontaktnih materialov na osnovi Ag-SnO2 in Ag-ZnO in primerjali med seboj te materiale s standardno izdelanimi materiali iste vrste.

2 Eksperimentalni del

Prahove, sestavljene iz srebra in kovinskega oksida, smo izdelali po prilagojeni metodi s šablono [9]. Uporabili smo trgovske nanodelce oksidov SnO_2, ZnO, In_2O_3, Bi_2O_3,

been developed. However, conventional powder mixing methods either wet or dry, reach their technical limit at powder particle sizes of about 1-2 microns due to more or less pronounced agglomerate formation [6]. Further improvement of metal oxide dispersion and thus homogeneity of microstructure of Ag-MeO contact materials is only possible by using alternative approaches in preparation of starting powder composites. For that purpose different electroless chemical methods based on chemical precipitation were investigated. In addition, modern polymer assisted inorganic nanocomposite formation and bio-casting methods that utilize different organic templates offer new possibilities for preparation of very uniform nanocomposite structures [5-8].

In view of that, in the current study an innovative attempt has been made to increase dispersion of Ag-MeO contact materials by introducing metal oxides nanoparticles in silver matrix and preparation of nanocomposite powders by template method using soluble starch as a template material. The effect of using metal oxide nanoparticles and method of their introduction in silver matrix on improvement of dispersion and uniformity of microstructure of the prepared composite powders were studied and discussed. In addition, structure dependent properties such as: density, porosity, hardness and electrical conductivity are demonstrated on the examples of the produced final Ag-SnO2 and Ag-ZnO contact materials and compared to each other and to conventionally prepared materials of the same type.

2 Experimental

Silver-metal oxide composite powders were produced by a modified template

in WO_3, velikosti 40–100 nm ter trgovski prah $AgNO_3$ kot prekurzorje. Uporabljena metoda sinteze uporablja škrob kot mehko organsko šablono in sloni na preprosti predpostavki, da se $AgNO_3$ za razliko od večine kovinskih nitratov pri segrevanju razkroji na elementno srebro namesto na ustrezen oksid. V prvi fazi smo dodali v destilirano vodo topni škrob in ga ob stalnem mešanju segreli na 40–50 ºC. Raztopino smo potem segreli na temperaturo vrelišča (~100 ºC) in jo pri tej temperature držali 15 min, potem pa smo jo ohladili na 50–70 ºC. Predhodno pripravljeno suspenzijo nanodelcev kovinskega oksida smo počasi dodajali. $AgNO_3$, delci kovinskega oksida pa so bili dodani v količini, potrebni, da se doseže želeno masno razmerje Ag:kovinski oksid v končnem materialu, tj. Ag-SnO_2 (92:8); Ag-ZnO (92:8); Ag-$SnO_2In_2O_3$ (89,1:8:2,9); Ag-SnO_2WO_3 (90:9,5:0,5) in Ag-$SnO_2In_20_3Bi_2O_3$ (89,2:8,7:0,5:1,6). Pripravljene mešanice smo sušili v sušilni komori pri 80 ºC, dokler ni voda izparela in so ostali trdni sestavljeni prahovi. Te prahove smo potem segreli in žarili v mufelni peči 4 ure pri 650 ºC. Pri tem je srebrov nitrat razpadel v elementno srebro, ki se je nahajalo med nanodelci kovinskega oksida. Škrobovo šablono smo nato odstranili. Vzorci končnih kontaktnih materialov Ag-SnO_2 in Ag- ZnO so bili izdelani iz dobljenih sestavljenih prahov po standardni metodi metalurgije prahov. Razmere pri izdelavi so podrobno opisane v [10]. Morfologijo in mikrostrukturo izdelanih sestavljenih prahov smo analizirali z vrstičnim elektronskim mikroskopom (SEM) JEOL JSM 6610LV in vrstičnim elektronskim mikroskopom s poljsko emisijo (FESEM) Tescan MIRA3. Gostoto vzorcev končnih električnih kontaktnim materialov smo ugotavljali s standardnimi metodami. Trdoto smo merili na poliranih vzorcih pri sobni temperaturi z merilnikom trdote po Vickersu z obtežbo 5 kp. Podane trdote predstavljajo

method [9] using commercial SnO_2, ZnO In_2O_3, Bi_2O_3, and WO_3 nanoparticles (40-100 nm) and commercial $AgNO_3$ powder as precursors. The applied synthesis method utilizes starch as a soft organic template and it is based on a simple principle that $AgNO_3$, unlike most metal nitrates, when heated thermally decomposes to elemental Ag instead of its respective oxide. In the first step soluble starch was added into distilled water, preheated at 40-50 ºC under continuous stirring. Solution was further heated up to boiling point (~100 ºC), where it was kept for 15 min, and then cooled down to 50-70 ºC. Previously prepared metal oxide nanoparticle suspensions were slowly added to solution during vigorous mixing. After few minutes $AgNO_3$ water solution was slowly added. Both $AgNO_3$ and metal oxide particles were added in quantities necessary to achieve desired Ag to metal oxide weight ratio in final material i.e. Ag-SnO_2 (92:8); Ag-ZnO (92:8); Ag-$SnO_2In_2O_3$ (89.1:8:2.9); Ag-SnO_2WO_3 (90:9.5:0.5) and Ag-$SnO_2In_20_3Bi_2O_3$ (89.2:8.7:0.5:1.6). The prepared mixtures were dried at 80 ºC in chamber dryer until water was evaporated and solid composites were obtained. The solid composites were subsequently burned and put into a muffle furnace pre-heated at 650 ºC, where they were calcinated for 4h. During the combustion and later calcination, silver nitrate was transformed to elemental Ag with embedded metal oxide nanoparticles and the starch template was removed. Samples of final Ag-SnO_2 and Ag-ZnO contact materials were prepared from the obtained composite powders via conventional powder metallurgy route. The processing conditions are given in more detail in [10]. Morphology and microstructure of the prepared composite powders were studied using JEOL JSM 6610LV scanning electron microscope (SEM) and Tescan MIRA3 XM field emission scanning electron

povprečje 5 meritev. Električno prevodnost preiskanih materialov smo merili na vzorcu s premerom 8 mm z merilnikom na vrtinčne tokove Foerster SIGMATEST 2.069.

3 Rezultati in razprava

Morfologijo sestavljenega prahu srebro-kositrov oksid, izdelanega po metodi s škrobovo šablono, prikazujejo SEM-mikroposnetki na sliki 1.

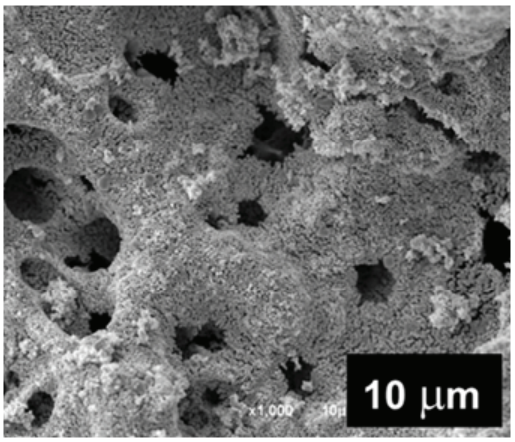
Dobljene sestavljene zgradbe spominjajo na intersticijski prostor v strjeni škrobovi osnovi, ki ga je prvotno zasedala mešanica prekurzorjev, a bi se lahko nanašal tudi na sprostitev plinov med odstranjevanjem šablone. Očitno smo dosegli zelo dobro mešanje in veliko disperzijo nanodelcev v srebrovi osnovi. Če vzamemo, da proces ni preveč dolgotrajen, da odstranjevanje škroba ni preveč zapleteno in da ni ovir za istočasno uvajanje različnih kovinskih oksidov, smo ta material za šablono izbrali pri nadaljnji izdelavi sestavljenih prahov.

microscope (FESEM). Density of the samples of final electrical contact materials was determined by standard methods. Hardness measurements were carried out on polished samples at room temperature using a Vickers hardness tester applying load of 5 kp. The reported hardness values are an average of five readings. Electrical conductivity of the investigated materials was measured using Foerster SIGMATEST 2.069 eddy current instrument with an 8 mm diameter probe.

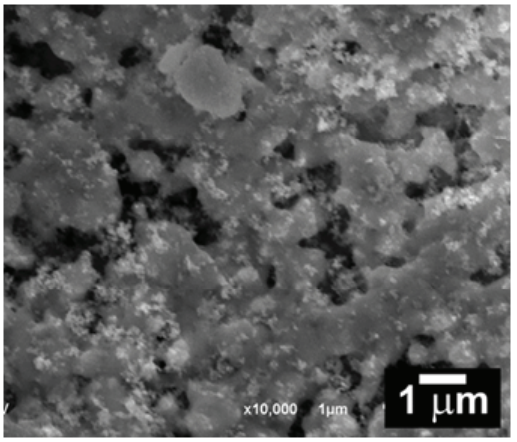
3 Results and discussion

Morphology of the silver tin-oxide composite powder prepared by template method using starch as a template material is illustrated by SEM images presented on Fig 1.

The obtained composite structure resembles the interstitial space within the solid starch matrix that was initially occupied by the precursor mixture but it can also be related to the release of gases during the template removal stage. It is evident that



×1000



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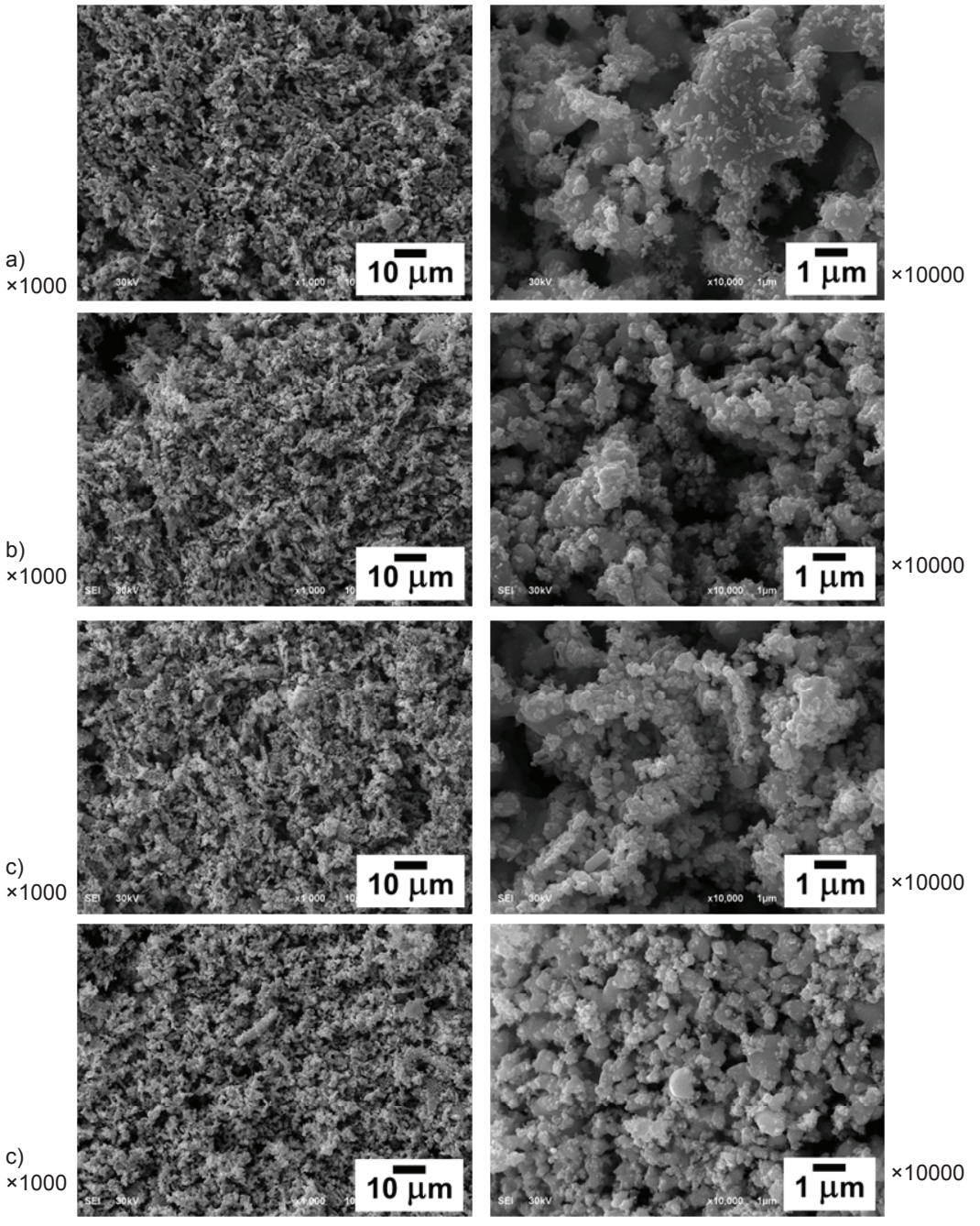
Slika 1. Morfologija sestavljenega prahu Ag-SnO_2, izdelana po metodi z uporabo šablone

Fig.1. Morphology of the Ag-SnO_2 composite powder prepared using template method

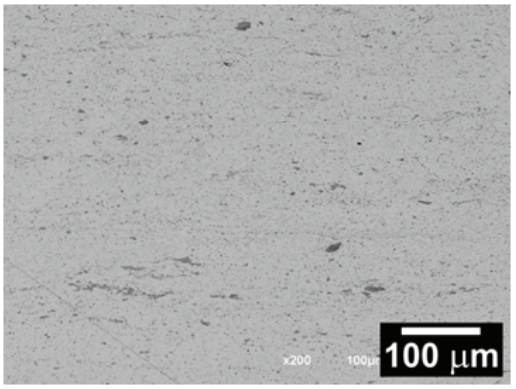


Slika 2. Morfologija sestavljenih prahov Ag-MeO, izdelanih po šablonski metodi:
a) Ag-ZnO, b) Ag-$SnO_2In_2O_3$, c) Ag-SnO_2WO_3, d) Ag-$SnO_2In_2O_3Bi_2O_3$

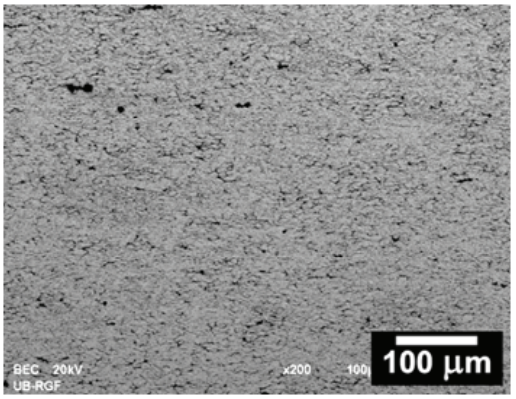
Figure 2. Morphology of the Ag-MeO composite powders prepared using template method:
a) Ag-ZnO, b) Ag-$SnO_2In_2O_3$, c) Ag-SnO_2WO_3, d) Ag-$SnO_2In_2O_3Bi_2O_3$

SEM posnetki na sliki 2 kažejo morfologijo izdelanih in preiskanih sestavljenih prahov Ag-MeO. Tako smo prikazali in potrdili prednosti uporabljene metode s šablono: dobro mešanje, veliko disperzijo nanodelcev kovinskega oksida in veliko enakomernost. Rezultati SEM-analize kažejo, da je metoda s šablono zelo primerna za istočasno uvajanje nanodelcev več kovinskih oksidov v srebrovo osnovo. To je pomembno predvsem zato, ker so pri uporabljenem postopki s šablono oksidni delci inkapsulirani v srebrovi osnovi in se izognemo izcejam. Zato predstavljajo izdelani sestavljeni prahovi zelo primerne prekurzorje za izdelavo električnih kontaktnih materialov.

a)



b)



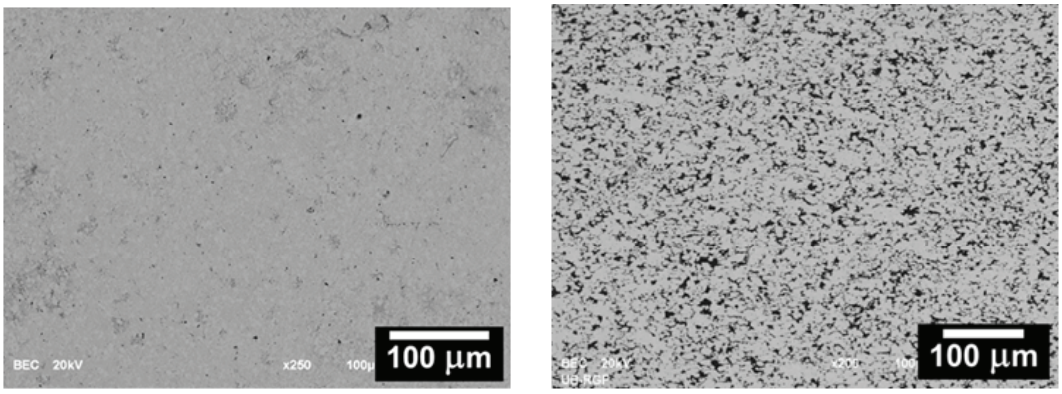
a very good mixing and high dispersion of nanoparticles in silver matrix were obtained. Given that the process is not too time demanding and that removal of starch is not too complicated, and that there are no obstacles to introduction of different metal oxide nanoparticle at the same time, this template material was adopted for further preparation of composite powders.

The SEM images presented on Fig. 2 illustrate morphology of the prepared and investigated Ag-MeO composite powders and demonstrate and confirm the advantages of the applied template method such as: good mixing, high dispersion of metal oxide nanoparticles and high uniformity. The results of SEM analysis show that the template method is very suitable for simultaneous introduction of multiple metal oxide nanoparticles into silver matrix. This is primarily because in course of the applied template process oxide particles are encapsulated within silver matrix and thus segregation is avoided. Accordingly, the prepared composite powders represent very favorable precursors for the production of electrical contact materials.

In order to illustrate the benefits of using composite powders for production of electrical contacts the samples of Ag-SnO_2 and Ag-ZnO contact materials were prepared by powder metallurgy method and for comparison corresponding microparticle materials were prepared by using conventional mixing and the same powder metallurgy route.

Slika. 3. Mikrostruktura končnega kontaktnega materiala na osnovi Ag-SnO_2 po:
a) metodi s šablono in b) s klasičnim mešanjem

Figure 3. Microstructure of the final Ag-SnO_2 contact material prepared using:
a) template method and b) conventional mixing



a) b)

Slika 4. Mikrostruktura končnega kontaktnega materiala na osnovi Ag-ZnO, izdelanega po: a) metodi s šablono in b) s klasičnim mešanjem

Figure 4. Microstructure of the final Ag-ZnO contact material prepared using: a) template method and b) conventional mixing

Da bi ponazorili prednosti uporabljenih sestavljenih prahov pri izdelavi električnih kontaktov, smo vzorce kontaktnih materialov iz Ag-SnO_2 in Ag-ZnO izdelali po postopkih metalurgije prahov in jih primerjali z materiali iz nanodelcev, ki so bili izdelani s standardnim mešanjem prahov in nato izdelani po postopkih metalurgije prahov.

Mikrostrukture izdelanih končnih kontaktov na osnovi Ag-SnO_2 in Ag-ZnO po sintranju, strojni obdelavi in dodatnem žarjenju prikazujejo SEM-metalografski posnetki poliranih obrusov na slikah 3 in 4.

Prikazane slike materiala na osnovi Ag-SnO_2 (slika 3) kažejo bistvene razlike v enakomernosti dobljenih materialov. Očitno je v vzorcih, izdelanih po šablonski metodi (slika 3a), disperzija oksida občutno večja, medtem ko je mikrostruktura vzorca, izdelanega s klasičnim mešanjem (slika 3b) manj drobna in bolj porozna, pri čemer se delci MeO nahajajo predvsem na mejah srebrovih zrn.

Praktično enak rezultat smo dobili pri materialu Ag-ZnO (slika 4). Predpostavili smo, da opažene razlike pri mikrostrukturi najverjetneje vplivajo na strukturno odvisne

Microstructures of the prepared final Ag-SnO_2 and Ag-ZnO contacts after sintering, mechanical treatment and subsequent annealing are illustrated by SEM metallographic images of polished cross-sections presented on Fig. 3 and Fig. 4, respectively.

The presented images of the Ag-SnO_2 material (Fig. 3) demonstrate substantial differences in uniformity of the obtained materials. It is evident that the sample prepared by template method (Fig. 3a) has significantly higher oxide dispersion whereas microstructure of the sample prepared by conventional mixing route (Fig. 3b) appears to be less fine and more porous with MeO particles predominantly situated on silver grain boundaries.

In case of Ag-ZnO material (Fig. 4) practically the same result was obtained. It can be assumed that the observed differences in microstructure will most certainly affect structure dependent properties of the prepared Ag-MeO electrical contacts. For that reason the comparative presentation of the physical properties of the

Razpredelnica 1. Primerjava fizikalnih lastnosti električnih kontaktnih materialov na osnovi Ag-MeO

Table 1. Comparative overview of physical properties of the prepared Ag-MeO electrical contact materials

Metoda izdelave / Preparation method	Sestava / Composition	Gostota / Density [g/cm^3]	Trdota / Hardness [HV5]	Prevodnost / Conductivity	
				[MS/m]	[%IACS]
Metoda s šablono/ metalurgija prahov / Template method / powder metallurgy	Ag-SnO2 (92:8) nano	9,86	123	38,92	67
Klasično mešanje/ metalurgija prahov / Conventional mixing / powder metallurgy	Ag-SnO2 (92:8) mikro	9,53	87	44,75	77
Metoda s šablono/ metalurgija prahov / Template method / powder metallurgy	Ag-ZnO (92:8) nano	9,55	96	35,27	61
Klasično mešanje/ metalurgija prahov / Conventional mixing / powder metallurgy	Ag-ZnO (92:8) mikro	9,49	82	38,65	67

lastnosti električnih kontaktov na osnovi Ag-MeO. Zato smo pripravili primerjavo fizikalnih lastnosti izdelanih električnih kontaktnih materialov, kar prikazuje razpredelnica 1.

Rezultati v razpredelnici 1 kažejo, da imajo materiali na osnovi Ag-SnO_2 in Ag-ZnO, izdelani po šablonski metodi, večjo gostoto in trdoto kot klasično izdelani iz mikrodelcev. Na osnovi videnih mikrostruktur smo privzeli, da smo se z uporabo šablonske metode izognili skepljenju in ločenju oksidnih delcev ter s tem dobili drobnejše in bolj enakomerne mikrostrukture, ki so omogočile večje disperzijsko utrjevanje. Takšne izboljšane lastnosti so želene za uporabo materialov glede na boljšo obrabno trdnost, večjo odpornost proti zvarjenju kontaktov in s tem daljšo življenjsko dobo. Nasprotno pa lahko višje vrednosti električne prevodnosti pripišemo manj enakomernim mikrostrukturam in prisotnosti con brez oksidov, kar omogoča boljšo povezanost srebrovih zrn. Nekoliko manjša prevodnost materialov, izdelanih s šablonsko metodo, je bila pričakovana, ker se na splošno električna prevodnost zmanjšuje z manjšanjem oksidnih delcev in povečevanjem njihove disperzije zaradi spremembe srednje proste poti prevodnih

prepared final silver-metal oxide electrical contact materials is given in Table 1.

From the presented results (Table 1) it can be seen that Ag-SnO_2 and Ag-ZnO materials prepared via template method have higher density and hardness compared to conventionally prepared microparticle ones. In line with the observed microstructures, it can be assumed that this is due to the fact that by using template method agglomeration and segregation of oxide particles were avoided finer and more uniform microstructures were obtained that enabled greater dispersion hardening. Such improved properties are desired from the application point of view in terms of better wear resistance, better anti-welding behavior and thus longer exploitation life. In contrast, higher values of electrical conductivity of the microparticle materials can be ascribed to the less uniform microstructures and presence of oxide free zones that provide better connectivity of the silver grains. Somewhat lower conductivity of the materials prepared by template method is expected since electrical conductivity generally decreases with reduction of oxide particle size and increase of their dispersion due to changes

elektronov [5,6,11]. Kljub temu so izmerjene vrednosti električne prevodnosti še v zahtevanih mejah za trgovske električne kontakte te vrste.

4 Sklepi

Preučevali smo inovativni pristop za izboljšanje disperzije kovinskega oksida v materialih za električne kontakte na osnovi srebro/kovinski oksid in o njem razpravljali. Uporabili smo prilagojeno šablonsko metodo s škrobom kot mehko organsko šablono za uvajanje nanodelcev kovinskega oksida v srebrovo osnovo in za izdelavo začetnih sestavljenih prahov Ag-MeO. Ugotovili smo, da imajo izdelani sestavljeni prahovi Ag-MeO veliko stopnjo disperzije oksidnih delcev in zelo enakomerno njihovo porazdelitev. Dodatne prednosti uporabljene sintezne metode je možnost uvajanja različnih kovinskih oksidov tudi istočasno in primernost za uvajanje nanodelcev kovinskega oksida. Dobljeni sestavljeni prahovi Ag-MeO se lahko uspešno uporabijo za izdelavo končnih električnih kontaktnih materialov z izboljšano disperzijo in s tem izboljšanimi mehanskimi lastnostmi, ker se z uporabljeno šablonsko metodo izognemo skepljenju in razmešanju oksidnih delcev. Primerjava z ustreznimi kontaktnimi materiali, izdelanimi na klasičen način, kaže pri materialih, izdelanih s šablonsko metodo s topnim škrobom, finejšo disperzijo kovinskih oksidov, večjo gostoto, večjo trdoto, manjšo poroznost in rahlo zmanjšano električno prevodnost, vendar pa so lastnosti še vedno primerljive z lastnostmi električnih kontaktnih materialov te vrste, izdelanih iz standardnih trgovskih mikrodelcev.

in mean free path of conduction electrons [5,6,11]. Nevertheless, the measured values of electrical conductivity are still in the required range for commercial electrical contacts of this type.

4 Conclusion

The innovative approach on improvement of metal oxide dispersion in cadmium-free silver/metal oxide electrical contact materials was studied and discussed. The modified template method using starch as a soft organic template was applied for introduction of metal oxide nanoparticles into silver matrix and for preparation of starting Ag-MeO composite powders. It was found that the prepared Ag-MeO composite powders exhibit high degree of dispersion and very uniform distribution of oxide particles. Additional advantages of the applied method of synthesis are possibility of introduction of different metal oxides even at the same time and suitability for introduction of metal oxide nanoparticles. The obtained Ag-MeO composite powders can be successfully used for further production of final electrical contact materials with improved dispersion and hence improved mechanical properties due to the fact that by using template method for preparation of composite powders agglomeration and segregation of oxide particles were avoided. Compared to corresponding contact materials prepared by conventional route, contact materials prepared by template method using soluble starch as a template, exhibit finer dispersion of metal oxides, higher density, higher hardness, lower porosity and slightly reduced electrical conductivity, however still comparable with those of conventional commercial microparticle electrical contact materials of this type.

Zahvala

Ta prispevek je finančno podprlo Ministrstvo za izobraževanje, znanost in tehnološki razvoj republike Srbije v okviru projektov OI 172037 in TR 34023.

Acknowledgement

This work has been supported by the Ministry of Education, Science and Technological Development of the Republic of Serbia under Projects OI 172037 and TR 34023.

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Meritve dimenzijskih sprememb med strjevanjem Al-Si zlitin

Measurement of dimensional changes of AlSi alloys during solidification

Povzetek

Namen prispevka je raziskati dimenzijske spremembe ulitka med strjevanjem. Analizirani so bili štirje vzorci. Prvi je bil iz zlitine AlSi12 brez dodatkov, drugi je bil udrobnjen s predzlitino AlTi5B1, tretji modificiran s predzlitino AlSr10 ter četrti udrobnjen in modificiran hkrati. Mikrostrukture vseh vzorcev so bile različne v smislu velikosti kristalnih zrn α_{Al} ter velikosti delcev evtektskega β_{Si}. Dilatometrijska analiza je pokazala razlike v krčenju vzorca iz zlitine AlSi12 brez dodatkov in modificiranih vzorcev, kjer je krčenje manjše. Strjevanje AlSi12 zlitine je bilo opisano z uporabo različnih metod, kot so termična analiza, dilatometrijska analiza v povezavi s termodinamskim izračunom faznih ravnotežij izvedeno s Thermo-Calc računalniškim orodjem. Metalografska analiza je bila izvedena z uporabo optične in elektronske mikroskopije (SEM). Analize mikrostrukturnih sestavin so bile izvedene z uporabo energijsko disperzijske spektrometrije (EDS).

Ključne besede: strjevanje, Al-Si zlitine, dimenzijske spremembe

Abstract

Solidification of AlSi12 alloy was described using different techniques such as thermal analysis, dilatometric analysis in accordance with thermodynamic calculation carried out by Thermo-Calc software. Metallographic investigation was made using optic and scanning electron microscopy (SEM). Phase analysis was done by energy dispersive spectroscopy (EDS). The aim of the paper is to investigate dimensional changes of a casting during solidification. Four specimens were analysed. First one was AlSi12 alloy without additions, second is grain refined with AlTi5B1 master alloy, third one modified with AlSr10 master alloy and the last one with both master alloys. Microstructures were different in terms of grain size of α_{Al} phase and size of β_{Si} particles. Dilatometric analysis has shown differences in shrinkage of basic alloy and modified alloys where shrinkage is lower.

Key words: Solidification, Al–Si alloy, Shrinkage

1 Uvod

Strjevanje zlitine AlSi12 je opisano z različnimi tehnikami, kot so termična analiza ter dilatometrijska analiza v skladu s termodinamskim izračunom faznih ravnotežij, izvedenim z računalniškim

1 Introduction

Solidification of AlSi12 alloy was described using different techniques such as thermal analysis, dilatometric analysis in accordance with thermodynamic calculation carried out by Thermo-Calc software. Metallographic

orodjem Thermo-Calc. Metalografske preiskave so bile izvedene z optičnim mikroskopom in vrstičnim elektronskim mikroskopom (SEM) opremljenim z energijsko disperzivno rentgensko spektroskopijo (EDS) za določevanje mikrostrukturnih sestavin.

Cilj članka je ugotoviti dimenzijske spremembe ulitka med strjevanjem. Analizirani so bili štirje vzorci iz zlitine AlSi12. Uporabljeno je bilo tudi udrobnjevanje in modificiranje taline. Za udrobnjevanje je bila uporabljena predzlitina AlTi5B1, za modificiranje pa predzlitina AlSr10. Prvi vzorec je bil izdelan brez predzlitin, v drugega je bilo dodana predzlitina s titanom, v tretjega predzlitina s stroncijem in v četrtega obe. Mikrostrukture so se razlikovale v smislu velikosti mikrostrukturnih sestavin, predvsem dendritnih zrn α_{Al} ter delcev evtektske faze β_{Si}. Dilatometrijska analiza je pokazala razlike v krčenju vzorcev brez dodatkov in vzorcev z dodatkom stroncija, kjer je krčenje manjše.

Strjevanje Al–Si zlitin, opisano s pomočjo termične analize, je dobro dostopno v literaturnih virih [1]. Strjevanje ulitkov je povezano z dimenzijskimi spremembami oziroma krčenjem med strjevanjem, kar lahko privede do livarskih napak, kot je krčilna poroznost. Poroznost je posledica krčenja kovin med strjevanjem. Krčenje med strjevanjem aluminija je kar 7 vol. %, pri zlitinah s silicijem pa se krčenje lahko zmanjša. V literaturi je težko zaslediti poročanja o meritvah dimenzijskih sprememb ulitkov med strjevanjem. Nekateri avtorji poročajo o meritvah dimenzijskih sprememb v ulitku iz merilne celice v povezavi z deformacijami merilne celice in nastanka zračne reže med ulitkom in formo [2].

investigation was made using optic and scanning electron microscopy (SEM). Phase analysis was done by energy dispersive spectroscopy (EDS). The aim of the paper is to investigate dimensional changes of a casting during solidification. Four specimens were analysed. First one was AlSi12 alloy without additions, second is grain refined with AlTi5B1 master alloy, third one modified with AlSr10 master alloy and the last one with both master alloys. Microstructures were different in terms of grain size of α_{Al} phase and size of β_{Si} particles. Dilatometric analysis has shown differences in shrinkage of basic alloy and modified alloys where shrinkage is lower.

Solidification of aluminium alloys is well described in literature in terms of thermal analysis [1]. Solidification of castings is connected with dimensional changes or shrinkage during solidification of the castings, which can produce defects such as shrinkage porosity. Shrinkage porosity is a consequence of shrinkage of metal during solidification, which can be up to 7 % (volume fraction) in case of aluminium and is decreased with higher amount of Si in Al–Si alloys. Not many authors have reported about dimensional change measurements of castings during solidification. Some have reported about dilatometry of casting in measuring cell and relationship between casting and mould connected with gap formation [2].

2 Experimental

In order to study such phenomena the permanent measuring cell from calcium silicate brick was produced. The casting from the cell was a 220 mm long bar with the square cross-section of 295 mm^2. At the end of the bar two quartz rods were inserted into measuring cell connected to two linear

2 Eksperimentalno delo

Z namenom študija pojava krčenja ulitka je bila izdelana merilna celica iz silikatne opeke. Ulitek iz merilne celice je imel kvadratni presek z 295 mm^2 ter dolžino 220 mm. Na koncih ulitka sta bili v livno votlino vstavljeni dve kvarčni cevčici, ki sta bili povezani na induktivna merilca pomika, ki sta merila deformacije med strjevanjem in nadaljnjim ohlajanjem. Istočasno je bila v toplotnem središču ulitka izvedena preprosta termična analiza. Štirje vzorci iz zlitine AlSi12 so bili pripravljeni. Uporabljeno je bilo tudi udrobnjevanje in modificiranje taline. Za udrobnjevanje je bila uporabljena predzlitina AlTi5B1, za modificiranje pa predzlitina AlSr10. Prvi vzorec je bil izdelan brez predzlitin in je bil označen z 12, v drugega je bilo dodana predzlitina s titanom in je bil označen z 12T, v tretjega predzlitina s stroncijem in je bil označen z 12S in v četrtega obe, označen z 12TS.

3 Rezultati in diskusija

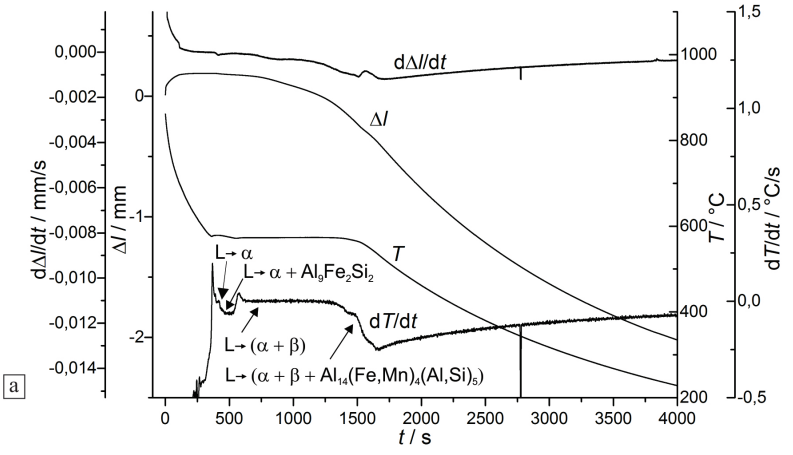
Rezultati preproste termične analize ulitka iz merilne celice so predstavljeni na sliki 1a. Ohlajevalna krivulja (*T*) vzorca 12 prikazuje potek strjevanja zlitine AlSi12. Ohlajevalna krivulja je v skladu s termodinamskim izračunom faznih ravnotežij, ki je prikazano na sliki 2, in prikazuje delež faz v odvisnosti od temperature. V prvem delu strjevanja se strjujejo dendritno izoblikovani kristali trdne raztopine α_{Al}, temu pa sledi strjevanje faze $Al_9Fe_2Si_2$. V evtektskem delu ohlajevalne krivulje se strjuje evtektik (α_{Al} + β_{Si}), strjevanje pa se zaključi s strjevanjem faze $Al_{14}(Fe,Mn)_4(Al,Si)_5$. Reakcije, ki potekajo med strjevanjem, so prikazane tudi na sliki 1a. Vse omenjene faze so bile najdene v mikrostrukturi in z EDS analizo identificirane. Mikrostrukture vzorcev 12 in

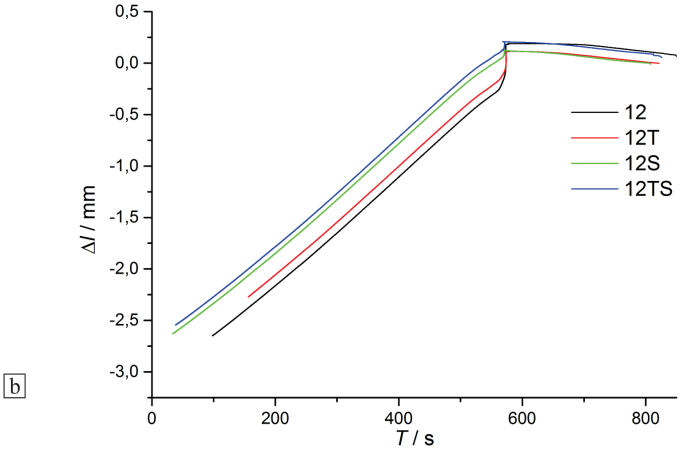
variable differential transformers (LVDT), which were measuring the dimensional changes during solidification and cooling. At the same time the thermal analysis was carried out in the thermal centre of the casting. Four samples of AlSi12 alloy were prepared. One was without additions and at others the Ti, Sr and both of additions were added simultaneously in form of master alloys AlTi5B1 and AlSr10. Samples were marked as 12, 12T for Ti addition, 12S for Sr addition, 12TS for Ti and Sr addition.

3 Results and Discussion

Results of thermal analysis are presented in Fig. 1a. Cooling curve (*T*) of sample 12 is showing the solidification of AlSi12 alloy. Cooling curve is in accordance with thermodynamic calculation of solidification presented in Fig. 2. In the first sequence of solidification the α_{Al} precipitates followed by $Al_9Fe_2Si_2$ phase precipitation. At eutectic part of the curve the solidification of (α_{Al} + β_{Si}) eutectic takes place and solidification is finished by $Al_{14}(Fe,Mn)_4(Al,Si)_5$ phase precipitation. The reactions of solidification are shown in Fig. 1a. All phases were detected in microstructure and analysed by EDS-analysis. Microstructure with marked phases is presented in Fig. 3. It can be seen that in modified samples the Al_2SrSi_2 phase is formed as a consequence of Sr addition [3, 4].

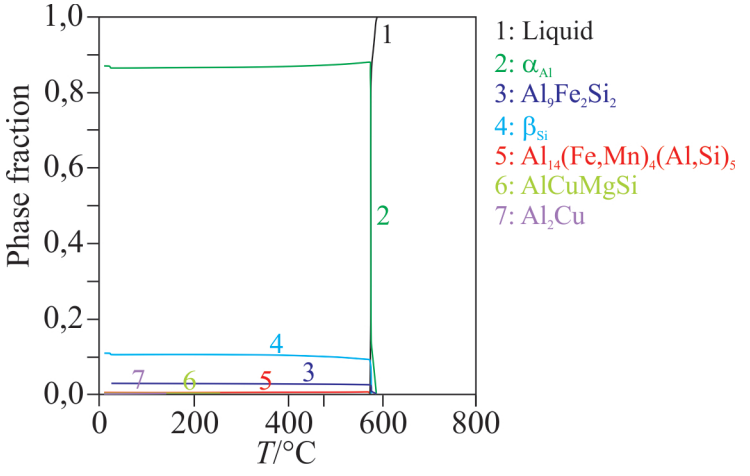
The measurement of dimensional changes of sample 12 is presented in Fig. 1b. It is seen on dilatometric curve (Δl) that casting is first expanding due to measuring cell expansion because of warming of a cell. Casting is still liquid at the time. When the solid skin is formed in the casting it starts to move the quartz rods and shrinkage of casting starts and takes place till the room temperature. Derivative dilatometric curve





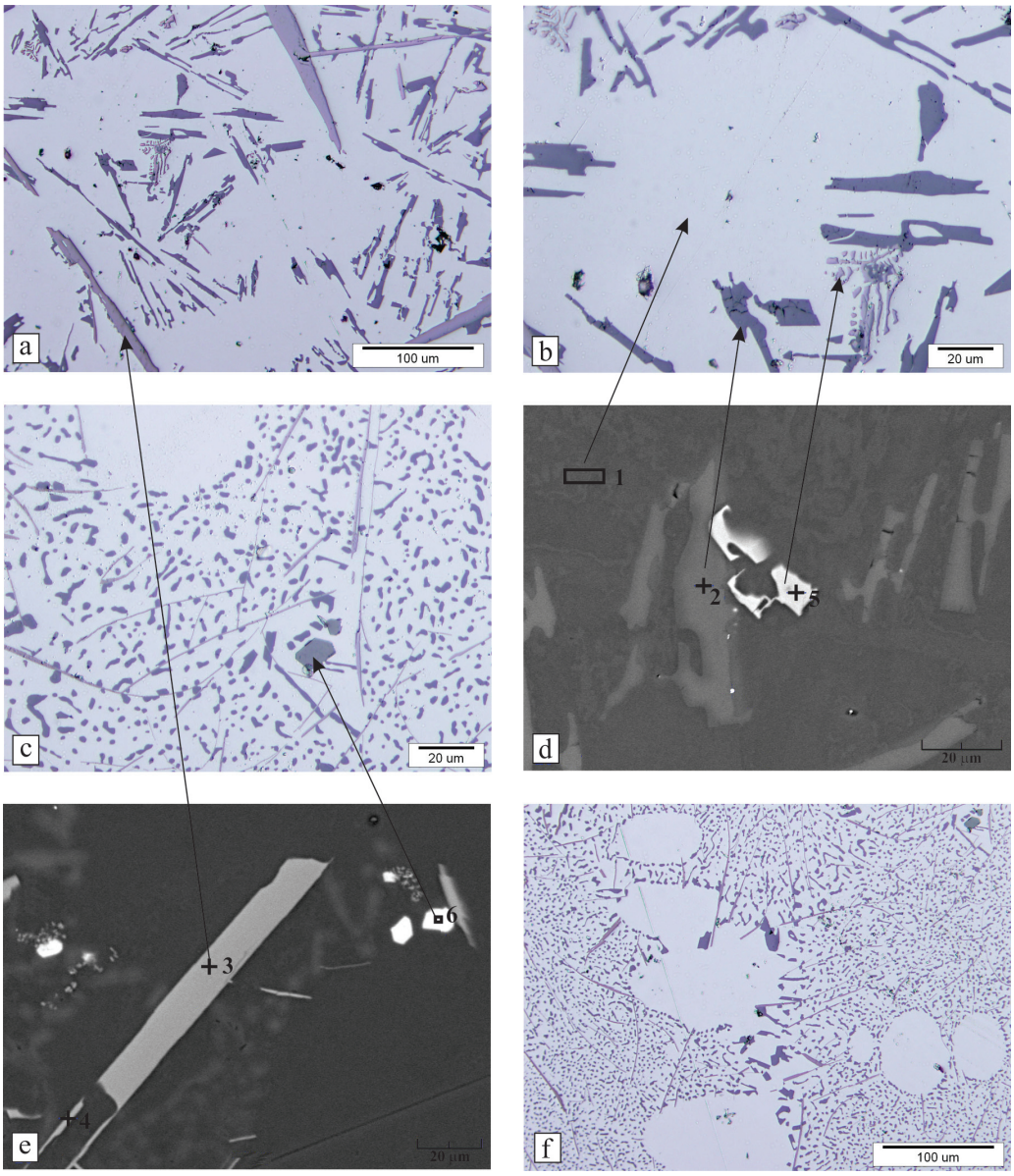
Slika 1: Rezultati meritev vzorca 12 z ohlajevalno krivuljo, dilatometrijsko krivuljo in obema diferenciranima krivuljama (a) in dimenzijske spremembe v odvisnosti od temperature za vse vzorce (b)

Figure 1: Results of measurement of sample 12 with cooling curve, dilatometric curve and both differentiated curves (a) and dimensional changes of all samples versus temperature (b)



Slika 2: Termodinamski izračun deležev faz v odvisnosti od temperature za zlitino AlSi12

Figure 2: Thermodynamic calculation of phase fractions dependent on temperature for AlSi12 alloy



Slika 3: Mikrostruktura zlitine AlSi12: svetlobnomikroskopski posnetek vzorca 12 z označenimi fazami(a, b), svetlobnomikroskopski posnetek vzorca 12S (c), SEM posnetek vzorca 12 z označenimi mesti EDS analiz (d, e) in svetlobnomikroskopski posnetek vzorca 12S (f); 1 - α_{Al}, 2 - β_{Si}, 3, 4 - $Al_9Fe_2Si_2$, 5 - $Al_{14}(Fe,Mn)_4(Al,Si)_5$, 6 – Al_2SrSi_2

Figure 3: Microstructure of AlSi12 alloy: optic micrograph of sample 12 with marked phases (a, b), optic micrograph of sample 12S (c), SEM micrographs of sample 12 with spots of EDS-analysis (d, e) and optic micrograph of 12S sample (f); 1 - α_{Al}, 2 - β_{Si}, 3, 4 - $Al_9Fe_2Si_2$, 5 - $Al_{14}(Fe,Mn)_4(Al,Si)_5$, 6 – Al_2SrSi_2

12S z označenimi fazami so prikazane na sliki 3. Opazi se, da se v vzorcu 12S pojavi faza Al_2SrSi_2 kot posledica dodatka Sr [3, 4].

Meritev dimenzijskih sprememb za vzorec 12 je prikazana na sliki 1a. Iz dilatometrijske krivulje (Δl) je videti, da se ulitek najprej širi zaradi segrevanja in širjenja merilne celice. V tem času je ulitek še tekoč. Ko se v ulitku ustvari trdna skorja, le-ta začne pomikati kvarčne cevke. Krčenje se začne in poteka do sobne temperature. Diferencirana dilatometrijska krivulja ($d\Delta l/dt$) kaže, da med ohlajanja prihaja do pojavov, kar je tudi v skladu s pojavi na diferencirani ohlajevalni krivulji (dT/dt), ki prikazuje potek strjevanja. Slika 1b prikazuje krčenje ulitka v odvisnosti od temperature. Očitno je, da z modificiranjem zlitine AlSi12 pride do razlik v krčenju ulitka. Vzorci modificirani s Sr se med strjevanjem krčijo manj kot vzorci brez dodanega Sr. Vzorec 12TS se krči za 60,9 % manj, vzorec 12S pa za 82,5 % manj kot vzorec iz zlitine AlSi12. Razlog za zmanjšano krčenje je najverjetneje spremenjena morfologija strjevanja in sposobnost napajanja ulitka. Znano pa je tudi, da dodatek St Al–Si zlitinam povečuje naplinjenost taline, kar povzroči nastanek plinske poroznosti, kompenzira krčenje med strjevanjem.

4 Zaključki

V delu je pojasnjeno strjevanje zlitine AlSi12 in določne mikrostrukturne sestavine. Ugotovljeno je, da je bilo modificiranje taline uspešno, saj so delci evtektske faze β_{Si} finejše izoblikovani. Modificiranje zlitine AlSi12 s Sr vpliva na krčenje pri strjevanju. Dilatometrijska analiza je pokazala, da je krčenje modificirane zlitine za 82,5 % manjše kot pri osnovni zlitini.

($d\Delta l/dt$) is showing that some effects on the curve are seen. These effects are in accordance with effects on derivative cooling curve (dT/dt) which presents the path of solidification. Fig. 1b is showing shrinkage of casting versus temperature and it is clearly seen that modification of AlSi12 alloy has an effect on shrinkage of casting. The samples with addition of Sr are shrinking less then castings without Sr addition. The shrinkage of 12TS sample is 60,9 % lower and 82.5 % lower in 12S sample than in basic alloy. The reason for lower shrinking must be found in the different solidification morphology of an alloy and ability of feeding of casting. At the same time the Sr addition can cause higher gas porosity in casting which can compensate shrinkage.

4 Conclusions

Solidification of AlSi12 alloy is described and microstructural constituents determined. It is seen that the modification was successful since β_{Si} particles are much finer in modified alloy. Shrinkage of an AlSi12 alloy is affected by modification. Dilatometric analysis showed that shrinkage of modified alloys is up to 82,5 % lower than in basic alloy.

Literatura / Literature

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AKTUALNO / ACTUAL

Ob 10. obletnici »zbirke ulitkov prof. dr. Milana Trbižana« odkritje njegovega doprsnega kipa

Doprsni kip
prof. dr. Milana Trbižana

Zasl. prof. dr. Milan Trbižan je umrl 20.06.2015 v svojem enainosemdesetem letu. Pred tem je načrtoval srečanje s kolegi iz Univerze in industrje na katerem je želel poročati o globalnem pomenu J. V. Valvasorja in izvesti druženje. Glede na dogodke smo se člani Oddelka za materiale in metalurgijo (OMM), Naravoslovnotehniške fakultete (NTF), Univerze v Ljubljani (UL), industijski partnerji iz Termita in Exoterma s katerimi je profesor sodeloval ter družina prof. dr. Milana Trbižana odločili, da kljub temu izvedemo srečanje z druženjem z enakim programom pri čemer smo izvedli tudi otvoritev njegovega doprsnega kipa. Le ta je delo kiparja Mihe Kača, ulitek pa je izdelal livarski mojster Roman Kamšek. Dogodek je bil v prostorih OMM, NTF, UL, dne 13.09.2016.

Program je obsegal glasbeni uvod, nagovor dekanje NTF-UL prof. dr. Eve Petre Forte Tavčar, predavanje o delu prof. Milana Trbižana in globalnem pomenu J. V. Valvasorja je pripravil predstojnik OMM prof. dr. Primož MRVAR, sledil je nagovor akademika prof. dr. Boštjana Žekša, ki je zatem odkril njegov doprsni kip. V imenu družine prof. dr. Milana Trbižana je na koncu njegova žena ga. Marija Trbižan izrekla besede zahvale udeležencem in sodelujočim. Sledilo je druženje kot si ga je zamislil prof. Milan Trbižan ob pijači in jedači, ki je bilo sproščeno in veselo, gostje pa so se kar dolgo zadržali.

Srečanja se je udeležilo približno 100 gostov tako iz Slovenije kot tudi dolgoletni livarski kolegi in prijatelji iz Nemčije, Poljske in Hrvaške.

Udeleženci slovestnega srečanja

Prof. dr. Primož Mrvar,
Predstojnik Katedre za livarstvo, NTF

AKTUALNO / ACTUAL

Livarsko srečanje »Deutscher Giessereitag 2016« v Magdeburgu

V organizaciji Društva nemških livarjev (VDG), Zveznega združenja nemške livarske industrije (BDG) in Društva raziskovalcev za livarstvo (FVG) je od 14.-15.04.2016 v Magdeburgu potekalo livarsko srečanje »Deutscher Giessereitag 2016«. Prireditve smo se udeležili kot gostje na povabilo BDG.

Odlično organiziranega srečanja se je udeležilo več kot 600 udeležencev, v pretežni meri iz Nemčije. Prvi dan srečanja so v dopoldanskem času potekali ogledi nemških livarn. Odločili smo se za ogled livarne TRIMET Aluminium Harzgerode. Gre za Al-livarno visokotlačnega in gravitacijskega kokilnega litja. Livarna ima tlačne stroje od 1400 - 4100 ton zaporne sile in 4 naprave za gravitacijsko kokilno litje, 22 obdelovalnih naprav, 20 CNC obdelovalnih strojev, 4 peči za toplotno obdelavo, naprave za pranje, luženje in konzerviranje. Letno proizvedejo 4,5 mio ulitkov. Glavni odjemalci njihovih ulitkov so na avtomobilskem in solarnem področju. Zaposlujejo 480 sodelavcev in 60 pripravnikov.

Eno uro pred uradnim pričetkom posvetovanja, pa so predsedniki organizatorjev posvetovanja skupaj z rektorjem Univerze Otto-von-Guericke Magdeburg uradno pozdravili in sprejeli skupino študentov s študija livarstva.

Po otvoritvi so sledila plenarna predavanja:

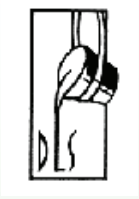
1. Digitalizacija v proizvodnji - Možnosti in izzivi, z avtorji M. Schenk in S. Leye, Fraunhofer-Institut für Fabrikbetriebe und Automatisierung IFF, Magdeburg
2. Trendi in izzivi za nemško livarsko industrijo z vidika akademskih raziskav, avtorja W. Volk, Technische Universität München in A. Bührig-Polaczek, RWTH Aachen

Potem je sledila še slovesna podelitev nagrad za inovacije v nemški livarski industriji.

Drugega dne je posvetovanje potekalo s strokovnim programom predavanj s področja tehnologije s poudarkom na kakovosti, materialih in produktih ter strokovnim programom s področja gospodarstva, kjer so predstavljena predavanja o strategiji in osnovnih pogojih za razvoj in delovanje nemškega gospodarstva ter kadrih.

Za razliko od prejšnjih posvetovanj so to pot pripravili tudi zbornik predavanj, ki je na voljo v knjižnici Društva.

Poročala: mag. Mirjam Jan-Blažić



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