Additional heat treatment of non-porous coatings obtained on medium carbon steel substrates by electron beam cladding of a 'Ti-Mo-C' powder composition

D O Mul, E A Drobyaz, T A Zimoglyadova, V A Bataev, D V Lazurenko and L I Shevtsova
Novosibirsk State Technical University, 20, Karla Marksa st., Novosibirsk, 630092, Russia

E-mail: ddariol@yandex.ru

Abstract. The structure and microhardness of surface layers, obtained by non-vacuum electron beam cladding of Ti-Mo-C powder mixture on a steel substrate after different types of heat treatment, were investigated. After cladding samples were heat treated in a furnace at 200…500 °C, as well as quenched at 860 ° C and then underwent high-temperature tempering. Heat treatment of cladded coatings induced tempering of martensite and precipitation of cementite particles (Fe_3C). Transmission electron microscopy of the samples after heating and holding at 300 ° C revealed precipitation of nanosized cubical TiC particles. The formation of hard nanosized particles led to the surface layer microhardness growth. The highest level of microhardness (which was 1.2…1.5-fold higher in comparison with coating microhardness after heat treatment) was achieved after heating of the claded material at 300 °C and 400 °C Additional quenching of samples at 860 °C did not increase the microhardness level.

1. Introduction

Recently the surface-modified metallic materials fabricated by using high-energy laser, electron or ion beams have been widely investigated [1-6]. Irradiation of metallic surfaces covered with powders (carbides, borides or carbide forming metals) by a high-energy beam leads to the formation of a melt, where added or synthesized ceramic compounds are distributed. After crystallization of such melt the structures, which provide enhanced microhardness and wear resistance of cladded materials compared to a substrate, can be formed. Subsequent heat treatment contributes to improvement of the structure and properties of cladded layers obtained on steel surfaces by cladding. Residual internal stresses can be relieved by temper of materials obtained by the methods of cladding [7, 8]. Moreover, quenching and temper of surface-hardened workpieces can be applied to improve the structure and properties.

It is well-known that temper of quenched steels alloyed with tungsten, vanadium and molybdenum at 500…600 °C is accompanied by precipitation of fine specific carbides. The decrease of the alloying degree of retained austenite provides a more complete martensitic transformation. Secondary hardening of martensite contributes to increase of hardness and thermal resistance of materials used at the temperatures up to 500 °C.

The current investigation is aimed at revealing the mechanisms of improvement of surface properties of mild-carbon steels treated by electron beam followed by heating in the temperature range between 200 and 500 ^{o}C, as well as post-quenching at 860 °C and subsequent temper at 500 °C.

2. Materials and methods

At the preliminary stage the workpieces with a size of 16×100×50 mm were cut out of the mild-carbon steel (0.41 % C, 1.11 % Cr, 0.86 % Mn, 0.32 % Si, 0.02 % S, 0.03 % P). Further these workpieces were covered by a powder mixture consisted of titanium, molybdenum, graphite and a flux (CaF_2 + LiF). The weight ratio of the aforementioned components was as follows: 41.8:4.4:13.8:33.3:6.7. Surface density of a powder mixture was 0.33 g/cm^2. Electron beam cladding was carried out using the electron accelerator ELV-6 (Novosibirsk). A beam energy value was 1.4 MeV. The distance from the outlet to a workpiece was 90 mm. The diameter of the electron beam correspondng to this distance was 12 mm. A workpiece was moved relatively the outlet with a rate of 10 mm/s. An increase of a treated square was reached by applying a scanning regime. The electron beam scanned a surface in the range of the workpiece width (50 mm) with a frequency of 50 Hz. A current beam was 28 mA.

Heat treatment of samples with coatings was carried out in a SNOL7.2/1300 muffle furnace. Heat addition was carried out at the temperatures of 200 °C, 300 °C, 400 °C and 500 °C during three hours, and oil quenching was conducted at 860 °C with subsequent drawing-back at 500 °C.

The structure of obtained materials was studied by the methods of optical microscopy, scanning electron microscopy (SEM) and X-ray diffraction (XRD) analysis. Metallographic investigations were carried out using an Axio Observer Z1m optical microscope. The study of the structure of the cladded and heat treated layers at higher magnifications and estimation of the elemental composition of local microvolumes of materials was carried out using a Carl Zeiss EVO 50 XVP scanning electron microscope equipped with a EDX X-Act microanalyzer. Transmission electron microscopy (TEM) was carried out using a FEI Tecnai G2 20 TWIN microscope. The phase composition of coatings was investigated by an ARL X'TRA diffractometer.

3. Results and discussion

3.1. The structure of cladded materials after heat treatment

Cladding of titanium, molybdenum and graphite on a steel workpiece led to the formation of a non-porous modified layer with a thickness of 2.4 mm (Figure 1).

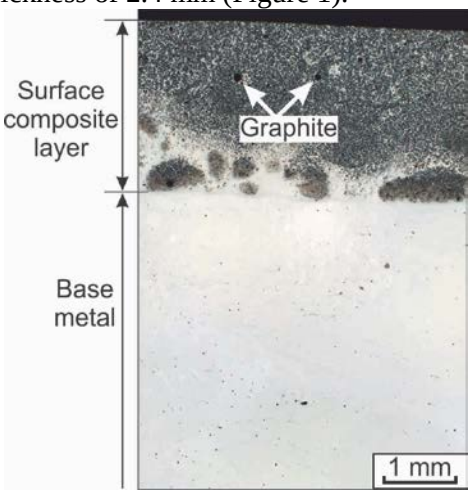

Figure 1. Cross section of samples after non-vacuum electron-beam cladding.

During the cooling of the cladded material the segregations of carbides were formed in the microvolumes enriched by titanium and carbon. These conglomerates appear as black spots on the bright background in the microphotograph. Moreover, in the cross section of the coating a small number of microvolumes with a size of 50 μm (or less) corresponded to undissolved graphite was revealed. The more detailed investigations showed that the structure of the coating was represented by a martensitic matrix, precipitated carbide particles (TiC) and retained austenite (Figure 2a). The volume fraction of carbides in the coating was equal to 30 %. The formation of such a matrix was

caused by the chemical composition of the material and crystallization conditions. A cooling velocity of the material was about 200…1000 °C per second. Carbon content in the coating was higher than 2 % (taking into account the losses occurring during the electron beam treatment). Such carbon percentage in combination with alloying elements caused formation of the enhanced quantity of retained austenite.

Quenching at 860 °C with subsequent high tempering induced the formation of a fine martensitic structure with retained austenite and a lot of carbide particles in the coating (Figure 2b).

Heating a cladded material in the temperature range between 200 and 500 °C during three hours was accompanied by martensite temper (Figure 2c-f). Wherein the structure of retained austenite was also transformed and a carbide phase was modified partially. White crystals were precipitated in the coatings. Many of them had a lamellar shape (in Figure 2 these particles are shown by arrows). The size of these precipitations equaled to 10…15 μm. The temperature rise induced the increase of the thickness of lamellar particles.

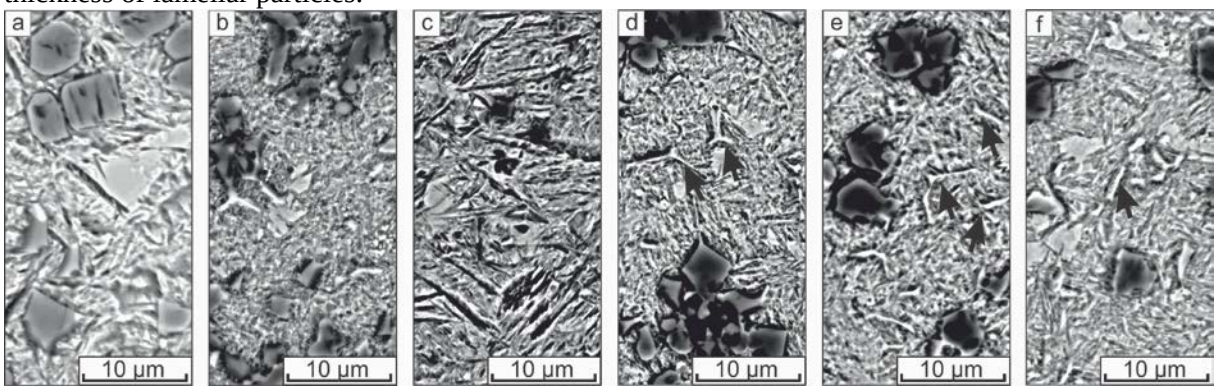

Figure 2. Scanning electron microscopy of the surface layer after cladding (a), after cladding and quenching at 860 °C with subsequent temper at 500 °C (b), after cladding and heating at 200 °C (c), 300 °C (d), 400 °C (e), 500 °C (f).

XRD analysis of heat treated coating revealed the presence of the Fe_3C phase (Figure 3). The austenite amount in the coating after additional heating was significantly reduced which was evidenced by decrease of the corresponding diffraction maximums. The same conclusion can be made based on the results of the metallographic analysis (Figure 2). Cementite precipitation is associated with the processes of austenite and martensite decomposition.

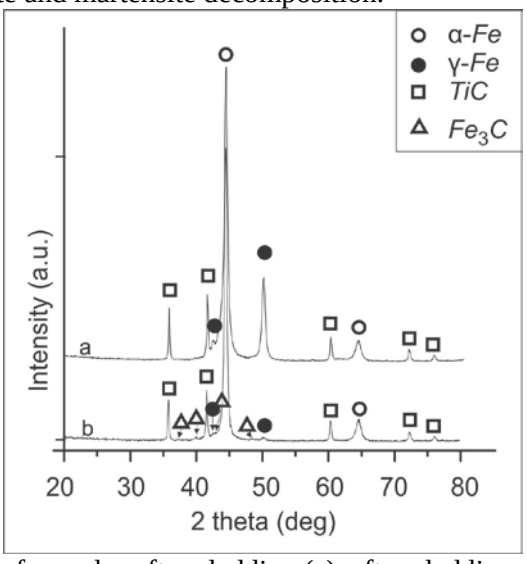

Figure 3. XRD analysis of samples after cladding (a), after cladding and heating at 300 °C (b)

A more sophisticated and nuanced view of the structure and a phase composition of the cladded layer before and after heat treatment at 300 °C was obtained by the electron diffraction method (Figure 4). The diffraction analysis showed that the matrix structure of the coating obtained by the electron beam treatment was represented by the mixture of martensite and retained austenite (Figure 4a, b). Titanium carbides precipitated during crystallization acted as a strengthening phase.

Additional heating at 300 °C during three hours led to the structural transformations of austenite accompanied by precipitation of the carbide particles of two types. Except the lamellar iron carbides, cubic-shaped carbide particles were revealed by TEM (Figure 4c, d). These particles distributed in *α*-Fe were characterized by the size of 5…10 nm and the phase composition of TiC.

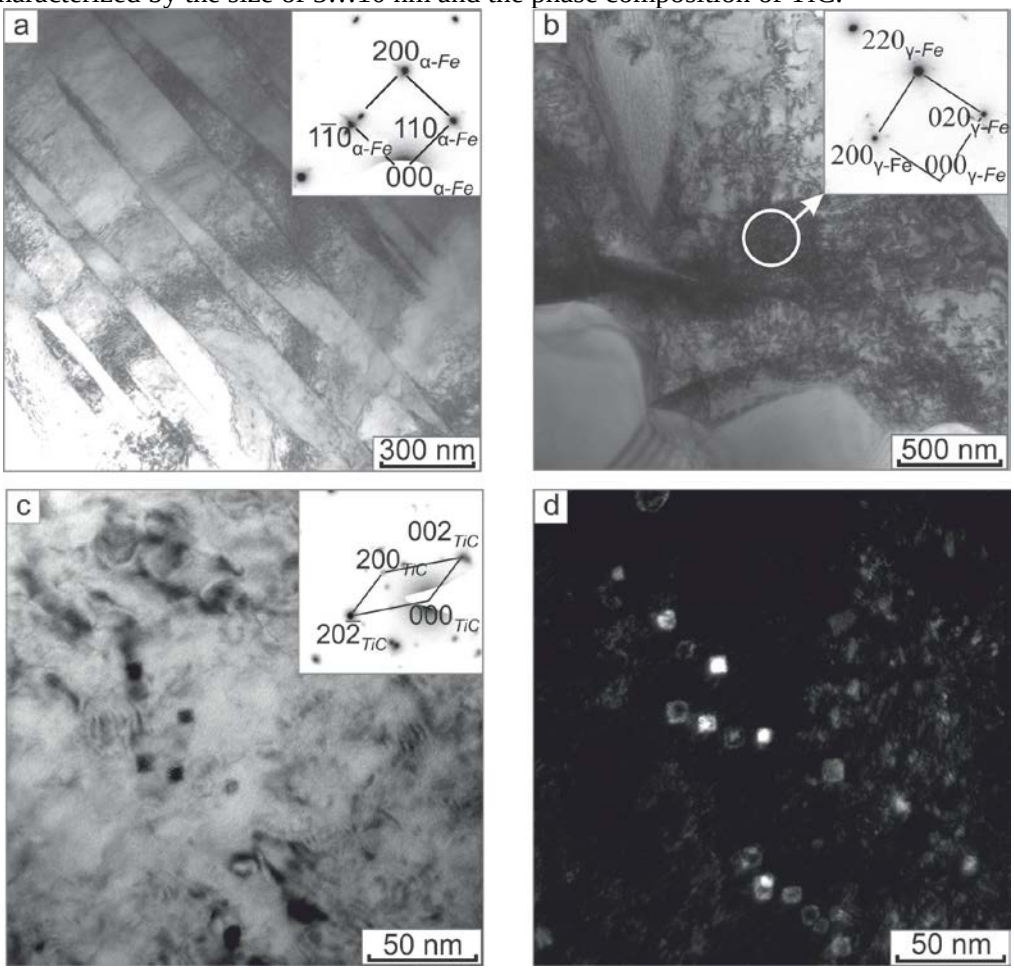

Figure 4. The fine structure of surface layers, obtained by cladding of titanium, molybdenum and graphite on steel substrate (a, b) after cladding followed by heating at 300 ° C (c, d), the dark-field image of carbide particles (d) obtained from electron-diffraction pattern is shown in Figure 5 (c) in 002 reflex.

3.2. Influence of heat treatment on microhardness of surface-hardened layer.

Microhardness of the cladded layer, heat affected zone and a base material of samples with cladded layers were measured after additional heat treatment of materials at 200 °C, 300 °C, 400 °C and 500 °C during three hours. The analysis of the results obtained revealed that the additional heating of materials at the temperatures of 300 °C and 400 °C induced 1.2…1.5-fold increase of microhardness in comparison with untreated coatings (Figure 5). A heat affected zone was characterized by the martensite temper. Increase of the temperature caused decrease of microhardness of these areas.

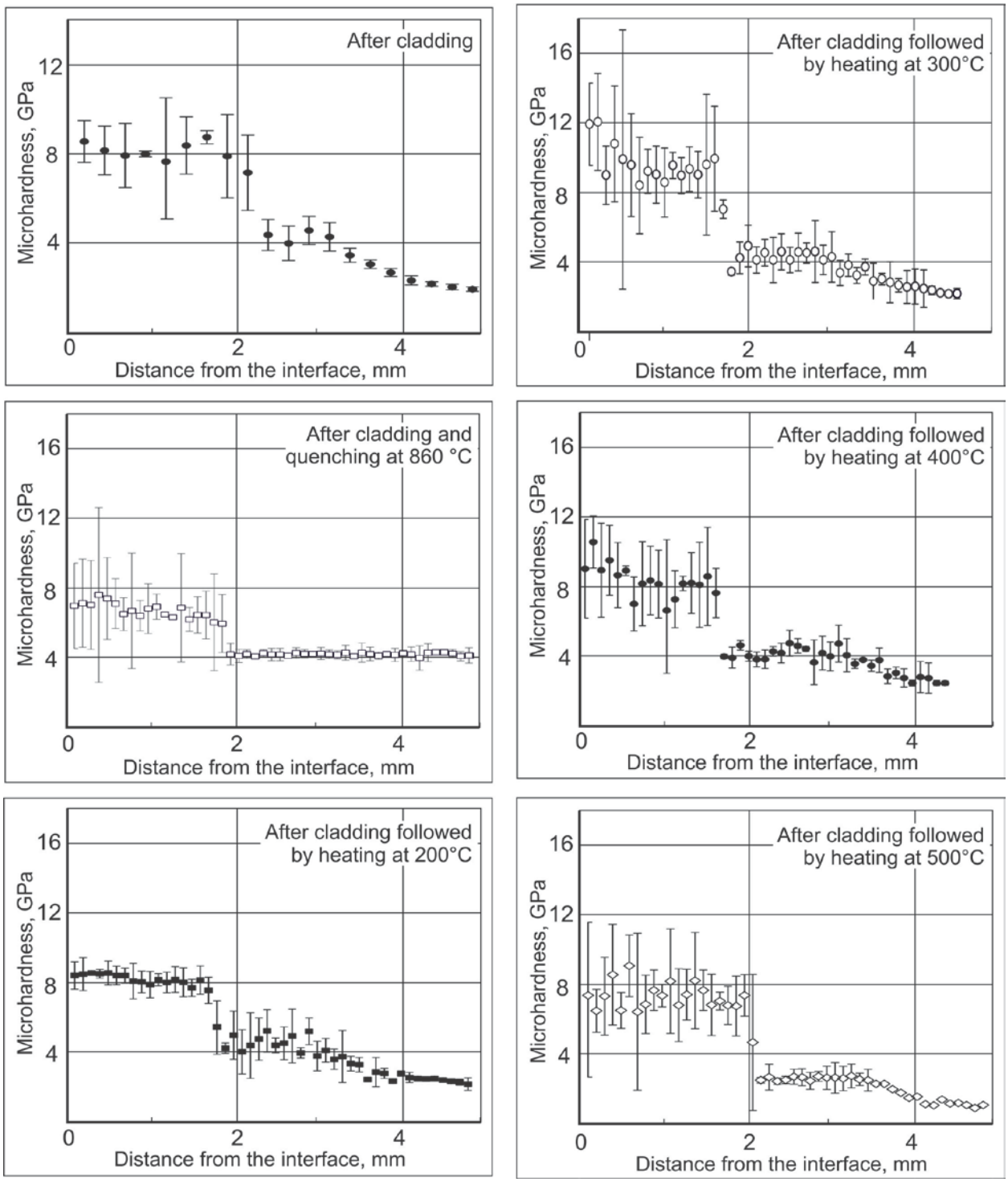

Figure 5. Microhardness distribution in depth of a layer, obtained by non-vacuum electron beam cladding of Ti-Mo-C powder mixture, after cladding and additional heat treatments.

The multiplicity formation of fine nanosized hard TiC particles possessed high hardness contributed in an increase of the cladded layer microhardness. The maximum microhardness level was reached after heating materials at 300…400 °C (Figure 5). The presence of hard nanosized TiC particles in the matrix can promote stabilization of a grain growth at higher temperatures.

The enhanced heating temperature (500 °C) initiated growth of the secondary carbides Fe_3C; the degree of the structure dispersity decreased and consequently microhardness of the coating dropped (Figure 5).

It should be emphasized that subsequent quenching of the cladded material from 860 °C did not induce increase of its microhardness (Figure 5). It can be explained by the fact that the temperature optimum for quenching of medium carbon steel (860 °C) does not allow forming homogeneous austenite in the cladded layer. The aforementioned temperature is corresponded to an intercritical temperature range for the cladded material.

4. Conclusions

The peculiarities of structural transformations of the surface alloyed layer obtained by non-vacuum electron beam cladding of the powder mixture consisted of titanium, molybdenum and graphite on the mild-carbon steel and additional heat treatments were investigated. The following characteristics of the coating heated at the temperature range between 200 and 500 °C were revealed:

Heating of the surface-hardened material at the temperatures from 200 to 500 °C was accompanied by precipitation of carbide particles of two types in the cladded layers. The first-type carbides had a lamellar shape; their length was 10…15 μm. The shape of the second-type carbides was cubic; the size was about 5…10 μm. XRD analysis and TEM investigations revealed that the phase composition of cubic particles corresponded to TiC and the lamellar particles were represented by cementite. Decomposition of martensite and retained austenite was the main factor contributed in the formation of carbides in the cladded layers.

Decomposition of the oversaturated solid solution occurring during the additional heat treatment of surface modified layer was accompanied by an increase of microhardness of the cladded material. The maximum microhardness growth (1.2…1.5-fold) was observed after heating the material at 300…400 °C during 3 h.

The presence of nanosized titanium carbide particles homogeneously distributed in the iron matrix can be considered as a factor, which induced stabilization of the grain growth in materials working at high temperatures.

Quenching of the obtained materials from the temperature of 860 °C and subsequent temper at 500 °C was accompanied by a decrease of the retained austenite volume fraction and precipitation of a large number of carbide particles in the cladded layer. It led to a decrease of the microhardness value from 8.5 GPa to 7 GPa.

Acknowledgements

This study was supported by the Russian Science Foundation (project No. 15-19-00230).

Reference

[1] Oh J C, Yun E, Golkovski M G and Lee S 2003 *Mater. Sci. Eng. A* **351** 98-108
[2] Yilbas B S, Patel F and Karatas C 2015 *Laser Surface Engineering Waugh J.L.G. Woodhead Publishing* 97-105
[3] Leyens C and Beyer E 2015 *Laser Surface Engineering Waugh J.L.G. Woodhead Publishing* 181-192
[4] Kusinski J et al 2012 *Bulletin of the Polish Academy of Sciences: Technical Sciences* **60** 711-728
[5] Nam D, Do J and Lee S 2009 *Scripta Mater.* **60** 695-698
[6] Golkovsky M G et al 2011 *Guangzhou* **314-316** 23-27
[7] Euh K et al 2003 *Mater. Sci. Eng. A* **346** 228-236
[8] Lee D G, Lee K and Lee S 2006 *Surf. Coat. Technol*. **201** 1296-1301