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# Laser production of B–Ni complex layers

# Aneta Bartkowska ⁎, Aleksandra Pertek

Poznan University of Technology, Institute of Materials Science and Engineering, Pl. M. Sklodowskiej-Curie 5, 60-965 Poznan, Poland

### article info abstract

Article history: Received 9 November 2013 Accepted in revised form 13 March 2014 Available online 27 March 2014

Keywords: Nickel plating Boronizing Laser remelting Laser alloying Microhardness Wear resistance

The paper presents the results of modification with nickel and laser treatment of boronized layers produced on C45 steel. The objective of the study was to investigate the microstructure and selected properties of the new layer. The boronized layer modified with nickel was produced by the galvanic-diffusion method. Galvanic coating was produced first; next the galvanic-coated sample was diffusion boronized at 950 °C for 4 h. This resulted in a two-zone microstructure consisting of a continuous subsurface zone and underlying needle-like iron borides. In the continuous zone microhardness was in the range of 1100–1200 HV0.1 while in the second zone of the needle-like microstructure the microhardness was between 1600 HV0.1 and 1400 HV0.1. Galvanic-diffusion boronickelized layers were modified by using  $CO<sub>2</sub>$  laser technology. As a result of the laser beam impact the presence of three zones was observed: remelted (MZ), heat affected (HAZ) and the substrate. Laser heat treatment was carried out by two methods. The first method consisted of remelting the galvanic-diffusion layer. The second method consisted of alloying galvanic coating with boron. Microhardness measurements of the surface layers were made before and after laser modification. An advantageous effect of laser modification on the microhardness of boronickelized layers was found. Microhardness profiles of boronickelized layers after laser modification were characterized by a smooth transition from the remelted zone through the heat affected zone to the substrate. In the remelted zone the microhardness was approximately 1100 HV0.1. In this study, also an X-ray phase analysis was carried out. Nickel boride phases  $Ni<sub>3</sub>B$  and  $Ni<sub>4</sub>B<sub>3</sub>$  were detected in the nickel modified boronized layer. In addition, the nonequilibrium phase of iron boride Fe<sub>3</sub>B occurred in the laser-modified layers. Laser modification had a positive effect on the wear resistance of nickel modified boronized layers.

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# 1. Introduction

One of the modern and highly efficient technologies of forming materials is laser technology [\[1,2\]](#page--1-0). Currently on the consumer market innovation, quality and price of the product play a major role. Nowadays materials produced are based on newer and newer technologies that compete with the existing ones, and which are at the same time environmentally friendly. Laser technologies such as laser hardening [\[2\]](#page--1-0), laser remelting [\[1,3](#page--1-0)–13] or laser alloying with elements (e.g. B, Ni, Cr) or intermetallic phases [\[14](#page--1-0)–18] enable shaping of materials, giving them new properties, like increased durability and efficiency. Lasers are increasingly being used in surface engineering. Various elements, phases or alloys are used to modify layers with laser beam. Two such elements which can be used to modify the layer can be boron and nickel. Boron is used to produce boronized layers [\[10,19,20\]](#page--1-0) which have a needle-like microstructure and are characterized by many advantageous properties such as: high hardness of up to 1800 HV while maintaining good adhesion to the substrate, high wear resistance [\[10\],](#page--1-0) high heat resistance up to 800 °C, as well as good corrosion resistance in

Corresponding author. Tel.:  $+48616653572$ .

E-mail address: [aneta.bartkowska@put.poznan.pl](mailto:aneta.bartkowska@put.poznan.pl) (A. Bartkowska).

many acid and alkali solutions [\[21\],](#page--1-0) and in liquid metal [\[22\].](#page--1-0) Despite many advantages, boronized layers have some disadvantages, among which there is brittleness, which may occur in the subsurface zone. The brittleness of boronized layers may be manifested by cracking, and sometimes as spalling and flaking of the layers from the substrate. Accordingly, solutions are sought to prevent brittleness of the layers, thus contributing to longer exploitation. Modification of boronized layers has been discussed in numerous publications [3–[8,10](#page--1-0)–13, 23–[41\].](#page--1-0) The effect of various methods of boronized layer modification with elements such as Ni, Cr, Cu, C, and N has been analyzed by many authors [4–8,11–13,23–[26,28,29,35,40,41\].](#page--1-0) Currently surfaces are modified with boron, both on iron alloys-stainless steel [\[33\],](#page--1-0) cast iron [\[15,34\]](#page--1-0) as well as on other metal alloys, e.g. nickel [\[37,42](#page--1-0)-44], titanium [\[45\],](#page--1-0) niobium [\[45\],](#page--1-0) tungsten [\[45\]](#page--1-0) or chromium [\[45\].](#page--1-0)

In this study laser modification of a layer containing nickel and boron was carried out. The influence of the two methods (laser remelting and laser alloying) of laser treatment on the microstructure, phase composition and selected properties of the layers such as microhardness and wear resistance was analyzed.

It appears particularly suitable to use boron and nickel laser modification to harden machine elements working in dusty conditions without lubrication such as pin chains in agricultural tools or caterpillar chains in building machines.

# 2. Experimental procedure

# 2.1. Materials

The material investigated was C45 steel and its chemical composition is given in Table 1. The chemical composition was determined on the basis of spectral analysis with a spectrometer Spectrovac 1000's Baird. The ring-shaped specimens (external diameter 20 mm, internal diameter 12 mm and height 12 mm) were used for the study.

# 2.2. Diffusion boronizing and galvanic-diffusion boronickelizing processes

The boronickelizing process consisted of nickel plating, diffusion boronizing and heat treatment, in this order. Nickel modified boronized layers were called 'boronickelized'. For nickel plating Watts bath was used, which uses a combination of nickel sulfate NiSO<sub>4</sub> ⋅ 7H<sub>2</sub>O and nickel chloride NiCl<sub>2</sub> 6H<sub>2</sub>O, along with boric acid H<sub>3</sub>BO<sub>3</sub> as well as preservatives and initiating additives. The bath temperature was 25 °C at the current density of 2.5 A/dm<sup>2</sup>. Nickel coatings deposited on C45 steel had 5 μm thicknesses and their average microhardness was 475 HV0.05. Diffusion boronizing was performed at 950 °C (1223 K) for 4 h using the gas-contact method. The powder used in the boronizing process contained: amorphous boron,  $KBF<sub>4</sub>$  as activator and carbon black (Grade N326) as filler. Boronized and boronickelized specimens were hardened in water from 850 °C (1123 K) to room temperature 25 °C (298 K) and tempered at 570  $^{\circ}$ C (843 K) for 1 h.

### 2.3. Laser modification of boronickelized layer

Laser heat treatment (LHT) of B–Ni complex layers was carried out with two methods ([Figs. 1 and 2\)](#page--1-0). The first method consisted of laser remelting of the galvanic-diffusion B–Ni layer. The second one consisted of laser alloying the nickel galvanic coating with boron. The boron coating was applied in paste form, which consisted of amorphous boron, water glass and distilled water. In the second method prior to applying the coating the sample was hardened from 850 °C in water and tempered at 570 °C for 1 h. Laser heat treatment (LHT) was carried out with the TRUMPF TLF 2600 Turbo  $CO<sub>2</sub>$  laser of nominal power 2.6 kW. Parameters used in the experiment were as follows: laser beam power  $P = 1.04$  kW, laser beam radiation density  $q =$ 33.12 kW/cm<sup>2</sup>, scanning laser beam velocity  $v = 2.88$  m/min, which resulted from the rotational speed n (61.15 min<sup>-1</sup>). The multiple mode laser beam (TEM $_{01^*}$ ) of circular shape with a 2 mm diameter was applied. Throughout the laser treatment the samples were cooled in atmospheric air. Laser tracks were arranged as multiple tracks with a distance  $f = 0.5$  mm between them [\(Fig. 3\)](#page--1-0), where f was the distance between the axes of adjacent tracks. The required scanning velocity v resulted from the rotational speed *n* and rate of feed  $v_f$  ([Fig. 3](#page--1-0)). The distance from the focusing mirror to the laser heat-treated surface was 106.8 mm and was longer than the focal length. The parameter  $f$ affects the distribution of the tracks on the workpiece surface. It was selected in order to completely cover the treated surface with tracks, so that wear resistance of the surfaces could be tested.

The flowcharts of production of boronickelized layers after laser modification are presented in [Figs. 1 and 2.](#page--1-0) [Fig. 1](#page--1-0) shows a flowchart of laser heat treatment of the layer formed by the first method—laser remelting. The flowchart of boronickelized layer production using the first method is composed of: galvanic treatment (Step 1), diffusion boronizing (Step 2), and laser modification (Step 3). [Fig. 2](#page--1-0) shows a flowchart of laser heat treatment of the layer formed by the second





method—laser alloying. Boronickelized layer production using the second method is composed of: galvanic treatment (Step 1), coating with boron paste (Step 2), and laser modification (Step 3). In the first and the second methods amorphous boron was introduced into the surface layer. In the first method the diffusion boronizing process was carried out at a temperature of 950 °C in 4 h. In the second method the sample was covered with boron coating 40 μm thick. The coating thickness was determined based on the average of 10 measurements on a sample.

The coating thickness was determined based on the average of 10 measurements on a sample by means of ultrameter A2002M, where the results are in the range of  $+/-5$  µm from the mean average.

# 2.4. Microstructure, phase analysis and microhardness profiles

Microstructure observations were carried out on polished and etched cross-sections of specimens by using a light microscope Metaval Carl Zeiss Jena with a camera 2300 3.0 MP and Live Motic Images Plus 2.0 Resolution software. The samples were first polished by using abrasive papers of different granularities, and, finally with  $Al_2O_3$ . Specimens were etched in 2% HNO<sub>3</sub> solution.

The phase analysis of the boronickelized layer was performed on EMPYREAN PANalytical X-Ray diffractometer using Mo  $K_{\alpha}$  radiation. To determine the microhardness profiles a ZWICK 3212 B Vickers hardness tester was used. An indentation load of 100 G and a loading time of 15 s were used in this study.

#### 2.5. Wear resistance

A ring-shaped sample as specimen and sintered carbide plate as counterspecimen were used to examine wear resistance. The composition of S20S sintered carbide complied with the norm PN-88/H-89500, and was as follows: 58 wt.% of WC, 31.5 wt.% of (TiC + TaC + NbC), and 10.5 wt.% of Co. Its hardness was equal to 1430 HV. Wear resistance tests were carried out under the load  $P = 147$  N and at a specimen speed of 0.26 m/s, in dry friction conditions (unlubricated sliding contact).

Wear resistance was evaluated by specimen mass loss per friction surface in a time unit. The wear intensity coefficient (Iw) was defined as the straight line curve in the frictional wear diagram and was determined from the equation:  $Iw = \Delta m/(S \cdot t)$  [mg / (cm<sup>2</sup> h)], where:  $\Delta m$  - mass loss [mg], S - friction surface [cm<sup>2</sup>], t - friction time [h].

Wear resistance of boronized layers was compared with galvanicdiffusion layers and laser-modified layers.

## 3. Results and discussion

#### 3.1. Microstructure and phase analysis

As a result of the boronizing process at 950 °C for 4 h, the layer was about 90 μm thick and was composed of needle-like iron borides [\(Fig. 4\)](#page--1-0). The boronized layer was composed of two phases: iron boride FeB nearer the surface, and iron boride  $Fe<sub>2</sub>B$  situated deeper.

Iron boride FeB has high microhardness but it is brittle and therefore the boronized layer should be modified by various methods to eliminate this.

The boron-rich FeB phase is considered undesirable, partly because FeB is more brittle than the Fe<sub>2</sub>B [\[10,19\]](#page--1-0). Another reason why the FeB formation is also undesirable is because FeB and  $Fe<sub>2</sub>B$  are formed under tensile and compressive residual stresses, respectively, which are due to differences in expansion/contraction during cooling from the boronizing temperature resulting from different coefficients of thermal expansion [\[10,19\]](#page--1-0).

The samples boronickelized at 950 °C for 4 h had two zones [\(Fig. 5\)](#page--1-0). The first zone  $(z_1)$  was continuous and its appearance resembles that of a galvanic coating. The zone situated below was well-connected with the substrate because it had a needle-like microstructure. The first

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