

RESEARCH PAPER

THE INFLUENCE OF ANNEALING TEMPERATURE ON THE MAGNETIC PROPERTIES OF CRYO-ROLLED NON-ORIENTED ELECTRICAL STEEL

Tibor Kvačkaj^{1}, Ivo Demjan², Peter Bella³, Róbert Kočíško¹, Patrik Petroušek¹, Alica Fedoriková⁴, Jana Bidulská¹, Miloslav Lupták¹, Petr Jandačka⁵, Marcela Lascsáková⁶*

¹ Technical University of Kosice, Faculty of Metallurgy, Materials and Recycling, Dpt. of Plastic Deformations and Process Simulations, Letna 9, 042 00, Slovakia

² Technical University of Kosice, Faculty of Civil Engineering, Letna 9, 042 00, Slovakia

³ ŽP VVC s.r.o., Kolkáreň 35, 976 81, Podbrezová, Slovakia

⁴ Reseach Centre Rež, Hlavní 130, Husinec Rež

⁵ Department of Game Management and Wildlife Biology, Faculty of Forestry and Wood Sciences, Czech University of Life Sciences, Kamýcká 129, 165 21 Prague 6, Czech Republic

⁶ Technical University of Kosice, Faculty of Mechanical Engineering, Letna 9, 042 00, Slovakia

*Corresponding author: tibor.kvackaj@tuke.sk, tel.: +421556024258, Technical University of Kosice, Faculty of Metallurgy, Materials and Recycling, Dpt. of Plastic Deformations and Process Simulations, 040 01, Letna 9, Kosice, Slovakia

Received: 13.04.2022

Accepted: 18.05.2022

ABSTRACT

The paper is focused on a comparison of the magnetic properties of the non-oriented isotropic electrical steel containing 3.5% Si. The material was processed by conventional rolling at ambient temperature and progressive rolling before which the samples were undercooling in liquid nitrogen. Deformations of the samples in both thermal conditions were in the interval $\varepsilon \in <5;35>[\%]$. Subsequently, the samples were heat treated at temperatures $T \in <900;1100> [^{\circ}\text{C}]$. Measuring of the magnetic properties was carried out in an alternating magnetic field at frequencies $f = 50; 100; 150$ Hz. At a frequency of 50 Hz were achieved smallest magnetic losses and therefore further measurements were made at a given frequency. Followed measurements of the magnetic induction were conducted at different intensities of the magnetic field. EBSD analyses were performed to obtain the IPF maps on which the resulting structure was evaluated after processing of the material. The specific magnetic losses were compared to different processing methods. The best magnetic properties defined as by minimal values of core losses were reached after samples rolled at cryogenic temperature followed by subsequently annealed. Also, higher proportion of cubic texture was archived after rolling at cryogenic temperature with compared to samples processed at ambient temperature.

Keywords: magnetic properties, deformation, cryo-rolling, non-oriented electrical steel

INTRODUCTION

Improvements in the performance of electrical devices and creating new high-quality applications have led to the optimization of existing magnetic materials. Electrical steels belong to a group of soft magnetic materials and are known for their excellent magnetic properties such as high electrical resistance and low magnetic losses [1-3]. Non oriented isotropic electrical steels (NGOES) are characterized by approximately the same magnetic properties in all directions of the plane of the sheet. Therefore, it is optimal if directional isotropic magnetic properties of non-oriented electrical steels are secured by cube crystallographic orientation is $\{100\} <001>$ with Goss texture is $\{110\} <001>$ [4, 5].

Improvement of electrical steels magnetic properties involves optimizing physical and metallurgical characteristics such as grain size, crystallographic texture, construction of grain boundaries and secondary inclusions. Movement of grain boundaries interacts with the precipitation presents basic physical happening in the evolution of the microstructure and texture during the thermo-mechanical processes of production of electrical steel [6]. The effect of plastic deformation on the properties of non-oriented electrical steels has a significant

difference under the cryo-rolling and ambient conditions. The progressive methods for this material, such as rolling under the cryogenic conditions, are currently not explored [7-9].

Required magnetic properties such as low total magnetic losses, high magnetic induction, and low coercive force are dependent on microstructure and texture evolution during thermomechanical processing. Therefore, it is important to control the rolling process of non-oriented electrical steels and improve its magnetic properties which are strongly depending on structural characteristics [10-17].

In cold-rolled sheets, recovery, recrystallization, grain growth and texture formation take place during the final annealing process. The study reported by Cunha et al. [18] demonstrated that recovery and recrystallization lead to remarkable improvements in magnetic properties, with grain growth resulting in a decrease in iron loss and a decreased permeability, due to the strengthening of the γ -fiber. The increase in grain size during grain growth is often associated with changes in texture. Two fairly conflicting results concerning the effect of grain growth on texture development were published [19]. Hutchinson et al. [20] reported that the $\{111\}$ texture component during grain growth is strengthened. In contrast, Jong-Tae

et al. [21] found that Goss texture and γ -fiber components are weakened during grain growth.

Based on previous studies, few researches had reported the microstructure and texture of thin-gauge non-oriented silicon steel during recrystallization annealing [22-26]. A more in-depth analysis of the influence of recrystallization annealing temperatures on microstructure, texture, and magnetic properties is subsequently required.

The main aim of the experiment is to describe the effect of plastic deformation carried out under different temperature conditions to the resulting magnetic properties of selected material.

MATERIAL AND METHODS

For experiment commercially produced non-oriented electrical steel with input thickness 2 mm was used. The chemical composition measured by spectrometry is as follows: 3.5%Si (in wt.%), 0.034 %C, 0.25%Mn, 0.015%P, 0.004%S, 0.021%Cu, 0.433% Al.

Before laboratory rolling, the homogenizing annealing at 900°C was performed. On samples surface, the layer of ceramic lubricant was applied to oxidation prevent. Samples were rolling under ambient and cryogenic conditions on the rolling mill DUO 210. To achieve the cryogenic rolling temperature were the samples in the liquid nitrogen for 30 minutes immersed. After cryogenic rolling, measured temperature of samples was -35°C. The total strain was $\varepsilon = <7;35> [\%]$. Next step was annealing in air ambience for 40 minutes at three different temperatures: $T \in <900; 1000; 1100^\circ\text{C}>$.

The crystallographic orientation and components texture were obtained by EBSD (Electron backscatter diffraction) methods in cross-section of rolled sheets. The obtained EBSD grain textures were determined by IPF maps.

Basic dimensions of the samples for measurement of magnetic properties were $l = 280$ mm, $b = 30$ mm and $h = 1.3$ mm. The samples were taken from rolled sheet in the direction of rolling. Measuring was carried out on the device SST, which uses a system Remacomp C200. The measurements were made at frequencies of $f = 50$ Hz, $B_{\max} = 1.5$ T.

RESULTS AND DISCUSSION

In Fig.1. a significant difference in microstructure are visible. The rolling direction is represented by an arrow. EBSD analysis shows the heterogeneous distribution of observed three planes, especially for ambient rolled sample. The central part of the sample rolled at ambient temperature consists from elongated grains in the rolling direction with a predominantly cubic crystallographic orientation $\{100\}$ and more dominant orientation $\{111\}$ in surface parts. After cryorolling the presence of deformation twins is visible and grains with Goss texture almost disappeared. It is caused by retarded dynamic recovery of material under cryogenic conditions. Elongation of the grains is better visible after rolling at ambient temperature. Reduction of grains length in case of cryogenic rolling is compensated by the formation of deformation twins in grains with orientation $\{111\}$. Deformation twins also contribute the growth of the grains with a preferred orientation in the plane $\{100\}$ during the recrystallization process. Samples rolled under cryogenic conditions after annealing showed a higher percentage of the cubic components, what is advantageous regarding the magnetic properties. Based on EBSD analysis it is clear that the sample rolled at ambient temperature has better magnetic properties because of the following reasons: the area of the hysteresis loop is smaller so the sample exhibits lower losses in alternating current magnetization and lower coercivity. Another reason is the high proportion of deformation twins

under cryogenic conditions what prevent passage of magnetic domains and other lattice defects in the material caused by cryorolling, also. On the other hand, recrystallization annealing of non-oriented electrical steel has positive impact to the final magnetic properties. Effect of annealing temperature on the magnetic properties of cryorolled non-oriented electrical steel is presented in the Fig.2. The total magnetic losses are determined by the same value of magnetic induction $B = 1.5$ T. Magnetic polarization was measured with frequency $f = 50$ Hz in term of industry requirements and different field magnetic polarization. From the Fig. 2 and Fig.3 it is clear that the lowest value of magnetic losses was achieved by annealing at 900 °C, then at $T = 1000$ °C.

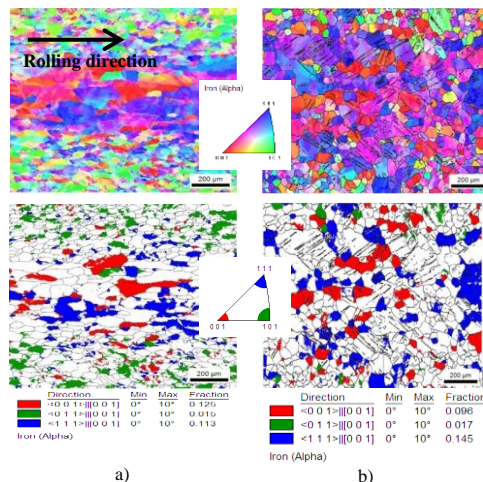


Fig. 1 Maps of inverse pole figures for samples of rolled samples without annealing with total strain $\varepsilon = 35\%$: a) ambient b) cryogenic conditions.

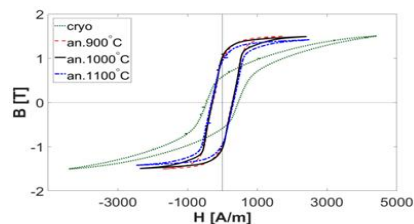


Fig. 2 The comparison of the hysteresis loop after cryo-rolling with deformation $\varepsilon=7\%$ and after annealing at 900, 1000 and 1100°C/40min

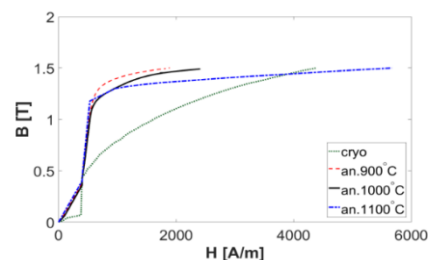


Fig. 3 The comparison of the permeabilities after cryo-rolling with deformation $\varepsilon=7\%$ and after annealing at 900, 1000 and 1100°C

The comparison of permeability between cryorolled samples annealed at various temperatures is shown in Fig. 3.

The dependence of the core losses on grain diameter after annealing at 900°C is shown in Fig. 4, from which is resulting that the minimal level of core losses was achieved after cryorolling with diameter of ferrite grain 106 µm. Comparison measured values after materials processing at ambient and cryorolling is listed at Table 1.

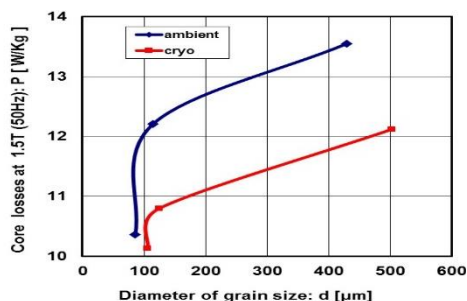


Fig. 4 The dependence of core losses on grain diameter after annealing at 900°C

A comparison of the measured values after material rolling at ambient and cryogenic temperatures is listed at Table 1. From measured data is resulting that the cryorolled samples achieved lower core losses under the same annealing temperature compared to samples rolled at room temperature.

Table 1 Magnetic losses

Rolling conditions	Annealing T [°C]	Specific magnetic losses at B=1.5T P [W/kg]	Magnetic polarization at f=50Hz		
			H=2500 A/m [T]	H=5.10 ³ A/m [T]	H=10 ⁴ A/m [T]
Ambient	900	10.36	1.56	1.64	1.75
Cryo	900	10.13	1.54	1.62	1.72
Ambient	1000	10.82	1.55	1.63	1.73
Cryo	1000	10.55	1.56	1.64	1.74
Ambient	1100	13.5	1.42	1.5	1.61
Cryo	1100	12.2	1.49	1.58	1.69

Cryorolling is considered to be the prominent processing method to develop high strength materials. Even though considerable work is available on mechanical properties of cryorolled materials, mainly light alloys, titanium, and steels [27-38]. Cryorolling can improve strength and ductility therefore opening up further opportunities to applications in even more strength critical applications. However, some critical factors related to the process phenomena are still needs further studies on improvement in its material properties.

CONCLUSIONS

Based on the results obtained from the rolling of NGOES material in ambient and cryogenic temperature conditions, the following conclusions can be drawn:

- samples rolled at cryogenic temperature and subsequently annealed showed a higher proportion of cubic texture than samples processed at ambient temperature

- the best magnetic properties defined as core losses of the observed material were obtained after cryorolling, followed by annealing process at temperature 900°C with holding time 40min with value P=10.13 W/kg
- the value P=10.13 W/kg is corresponding to the ferrite grain diameter of d=106 µm with cubic texture.

REFERENCES

1. R. Bidulský, M.A. Grande, L. Ferraris, P. Ferraris, J. Bidulská: Acta Physica Polonica A, 118, 2010, 802-803. <https://doi.org/10.12693/APhysPolA.118.802>.
2. R. Bidulský, J. Bidulská, M.A. Grande, L. Ferraris: Acta Metallurgica Slovaca, 20, 2014, 271-278. <https://doi.org/10.12776/ams.v20i3.351>.
3. T. Kvačkaj, J. Bidulská, R. Bidulský, A. Kováčová, R. Kočiško, P. Bella, M. Lupták, J. Bacsó: Acta Physica Polonica A, 126, 2014, 184-185. <https://doi.org/10.12693/APhysPolA.126.184>.
4. J. Liu, Y. Sha, K. Hu, F. Zhang, L. Zuo: Metallurgical and Materials Transactions A, 45, 2014, 134-138. <https://doi.org/10.1007/s11661-013-1917-2>.
5. I. Gutierrez-Urrutia, A. Böttcher, L. Lahn, D. Raabe, J.: Materials Science, 49, 2014, 269-278. <https://doi.org/10.1007/s10853-013-7701-2>.
6. T. Kvačkaj, J. Bacsó, J. Bidulská, M. Lupták, I. Pokorný, M. Kvačkaj, M. Vlado: Acta Metallurgica Slovaca, 16, 2010, 268-276.
7. X. Lu et al.: Journal of Magnetism and Magnetic Materials, 404, 2016, 230-237. <https://doi.org/10.1016/j.jmmm.2015.12.043>.
8. D. Bublíková, H. Jirková, K. Rubešová, M. Pekovič, J. Volkmannová, M. Graf: Acta Metallurgica Slovaca, 25, 2019, 93-100. <https://doi.org/10.12776/ams.v25i2.1266>.
9. P. Prislupčák, T. Kvačkaj, J. Bidulská, P. Záhumenský, V. Homolová, V. Zimovčák: Acta Metallurgica Slovaca, 27, 2021, 207-209. <https://doi.org/10.36547/ams.27.4.1306>.
10. J. Sidor, L. Kestens: Scripta Materialia, 68, 2013, 273-276. <https://doi.org/10.1016/j.scriptamat.2012.10.039>.
11. G. Anand, K. Barai, R. Madhavan, P.P. Chattopadhyay: Materials Science and Engineering: A, 638, 2015, 114-120. <https://doi.org/10.1016/j.msea.2015.04.034>.
12. T. Konkova, S. Mironov, A. Korznikov, S.L. Semiatin: Materials Science and Engineering: A, 528, 2011, 7432-7443. <https://doi.org/10.1016/j.msea.2011.06.047>.
13. T. Kvačkaj et al: Acta Physica Polonica: A, 131, 2017, 1105-1107. <https://doi.org/10.12693/APhysPolA.131.1105>.
14. M. Mehdi, Y. He, E.J. Hilinski, L. Kestens, A. Edrissy: Acta Materialia, 185, 2020, 540-554. <https://doi.org/10.1016/j.actamat.2019.12.024>.
15. H. Jiao et al: Acta Materialia, 199, 2020, 311-325. <https://doi.org/10.1016/j.actamat.2020.08.048>.
16. Z.H. Li, S.K. Xie, G.D. Wang, H.T. Liu: Journal of Alloys and Compounds, 888, 2021, 161576. <https://doi.org/10.1016/j.jallcom.2021.161576>.
17. H. Naumoski, B. Riedmüller, A. Minkow, U. Herr: Journal of Magnetism and Magnetic Materials, 392, 2015, 126-133. <https://doi.org/10.1016/j.jmmm.2015.05.031>.
18. M.A.D. Cunha, S.C. Paolinelli: Journal of Magnetism and Magnetic Materials, 5, 2002, 379-381. <https://doi.org/10.1590/S1516-14392002000300024>.
19. J. Qiao, L. Chuanxing, G. Feihu, X. Li, Q. Shengtao, W. Haujun: Journal of Materials Research and Technology, 116, 2019, 412. <https://doi.org/10.1051/metal/2018123>.
20. F.J. Humphreys: Acta Materialia, 45, 1997, 4231-4240. [https://doi.org/10.1016/S1359-6454\(97\)00070-0](https://doi.org/10.1016/S1359-6454(97)00070-0).

21. P. Jong-Tae, J.A. Szpunar: ISIJ International, 45, 2006, 743-749. <https://doi.org/10.2355/isijinternational.45.743>.
22. H. Mun, S. Leen, Y.M. Koo: ISIJ International, 57, 2017, 1241-1245. <https://doi.org/10.2355/isijinternational.ISIJINT-2016-564>.
23. H.-P. Yang, Y.H. Sha, F. Zhang, L. Zuo: Journal of North-eastern University, 34, 2013, 658-662.
24. I. Petryshynets, F. Kovac, B. Petrov, L. Falat, V. Puchy: Materials, 12, 2019, 15. <https://doi.org/10.3390/ma12121914>.
25. S. Oktay, M.K. Sesen, P.E. Di Nunzio: Acta Metallurgica Slovaca, 26, 2020, 11-16. <https://doi.org/10.36547/ams.26.1.449>.
26. I. Petryshynets, F. Kovac, L. Falat, V. Puchy, M. Sebek: Acta Physica Polonica A, 133, 2018, 1065-1068. <https://doi.org/10.12963/APhysPolA.133.1065>.
27. H. L. Yu et al.: Advanced Engineering Materials, 18, 2016, 754-769. <https://doi.org/10.1002/adem.201500369>.
28. K.S. Panigrahi, R. Jayaganthan: Materials Science and Engineering A, 480, 2008, 299-305. <https://doi.org/10.1016/j.msea.2007.07.024>.
29. S.V. Zherebtsov et al.: Acta Materialia, 61, 2013, 1167-1178. <https://doi.org/10.1016/j.actamat.2012.10.026>.
30. P. Petrousek, T. Kvačák, R. Kocisko, J. Bidulská, M. Luptak, D. Manfredi, M. Actis Grande, R. Bidulsky: Acta Metallurgica Slovaca, 25, 2019, 283-290. <https://doi.org/10.12776/ams.v25i4.1366>.
31. R. Bidulský, J. Bidulská, F.S. Gobber, T. Kvačák, P. Petroušek, M. Actis-Grande, K.-P. Weiss, D. Manfredi: Materials, 13, 2020, 3328. <https://doi.org/10.3390/ma13153328>.
32. K. Luo, Y. Wu, Y. Zhang, G. Lei, H. Yu: Metals, 12, 2022, 625. <https://doi.org/10.3390/met12040625>.
33. M.V. Markushev, R.R. Ilyasov, S.V. Krymskiy, I.S. Valeev, O.S. Sitdikov: Letters on Materials, 11(4), 2021, 491-496. <https://doi.org/10.22226/2410-3535-2021-4-491-496>.
34. Y. Tan, W. Li, A. Li, X. Shi: Scientific Reports, 11, 2021, 20893. <https://doi.org/10.1038/s41598-021-99706-x>.
35. H. Xiong, L. Su, Ch. Kong, H. Yu: Advanced Engineering Materials, 23, 2021, 2001533. <https://doi.org/10.1002/adem.202001533>.
36. G.V.S. Kumar et al.: Scientific Reports, 10, 2020, 354. <https://doi.org/10.1038/s41598-019-57208-x>.
37. T. Kvačák, J. Bidulská, R. Bidulsky: Materials, 14(8), 2021, 1988. <https://doi.org/10.3390/ma14081988>.
38. T. Trzepieciniski, V. Oleksik, T. Pepelnjak, S.M. Najm, I. Paniti, K. Maji: Metals, 11, 2021, 1188. <https://doi.org/10.3390/met11081188>.