Synthesis of Ni-Al intermetallic surface alloys produced by using a low-energy high-current electron beam

E V Yakovlev, A B Markov, D A Shepel, A V Solovyov and V I Petrov

Tomsk Scientific Center SB RAS, 10/4 Akademicheskiy Ave., Tomsk, 634055, Russia

E-mail: yakov_e@mail.ru

Abstract. Ni-Al surface alloy electron beam synthesis results are presented. Surface alloy was formed on a carbon steel substrate by multilayer Ni-Al system deposition and subsequent one pulse low-energy high-current electron beam (LEHCEB) treatment in single vacuum cycle. It is shown that surface alloy containing mainly intermetallic phase NiAl is formed as a result of one pulse LEHCEB treatment. It is established that NiAl surface alloy microhardness and wear resistance are higher in comparison to steel substrate parameters.

1. Introduction

Carbon steels are widely used in industry for constructing, machines and parts due to its workability and relatively low price [1]. However it has also significant disadvantages low wear resistance, corrosion resistance and oxidation resistance [2]. Coating deposition is a method to improve this properties. Nowadays composite coatings made of high temperature intermetallic compounds are widely studied [1, 2]. NiAl compound combine metallic and ceramic properties, high melting temperature, thermal conductivity, oxidation resistance and high temperature corrosion resistance as well as low density [3]. Thereby intermetallic NiAl is suitable for coatings of parts which are used at high temperature in aggressive environments, for example, airplane engine blades or industrial steam turbine blades and others [4].

The aim of the work is to improve carbon steel operational characteristics by forming NiAl intermetallic surface alloy on a carbon steel substrate. Ni-Al system temperature fields numeric calculations were conducted, formed Ni-Al surface alloys structure and properties were investigated.

2. Experimental

Electron beam machine RITM-SP combines magnetron sputtering system and LEHCEB source. Electrons energy varies from 10 to 30 keV, current is up to 25 kA, pulse duration 2–4 μs, beam diameter is up to 80 mm. RITM-SP allows to conduct deposition and mixing processes in a single vacuum cycle [5].

Steel “St3” (0.17 C, 0.04 P, 0.04 S, 1.4 Mn, 0.4 Si, Fe – rest, weight.%) were used to prepare substrate samples. Samples dimensions are 15×15×2 mm, virgin roughness Ra = 0.23 μm. Surface alloy was formed by multilayer Ni-Al system one pulse LEHCEB treatment. Ni-Al system Ni (0.11 μm) – Al (0.167 μm) –…– Al(0.167 μm) – Ni (0.11 μm) consists of 19 layers with 2.5 μm overall thickness. Ni and Al layers thickness ratio is chosen to get system equiatomic composition. Layers were deposited by Ni (99.95 weight.%) and Al (99.95 weight.%) targets magnetron sputtering,
sputtering rates were $10.9\pm0.6$ and $9\pm0.5\,\mu\text{m}\cdot\text{h}^{-1}$, respectively. Virgin substrates were LEHCEB treated to clean surface before deposition. LEHCEB energy density $E_s = 4.2\pm0.9\,\text{J}\cdot\text{cm}^{-2}$ for cleaning and mixing was numerically calculated. LEHCEB parameters are as follows: electrons energy 20 keV, pulse duration 2.5 $\mu\text{s}$, pulse number 30 for cleaning and 1 after deposition. Morphology and topography were investigated by scanning electron microscopy (PhilipsSEM-515). Elemental composition was investigated by energy dispersion x-ray analysis (EDS). An investigation of the phase composition of the specimens was carried out using the X-ray diffraction analysis in a Shimadzu XRD 6000 diffractometer in a grazing incidence diffraction geometry at the incident angle $\omega = 5^\circ$. Microhardness was investigated by Wickers method (PMT-3M). Wear resistance «pin-on-disc» tests were conducted using TRIBOtester, 3 mm radius 100Cr6 ball was used as a counterbody. Specimen load was 4 N, friction distance 50 m, track radius 2 mm and friction velocity 25 mm s$^{-1}$. Tests were conducted in normal conditions without lubricants.

### 3. Results and discussion

Multilayer samples surfaces before and after LEHCEB treatment are represented in figure 1. It is seen from figure 1a that multilayer Ni-Al coating homogenous and defect free. Surface roughness $R_a = 0.19\,\mu\text{m}$. Virgin substrate roughness is higher but it smoothed during LEHCEB substrate cleaning [18]. Ni-Al coating one pulse LEHCEB treatment led to surface layer melting and more rough structure is formed, roughness after LEHCEB treatment $R_a = 0.34\,\mu\text{m}$. Morphology is also changed, there are cracks on the surface. There is Ni (46.2); Al (45.7); Fe (8.1 weight.%) in Ni-Al surface layer after one pulse LEHCEB treatment. Significant Fe concentration indicates that substrate was also melted and diffused into surface layers. Thereby intense all the layers and substrate mixing was observed.

![Figure 1](image1.png)

**Figure 1.** SEM-images of the surface sample of Ni-Al multilayer coatings before (a) and after (b) LEHCEB treatment.

Multilayer samples cross-sections before and after LEHCEB treatment are represented in figure 2. It is seen from figure 2a that multilayer Ni-Al coating consists of alternating thin layers. Average Ni-Al system thickness is $2.6\pm0.1\,\mu\text{m}$, with the maximum and minimum $\sim 2.7$ and $\sim 2.5\,\mu\text{m}$, respectively. Separate Ni and Al layers are seen on the picture, their thicknesses are $\sim 0.1$ and $\sim 0.16\,\mu\text{m}$, respectively. We are unable to measure chemical composition of single layer due to used method limits (analysis area is $2--5\,\mu\text{m}$).

Multilayer Ni-Al system is mixed and surface alloy is formed as a result of LEHCEB treatment. It is seen from figure 2b that surface alloy average thickness is $2.8\pm0.4$ is, with the maximum and minimum $\sim 3.2$ and $\sim 2.2\,\mu\text{m}$, respectively. Surface alloy cross-section shows homogenous structure with cracks up to $2\,\mu\text{m}$ deep.
Steel substrate sample, Ni-Al multilayer system on steel substrate sample (before LEHCEB treatment) and surface alloy sample (Ni-Al multilayer system on steel substrate sample after LEHCEB treatment) surface layer phase compositions were investigated. X-ray graphs are shown in figure 3. Steel substrate sample phase composition (figure 3a) consists of single \( \alpha \)-Fe phase, as expected. Ni and Al peaks as well as \( \alpha \)-Fe peaks are present in Ni-Al multilayer system on steel substrate sample (figure 3b). One pulse LHCEB treatment leads to significant changes in phase composition. There are NiAl intermetallic phase peaks instead of Ni and Al peaks in surface alloy sample phase composition (figure 3c). Thereby it can be concluded that intermetallic phase NiAl is formed as a result of equiatomic composition Ni-Al multilayer system one pulse LEHCEB treatment.

Aluminides Fe\(_2\)Al, FeAl\(_x\), FeAl\(_3\) peaks are not observed. FeAl and Fe\(_3\)Al diffraction maximums are close to high temperature NiAl phase maximum but have a 0.8–1° shift, so significant FeAl or Fe\(_3\)Al amount would lead to peaks bifurcation but it is not observed. In other words mainly intermetallic phase NiAl is formed, aluminides are not formed or their amount is insignificant.

**Figure 2.** SEM image of cross-section of Ni-Al multilayer coatings before (a) and after (b) LEHCEB treatment.

**Figure 3.** XRD patterns obtained in the grazing incidence geometry. Initial specimen (a) and Ni-Al multilayer coatings before (b) and after (c) LEHCEB treatment, respectively.

**Figure 4.** Microhardness and wear coefficient for initial specimen (1) and Ni-Al multilayer coatings before (2) and after (3) LEHCEB treatment, respectively.
Steel substrate sample, Ni-Al multilayer system on steel substrate sample (before LEHCEB treatment) and surface alloy sample (Ni-Al multilayer system on steel substrate sample after LEHCEB treatment) tribologic tests and microhardness of surface measurements results are shown in figure 4. Steel substrate wear coefficient is $8 \times 10^{-5}$ mm$^3$·(N·m)$^{-1}$, microhardness is HV = 2.8 GPa. After Ni-Al multilayer system deposition wear resistance is approximately 3 times lower (wear coefficient is $23 \times 10^{-5}$ mm$^3$·(N·m)$^{-1}$). It can be explained by alternating relatively hard Ni and relatively soft Al layers. During the test top thin Ni layer is destructed into small abrasive pieces which leads to wear increasing. At the same time microharness of surface is little bit higher HV = 3.1 GPa. Ni-Al surface alloy wear coefficient is $7.3 \times 10^{-5}$ mm$^3$·(N·m)$^{-1}$, microhardness of surface is 2 times higher HV = 5.5 GPa. It is clear that mechanical properties are improved due to formed hard and wear resistant intermetallic phase NiAl.

4. Conclusions
A Ni-Al Surface alloy was formed on a carbon steel substrate by 2.5 μm thick multilayer Ni-Al system deposition and subsequent one pulse low-energy high-current electron beam (LEHCEB) treatment. It is shown that surface alloy containing mainly intermetallic phase NiAl is formed as a result of one pulse LEHCEB treatment. It is established that NiAl surface alloy microhardness and wear resistance are higher in comparison to steel substrate parameters.

References
[1] Bafandeh M R, Omidi A and Irankhah A 2017 Surf. Coat. Technol. 315 268
[2] Sierra C and Vazquez A J 2005 Solar Energy Materials & Solar Cells 86 33
[3] Dercz G, Pajak L and Formanek B 2006 Journal of Materials Processing Technology 175 334
[4] Wen J, Cui H, Wei N, Song X, Zhang G, Wang C and Song Q 2017 Journal of Alloys and Compounds 695 2424
[5] Markov A B, Mikov A V, Ozu G E and Padei A G 2011 Instrum. and Experim. Tech. 54 862