

Effect of structural steel ion plasma nitriding on material durability in pulsed high magnetic fields

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Abstract. The work was aimed to study the influence of plasma nitriding on electrical and mechanical properties of structural steels and their durability in pulsed high magnetic field. The plates and cylindrical magnetic flux concentrators were made of several steel grades (30KhGS, 40Kh, 50KhGA, 38Kh2MYuA, and U8A), heat-treated, and subjected to the low-temperature (400, 500°C) plasma nitriding. Electrical and mechanical properties of materials, phase composition of steel surface layer, microstructure and microhardness profiles were investigated on the plates before and after plasma treatment. Microstructure and microhardness profiles across the subsurface layer of plasma treated and untreated concentrators applied for high magnetic field generation were also studied. Magnetic field of 50 T under tens of microseconds in duration inside the flux concentrators was generated by long-life outer coil.

1. Introduction

As is known, tool coils (inductors) for magnetic pulse welding (MPW) of hard metals have poor durability under the application of magnetic fields higher than 30 – 40 T. The features of pulsed high magnetic field generation and MPW process are well described in the literature [1-3]. During MPW metallic workpieces are placed inside an inductor and rapidly deformed under the action of electromagnetic Lorentz force caused by the magnetic field and eddy current induced in the workpiece surface. At magnetic field of 40 T the acting pressure is 0.6 GPa.

MPW mostly employs single-turn coils due to their easy manufacturing and high strength. Such a coil corresponds to a massive conductor having a cylindrical hole and through-the-wall slit as shown in Figure 1a. It allows flowing circular current around the hole and generating an axial magnetic flux. Figure 1b demonstrates another design that represents a pulse transformer modification. It contains a primary massive coil and a secondary coil usually called as a magnetic flux concentrator or a field shaper. This design is more preferable in practice because a damaged secondary coil can be quickly replaced; nevertheless this construction has lower efficiency of magnetic energy delivery.

When a pulsed current flows through an inductor (e.g. at a capacitor bank discharge), it tends to concentrate inside a thin surface layer of the inductor (skin-effect). In real conductors having finite electrical conductivity some diffusion of magnetic flux induced by pulsed current into conductor interior takes place. To restrict magnetic field diffusion the current pulse duration (half-period) must be as short as tens of microseconds. In such a fast rising magnetic field inductor material due to the current concentration inside the skin-layer suffers high thermo-mechanical stresses induced by electromagnetic force and Joule heating. These are the main factors which cause inductor damage.



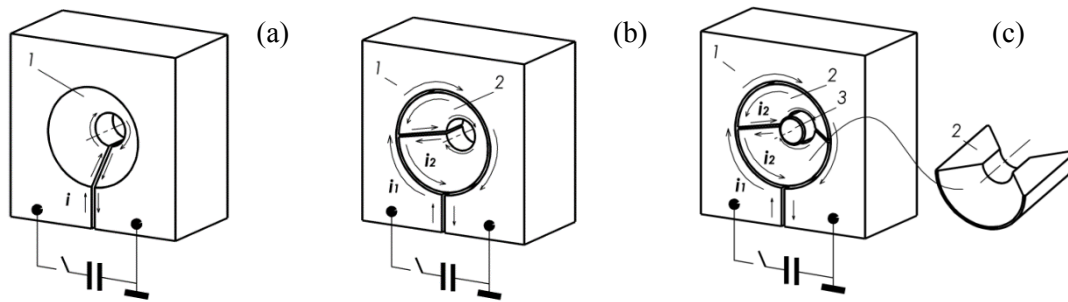


Figure 1. Diagram of a single-turn inductor (a) and inductors with single (b) and duplex (c) flux concentrator: 1 – primary coil, 2 – secondary coil (magnetic flux concentrator), 3 – rod.

In the literature one can find papers devoted to the theoretical analysis of thermo-mechanical stresses in thick coils, e.g. [4,5]. It was shown that the thermo-mechanical stresses in fast rising magnetic fields usually exceed the magnetic field pressure applied. Some solutions of magnetic field diffusion equations indicate at least two ways to reduce the Joule heating of the conductor surface [6,7]. The first way is to change the waveform of magnetic field pulse. The current pulse waveforms $[\sin, (1-\cos)]$ which are the most common in pulse power technology provide higher surface energy release than linearly increasing magnetic field. However, this waveform is not applicable for MPW. The second way is to use a conductor whose conductivity increases with depth allowing to reduce Joule heating at the surface by the factor of 2 – 4 compared to uniform conductivity case [6,7].

Thus, modifying the inductor current-carrying layer, changing its conductivity and strength, may be the useful way to improve inductor performance. Nowadays structural steels are alternative inductor materials instead of strong copper-based alloys due to lower cost, higher strength, and applicability to modification by, e.g. plasma nitriding. This process for steel modification is well known in the literature [8,9], but specific application of steel for inductor production leaves open the questions on electrical properties and inductor durability.

The work is aimed to study the influence of plasma nitriding on electrical and mechanical properties of structural steels and their durability in pulsed high magnetic field of 50 T in amplitude and about 6 μs in rising time. In the present work we used the coil assemblage with a duplex concentrator (figure 1c) considering its easier nitriding and characterization of concentrator inner surface after each magnetic field pulse.

2. Experimental

2.1. Materials and sample preparation

Several domestic structural steel grades were chosen for study according to their appropriate thermal and mechanical properties. Nomenclature and chemical composition of steels are listed in Table 1. They include 30KhGS, 40Kh, 50KhGA, 38Kh2MYuA with medium carbon content of 0.3 – 0.5% and chromium content of 0.8 – 1.7%. A high carbon instrumental steel U8A was also used.

Three types of samples were prepared. First, thick enough plates used for the nitriding study, with dimensions of 10x20x2 mm. Second, thin plates used for the electrical conductivity measurements, with dimensions of (6 – 8)x(25 – 30)x(0.35 – 0.50) mm. Third, cylindrical flux concentrators with cone ends (field shapers) having 30 and 10 mm outer and inner diameters, 30 mm length, and inner channel length of 10 mm were used for generating the high magnetic field. The samples were studied after quenching with tempering and after nitriding the quenched samples. The plates and concentrators quenching was performed in the temperature range of 850 – 900°C for medium carbon steels and at 780°C for U8A. The tempering was performed in air at 400 and 200°C during 2 hours for untreated reference and to be nitrided samples. Some thin plates were subjected to isothermal annealing in vacuum at 770°C for U8A and 850°C for the tests to characterize the equilibrium conductivity.

Ion plasma treatment was performed using an electron source with a plasma cathode based on low-

pressure glow discharge described in detail elsewhere [9]. Electrons emitted by the plasma cathode were accelerated in a double layer of a space charge formed in meshes of the plasma cathode grid. Initial energy of fast electrons was determined by the voltage of 150 V applied between the grid and plasma chamber. Gas ionization by the electron beam provided plasma generation. The working chamber was evacuated by a turbomolecular pump to the residual pressure of 0.002 Pa. The argon-nitrogen mixture with the Ar/N₂ ratio of 0.8 and the total pressure of 3 Pa was used. The gas ions accelerated by bias voltage (200 V) applied to the specimen provided a sample surface bombardment and heating as well. The measured ion beam current density was 2 – 3 mA/cm². The temperature of the specimens was controlled by a thermocouple and was fixed at 400 or 500°C by adjusting the beam current in the range of 2.0 – 3.3 A. Figure 2 shows arrays of the plates and the concentrators for nitriding. The duration of nitriding process was 4 hours both for the thick plates and the concentrators. The samples for resistivity measurements were subjected to a double-sided nitriding at 500°C during 5 h.

Table 1. Data sheet chemical composition.

Grades	Content, wt. %								
	C	Cr	Mn	Si	Al	Mo	Cu	Ni	Fe
30KhGS	0.31	1.01	1.01	1.14		0.01	0.23	0.08	bal.
40Kh	0.38	0.82	0.57	0.26		0.02	0.22	0.11	bal.
50KhGA	0.49	1.06	0.93	0.23			0.16	0.13	bal.
38Kh2MYuA	0.38	1.69	0.48	0.22	0.86	0.19	0.12	0.22	bal.
U8A	0.80	0.27	0.21	0.22			0.21	0.11	bal.

Table note: in steel designation: Kh – chromium, G – manganese, S – silicon, M – molybdenum, Yu – aluminum.

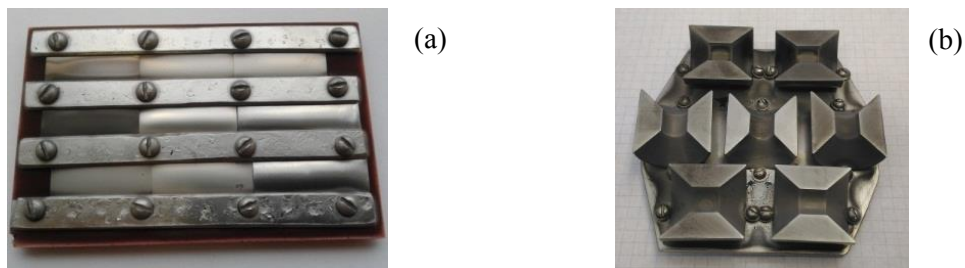


Figure 2. View of arrays of the plates (a) and flux concentrators (b) for nitriding.

2.2. Electrical conductivity measurement

The resistivity of the thin plates was measured by 4-probe method using digital DC milliohm meter GOM-802 (Good Will Instrument) at ambient temperature of 25°C. Special brass contactors with fixed distance between the potential probes were applied to measure the resistance of the narrow thin plates. The long edges of nitrided samples were cut after treatment to remove untreated regions. Curvature of the nitrided samples was taken into account at resistivity evaluation. The accuracy of resistance measuring (9 times per every sample) at 1 A test current was 0.5%. The accuracy of sample geometry and potential probes distance measurements was accordingly 0.2 and 0.3%. The summary error for resistivity was established at 0.8%. The resistivity was measured on three types of thin plates: after annealing, after quenching, and after nitriding.

2.3. Magnetic flux concentrators (field shapers) testing

The experiment aimed to investigate the effect of fast-rising high magnetic field on steel properties was carried out using a pulsed current generator based on a capacitor storage ($C = 435 \mu\text{F}$, $U_{\text{max}} = 25 \text{ kV}$). The generator short circuit current peaks at 1.5 MA with 5 – 6 μs rising time. In our experiments the generator was loaded to a single-turn inductor with magnetic flux concentrator as shown on figure 3 (on the photo the concentrator and rod are slightly extracted). We used the duplex

concentrator considering its easier nitriding and characterization of inner surface after each current pulse. Both parts of duplex concentrators were well insulated from each other and from the primary coil using the Kapton® tape (0.05 mm thick) having high electrical and thermal strength. A tempered beryllium bronze rod of 9 mm in diameter was inserted inside the channel to increase magnetic flux density. The self-inductance of the coil assemblage was about 16 nH.

To establish the magnetic field of 50 T in amplitude a circular gap arranged pick-up coil was used. The magnetic field in the space between the concentrator and the rod in their middle plane was measured at different rod diameters. Figure 4a demonstrates the peak field to peak current ratio (inductor geometrical factor) depending on the rod diameter. It is seen that different current load results in the geometrical factor variation due to Joule heating. Using the dependence at the highest current, the geometrical factor of 45 T/MA was determined for working rod diameter. This factor value was used to calculate the working current to be 1050 kA.

The figure 4b demonstrates the time dependence of discharge current measured by a Rogovsky coil and magnetic field measured by the pick-up coil with the rod of 8.5 mm in diameter at 13 kV generator charge voltage. The magnetic field rising time and half period were 5.5 and 12.5 μ s, respectively. After the testing at different pulse numbers: zero pulses (initial state), 5 pulses, and after damage, the parts of concentrators were cut across the middle plane and polished for examination.

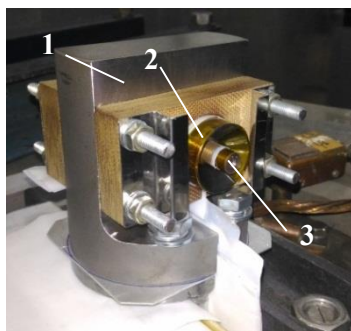


Figure 3. Coil assemblage:
1 – primary coil, 2 – magnetic
flux concentrator, 3 – rod.

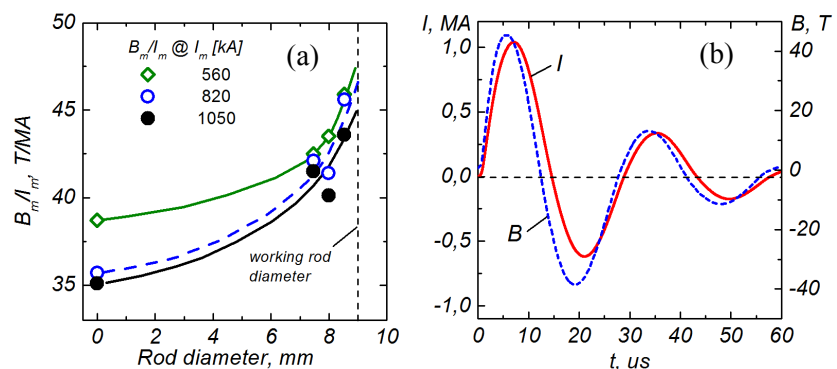


Figure 4. Inductor geometrical factor vs. rod diameter at different current load (a) and time dependence of current and magnetic field at $U_0 = 13$ kV ($C = 435$ μ F).

2.4. Micromechanical testing

Micromechanical testing was performed using Nanotest 600 instrument with a diamond indenter. The profiles of Vickers microhardness H_v were measured both on cross-sectioned polished flat plates, through-the-thickness, and on flux concentrators towards the radial direction. Indenter load of 100 mN was used to provide the spatial resolution of tens of micrometers.

2.5. Structural and microstructural investigation

Phase composition of nitrided thick plates surface was studied using X-ray diffractometer D8 Discover (Bruker AXS) with Cu $K\alpha_{1,2}$ radiation and graphite monochromator. A Rietveld program TOPAS 3 was used to analyze the X-ray patterns. Polished and chemically etched (nitric acid alcohol solution) cross sections of the plates and the concentrators were analyzed by Olympus BX41RF metallograph.

3. Results and Discussion

3.1. Micromechanical and structural properties of the flat samples

The microhardness profiles measured on nitrided thick plates are presented in figure 5. The modified layer of 70 – 100 μ m and 150 – 200 μ m thick was obtained after the nitriding at 400 and 500°C respectively for all the steels except U8A. The steels with 0.8 – 1%Cr have similar H_v profiles. The

value of H_v at the surface was 6.8 – 7.4 GPa and 8.6 – 8.9 GPa after nitriding at 400 and 500°C, respectively. The high surface saturation with nitrogen was inherent to the higher chromium steel 38Kh2MYuA (1.7% Cr) that resulted in significant increase in surface microhardness that was 11.3 and 13.5 GPa after nitriding at 400 and 500°C, respectively. Weak nitriding effect was obtained on high carbon steel U8A with its near-surface H_v value only 30% higher than that in the bulk. Hatched rectangles on both graphs represent the range of H_v for all the steels before nitriding. One can conclude that nitriding process temperature resulted in decreasing the bulk strength of steels due to the tempering process. Thus, the temperature of 500°C was suggested to be the maximal (optimal) to produce flux concentrators with modified layer of 150 μm thick in the average and strength enough bulk material that is important for magnetic pulse tool.

X-ray analysis revealed that the main surface phase at 400°C nitriding temperature was $\alpha\text{-Fe}$ (cubic, S.G.: Im-3m) with the highest content of 86 – 96 wt.% for the steels 30KhGS, 40Kh, and 50KhGA and the Fe_2N phase in the rest. In contrast to them the 38Kh2MYuA steel at this nitriding temperature contained the Fe_2N as high as 62 wt.% and 13 wt.% of $\text{FeN}_{0.88}$ (tetragonal, S.G.: I4/mmm). X-ray data for 500°C nitriding temperature are listed in table 2.

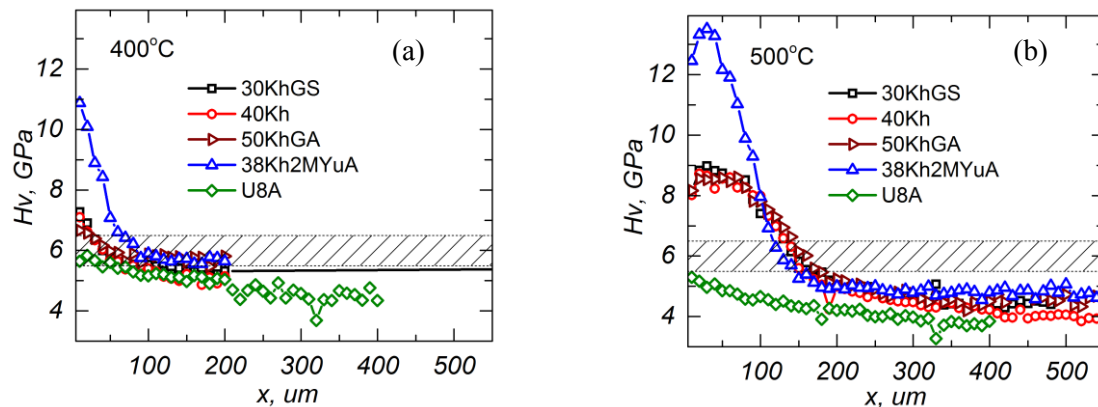


Figure 5. Vickers microhardness profiles on cross-sectioned plates after nitriding at 400°C (a) and 500°C (b). Indentor load was 100 mN.

Table 2. Steel surface phase composition after nitriding at 500°C.

Grades	Content, wt. %		
	Fe_2N (orthorhombic, S.G.: Pbcn)	$\gamma\text{-Fe}_4\text{N}$ (cubic, S.G.: Pm-3m)	$\epsilon\text{-Fe}_3\text{N}_{1+x}$ (hexagonal, S.G.: P6322)
30KhGS	96	4	-
40Kh	54	11	35
50KhGA	83	17	-
38Kh2MYuA	44	26	30
U8A	49	51	-

3.2. Electrical properties of steel samples

Table 3 summarizes the resistivity of steels measured on thin plates for three states: after annealing, after quenching with subsequent tempering at the temperature of 200°C/2 h, and after plasma nitriding the quenched and tempered samples. Copper grade Cu-DHP was as a reference material: the measured resistivity, $2.05 \pm 0.05 \mu\Omega\cdot\text{cm}$, was in good agreement with Cu-DHP data sheet.

With the reference to Table 1 it can be concluded that the resistivity of the steels after annealing and quenching is greatly influenced by the chromium content. However, after the quenching the resistivity increases accordingly to carbon content as well, which is clear from the ratio ρ_q/ρ_a . It is probably associated with the recrystallization of ferric carbide precipitates of small size on the grain

boundaries during the samples tempering. An unexpected result was obtained on nitrided samples for which an enhanced conductivity was observed. It becomes clear from the ratio ρ_n/ρ_a . The conductivity was occurred to be up to 27% higher than that of the equilibrium one. In general, the effect was as high as the chromium content was low except for 38Kh2MYuA. This increase in conductivity is difficult to attribute to any certain phase in the “iron-nitrogen” system since the nitrided plates have gradient structure according to the X-ray data: thin surface regions with iron nitride phases and a bulk region which is a solid solution of nitrogen in iron base. The chromium and carbon binding by the nitrogen is also a possible reason of the conductivity increase. To explore this effect a low carbon (0.06%) and chromium (<0.1%) steel must be examined.

Table 3. Resistivity of the steels before and after plasma treatment.

Grades	Resistivity@25°C, 10 ⁻⁸ Ohm·m			ρ_q / ρ_a	ρ_n / ρ_a
	ρ_a (annealed)	ρ_q (quenched)	ρ_n (nitrided)		
30KhGS	35.6±0.6	42.5±0.3	31.4±0.3	1.19	0.88
40Kh	24.5±0.3	31.6±0.4	20.9±0.4	1.29	0.85
50KhGA	24.5±0.2	34.1±0.3	21.5±0.2	1.39	0.88
38Kh2MYuA	36.7±0.6	44.3±0.3	28.8±0.3	1.21	0.78
U8A	20.4±0.2	27.3±0.3	20.1±0.3	1.34	0.98

Figure 6a shows a view of the nitrided plates (from the left: 30KhGS, 40Kh, 50KhGA, 38Kh2MYuA, and U8A). One can see that the samples bending of different degree takes place after the nitriding. At that, correlation between the curvature and the chromium content can also be found. The higher chromium content resulted in stronger bending in contrast to U8A plate which was almost flat. The degree of nitrogen saturation through the plate thickness was indirectly confirmed by the Hv profile. The profiles for the steels 50KhGA, 40Kh, and 38Kh2MYuA are presented in figure 6b.

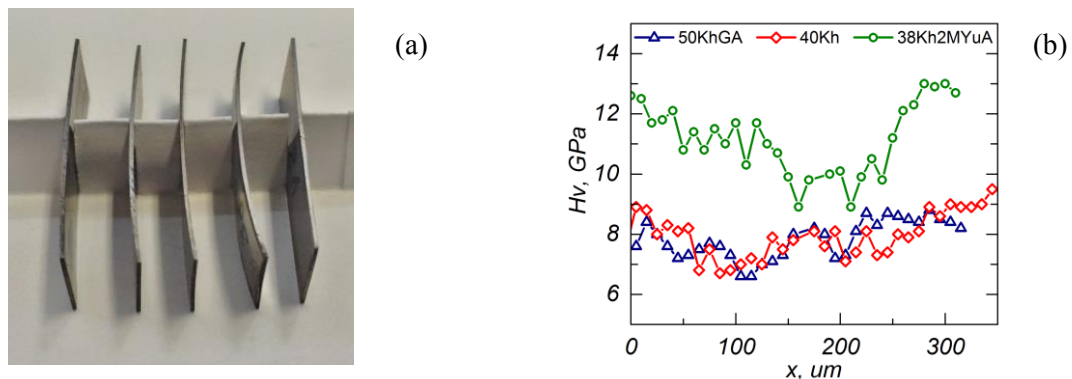


Figure 6. View of thin plates for resistivity measurements (a) and Hv profiles through-the-thick (b).

3.3. Micromechanical and microstructural properties of flux concentrators of 30KhGS steel before and after magnetic field generation

The preliminary results of 30KhGS steel flux concentrators durability testing and investigation of micromechanical and microstructural properties evolution are presented here. Plasma treated and untreated concentrators without magnetic field treatment were taken as reference samples.

Figure 7a represents a cross midsection photo of the nitrided (1N.1) and untreated (1.2) concentrators parts after damage, i.e. after the formation of visual longitudinal radial cracks. The figure 7b shows the microhardness radial profiles measured near the inner channel surface for three different untreated half concentrators: “initial” – without magnetic field generation, after 5 pulses of high magnetic field and after damage (47 pulses). The figure 7c shows the same for three nitrided half concentrators. It is seen that initially the untreated sample (figure 7b) has practically uniform Hv

distribution. After the following pulses the surface strength gradually decreases due to the tempering effect with the bulk strength maintenance. On plasma treated sample an expected Hv radial distribution in the initial state is observed. The behavior of Hv(r) after 5 and 15 pulses is practically the same. Despite of lower microhardness, which was detected for the sample after 5 pulses, the modified layer is characterized by a high heat-resistance.

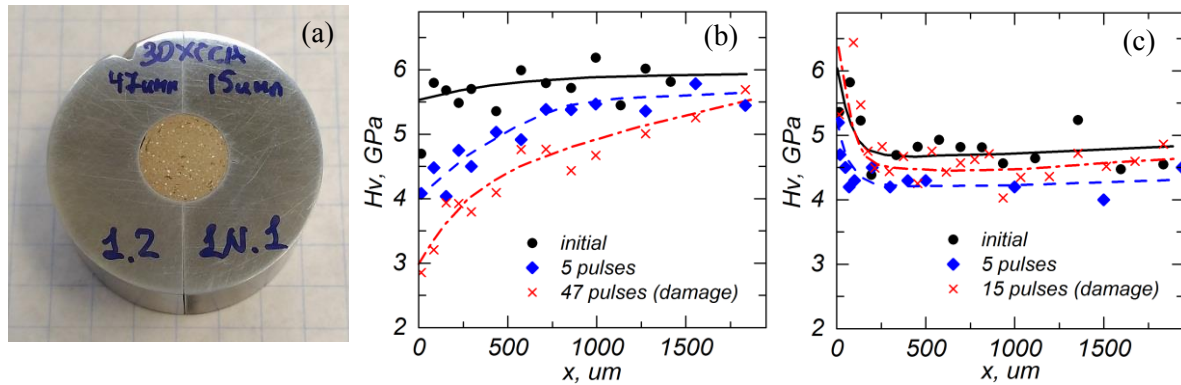


Figure 7. View of the cross midsections of nitrided and untreated concentrators (a) and Hv radial profiles of untreated (b) and nitrided (c) concentrators.

It was observed that the formation of a hard nitrided surface layer, which is brittle enough, results in rapid crack growth. The crack formation mechanism is the same as described in [2]. Due to the high strength of nitrided layer the cracks with sharp edges are formed during the skin-layer cooling under the action of tensile stresses. Further increase in crack depth is enhanced by the so-called “saw effect” and accompanied by plasma sparks ejection. In untreated concentrators longitudinal corrugations at inner channel surface was formed due to the plastic deformation, especially at the channel edges. It was directly confirmed by the surface viewing after several pulses. Finally, the number of ultimate pulses for plasma treated 30KhGS steel concentrator was 15 in contrast to untreated one which was tested 47 times and the crack nuclei formation occurred only at channel edges.

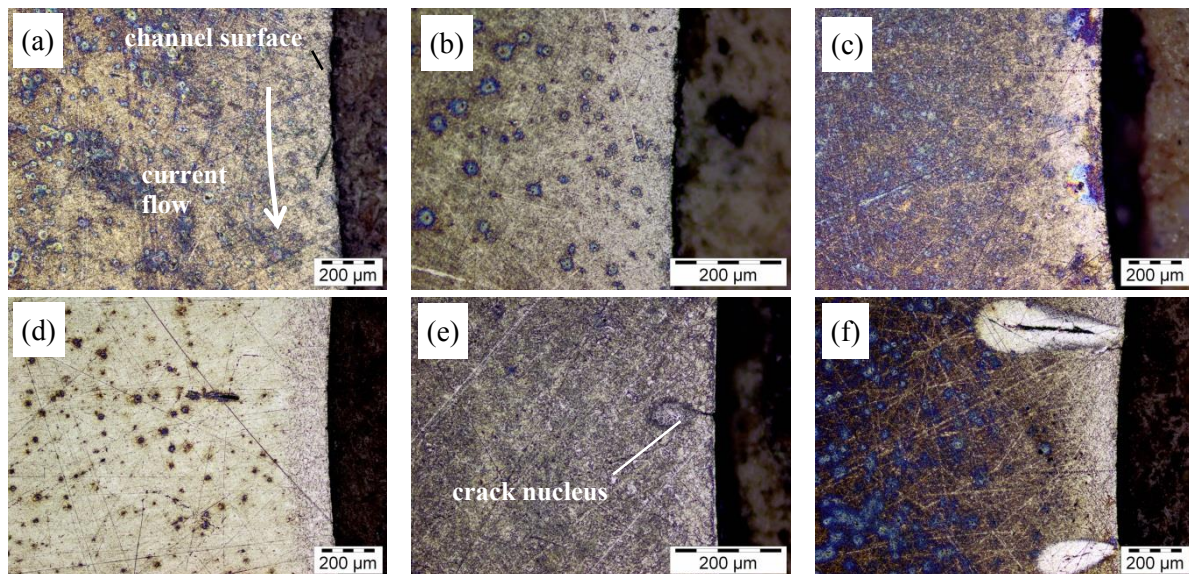


Figure 8. Microimages of inner channel region of untreated (a – c) and nitrided (d – f) concentrators at different numbers of 50 T magnetic field pulses: a, d – zero, b, e – 5, c – 15, f – 47.

Figure 8 represents the microimages of cross midsections of the plasma treated and untreated concentrators close to the channel surface. The steel structure after the quenching is determined to be mainly acicular martensite. Under the magnetic field treatment, pulse by pulse, the martensite phase undergoes a transformation at a surface layer of 150 – 200 μm thick (heat-affected area). In plasma treated samples the modified layer of about 170 μm thick is well defined and characterized by grains refinement due to the precipitation of nitride phases and iron base saturation with nitrogen. No significant microstructure transformation was detected for these samples under the magnetic field treatment. Only the cracks nuclei and cracks enhanced through the melting and spark plasma ejection were observed after 5 and 15 pulses, respectively.

4. Conclusion

The influence of low-temperature (400, 500°C) plasma nitriding on electrical and mechanical properties of structural steels, including 30KhGS, 50KhGA, 40Kh, 38Kh2MYuA, and U8A, has been investigated. The examination of the samples treated in argon-nitrogen plasma revealed the common behavior of microhardness profiles. The modified layers with thicknesses of 70 – 100 μm and 150 – 200 μm have been obtained after nitriding at 400 and 500°C respectively for all the steels except the high carbon steel U8A. The annealed steels resistivity was determined in the range of 20 – 37 $\mu\text{Ohm}\cdot\text{cm}$ depending on the steel grade. An increase in resistivity was established for all the steels after the quenching; at that, the higher carbon content resulted in higher resistivity increase by 1.19 – 1.34 times. An increase in conductivity was observed after the steels ion plasma nitriding. The conductivity was occurred to be up to 27% higher than that of the equilibrium one. The durability tests of 30KhGS magnetic flux concentrators in pulsed high magnetic field of 50 T in amplitude and 5.5 μs in rising time have been performed. The ultimate number of pulses for nitrided 30KhGS steel concentrator was 15 in contrast to untreated one that withstood 47 pulses without significant cracking. Micromechanical and microstructural properties evolution of plasma treated and untreated concentrators of 30KhGS have been studied as well. It was observed that the decrease of surface strength occurred from pulse to pulse in quenched samples. On the contrary, quenched steel surface modification by the nitriding resulted in stability of microstructure owing to higher heat-resistance. The formation of hard surface layer in nitrided concentrator which is brittle enough resulted in rapid crack growth. The further experiments on durability of 30KhGS steel concentrator having an enhanced surface conductivity and strength under the action of moderate of 30 T magnetic fields (a fatigue failures testing) will allow understanding the applicability of surface modification by plasma nitriding.

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