Combined treatment of steel, including electrospark doping and subsequent irradiation with a high-intensity electron beam

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Abstract. A thermodynamic analysis of phase transformations taking place during doping of steel with tungsten and titanium has been performed. The studies on the surface layer of steel modified using the combined method (electrospark doping and the subsequent electron-beam treatment) have been carried out. Formation in the surface layer of a multi-phase submicrocrystalline structure with high strength properties has been revealed.

1. Introduction
The method of electrospark doping (ESD), based on the phenomenon of electrical erosion and polar transfer of the anode material to a cathode (a work piece) during a pulsed electric discharge between them in a gaseous medium, differs from other surface treatment methods in its simplicity, reliability, and cost-effectiveness [1]. Depending on the anode material, an extended surface layer with high strength, tribological, and other properties is formed on the work piece. The disadvantages of ESD are as follows: high surface roughness of the treated surface of the work piece, the presence of cracks, discontinuities, and micropores. To reduce the roughness introduced by electrospark doping, the methods based on the mechanical impact on the modified surface are used (surface-plastic strain, running the ball, nonabrasive ultrasonic treatment, etc.) [2, 3], as well as treatment with concentrated energy flows (plasma flow, electron and ion beams, laser beams) [4, 5].

The paper is devoted to the analysis of the structure and properties of the surface layer of carbon steel subjected to electrospark doping and subsequent treatment with a high-intensity pulsed electron beam.

2. Material and methods of study
The material of the study was carbon steel 65G. Samples with dimensions of 25x25x50 mm were hardened at the temperature from 820 °C in oil and tempered at 350 °C. The electrospark treatment...
was carried out in air using installation ‘Elitron 22A’ at the discharge current of 2 A and the voltage on the doping electrode of 33 V. Technically pure tungsten was used as an electrode. The electron-beam treatment was performed on the basis of installation ‘SOLO’ [5]. The studies on the structure of the modified layer were carried out using methods of optical (NEOFOT-32) and scanning electron (SEM-515 Philips) microscopy, the X-ray diffraction analysis (diffractometer XRD 6000). The microhardness (PMT-3, the load on the indenter is 0.1 N) and the durability (Tribometer CSEM, Switzerland) were also measured.

3. Research results and their discussion

Doping of steel with tungsten allows a significantly improvement of its durability. This is due to the fact that doping of wolfram (W) increases the volume share of tungsten carbides during quenching at normal temperature. It is important to note that an increase in the volume fraction of tungsten carbides leads to formation of a finer grain and, therefore, to the improvement of mechanical properties, in particular higher viscosity.

In system Fe-W-C, the atoms of Fe and W belong to transition metals of VI$\text{A}$ group, and carbon is a nonmetal [9]. In system W-C, the formation of three compounds has been established: $\text{W}_2\text{C}$(β) (prototype Fe$\text{2}N$, space group (sp. gr.) $P63/mmc$), $\gamma\text{WC}_1\text{−}X$ (prototype NaCl, sp. gr. $Fm\overline{3}m$) and $\text{WC}$(δ) (prototype WC, sp. gr. $P6m2$) (Figure 1).

![Figure 1. Binary diagrams of systems Fe-W, Fe-C, WC [7] and the isothermal section of ternary system Fe-W-C at 1000 °C [8].](image)

In system Fe-C the following has been revealed: the formation of stable compound $\text{Fe}_3\text{C}$ (prototype Fe$\text{3}C$, sp. gr. $Pnma$) and solid solutions ($\alpha\text{-Fe}$) and ($\gamma\text{-Fe}$), and, at increased pressures, the stabilization of compound $\text{Fe}_7\text{C}_3$ (prototype Cr$\text{7}C_3$, sp. gr. $Pnma$) take place. Carbon is a nonmetal and belongs to group IV$\text{B}$ of the periodic table. Carbon atoms have four electrons on outer shells $s$- and $p$- $(2s^22p^2)$. As compared to the main alloy-forming metals, carbon atoms are much smaller and when alloyed with
metals they form interstitial phases \((R_C = 0.0916 \text{ nm}, \ \delta_R = (R_W - R_C)/R_W = 0.38 \text{ and} \ \delta_R = (R_W - R_C)/R_W = 0.28)\). Since treatment of the material with an electron beam may lead to formation of new metastable compounds, we shall briefly mention metastable iron-based carbides. Thus, in [7, 9] it has been found that higher carbides \(\text{Fe}_7\text{C}_3\) and \(\text{Fe}_2\text{C}\) stabilize at increased pressures. Carbide \(\text{Fe}_7\text{C}_3\) has hexagonal structure \(D10_1\) (prototype \(\text{Cr}_7\text{C}_3\), sp.gr. \(Pnma\)). Compound \(\text{Fe}_2\text{C}\) is metastable with a hexagonal structure (\(\epsilon\)-carbide, sp. gr. \(P6_3m\)) and is released during tempering of steel [10]. Moreover, [9] represents the data on the release of metastable carbides \(\eta\text{Fe}_2\text{C}\) (sp. gr. \(Pnm\)) and \(\chi\text{Fe}_5\text{C}_2\) (prototype \(B_2\text{Pd}_5\), sp. gr. \(C2/c\)), which are found during tempering of steel.

In system Fe-W the following has been discovered: three intermediate phases \(\lambda(\text{Fe}_2\text{W})\) (\(P6_3/mmc\), prototype \(\text{MgZn}_2\)), \(\mu(\text{Fe}_7\text{W}_6\) (prototype \(\text{Fe}_7\text{W}_6\), sp. gr. \(R\frac{3}{m}\)), \(\gamma(\text{FeW})\) (prototype \(\text{MoNi}\), sp. gr. \(P2_12_1\)), and solid solutions (W), (\(\alpha\)-Fe) and (\(\gamma\)-Fe). In this system, alloy-forming elements have size factor \(\delta_R = 0.139 (R_W = 0.148 \text{ nm}, R_C = 0.1274 \text{ nm})\), which is significantly less than 0.2; however, during alloying metals Fe and W form solid solutions with a very limited solubility. The solubility in W (\(\alpha\)-Fe) with a decrease in the temperature reduces from 14.3 % (at.) at 1548 °C up to 4.6 % (at.) at 1190 °C [7]. The solubility of Fe in (W) < 2.6 % (atm.) is at a temperature of 1677 °C [7].

In ternary system Fe-W-C a whole range of intermediate stable and metastable compounds with complex crystalline structures that exist in different temperature and concentration ranges has been discovered (Figure 1).

![Figure 2](image_url)

**Figure 2.** Binary diagrams of systems Fe-Ti, Fe-C, Ti-C [7] and the isothermal section of ternary system Fe-Ti-C at 1000 °C [12, 13].

Two intermediate phases, TiFe\(_2\) (structure \(C14\), prototype \(\text{MgZn}_2\), space group \(P6/mmc\)) and TiFe (structure \(B2\), prototype \(\text{CsCl}\), sp. gr. \(Pm\frac{3}{m}\)), are formed in system Fe-Ti (Figure 2). In regard to initial components, there are solid solutions based on Fe and Ti. Solid solutions based on Fe and Ti exhibit transformations which are caused by polymorphic transitions in pure metals Fe and Ti [7].

In system Ti-C there is one compound of carbide TiC(\(\delta\)) (structure \(B1\), prototype \(\text{NaCl}\), sp. gr. \(Fm\frac{3}{m}\)) with a wide range of homogeneity. Compound TiC(\(\delta\)) melts congruently at 3073 ± 25 °C. The
homogeneity range of phase TiC near the solidus phase occupies from 32 up to 50 % (at.) [7]. From a structural point of view, compound TiC(δ) is a solid solution of non-metal atoms in octa-interstitial sites of the FCC-lattice of metal [11]. Consequently, in the literature carbide TiC (δ) is often referred to as TiC_x.

Within the isothermal section of system Fe-Ti-C there are no ternary compounds formed based on binary compounds of corresponding binary systems. It is important to note that in system Fe-Ti-C there are no intermediate ternary compounds, as it occurs in ternary system Fe-W-C.

Electrospark doping of steel, as mentioned above, is accompanied by formation of a highly-defected surface layer containing microcraters and microcracks, as well as buildups of metal. Our case has also shown formation of a similar structure of the doping surface (Figure 3a and Figure 4a).

![Figure 3](image1.jpg)

**Figure 3.** The structure of the steel surface subjected to electrospark doping with tungsten (a) and the subsequent irradiation with a high-intensity pulsed electron beam (b).

The subsequent irradiation of the electroexplosive doping surface with a high-intensity electron beam leads to melting of the surface layer with a thickness of up to 30 microns. The doping surface smooths microcraters and buildups of metal are not registered (Figure 3b and Figure 4b). In some cases, microcracks are detected on doping surfaces. High-rate crystallization of the molten layer is accompanied by formation of a submicrocrystalline structure, the characteristic image of which is shown in Figure 3b and Figure 4b. It should be noted that average sizes of crystallites formed in the surface layer of steel are as follows: doped with tungsten – ≈ 300 nm; doped with titanium alloy – ≈ 900 nm (Figure 3 and Figure 4).

![Figure 4](image2.jpg)

**Figure 4.** The structure of the steel surface subjected to electrospark doping with titanium (a) and the subsequent irradiation with a high-intensity pulsed electron beam (b).
The elemental composition of the doped layer has been studied using the electron microprobe analysis method. The studies have shown that after electrospark doping with tungsten, the concentration of the alloying element in the steel surface layer was ≈ 7 at. %; after doping with titanium – (30…40) at. %. Additional irradiation of the modified layer with a high-intensity electron beam led to remelting of the surface layer. Consequently, the concentration of the doping element in the surface layer of steel decreased and in system Fe-W-C it amounted to (3…5) at. %; in system Fe-Ti-C it was equal to (10…18) at. %

Mechanical and tribological tests of the modified steel have been carried out. It has been established that formation in the surface layer of a multiphase nano- and submicrocrystalline structure leads to a substantial increase in mechanical and tribological properties of steel.

4. Conclusion
A thermodynamic analysis of phase transformations taking place during doping of steel with tungsten and titanium has been performed. It has been shown that doping of steel with carbide-forming elements results in formation of solid solutions, inclusions of carbide phases, and metal compounds. The study of the surface layer of steel subjected to electrospark doping has been carried out. Formation of a high-relief modified layer containing microcraters and microcracks has been revealed. It has been shown that irradiation of the doped layer with a high-intensity electron beam leads to smoothing of the modification surface and to formation of a submicron and nanocrystalline structure. It has been established that formation in the surface layer of a multiphase submicrocrystalline structure leads to a substantial increase in mechanical and tribological properties of steel.

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