Avrupa Bilim ve Teknoloji Dergisi
Özel Sayı 39, S. 9-12, Temmuz 2022
© Telif hakkı EJOSAT'a aittir
Araştırma Makalesi

www.ejosat.com ISSN:2148-2683

European Journal of Science and Technology
Special Issue 39, pp. 9-12, July 2022
Copyright © 2022 EJOSAT
Research Article

Effects of Heat Treatment on Magnetic Properties of NdFeB Based Permanent Magnet Alloys

Muhammet Fatih Kilicaslan [1*], Yasin Yilmaz [2], Bekir Akgul [3]

[1*] Sivas University of Science and Technology, Faculty of Engineering, Departmant of Fundamental Sciences and Engineering, Sivas, Turkey, (ORCID: 0000-0001-8507-3900), fatihkilicaslan@sivas.edu.tr

[2] Sivas University of Science and Technology, Faculty of Aeronautics and astronautics, Departmant of Aeronautical Engineering , Sivas, Turkey, (ORCID: 0000-0003-0690-8004), yyilmaz@sivas.edu.tr

[3] Sivas University of Science and Technology, Faculty of Aeronautics and astronautics, Departmant of Aeronautical Engineering, Sivas, Turkey, (ORCID: 0000-0002-4744-9097), bekirakgul@sivas.edu.tr

(3rd International Conference on Applied Engineering and Natural Sciences ICAENS 2022, July 20-23, 2022)

(**DOI:** 10.31590/ejosat.1142054)

ATIF/REFERENCE: Kilicaslan, M. F., Yilmaz, Y. & Akgul, B. (2022). Effects of Heat Treatment on Magnetic Properties of NdFeB Based Permanent Magnet Alloys. *European Journal of Science and Technology*, (39), 9-12.

Abstract

In this study, the effect of flash annealing heat treatment applied at 680 °C for 5 minutes on the magnetic properties of NdFeB-based permanent magnet alloys produced by melt spinning method, which is one of the fast solidification methods, was investigated by using a very fast heating and cooling rate of 300 K/s. The obtained results showed that there was a remarkable improvement in the magnetic properties of the alloys with the applied heat treatment. Mainly, the hard magnetic properties were optimized by enhancing magnetic remanence from 21.64 emu/g to 55.76 emu/g, magnetic coercivity from 1184.15 Oe to 9146.30 Oe, and maximum energy product from 4.06 kJ/m^3 to 62.02 kJ/m^3 respectively.

Keywords: NdFeB alloys, Melt spinning, Heat treatment, Magnetic properties.

---

[*] Corresponding Author: fatihkilicaslan@sivas.edu.tr

1. Introduction

The development of NdFeB-based permanent magnets occurred in the early 1980s [1]. This type of permanent magnets, thanks to their superior magnetic properties [2], are becoming increasingly indispensable for societies in parallel with the developing technology day by day [3]. These magnetic properties are high remanence (B_r), high coercivity (H_c) [4], and these properties also enable them to have a high maximum energy product, $(BH)_{max}$ [1, 5]. Their superior magnetic properties; make it possible for them to be widely used in many fields such as hybrid electric vehicles [9] and energy storage systems [10], as well as in fields such as electronics [6], aviation [7], and medical [8]. In the production of NdFeB-based permanent magnets, methods such as rapid solidification (for example, melt spinning), atomization, HDDR process, mechanical alloying, severe plastic deformation, powder metallurgy are used [11]. The development of the application areas of NdFeB-based permanent magnets necessitates the optimization of their magnetic properties. For example, if they are used in hybrid engines, they should have a very high coercivity (approximately 2500 kA/m) and be able to maintain magnetic stability up to 150 °C [8]. It can be said that this situation increases the need for NdFeB magnets with high $(BH)_{max}$ and high H_c properties. Among the existing production methods, melt spinning method is a type of rapid solidification method using very high cooling rates (as high as 10^9 K/s), it provides amorphous and/or nanocrystalline structures [12,13] and thus superior magnetic properties such as high H_c and $(BH)_{max}$ unique to permanent magnets [1].

As is known, the modification of the magnetic properties of NdFeB-based magnets can be achieved with any applied heat treatment [14]. In this respect, the main purpose of our current study is to examine the effect of flash annealing heat treatment applied by rapidly heating NdFeB based alloys produced by melt spinning method to 680 °C temperature and cooling them rapidly after applying heat treatment for 5 minutes.

2. Material and Method

In order to produce NdFeB-based ribbon alloys, first of all, ingot alloys were produced under a high vacuum (approximately 10^{-6} mbar) and in the argon atmosphere (approximately 550 mbar partial pressure) with the help of a vacuum induction melting (VIM) furnace. Nominal chemical composition of the alloys was adjusted to 36% Nd, 62% Fe and 2% B by weight. For each ingot alloy, the melting process was repeated 5 times to ensure maximum homogeneity. The weight of each ingot alloy produced were 260 g. For producing NdFeB based alloys in the ribbon form in melt spinning process, by means of a nozzle with a cross section of 1x8 mm^2, the liquid metal re-melted under vacuum was ejected onto a copper wheel rotating at a speed of approximately 35 m/s. While the argon partial pressure used to eject the liquid metal onto the copper whell was 250 mbar, the distance between the whell and the nozzle was adjusted to 0.5 mm, and thus NdFeB alloys in the form of ribbon with a homogeneous thickness of 8 µm and a width of 25 mm were produced. X-ray diffraction (XRD) analysis (with Cu-Kα radiation) was performed to detect the crystalline and amorphous regions in the alloys. Vibrating sample magnetometer (VSM) analysis was performed to determine the magnetic properties of each alloy. In order to examine the effect of heat treatment on crystallization and magnetic properties, flash annealing heat treatment was applied at very rapid heating and cooling rates of 300 K/s for 5 minutes, at 680 °C, respectively. The description of each alloy produced within the scope of this study is summarized in Table 1.

Table 1. Description of melt spun NdFeB based alloys that are not heat treated and heat treated at 680 °C for 5 minutes

Melt spun alloys	Description
N0	Melt spun, non-heat treated
N680-5	Melt spun, heat treated at 680 °C for 5 minutes

3. Results and Discussion

In Fig. 1, the XRD patterns of the NdFeB based alloys produced by the melt spinning method are shown in the non-heat treated and heat treated state at 680 °C for 5 minutes. Accordingly, it can be clearly seen that each melt spun NdFeB alloy exhibits an amorphous structure with traces of crystalline structure. The rapid cooling used in the melt spinning method is the main reason for obtaining the amorphous structure [15]. The XRD pattern of the non-heat treated alloy is seen with the crystalline peak of the hard magnetic $Nd_2Fe_{14}B$ phase at an angle of about 2θ=30° and the peak of the soft magnetic α-Fe phase at an angle of 2θ=45°. With the applied flash annealing heat treatment, an increase in the intensity of the peaks belonging to the hard and soft magnetic phases was observed. In other words, a transformation from amorphous structure to crystal structure has taken place here [16]. A certain temperature and time are required for the atoms in the material to pass from the amorphous structure to the crystalline order [17]. At high temperatures and/or long periods of time, atomic mobility increases, so crystallization occurs more [18].

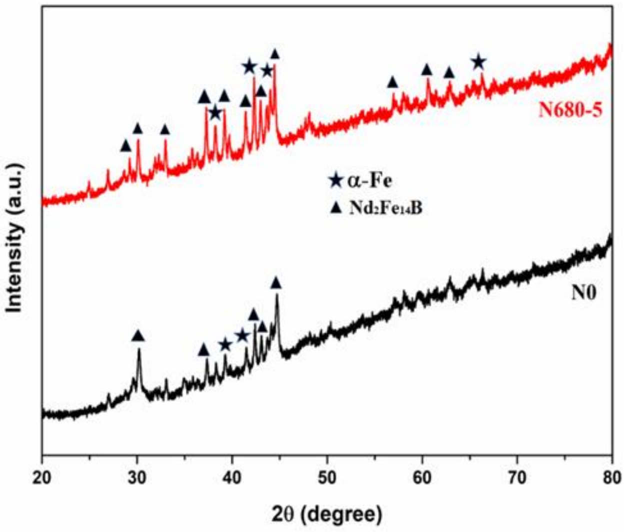

Fig 1. XRD patterns of melt spun NdFeB based alloys in the non-heat treated (N0) and heat treated condition (N680-5) at 680 °C for 5 min.

Fig. 2 shows the results of differential scanning calorimetry (DSC) analyzes applied to determine the Curie temperatures of melt spun NdFeB based alloys at non-heat treated and heat treated conditions at 680 °C for 5 min. According to the obtained DSC analysis results, the Curie temperatures (T_c) of melt spun alloys are 302 °C for the non-heat treated alloy and 310 °C for heat treated at 680 °C for 5 min. respectively. These results are consistent with those of Haiyang et al.'s study. [19] and a slight increase in T_c temperature occurred with the applied flash annealing heat treatment. Whereas, recrystallization temperatures are 380 °C for the non-heat treated alloy and 388 °C for the heat treated alloy at 680 °C for 5 min.

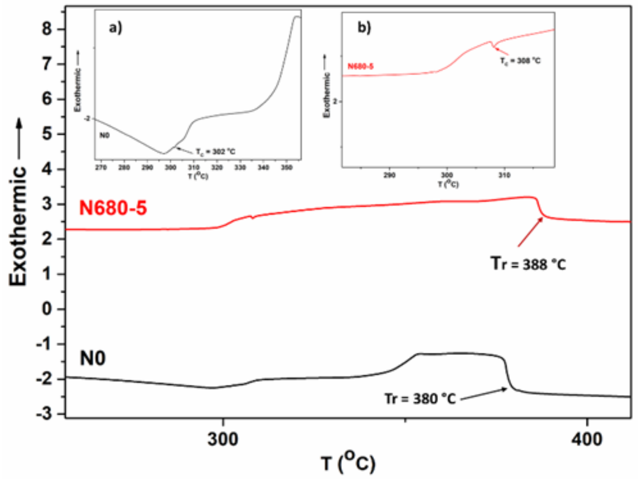

Fig 2. DSC curves of melt spun NdFeB based alloys in the non-heat treateded (N0) and heat treated (N680-5) condition at 680 °C for 5 minutes.

In Fig. 3 and 4, the demagnetization curves and second quadrant of the demagnetization curves of melt-spun NdFeB based alloys and are shown in the non-heat treated and heat treated condition at 680 for 5 minutes, respectively. As can be seen, in the non-heat treated alloy, magnetic coercivity (H_c), 1184.15 Oe, maximum energy product (($(BH)_{max}$), 4.06 kJ/m^3, magnetic remanance (B_r), 21.64 emu/g and saturation magnetization (B_s), 93.50 emu/g. With the heat treatment applied for 680 °C and 5 minutes, an increase was observed in H_c, $(BH)_{max}$, B_r and B_s properties, and an improvement was observed towards 9146.30 Oe, 62.02 kJ/m^3, 55.76 emu/g and 104.65 emu/g, respectively. This increase in magnetic properties can be explained by the emergence of more nanocrystalline α-Fe soft magnetic phase and $Nd_2Fe_{14}B$ hard magnetic phases with the effect of applied flash annealing heat treatment and the dominance of hard magnetic $Nd_2Fe_{14}B$ phase [18]. According to the exchange coupling mechanism that occurs between the hard and soft magnetic phases, the magnetic moment of the soft magnetic phase rotates along the boundaries of the hard magnetic phase, which provides an improvement in the permanent magnetic properties [20]. In Table 2, the magnetic properties of melt spun NdFeB based alloys, at non-heat treated and heat treated condition at 680 °C for 5 minutes, are summarized.

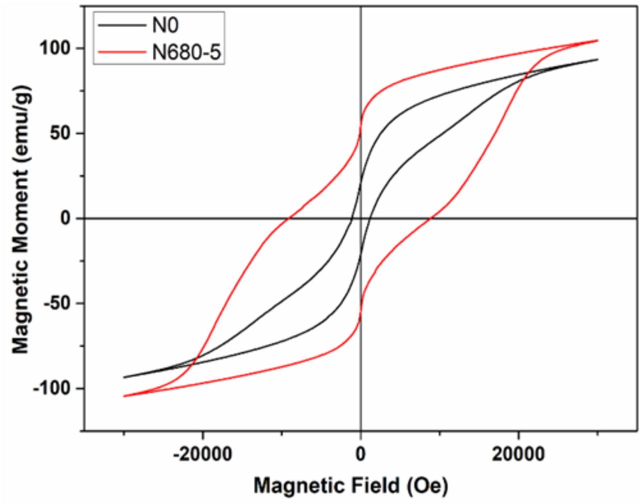

Fig 3. VSM curves of melt spun NdFeB based alloys in non-heat treated (N0) and heat treated (N680-5) condition at 680 °C for 5 minutes.

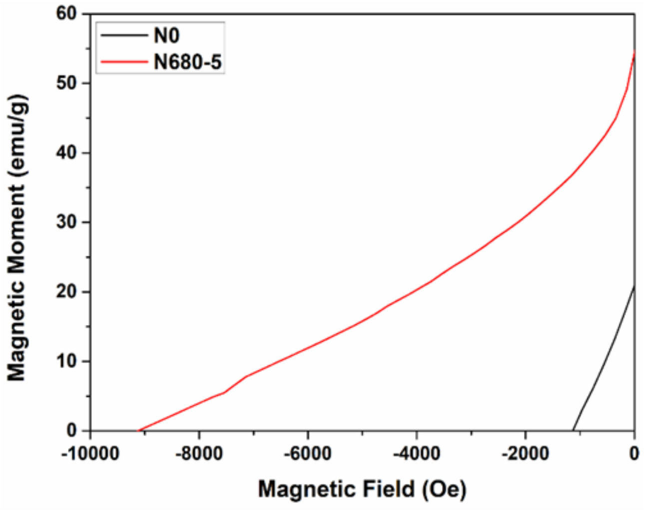

Fig 4. Second quadrant parts of VSM curves of melt spun NdFeB based alloys in the non-heat treated (N0) and heat treated (N680-5) condition at 680 °C for 5 minutes.

Table 1. Summary of the magnetic properties of melt spun NdFeB based alloys in the non-heat treated (N0) and heat treated (N680-5) condition at 680 °C for 5 min.

Melt spun alloy	**B_s (emu/g)**	**B_r (emu/g)**	**H_c (Oe)**	**$(BH)_{max}$ (kJ/m^3)**
N0	93.50	21.64	1184.15	4.06
N680-5	104.65	55.76	9146.30	62.02

4. Conclusions and Recommendations

Within the scope of our study, the effect of flash annealing heat treatment applied at 680 °C and for 5 minutes using very rapid heating and cooling rates on the magnetic properties of NdFeB based permanent magnet alloys produced using the melt spinning method by rapid solidification is discussed. The XRD pattern of the non-heat treated alloy reveals the coexistence of amorphous and nanocrystalline α-Fe soft magnetic and $Nd_2Fe_{14}B$ hard magnetic phases. Optimization was achieved in the hard magnetic properties, with the emergence of more soft and hard magnetic phases with the crystallization caused by the effect of the applied heat treatment, and the improvement in magnetic properties such as H_c, B_r and $(BH)_{max}$, especially with the dominance of the hard magnetic $Nd_2Fe_{14}B$ phase.

5. Acknowledge

This study was financed by the Scientific Research Projects Coordinator of Sivas University of Science and Technology with Project Number of 2020-TDP-Mühe-0001.

References

[1] S. J. Z. Haofeng, L. Hongmei, Influence of Heat Treatment on Microstructures and Properties of Nd8Fe78B6Co4 Alloy, J. Rare Earths. 2 (2006) 4–6.

[2] X. Lin, Y. Luo, H. Peng, Y. Yang, Y. Dou, Z. Wang, K. Xu, S. Diao, D. Yu, Phase structure evolution and magnetic properties of La / Ce doped melt- spun NdFeB alloys, J. Magn. Magn. Mater. 490 (2019) 165454. https://doi.org/10.1016/j.jmmm.2019.165454.

[3] H. Jin, P. Afiuny, T. Mcintyre, Y. Yih, J.W. Sutherland, Comparative Life Cycle Assessment of NdFeB Magnets: Virgin Production versus Magnet-to-Magnet Recycling, Procedia CIRP. 48 (2016) 45–50. https://doi.org/10.1016/j.procir.2016.03.013.

[4] E.E. Kaya, O. Kaya, S. Stopic, S. Gürmen, B. Friedrich, NdFeB Magnets Recycling Process: An Alternative Method to Produce Mixed Rare Earth Oxide from Scrap NdFeB Magnets, Metals (Basel). 716 (2021) 1–13. https://doi.org/https://doi.org/10.3390/met11050716.

[5] H.S.-A. K. Hono, Strategy for high-coercivity Nd – Fe – B magnets, Scr. Mater. 67 (2012) 530–535. https://doi.org/10.1016/j.scriptamat.2012.06.038.

[6] T. Elwert, D. Goldmann, F. Roemer, S. Schwarz, Recycling of NdFeB Magnets from Electric Drive Motors of (Hybrid) Electric Vehicles, J. Sustain. Metall. 3 (2017) 108–121. https://doi.org/10.1007/s40831-016-0085-1.

[7] D.P. M. M. Codescu, W. Kappel, M. Dumitrache, Corrosion tests on alloys and permanent magnets based on NdFeB, used in aerospace industry, J. Optoelectron. Adv. Mater. 10 (2008) 790–793.

[8] M. Lv, T. Kong, W. Zhang, M. Zhu, H. Jin, W. Li, Progress on modification of microstructures and magnetic properties of Nd- Fe-B magnets by the grain boundary diffusion engineering, J. Magn. Magn. Mater. 517 (2021). https://doi.org/10.1016/j.jmmm.2020.167278.

[9] K.B. Y. Yang, A. Walton, R. Sheridan, K. Güth, R. GauB, O. Gutfleisch, M. Buchert, B. Steenari, T. V. Gerven, P. T. Jones, REE Recovery from End-of-Life NdFeB Permanent Magnet Scrap: A Critical Review, J. Sustain. Met. (2017) 122–149. https://doi.org/10.1007/s40831-016-0090-4.

[10] B. Michalski, M. Szymanski, K. Gola, J. Zygmuntowicz, Experimental evidence for the suitability of the hydrogen decomposition process for the recycling of Nd-Fe-B sintered magnets, J. Magn. Magn. Mater. 548 (2022) 1–8. https://doi.org/10.1016/j.jmmm.2021.168979.

[11] D. Brown, B. Ma, Z. Chen, Developments in the processing and properties of NdFeb-type permanent magnets, J. Magn. Magn. Mater. 248 (2002) 432–440.

[12] M.F. Kılıçaslan, Y. Yılmaz, B. Akgül, H. Karataş, C.D. Vurdu, Effect of Fe-Ni Substitution in FeNiSiB Soft Magnetic Alloys Produced by Melt Spinning, Adv. Mater. Sci. 21 (2021) 79–89. https://doi.org/10.2478/adms-2021-0026.

[13] H.C. Hua, G.Y. Wang, C.H. Zheng, G.X. Huang, Q.Z. Xu, L.H. Wu, S.Y. Shi, Microstructure of melt-spun NdFeB magnet, Mater. Lett. 7 (1988) 65–67. https://doi.org/10.1016/0167-577X(88)90085-7.

[14] W.F. Li, T. Ohkubo, K. Hono, M. Sagawa, The origin of coercivity decrease in fine grained Nd-Fe-B sintered magnets, J. Magn. Magn. Mater. 321 (2009) 1100–1105. https://doi.org/10.1016/j.jmmm.2008.10.032.

[15] Z.Y. Zhang, L.Z. Zhao, X.C. Zhong, D.L. Jiao, Z.W. Liu, Phase precipitation behavior of melt-spun ternary Ce2Fe14B alloy during rapid quenching and heat treatment, J. Magn. Magn. Mater. 441 (2017) 429–435. https://doi.org/10.1016/j.jmmm.2017.06.028.

[16] M. Zhang, W. Zhang, F. Chen, Y. Guo, F. Li, W. Liu, Effect of Heat Treatment on Microstructure and Magnetic Properties of Ce-Doped NdFeB Ribbons, J. Supercond. Nov. Magn. (2018) 2811–2816. https://doi.org/10.1007/s10948-017-4553-z.

[17] W.D. Callister, D.G. Rethwisch, Materials Science and Engineering: An Introduction, 10th ed., Wiley, 2018.

[18] S. Caglar, M.F. Kilicaslan, A. Atasoy, H. Tiryaki, M. Erkovan, S. jik Hong, Effect of heat treatment on magnetic properties of nanocomposite Nd-lean Nd7Fe73B20 ribbons, J. Mater. Sci. Mater. Electron. 32 (2021) 2338–2345. https://doi.org/10.1007/s10854-020-04999-9.

[19] L.W. N. Haiyang, Z. Liqun, L. Huicong, Effect of Cathodic Hydrogen Evolution on the Coercivity and Thermal Stability of Sintered NdFeB Magnets, Xiyou Jinshu Cailiao Yu Gongcheng/Rare Met. Mater. Eng. 46 (2017) 3658–3662. https://doi.org/10.1016/s1875-5372(18)30053-5.

[20] W. Yupu, Y.O.U. Caiyin, W. Junwei, T. Na, L.U. Zhengxin, Coercivity enhancement of Nd 2 Fe 14 B / Į-Fe nanocomposite magnets through neodymium diffusion under annealing, J. Rare Earths. 30 (2012) 757–760. https://doi.org/10.1016/S1002-0721(12)60125-2.