

The effects of CO₂ laser and thulium-doped fibre laser scribing on magnetic domains structure, coercivity, and nanohardness of Fe-3.2Si grain-oriented electrical steel sheets

V. Puchý^{1*}, F. Kováč¹, L. Falat¹, I. Petryshynets¹, R. Džunda¹, M. Fides¹, M. Podobová¹, J. Mrázek², Y. Baravets², P. Honzátko², S. Vytykáčová²

¹*Institute of Materials Research, SAS, Watsonova 47, 040 01 Košice, Slovak Republic*

²*Institute of Photonics and Electronics, CAS, Chaberská 57, 182 51 Prague, Czech Republic*

Received 23 March 2018, received in revised form 23 July 2018, accepted 20 September 2018

Abstract

Two different laser sources, namely the CO₂ laser and continuous-wave thulium-doped fibre laser were individually used to study their effects on the resulting changes in magnetic domains structure, coercivity, and nanohardness of Fe-3.2Si grain-oriented electrical steel sheets. The CO₂ laser with a constant power of 30 W, constant frequency of 1 kHz and variable scribing speed in the range of 10–50 mm s⁻¹ was used to perform the first series of five different laser scribing treatments. Separately, the second series of next five laser scribing treatments was performed using thulium-doped fibre laser operating at a constant scribing speed of 2.4 mm s⁻¹ and variable laser power in the range of 2–10 W. The results showed that compared to the laser untreated material with a coercivity of 0.21 A m⁻¹, distinguishable improvements in soft magnetic characteristics (i.e., coercivity reduction) were obtained after the all laser treatments used. At the same time, lowered nanohardness values were indicated in central parts of laser scribed areas. The major effects responsible for the observed coercivity improvements are related to optimal refinement of magnetic domains structures by applied laser treatments.

Key words: CO₂ laser, thulium-doped fibre laser, electrical steel, laser scribing, magnetic domains, coercivity

1. Introduction

Grain-oriented (GO) electrical steels occupy a significant part in world-wide market of soft magnetic materials. They are widely used in the form of core-laminated products, e.g. in the construction of power transformer cores thanks to their excellent power capacity. During their magnetization under alternating current conditions, a part of the electric energy is known to be consumed which is attributed to the existence of the so-called core losses. The promising results have been shown in GO electrical steel sheets through the refinement of their magnetic domain structure as a result of mechanical and/or thermal stresses implementation [1, 2]. It is already known that the desired effects can be achieved by several processing techniques, e.g. mechanical scribing/scratching, electrical discharge machining, plasma flame treatment, chemi-

cal etching, and also by laser scribing [3–9]. The laser scribing technique is based on a rapid laser-induced increase of temperature on the sheet surface causing residual stresses during subsequent cooling that refines the magnetic domains. The mechanisms responsible for the improvement of soft magnetic properties include magnetic domain structure refinement, stress relaxation, and inhibition of domain-wall movement [10]. The industrially used laser scribing systems have been widely optimized for several other materials and applications, e.g. scribing of crystalline Si, resistor trimming or circuit repair [11, 12]. Compared to laser cladding [13] or laser surface alloying [14], the laser scribing of electrical steel sheets can be performed by applying only a very low laser power, e.g. up to 100 W. Thus any undesired effects related to surface deterioration of the treated material, e.g. by surface melting and ablation can be avoided. Our former stud-

*Corresponding author: e-mail address: vpuchy@saske.sk

Table 1. The chemical composition of the M165-35S grade of Fe-3.2Si electrical GO steel (wt.%)

Si	C	Mn	S	Cu	P	Al	N	Fe
3.15	0.006	0.21	0.009	0.44	0.007	0.013	0.011	Balance

ies [15–17] were focused on optimizing of neodymium-doped fibre laser scribing conditions of Fe-3.2Si electrical steel sheets for a single pulse, multipulse, and modulated multipulse laser scribing regimes. The present study deals with the effects of the CO₂ laser and thulium-doped fibre laser scribing treatments on the changes in the magnetic domains structure, coercivity, and nanohardness of the same experimental material.

2. Experimental material and procedure

The Fe-3.2Si electrical GO steel of M165-35S grade was manufactured by ArcelorMittal Frýdek-Místek, a.s. and the specimens with dimensions of 30 mm × 10 mm × 0.35 mm were used as experimental material. Its chemical composition is listed in Table 1.

The experimental specimens were produced by laser cutting with oxygen assist gas to assure better cutting performance and minimization of the introduction of residual stresses. The prepared specimens were annealed in a nitrogen gas atmosphere and afterward electrolytically polished using the Lectropol-5 electrolytic polishing machine in a solution of CH₃COOH and HClO₄ (20:1 per volume). The conditions of electrolytic polishing were: 49 V, 22°C. This procedure was used to avoid generation of residual stresses during preparation. The final cleaning of produced mirror-finished specimens was performed in deionized water and ethanol. The prepared smooth surfaces of GO silicon steel specimens were used for the magnetic domain structure observation. The prepared specimens were mounted into a specimen holder and put onto the experimental laser X-Y movable stage. Two individual sample series were subjected to several laser scribing regimes performed in air using either CO₂ laser (Coherent, model Diamond C-30) or thulium-doped fibre laser (experimental configuration) sources. The CO₂ laser in pulse width modulation (PWM) regime with a constant power of 30 W, constant frequency of 1 kHz, 10 % duty cycle and variable scribing speed in the range of 10–50 mm s⁻¹ was used to perform the first series of five various laser scribing treatments. The CO₂ laser scribing was performed using laser radiation with a wavelength of 9174 nm, and the focus diameter on the sample surface was 100 μm. The second series of the next five laser scribing treatments was performed using continuous wave thulium-doped fibre laser operating at constant scribing speed of 2.4 mm s⁻¹ and variable laser power in the range of 2–10 W. The thulium-doped fibre

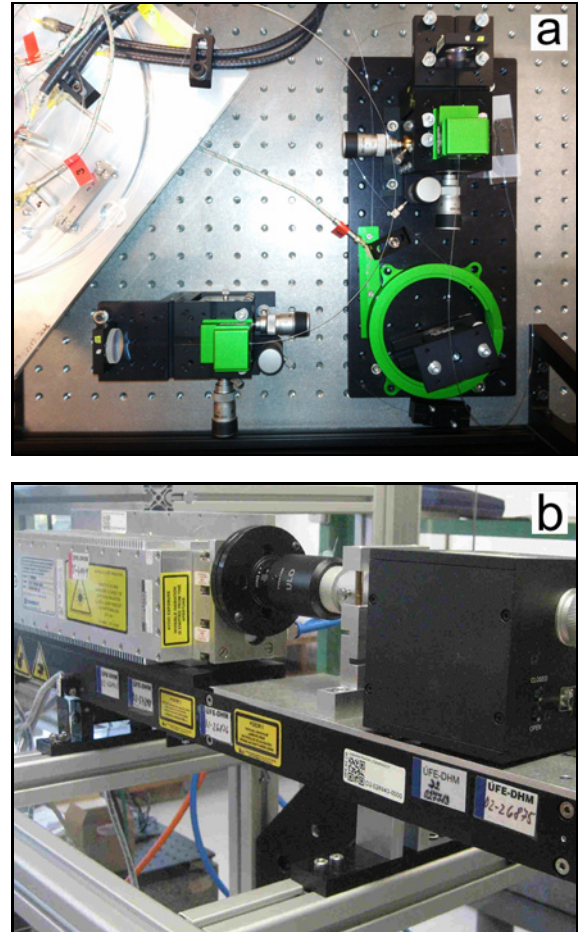


Fig. 1. Experimental setup of the used laser scribing systems: (a) thulium-doped fibre laser and (b) CO₂ laser.

laser scribing was performed using the radiation with a wavelength of 2039 nm. The focus diameter on the sample surface was 35 μm. For both used methods, the laser scribing was always oriented perpendicularly to the rolling direction (RD) of the investigated material, and the spacing of laser scribe lines was 4 mm. The experimental set-up of both used laser scribing systems is visualized in Fig. 1. Figure 2 schematically demonstrates the used laser processing regimes and their interaction with the studied Fe-3.2Si material. The microstructure and morphology of laser treated surfaces were observed by scanning electron microscope (SEM) JEOL JSM-7000F. The measurement of coercivity was performed using “Oersted type” coercivity-meter KPS-1C. The magnetic domains structures were observed using two visualization methods, namely Bit-

Table 2. Process variables for laser scribing of Fe-3.2Si electrical steel sheets and resulting soft magnetic and nanohardness characteristics

Laser source	Sample No.	Laser power (W)	Scribing speed (mm s^{-1})	Supplied laser energy (mJ)	Energy per length unit (J mm^{-1})	Coercivity (A m^{-1})	Magnetic domain width (μm)	HV _{2000nm} in the centre of laser track (GPa)
none	0	0	0	0	0	0.21	45 ± 4.2	2.45 ± 0.33
Tm-doped fibre laser	1	2	2.4	30	0.83	0.07	11.5 ± 1.2	2.27 ± 0.32
	2	4		60	1.67	0.11	14 ± 1.6	2.24 ± 0.30
	3	6		90	2.5	0.115	14.5 ± 1.5	2.22 ± 0.29
	4	8		120	3.33	0.13	24 ± 2.9	2.18 ± 0.25
	5	10		150	4.17	0.15	33 ± 4.0	2.05 ± 0.21
CO ₂ laser	6	30	50	60	0.6	0.114	27.5 ± 3.1	2.41 ± 0.51
	7		40	75	0.75	0.105	25 ± 2.7	2.35 ± 0.42
	8		30	100	1	0.097	20.5 ± 2.2	2.30 ± 0.39
	9		20	150	1.5	0.055	10.5 ± 1.3	2.28 ± 0.35
	10		10	300	3	0.08	17.5 ± 2.0	2.25 ± 0.34

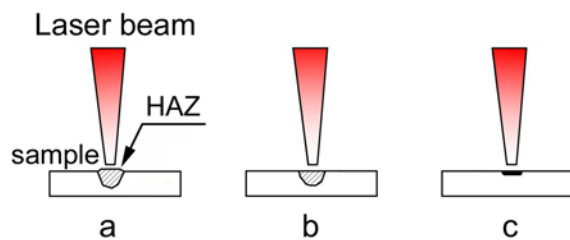


Fig. 2. Schematic visualization of the used laser processing regimes and their interaction with studied material: (a) thulium-doped fibre laser with maximal applied power generates melting with overflow of molten material, (b) thulium-doped fibre laser with medium power – no evidence of melting with smoothing of the surface geometry, (c) CO₂ laser with no melting and no change in surface geometry – macroscopically invisible heat-affected zone (HAZ).

ter technique in light optical microscopy and magnetic contrast visualization in SEM microscopy. The samples for Bitter microscopy were produced by electrolytic polishing and coating with colloid magnetic suspension Ferrofluid. Then, the magnetic domains were observed by light optical microscope Olympus GX71 using Nomarski differential interference contrast (DIC) similarly as also used, e.g. in [18]. The SEM observation of magnetic domains structures was carried out in accordance with specific conditions (tilt angle 5°, voltage 10 kV, probe current 7 nA) that were reported in [19]. The width of magnetic domains was measured using the *Image J* software for *Windows*. For this purpose, fifty readings were recorded for individual material states, followed by their statistical analyses. Nanohardness testing was carried out in the centre

of laser track using nanohardness tester *Agilent G200* with applied penetration depth up to 2000 nm. The samples were tested for coercivity and nanohardness before and after the laser processing. The most representative samples were chosen for the microstructure and texture analysis. The texture analysis was carried out by an electron backscattered diffraction (EBSD) method in the plane perpendicular to the transfer direction for the typical sample size 25 mm × 10 mm. The JEOL JSM 7000F FEG scanning electron microscope was used to perform the texture analysis. The patterns of the backscattered electrons were detected by the “Nordlys-I” EBSD detector. The data obtained were processed by the CHANNEL-5, HKL software package.

3. Results and discussion

The effects of laser scribing process variables on coercivity, nanohardness, and magnetic domains characteristics of Fe-3.2Si electrical steel sheets under investigation are summarized in Table 2. Laser scribing variables were input laser power in the case of thulium-doped fibre laser and laser scribing speed in the case of the CO₂ laser. The soft magnetic characteristics, i.e. the coercivity values of the samples were measured using a direct current (D.C.) coercivity measurement device. The laser modified samples were characterized in D.C. magnetic field along their axes. The samples were placed inside a solenoid which was then energized by a direct current. The samples were first demagnetized and then magnetized until the magnetic flux density reached its saturation. The acquired coercivity data were counted

from the coercimeter. The laser energy per length unit E_L is known to have a significant effect on the reduction of core losses. Thus it has been commonly used to agglomerate together most of the factors that affect magnetic domains refinement during laser heating as described by Rauscher et al. [9]. E_L in J mm^{-1} can be expressed by the following equation:

$$E_L = P_L/v_{\text{spot}}, \quad (1)$$

where P_L stands for laser power (W) and v_{spot} represents spot velocity, i.e., laser scribing speed (mm s^{-1}). In present work, the lowest coercivity values indicating lowest core losses were achieved at specific E_L values for both used laser beam sources (see Table 2). In the case of a CO_2 laser, at too low E_L value, the achieved coercivity has not been significantly improved (i.e., lowered) because of insufficient absorbed energy. It has been found out that the greatest coercivity reduction occurs at certain optimal E_L value, whereas further E_L increase or decrease deteriorates the material soft magnetic performance. In contrast, in the case of the thulium-doped fibre laser, a gradual increasing of E_L values causes moderate coercivity increase (i.e., soft magnetic properties deterioration). In conclusion, both laser power and spot velocity represent controlling parameters in obtaining desired coercivity and thus core loss reduction. The results in Table 2 clearly show that the coercivity can be improved, i.e. reduced by refining the magnetic domain size. Smaller domains manage themselves more easily to magnetization and thus eddy current losses also become smaller in the smaller sized domains [20]. Magnetic properties can also be affected by micro and substructural characteristics regarding dislocations, grain boundaries, inclusions, precipitates, and also by applied stress. The change in magnetic domain size depends on laser energy and laser scribing speed. In the case of the thulium-doped fibre laser, the coercivity was gradually decreasing with decreasing in laser input energy, whereas in the case of a CO_2 laser, the coercivity was gradually decreasing with decrease in laser scribing speed. The reduction in coercivity is primarily due to magnetic domain refinement caused by residual stresses. This mechanism generates the formation of subdomains which leads to the breakage of primary 180° magnetic domains. The stresses generated along the scribe lines also tend to increase the surface resistivity, which results in a reduction in total eddy current loss. It is in line with the fact that local deformation-induced dislocations enhance the resistivity, which gives evidence that coercivity is reduced due to the deformation causing enhancement in the resistivity of the materials [21]. It has been generally accepted that the lowering of core loss of commercial GO electrical steel sheets can be achieved up to $\sim 30\%$

[22]. The core loss improvement of 30% is not only caused by the laser treatment. First of all, the material improvement from conventional grain-oriented (CGO) steel to high grade oriented (HGO) steel is the core. The further improvement of the magnetic properties by laser treatment is the second step. A core loss decrease of $10\text{--}15\%$ by laser treatment is realistic. The decrease of static hysteresis losses only appears if weak defects are produced. This behaviour, i.e. the reduction of the coercive force and the power losses by the introduction of crystal defects, is known as the Brown's paradox [1]. Dynamic hysteresis loops of GO steels depend on magnetic flux density (T) and coercivity (A m^{-1}). In our present investigation, only coercivity values have been measured because the determination of core loss (W kg^{-1}) would require bigger-sized samples that were not available. Thus, in spite of such incomplete information about the core loss of presently investigated material, the obtained coercivity values represent strong indicative information about its magnetic properties in D.C. conditions.

To demonstrate the magnetic domains modifications by laser radiation, the magnetic suspension Ferrofluid was used to make the magnetic domain structure visible in the optical microscope by the Bitter method. Also, complementary SEM method was used to analyse the magnetic domain structure. Both the domain imaging techniques were used to correlate the observed domain structures with obtained coercivity and nanohardness characteristics of laser treated and untreated materials. Figure 3 demonstrates a comparison of magnetic domain structures of the studied electrical steel samples without laser modification (Fig. 3a) and with laser modification by 10 W thulium-doped fibre laser with a scribing speed of 2.4 mm s^{-1} (Fig. 3b). The magnetic particles of colloidal suspension typically decorate the magnetic domain walls. This method provides overall information about the size and shape of magnetic domain structures in magnetic materials. In the case of laser untreated material under present investigation, the 180° domain width was measured to be $45 \pm 4.2 \mu\text{m}$ (Fig. 3a), whereas, after the 10 W thulium-doped fibre laser treatment, the domain width was reduced to $33 \pm 4.0 \mu\text{m}$ (Fig. 3b). Due to laser irradiation of the material surface which produces the surface defects, additional pinning centres are formed in laser-irradiated tracks that enhance the decrease of coercivity by refining of magnetic domains. Also, it has been revealed that laser treatment creates internal tensile stresses between laser scribing lines, surface crystal defects and stray fields, which contribute to the process of magnetic domain refinement [23].

Another applied method for magnetic domain structure observation was a conventional scanning electron microscope magnetic contrast. The method is based on the principle of deflection of electrons moving

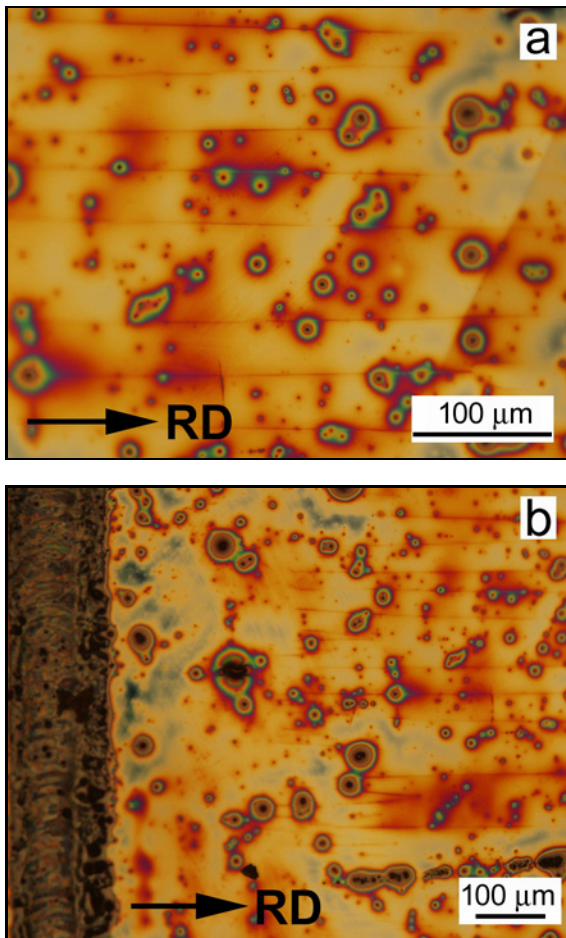


Fig. 3. Bitter technique visualization of magnetic domains structure of the investigated Fe-3.2Si electrical steel: (a) without laser modification and (b) near the laser track after the 10 W thulium-doped fibre laser modification using the scribing speed of 2.4 mm s^{-1} .

in a magnetic field due to the Lorentz force. Magnetic contrast arises from high energy backscattered electrons contrast, and it is related to the deflection of the incident and backscattered electrons by magnetic fields within the sample. The contrast of magnetic structures was maximized by using 10 kV beam energy and by tilting the sample surface to about 5° relative to the incident beam. Figure 4 shows the simple primary 180° magnetic domains between the laser tracks created by 8 W thulium-doped fibre laser (Fig. 4a) and the structure of the domain created by 30 W CO_2 laser using scribing speed of 20 mm s^{-1} .

The laser untreated and laser modified samples with the lowest obtained coercivity values were chosen for the microstructure and texture analysis. The texture analyses were carried out using electron backscattered diffraction (EBSD) method in the direction perpendicular to the transverse section of experimental samples. The crystallographic orientation of the experimental GO steel sample taken after the final box

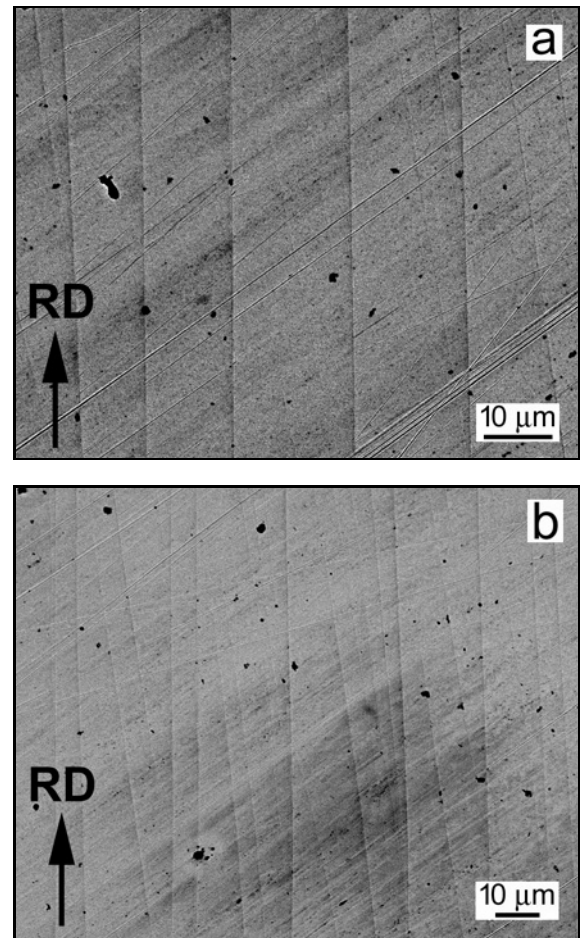


Fig. 4. SEM magnetic contrast visualization of magnetic domains structure of the investigated Fe-3.2Si electrical steel after the laser modification by: (a) 8 W thulium-doped fibre laser with 2.4 mm s^{-1} scribing speed and (b) 30 W CO_2 laser using the scribing speed of 20 mm s^{-1} .

annealing process is presented by Inverse Pole Figure (IPF) map in Fig. 5a. As one can see, the sample is characterized by the final coarse-grained microstructure with the average grain size of about some millimetres in size. The IPF map shows that the grain matrix is formed by the grains with preferable $\{110\} \langle 001 \rangle$ Goss crystallographic orientation. It means that the easy magnetization direction $\langle 001 \rangle$ is strongly parallel to the rolling direction (RD) of the sample. The IPF maps of the cross-section microstructures with thermally laser-affected zones that were induced by CO_2 laser and thulium-doped fibre laser are presented in Figs. 5b,c, respectively. Here it is evident that these IPF maps represent a part of the grains affected by a laser beam (marked by black dashed line) with characteristic Goss texture. It is visible that the laser scribing does not have any significant influence on the sharpness of the Goss texture. The results obtained in present study by using different laser sources aiming at magnetic domain structure modification revealed that

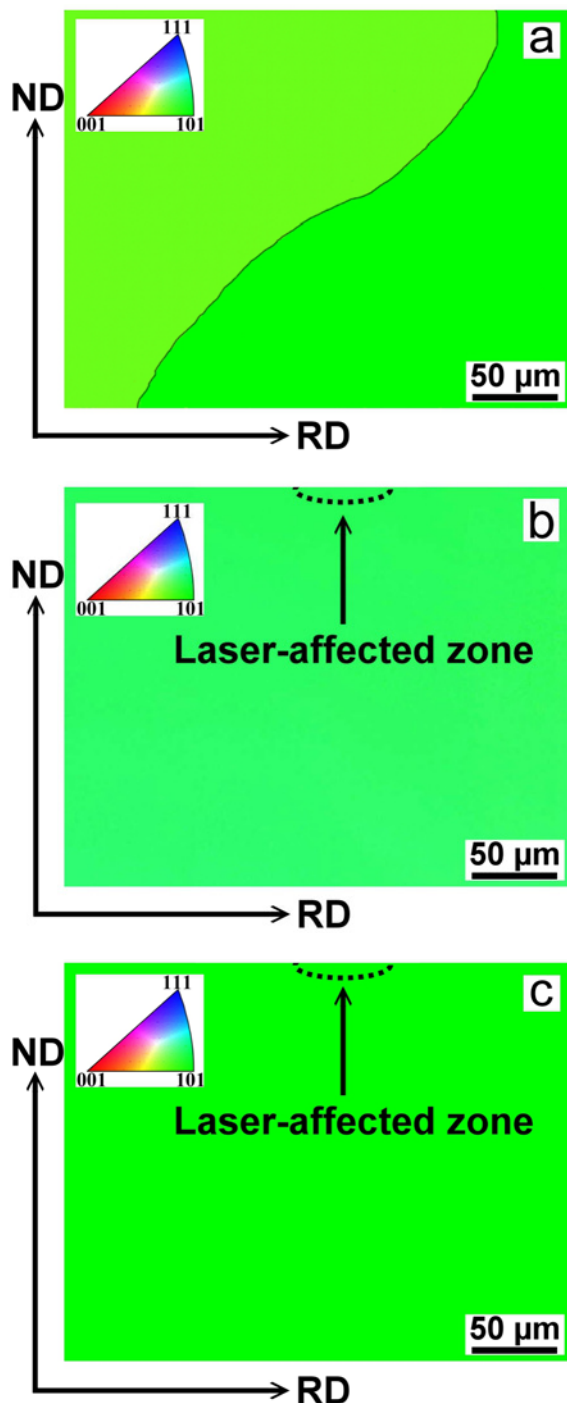


Fig. 5. IPF maps characterizing crystallographic orientation of experimental GO steel: (a) after final box annealing without laser scribing, (b) in grain with laser-affected zone obtained after CO₂ laser scribing and (c) in grain with laser-affected zone obtained after thulium-doped fibre laser scribing. (The key for the grain crystallographic orientation is located in the upper left corner of the figures.)

all the laser-scribed samples of Fe-3.2Si electrical steel sheets exhibited at the same time lower domain width

and remarkably reduced coercivity values compared to the original material without modification. Thus, it can be stated that both CO₂ laser and thulium-doped fibre laser scribing treatments represent energy efficient means in obtaining electrical steels for application in power transformers with lower core losses. Our future work will be focused on the investigation of experimental laser scribing treatments of electrical steel sheets also using nanosecond solid-state laser scribing systems.

4. Conclusions

Thulium-doped fibre laser and CO₂ laser scribing processes were carried out to investigate their effects on coercivity, nanohardness, and magnetic domains modifications of Fe-3.2Si grain-oriented electrical steel sheets. The obtained results can be summarized in the following conclusions:

- Compared to the laser untreated material, the coercivity of Fe-3.2Si grain-oriented silicon steel sheets was improved (i.e., reduced) by refining the magnetic domains structure using both CO₂ laser and thulium-doped fibre laser scribing processes.

- The best improvements related to the lowest coercivity values of 0.055 A m⁻¹ and 0.07 A m⁻¹ were obtained by the CO₂ laser treatment with a scribing speed of 20 mm s⁻¹ and the thulium-doped fibre laser treatment with a laser power of 2 W, respectively.

- All laser treated materials exhibited lower nanohardness values compared to the laser untreated material.

- The used laser processing regimes did not cause any significant changes in the crystallographic texture of the studied material.

Acknowledgements

This work was supported by the Slovak Research and Development Agency under the contracts Nos. APVV-15-0259 and APVV-0147-11, Slovak Scientific Grant Agency VEGA, grant No. 2/0066/18, and bilateral project SAV-AVCR 16-17. The work was also realized within the frame of the projects ITMS 26220220061, ITMS 26220220064, and ITMS 26220220186.

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