Investigation of Structure and Phase Composition of Zone of The Pulse Electron-Beam Liquidphase Mixing of Coating-Steel System[1]

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Abstract – By the methods of metallography, scanning and transmission diffraction microscopy we conduct the investigations of the structure formed as a result of electron-beam treatment with microsecond pulse duration of steel with preliminary deposited on it TiN coating. It was shown that the electron-beam treatment is accompanied by modification of both coating and substrate. High-speed heating and cooling lead to hardening of steel layer abutting to the coating with the concurrent doping by titanium and nitrogen. Coating modification is accompanied by recrystallization process and damage of columnar structure.

1. Introduction

Electron-beam treatment of metals and alloys is one of the most flexible method of influence on phase composition and defective substructure of metals and alloys [1, 2]. The main advantage of this treatment method is a possibility of wide using of electrons energy, depth of their penetration to the material and accordingly wide variation of thermal fields dynamics. All that makes pulsed electron beams high effective tool as for purposeful improvement of production operating characteristics, as for investigations of processes carrying in the material in thermal impact conditions.

One of modification method of structure and phase composition and consequently physic-chemical mechanical characteristics of metals and alloys is doping of production surface layers by liquid-phase mixing of coating-substrate system. The aim of this paper was study of phase composition and defective substructure of zone of coating – substrate system liquid-phase mixing modification by methods of modern material science.

2. Experimental Procedure

As a coating used the TiN compound obtained by arc discharge plasma-assisted evaporation of the titanium cathode in an atmosphere of nitrogen. Coating synthesis was carried out with using of evaporators with arc current of 50–100 A and "PINK" plasma-generator, providing in deposition working space the nitrogen plasma with a concentration of 10^9–10^{10} cm^{-3}. The substrate was 4140 steel in ferrite-perlite state. The coating-substrate system liquid-phase modification was conducted by pulse-periodic electron-beam treatment on the "SOLO" installation in the regime of melting of surface layer with a thickness of ~5μm (E_S=30 J/cm^2; τ=100 μs, N=1 pulse.). The investigations of phase composition and defective substructure were carried out by methods of metallography (device OLYMPUS GX71 with a DP70 digital camera), scanning electron microscopy (SEM-515 "Philips") and diffraction electron microscopy (ЭМ-125). The material mechanical characteristics were investigated by determination of microhardness on the ПМТ-3 device.

3. Results and Discussion

3.1. 4140 steel phase state and defective substructure before the irradiation

The structure of 4140 steel samples before the irradiation was formed due to anneal at temperature of 850 °C during two hours and following slow cooling with an oven. Metallographic and electron-microscopic investigations showed that due to this treatment the structure consisting of ferrite and perlite grains mainly of lamellar morphology (Fig. 1) is formed.

Ferrite grains and interlayer are defective- in both cases is observed a dislocation substructure in the form of chaos and/or networks with a dislocation scalar density of ~3,8·10^{10} cm^{-2} for ferrite grains and ~0,6·10^{10} cm^{-2} for perlite grains. Cementite plates are also defective: there are ferrite bridges, cementite plates are curved and have a variable thickness.

The formation of ferrite-perlite structure leads to formation of globular morphology cementite particles, located along ferrite and perlite grains boundaries, and in grain boundary junctions. Particle sizes are the micrometer tenth parts.

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3.2. The structure of coating before the electron-beam treatment

The TiN coating was formed at conditions of simultaneous deposition of gas (nitrogen) and metal (titanium) component plasma generated by low pressure discharges. As a result of deposition the TiN coating with a thickness of (1–2) m was formed on the steel surface. As rule formed coatings has a columnar structure with an open growth texture identifiable by SEM and TEM methods (Fig. 2). Crystallites sizes, which form coatings, change in range of 20–40 nm.

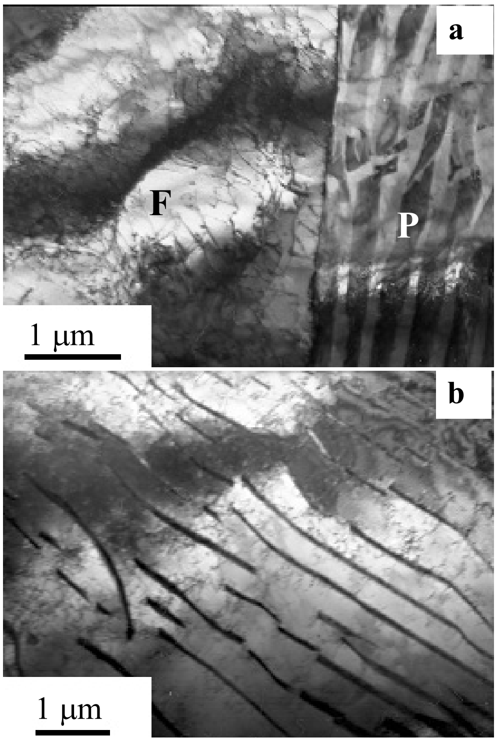

Fig. 1. TEM images of 4140 steel structure before the electron-beam treatment. F – ferrite grain; P – colony of lamellar perlite

3.3. The structure of 4140 steel after the electron-beam treatment

The electron-beam treatment of coating /substrate system (TiN/Steel) leads to numerous changes of composite material. First of all we will consider the modification of 4140 steel substrate phase composition and defective substructure. As it was mentioned above the substrate was in polycrystalline state formed by structurally free ferrite and plate perlite grains. The irradiation leads to high-speed heating, melting, crystallization and cooling of steel surface layer. Small times of thermal influence didn't allow in full measure to pass leveling carbon diffusion, and it lead to formation of the structure consisting of ferrite grains (Fig. 3, *a*) and grains with a volume containing the formed martensite structure (Fig. 3, *b*). Martensite by the morphological principle is divided into lath (the main structural component) and plate twinned martensite. Dislocation substructure is fixed in martensite plates and ferrite grains. In the first case (martensite crystals) is observed dense networks with a dislocation scalar density of $\sim 1 \cdot 10^{11}$ cm^{-2}. In ferrite grains observed networks, band and subgrain dislocation structure. Dislocation scalar density changes in range of $(2{,}4–4{,}5) \cdot 10^{10}$ cm^{-2}.

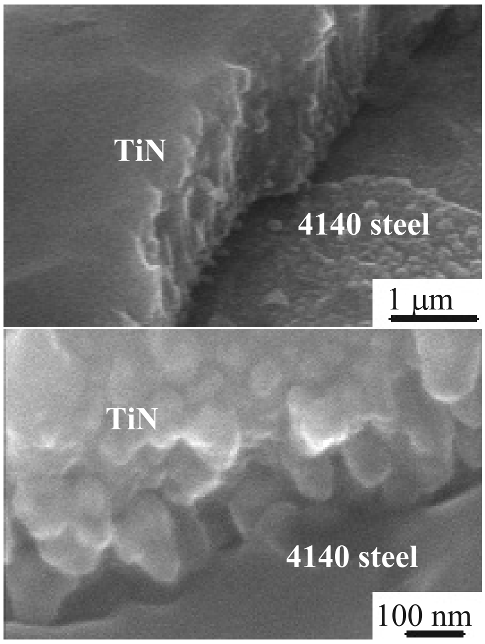

Fig. 2. SEM images of TiN coating structure before the electron-beam treatment

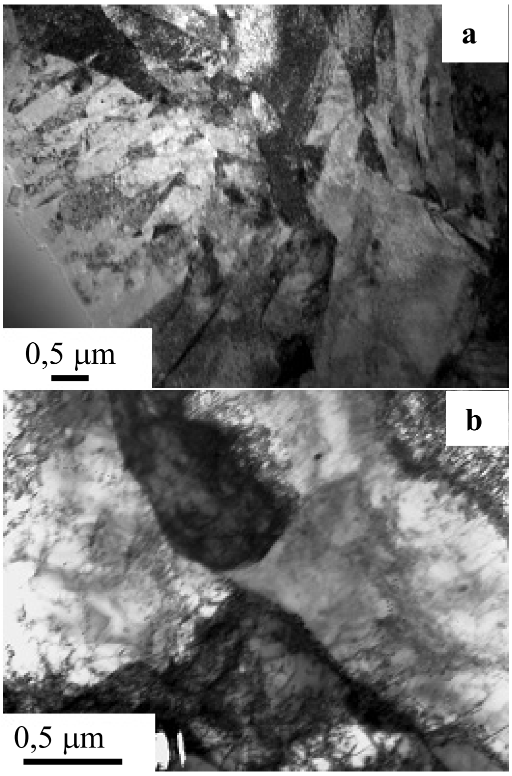

Fig. 3. TEM images of structure formed in 4140 steel as a result of the electron-beam treatment

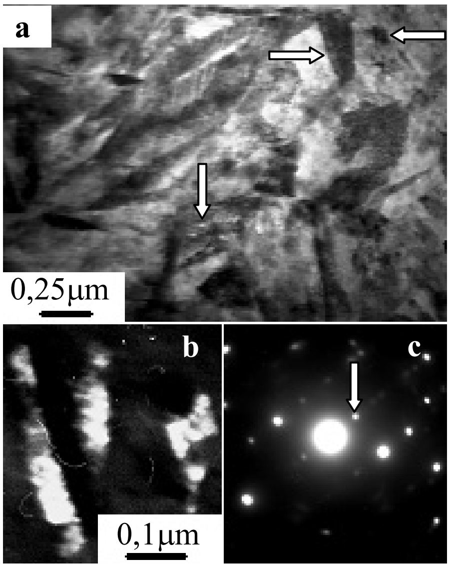

Fig. 4. TEM images of structure formed in 4140 steel as a result of the electron-beam treatment; a – bright field image; b – dark field image, obtained in [110]Ti_3N reflection; c – diffraction pattern. Arrows show: on (a) – regions containing titanium nitride particles; on (c) – dark field reflection

The electron-beam treatment leads to composition variety of steel carbide phase composition. If there was just iron carbide of Fe_3C (cementite) composition in the initial state, than after the irradiation the complete composition carbides are shown up, they contain chrome and iron atoms of $M_{12}C$ и $M_{23}C_6$, and titanium nitrides of Ti_3N composition (Fig. 4). $M_{12}C$ and $M_{23}C_6$ type carbides should be related to 4140 steel own carbides, and titanium nitride particles were formed due to steel doping by titanium and nitrogen from plasma formed as a result of evaporation and sputtering of coating taking place at electron-beam treatment of coating. At that a certain tendency is found out – titanium nitride particles have a plate form and form separate areas (subgrains).

3.4. Structure of TiN coating after electron-beam treatment

Electron-beam treatment has resulted to partial (in the centre of irradiation spot) damage of nitride coating (Fig. 5). It is shown that the coating is broken into separate fragments with sizes in range from 2 up to 50 μm (Fig. 5, *a*). Fragments in turn consist of areas with an average size of ~3,5 μm (Fig. 5, *b*). It is possible to show by TEM methods that areas are formed by crystals with sizes of ~20 nm, and it corresponds to the crystal sizes of coating initial state (Fig. 6). There are the crystallites with average sizes ~10 nm along region boundaries (Fig. 7). As rule the structure with two crystallite dimensional levels (10 and 20 nm) is emerged in coating layer abutting the substrate surface (4140 steel). This circumstance allowed expressing the next supposition. Crystallites formed in coating region are titanium nitrides of TiN composition; crystallites located along regions boundaries of coating can formed by titanium carbide of TiC composition. The premises for titanium carbide formation are – availability of carbon as in work chamber of installation, as carbon diffusion from steel volume initiated by high-temperature electron beam treatment. Interpretation of electron diffraction patterns obtained from these film region don't allow to separate the carbide and nitride phases reflections, that is caused by precision of TiN and TiC phases lattice parameters. However it should be mentioned that the reflections on the diffraction patterns have a visible broadening, and it can be caused by returns overlap from lattices of two phases (Fig. 7, *b*).

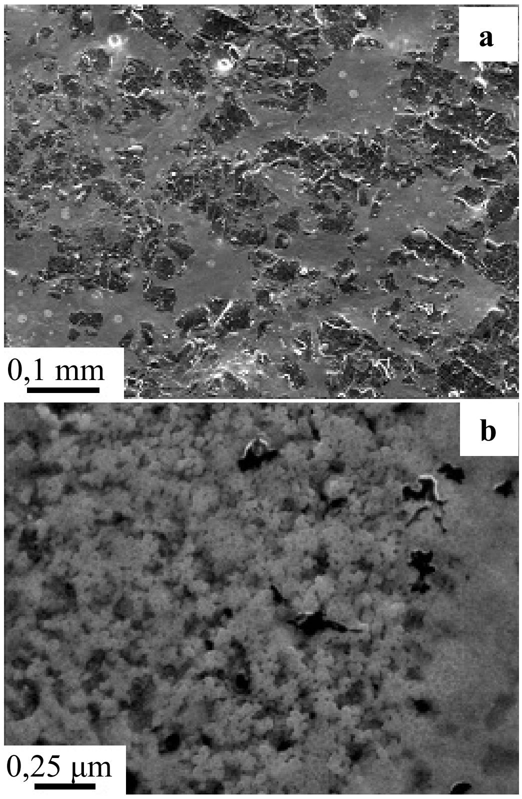

Fig. 5. SEM images of structure formed as a result of the electron-beam treatment of TiN coating

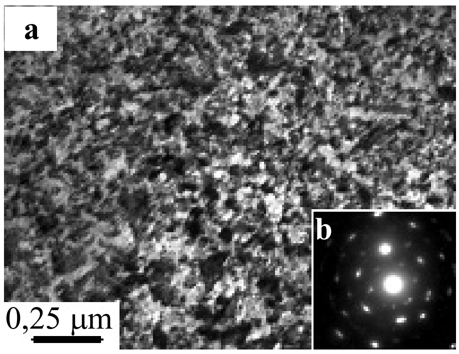

Fig. 6. TEM images of structure formed due to electron-beam treatment of TiN coating

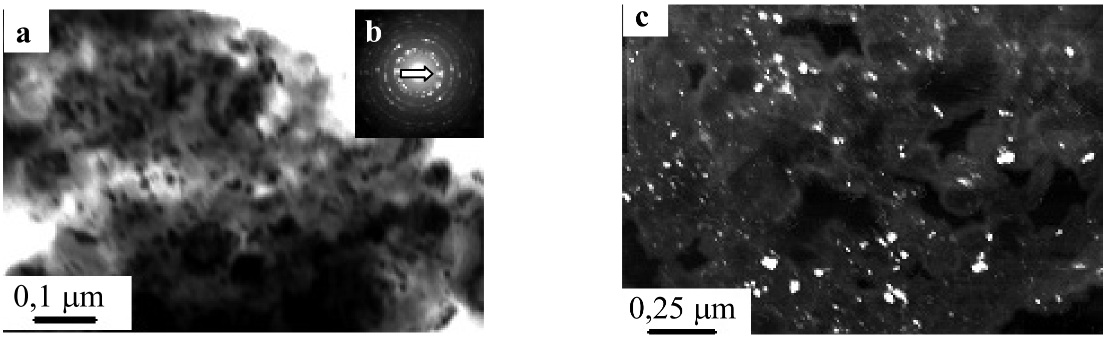

Fig. 7. TEM images of structure formed due to electron-beam treatment of TiN coating; a – bright field image; b –diffraction pattern (a dark field reflection is shown by the arrow); c – dark field image obtained in [110]TiN reflection

4. Conclusions

The results obtained in this work show that electron-beam treatment of coating/steel substrate composition is accompanied by steel allowing by coating elements, damage of coating columnar structure, separation of the second phase particles. This phase is conceivably carbide phase.

References

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