

## Phase Composition and Defect Substructure of Strengthening Layer Surfaced on Low Alloyed Steel

Yurii F. Ivanov<sup>1,2,a</sup>, Victor E. Gromov<sup>3,b\*</sup>, Sergey V. Konovalov<sup>4,c</sup>,  
Vassily E. Kormyshev<sup>3,d</sup>, Rongshan Qin<sup>5,e</sup>, Olga A. Semina<sup>3,f</sup>

<sup>1</sup>Tomsk National Research State University, Tomsk, 634050, Russia

<sup>2</sup>Institute of High-Current Electronics of Siberian Branch of Russian Academy of Sciences, Tomsk, 634021, Russia

<sup>3</sup>Siberian State Industrial University, Novokuznetsk, 654007, Russia

<sup>4</sup>Samara National Research University, Samara, 443086, Russia

<sup>5</sup>School of Engineering and Innovation, The Open University, Walton Hall, Milton Keynes MK7 6AA, UK

<sup>a</sup>yufi55@mail.ru, <sup>b</sup>gromov@physics.sibsiu.ru, <sup>c</sup>ksev@ssau.ru, <sup>d</sup>89236230000@mail.ru,  
<sup>e</sup>Rongshan.Qin@open.ac.uk, <sup>f</sup>syomin53@gmail.com

\*corresponding author

**Keywords:** nanostructure, phase composition, microhardness, electro arc surfacing, eutectic, boron carbides, niobium carbides.

**Abstract.** The microstructure and microhardness distribution in surface of low carbon Hardox 450 steel coated with alloyed powder wires of different chemical compositions are studied. It is shown that the microhardness of 6-8 mm thickness surfaced layer exceeds that of base metal by more than 2 times. The increased mechanical properties of surfaced layer are caused by the submicro and nanoscale dispersed martensite, containing the niobium carbides Nb<sub>2</sub>C, NbC and iron borides Fe<sub>2</sub>B. In the bulk plates a dislocation substructure of the net-like type with scalar dislocation density of 10<sup>11</sup> cm<sup>-2</sup> is observed. The layer surfaced with the wire containing B possesses the highest hardness. The possible mechanisms of niobium and boron carbides formation in surfacing are discussed.

### Introduction

Using of surface coatings opens up new possibilities to manipulate materials with requireable properties in particular environments. Protective coating layer can be achieved using thermal spraying, cladding and welding [1-3]. Among all the available methods, welding is considered to be an economical one as a variety of processes can be implemented to deposit the desired coatings. In most cases, a wire is used as an electrode or filler to form a protective layer on the surface of a base metal.

Among the weld cladding procedures, gas tungsten arc (GTA) surfacing process is a cost-effective approach applied when reactive materials (as coatings or substrates) are involved [4]. GTA surface modification by means of alloying is a process in which an alloy powder of a desirable composition and a thin surface layer of the substrate material are simultaneously melted, and then rapidly solidified to form a dense coating metallurgically bonded to the base material [5]. The solidification rate is very high during coating in this process. Moreover, the coated surface obtained by GTA technique has a potential to produce rapidly solidified fine microstructures that has high hardness and wear resistance synthesized on various traditional substrate materials [6].

The effects of GTA processing were used for developing wear resistance AISI 1020 steel substrate. Appropriate quantities of FeB powder and Hardox 450 steel were combined to create conditions that synthesize particles into reinforced Fe-based composite surface coating [7]. The studies concluded that Hardox 450 steel + 40 wt % FeB composite coating was the most appropriate combination in terms of hardness and wear performance [7].

Hardox 450 steel is used in order to generate desirable wear resistance and excellent cold bending property and weldability. High hardness Hardox 450 steel is obtained by quenching a sheet with special composition to generate martensite structure. To improve its technological properties further, the steel underwent additional modification including surface processing for obtaining the demanded properties [8-12].

The purpose of this research was to analyze the phase composition, defect substructure and mechanical properties of the layer surfaced on Hardox 450 steel with surfacing wire of different compositions.

### Material and Methods of Research

Hardox 450 steel with composition listed in Table 1 was used as base material. The wires for surfacing have composition shown in Table 2.

Table 1. Elemental composition of Hardox 450 steel (chemical composition of metal, wt%, balance Fe).

C	Si	Mn	Cr	Ni	Mo	B	P	S
0.23	0.70	1.60	0.25	0.25	0.25	0.004	0.025	0.010

Table 2. Elemental composition of electrode wire (wt%, balance Fe).

Wire mark	Elemental composition								
	C	Mn	Si	Cr	Nb	W	Ni	B	V
1	1.3	0.9	1.1	7.0	8.5	1.4	0.0	0.0	0.0
2	0.7	2.0	1.0	0.0	0.0	0.0	2.0	4.5	0.0
3	1.4	0.0	0.0	7.0	8.0	1.2	0.0	0.0	1.0

The formation of surfaced layer was processed in atmosphere gas (98%Ar and 2%CO<sub>2</sub>), welding current 250-300 A and arc voltage 30-35 V. The mechanical properties of the surface layer were determined using microhardness (device PMT-3, Vickers method) at indenter load of 5 N. The microstructure of the material was studied by optical microscopy (OM, Microvisor  $\mu$ Vizo-MET-221) and scanning electron microscopy (SEM, SEM-515 Philips). The elemental composition of the surface layer was determined by X-ray micro-spectrum analysis (EDAX ECON IV attached to the SEM). The phase constitution of the surface layer was analyzed using X-ray diffraction (XRD-7000s, Shimadzu, Japan). The defect substructure of Hardox 450 steel and the surfaced layer was analyzed using transmission diffraction electron microscopy (TEM), (EM-125).

### Results and Discussion.

*Hardox 450 steel – initial state.* TEM reveals the presence of martensite lamellar crystals. It is a result of shear  $\gamma \rightarrow \alpha$  transformation [13].  $\alpha$ -phase is iron-based solid solution with bcc crystal lattice. The  $\alpha$ -phase crystals are fragmental and separated into weakly disoriented. A small amount of sub-grains is present in the material. Some secondary phases are found inside the plates and also around its boundaries, at boundaries of fragments and sub-grains. Those particles are Fe<sub>3</sub>C according to the indexing of micro-electron diffraction patterns.

*Mechanical properties of the surfaced layer.* The microhardness profile of the steel surfaced layer was measured. The data plotted on Fig. 1 shows that as a result of surfacing formation (independent of surfacing wire grade) a high-strength surface layer not less than 6 mm thick with average value of microhardness  $\approx$  11.2 GPa is formed. When moving farther away from the surfaced layer the microhardness of the material drops quickly into a level of  $\approx$  5.8 GPa. Consequently, hardness of surfaced layer exceeds that of the base metal (Hardox 450 steel) by 1.9 times in a

thickness of 6.0-8.0 mm. The highest hardness is the layer surfaced with wire № 2 shown as the curve 1 in Fig. 3. In this case the hardened layer thickness is not less than 7.5 mm. Its microhardness varies between 10.5 and 12.5 GPa.

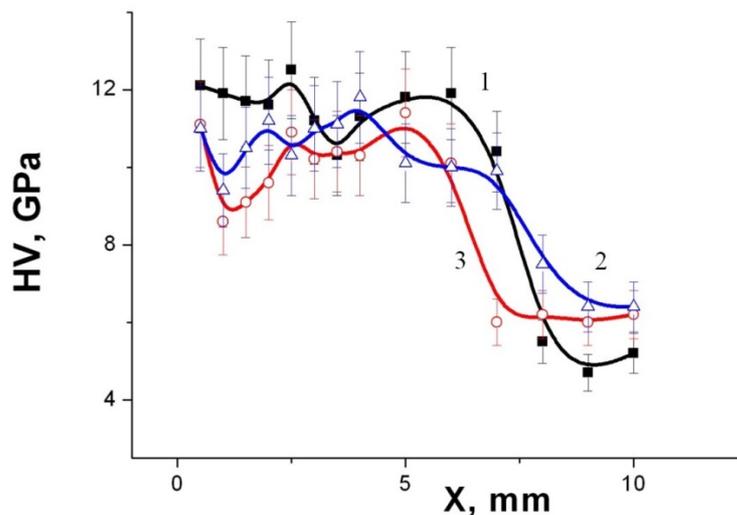


Fig. 1. Microhardness profile of “surfaced layer/(Hardox 450 steel) substrate”; curve 1 – surfacing wire №2; curve 2 – surfacing wire №3; curve 3 – surfacing wire №1.

*Phase constitution of the defect substructure in surfaced layer.* By analyzing of composition of surfacing wires presented in Table 2, it can be supposed that high strength properties of the layer surfaced with wire №2 are caused by hardening of the material with iron boride. Fe-B system contains the compounds of  $\text{Fe}_2\text{B}$ ,  $\text{FeB}$  and  $\text{FeB}_2$  [14].

The solid solution of boron in bcc (body-centered cubic) lattice of iron has microhardness of 3700 MPa, modified structure  $\alpha\text{-Fe} + \text{Fe}_3\text{B}$  has 6300 MPa, eutectic structures of dendrite in commercially pure iron has 6000-16000 MPa and 40Cr steel 10000-16000 MPa. The boride structures in 40Cr steel have the following microhardness:  $\text{Fe}_2\text{B}$ : 16800 MPa,  $\text{Fe}_2\text{B}+\text{FeB}$ : 16800-18900 MPa,  $\text{FeB}$ : 18900-21000 MPa [15]. Therefore, the high strength properties of the layer surfaced on Hardox 450 steel with wire №2 may be caused by the formation of eutectic on the base of iron boride  $\text{Fe}_2\text{B}$ . SEM study of the surfaced layer reveals the formation of lamellar type eutectic.

The effect of boron on the properties of iron is similar to that of carbon but is several times stronger. The addition of boron in the quantity of 0.02% (at.) facilitates the refinement of iron grain and gives possibility to use thermal treatment (quenching) for increasing its properties [15]. The results in support of the given thesis were obtained by us when analyzing  $\alpha$ -phase being formed in eutectic. Fig. 2 shows the characteristic image of surfaced layer metal demonstrating the structure of  $\alpha$ -phase interlayer. The lamellar structure is clearly defined. The transverse size of plates varies between 30 and 70 nm. A dislocation substructure of net-like type is observed in the volume of plates. Judging from the size of dislocation net cells the dislocation scalar density amounts to  $10^{11} \text{ cm}^{-2}$ . The high dislocation density and lamellar morphology of interlayer structure are indicative of the shear mechanism of  $\alpha$ -phase formations with production of ultra-fine martensite structure. Notice for comparison that the average transverse sizes of plates of packet martensite vary within 150-200 nm in quenched steel, the transverse ones of lamellar martensite crystals reach units of micrometers [16].

The less strong structure of the surfaced layer is formed on Hardox 450 steel in application of surfacing wire №3. Hardness of surfaced layer exceeds  $\approx 1.7$  fold that of the base metal (Hardox 450 steel) with the thickness of surfaced layer up to 8.0 mm.

When analyzing the data presented in Table 2, it may be supposed that the high strength properties of surfaced layer are caused by the strengthening of the material with niobium carbides. System Nb-C is characterized by the availability of Nb-based solid solution, two stable intermediate phases  $\text{Nb}_2\text{C}$  and  $\text{NbC}$ , and meta-stable phase  $\text{Nb}_3\text{C}_2$  [14].

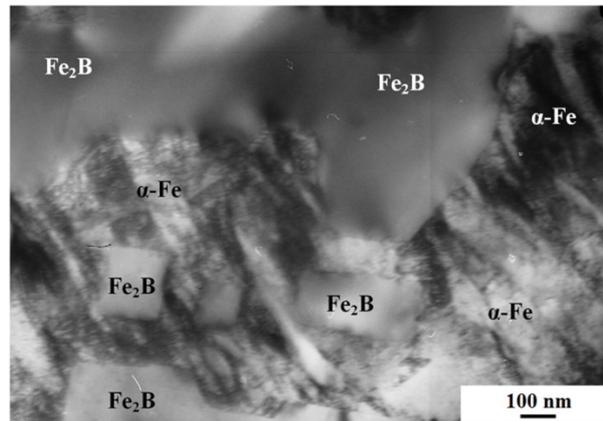


Fig. 2. Electron microscope image of structure of the layer surfaced with wire №2 on Hardox 450 steel.

The studies of the layer surfaced with wire №1 and №2 showed that its high strength properties were caused by the formation of quenched structure (martensite) and precipitation of the second phase particles as well. The characteristic feature of the quenched structure is a coarsening (in comparison with the structure of the layer surfaced with wire №2) martensite structure. The transverse size of which varies within 100-200 nm.

Indexing of micro-electron diffraction patterns obtained from martensite structure enabled to reveal the presence of retained austenite ( $\gamma$ -phase, solid solution on base of face-centered crystal lattice of iron). The retained austenite in the form of extended interlayers locates along the boundaries of martensite crystals.

The main ordered phase of the layer surfaced with wire №1 is niobium carbide particles. The size of inclusions vary within 0.2-1.5  $\mu\text{m}$ . The shape of niobium carbide particles is very diverse and it is determined by the place of particle location. In the volume of grains the niobium carbide particles have, largely, faceted shape (Fig. 3, a); along the boundaries the particles form the extended interlayers (Fig. 3, b); in the grain boundary junctions they are shaped into triple extended node (Fig. 4). In some cases, the reflections belonging to carbides of chromium and tungsten are revealed in micro-electron diffraction patterns.

The layer formed with surfacing wire №3 contains carbides of complex composition  $\text{M}_{23}\text{C}_6$  as a strengthening phase along with niobium carbide NbC. Following the results presented in Table 2 one might expect that carbide under discussion is formed by atoms of chromium, iron and tungsten and it has a composition  $(\text{Cr}, \text{Fe}, \text{W})_{23}\text{C}_6$ .

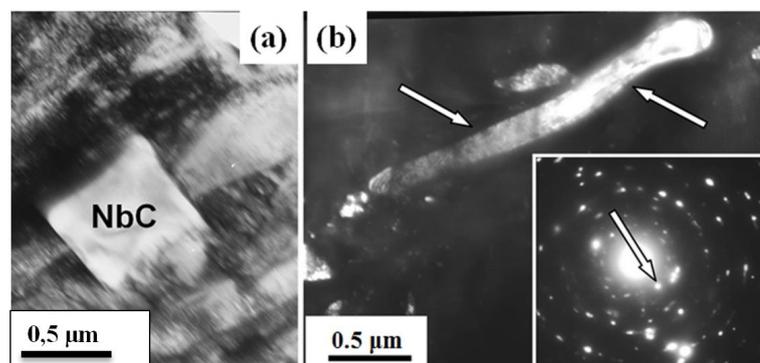


Fig. 3. Electron microscope image of surfacing structure applied with wire №1 on Hardox 450 steel; a – light field; b – dark field obtained in reflection [002] NbC (it is designated by the arrow in micro-electron diffraction pattern). In (b) niobium carbide is designated by arrows.

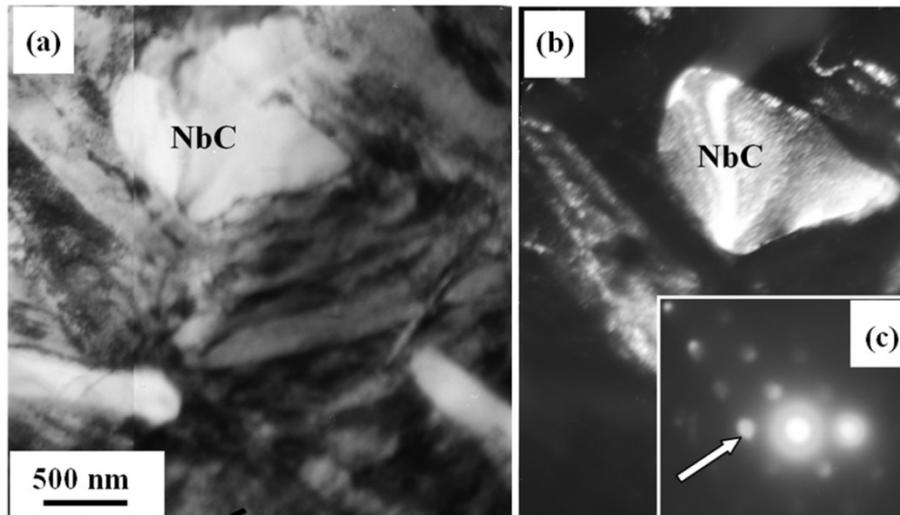


Fig. 4. Electron microscope image of surfacing structure applied with wire №1 on Hardox 450 steel; a – light field; b – dark field obtained in closely located reflections  $[002] \text{NbC} + [110] \alpha\text{-Fe}$  (the reflections are designated with arrow in (c)); c – micro-electron diffraction pattern, the arrow designates the reflections in which dark field was obtained.

## Conclusion

The investigation of phase composition, defect substructure and mechanical properties of the surface layer of Hardox 450 steel surfaced with surfacing wire №1, №2 and №3 in a single pass are performed. It is shown that the highest hardness is the one surfaced with wire №1. Its strengthened layer thickness is not less than 7.5 mm and its microhardness varies between 10.5 and 12.5 GPa. It is found that the increased mechanical properties of the surfaced layer are connected to the formation of multi-phase submicro and nanoscale structures. The strengthening is caused by the quenching-induced formation of ultra-fine martensite and the presence of the inclusions mainly of iron boride  $\text{Fe}_2\text{B}$  of submicron size and the formation of the eutectic of the lamellar structure (surfacing wire  $N > 2$ ). Niobium carbides NbC,  $\text{Nb}_2\text{C}$  and complex carbides are the strengthening particle for surface wires 1 and 3.

## Acknowledgement

Financial support of the research was provided in part under grant of Russian Science Foundation (project №15-19-00065).

## References

- [1] M. Kirchgaßner, E. Badisch and F. Franek, Behaviour of iron-based hardfacing alloys under abrasion and impact, *Wear*, 265 (2008) 772–779.
- [2] P. F. Mendez, N. Barnes, K. Bell, S. D. Borle, S. S. Gajapathi, S. D. Guest, H. Izadi, A. K. Gol and G. Wood, Welding processes for wear resistant overlays, *J. Manuf. Process.* 16 (2014) 4–25.
- [3] R. Zahiri, R. Sundaramoorthy, P. Lysz and C. Subramanian, Hardfacing using ferro-alloy powder mixtures by submerged arc welding, *Surface and Coatings Technology*, 260 (2014) 220–229.
- [4] W. Wu, L. Y. Hwu, D. Y. Lin and J. L. Lee, The relationship between alloying elements and retained austenite in martensitic stainless steel welds, *Scripta Materialia* 42 (2000) 1071–1076.
- [5] O. Yilmaz, M. Özenba and S. Buytoz, Microstructural characteristics of gas tungsten arc synthesised Fe-Cr-Si-C coating, *Mater. Sci. Technol.* 18 (2002) 1209–1216.

- 
- [6] A. Agarwal and N. B. Dahotre, Comparative wear in titanium diboride coatings on steel using high energy density processes, *Wear*, 240 (2000) 144–151.
- [7] T. Teker, S. Karatas and S. Osman Yilmaz, Microstructure and wear properties of AISI 1020 steel surface modified by HARDOX 450 and FeB powder mixture, *Protection of Metals and Physical Chemistry of Surfaces*, 50 (2014) 94–103.
- [8] E.V. Kapralov, S.V. Raikov, E.A. Budovskikh, V.E. Gromov, E.S. Vaschuk and Yu.F. Ivanov: Structural phase states and properties of coatings welded onto steel surfaces using powder, *Bulletin of the Russian academy of sciences. Physics*, 78 (2014) 1015–1021.
- [9] S.V. Raikov, E.V. Kapralov, Yu.F. Ivanov, E.A. Budovskikh and V.E. Gromov, Structure gradient in wear-resistant coating on steel, *Steel in Translation*, 45 (2015) 120–124.
- [10] S.V. Raikov, E.V. Kapralov, E.S. Vaschuk, E.A. Budovskikh, V.E. Gromov, Yu.F. Ivanov, A.D. Teresov and K.V. Sosnin, Structure of the surface layer of a wear-resistant coating after treatment with a high-intensity electron beam, *Journal of Surface Investigation. X-ray, Synchrotron and Neutron Techniques*, 9 (2015) 934–938.
- [11] N.A. Popova, E. Nikonenko, Yu. F. Ivanov, V.E. Gromov, E.A. Budovskikh, S.V. Raikov, E.V. Kapralov and E.S. Vaschuk, Structure and properties of wear-resistant weld deposit formed on martensitic steel using the electric-arc method, *Advanced Materials Research*, 1013 (2014) 194–199.
- [12] E.V. Kapralov, S.V. Raikov, E.S. Vaschuk, E.A. Budovskikh and V.E. Gromov, Structural-phase states and wear resistance of surface formed on steel by surfacing, *AIP Conference Proceedings*, 1623 (2014) 233–236.
- [13] V.G. Kurduymov, L.M. Utevskii and R.I. Entin, *Transformation in iron and steel*, Nauka, Moscow, 1977.
- [14] O.A. Bannykh, P.B. Budberg and S.P. Alisova, *State diagrams of binary and multi-component systems on base of iron*, Metallurgiya, Moscow, 1986.
- [15] V. Biryukov, Modification of surface with the help of laser radiation, *Fotonika*, 3 (2010)18–21.
- [16] Yu.F. Ivanov, E.V. Kornet, E.V. Kozov and V.E. Gromov, *Quenched structural steel: structure and mechanisms of hardening*. SibSIU, Novokuznetsk. 2010.