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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

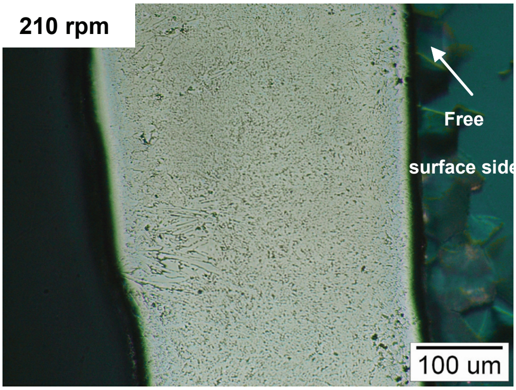
Iz rezultatov izmerjene kemijske sestave vzorcev po taljenju, ki so prikazani v tabeli 3, je razvidno, da pri kratkem času taljenja, 6 minut, ostane še določena količina železa neraztopljena. Kot posledica le-tega je delež izmerjenega železa v vzorcu 1 z ICP analizo nekoliko manjši. S podaljšanjem časa 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

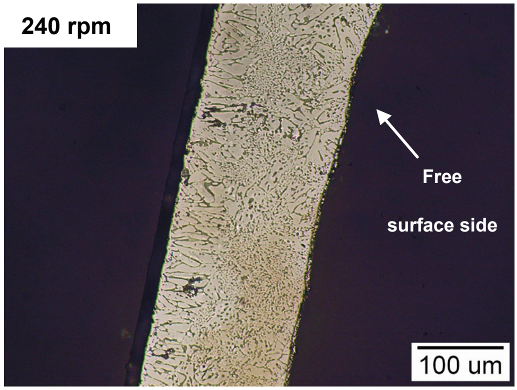
Tabela 3: Rezultati ICP-OES meritev

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



a)



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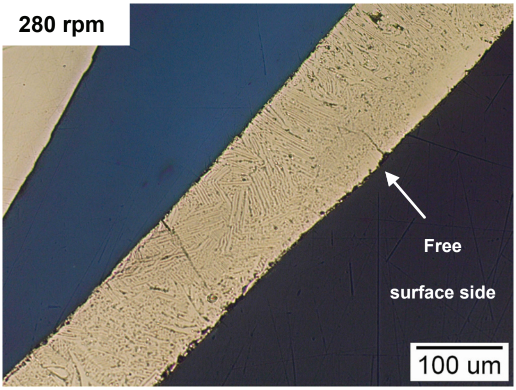


c)

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).

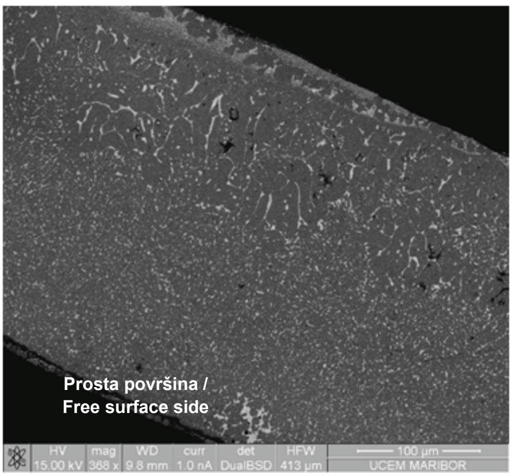
Slika 5 prikazuje magnetne lastnosti vzorcev, ki nakazujejo nedvoumno odvisnost med časom taljenja (povezano s raztopitvijo železa v talini), kot tudi pojavom nastanka α-Fe faze (povezano

surrounded by Nd-reach phase and suppresses the formation of soft magnetic α-Fe phase (fig. 3 and fig. 4).

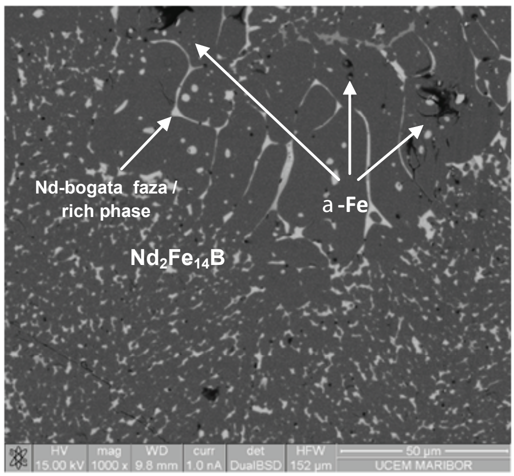
Tabela 5. Meritev nečistoč v luskah v litem stanju

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



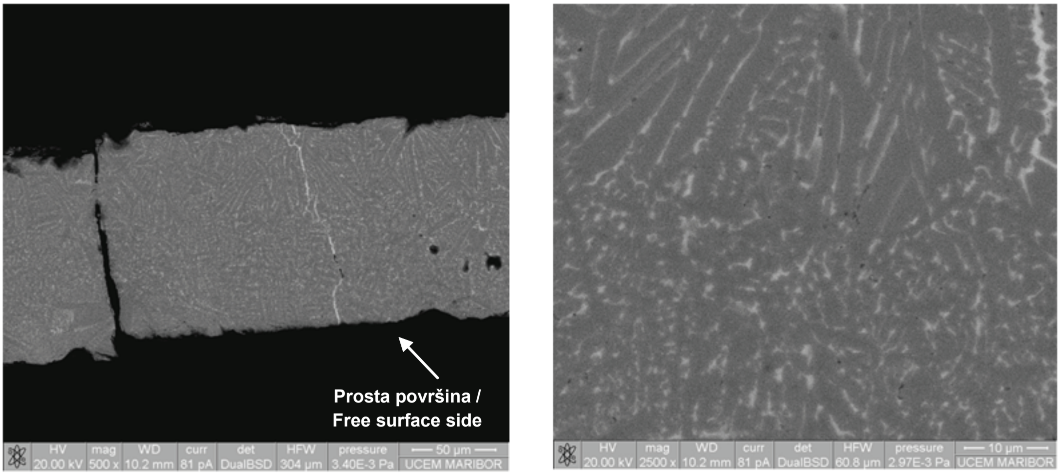
a)



b)

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

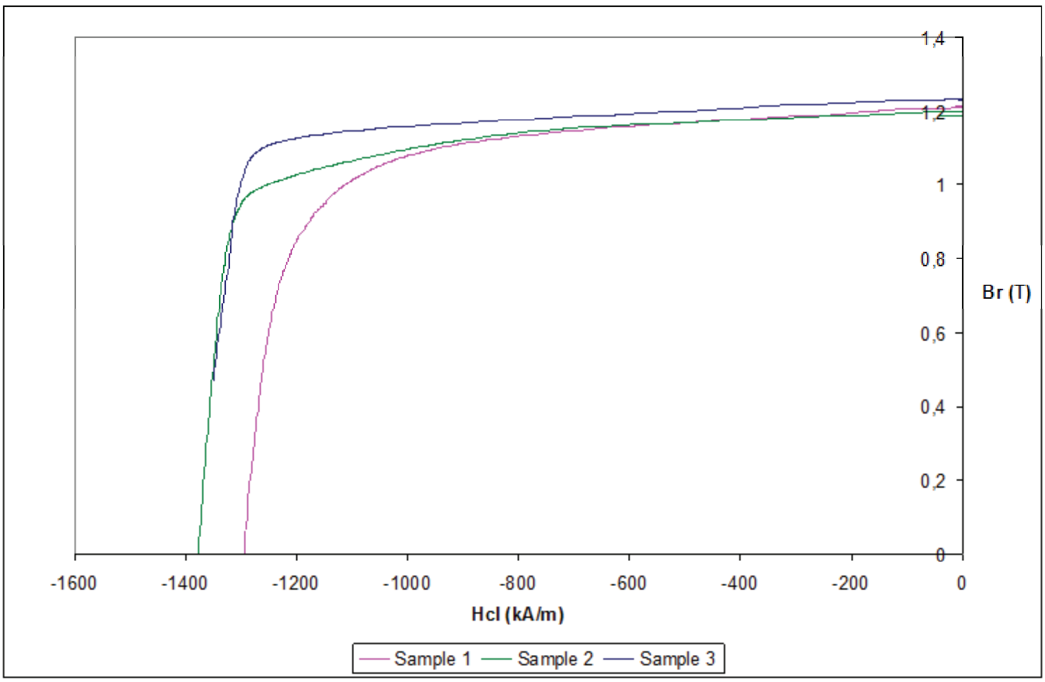
Figure 3: The morphology of the centrifugally atomized NdFeB flake at wheel speed of 210 rpm; SEM micrograph, polished cross section



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.

Literatura / References

[1] W. Kurz, D. J. Fisher, Fundaments of Solidification, Trans Tech Publications Brookfield, 1989

[2] H. Jones, Rapid Solidification of Metals and Alloys, Monograph No. 8, The institution of metallurgists, London, UK, 1982.

[3] H. H. Liebermann, Rapidly Solidified Alloys, CRC Press, 1993

[4] J. Bernardi, J. Fidler, M. Sagawa, Y. Hirose, J. Appl. Phys., 1998, 83, 11, 6396-6398.

[5] L. Q. Yu, M. Yan, J. M. Wu, W. Luo, X. G. Cui, H. G. Ying, Physica B, 2007, 393, 1-5.

[6] Z. Tian, S. Li, K. Peng, B. Gu, Y. Du, Materials Science and Engineering A, 2004, 380, 143-146

[7] D. Brown B. Ma, Z. Chen, Journal of Magnetism and Magnetic Materials, 2002, 248, 432-440.

[8] J. Xu et al., Journal of rare earths, 2006, 24, 306-309.