Ytterbium impulse laser for surface modification of tool steel with $B_4C$-Al powders

U L Mishigidorzhiiyn$^{1,2}$, N S Ulakhanov$^{1,2}$, A V Nomoev$^{1,3}$ and A B Lupsanov$^{1,3}$

$^1$Institute of Physical Materials Science of the Siberian Branch of the Russian Academy of Sciences, 6 Sakhyanovoi St., Ulan-Ude, 670047, Russia
$^2$East Siberia State University of Technology and Management, 40V Klyuchevskaya St., Ulan-Ude, 670013, Russia
$^3$Buryat State University, 24a Smolina St., Ulan-Ude, 670000, Russia

E-mail: druh@mail.ru

Abstract. The paper deals with a new application solution of Ytterbium Picosecond Pulsed Fiber Laser for surface modification of 3Kh2V8F hot-work tool steel (the analog of AISI H21 steel). Surface modification was conducted by $B_4C$-Al powders from preplaced pastes followed by laser heating. The ratio of $B_4C$-Al powders was taken as 5/1 by weight and the paste thickness was approximately 1 mm. Laser treatment was conducted according to the following parameters: 1070 nm of wavelength, 100 W of power, 1 mJ of pulse energy, 100 ns pulse duration, pulse frequency range from 50 kHz to 90 kHz. Several tracks with different widths were obtained as a result of treatment depending on velocity of the laser move. EDS analysis showed that $B_4C$ particles were not completely dissolved in the weld beads. However, an enhanced concentration of boron (8-12 wt.%) was revealed in the vicinity of $B_4C$ particles. The aluminum concentration was low (up to 0.79 wt.%) on the surface of the weld beads.

1. Introduction

Laser micro-machining is of particular interests due to its quality and cost efficiency. It is widely used in automotive and aerospace industries for industrial materials processing (steels, aluminum and titanium) [1]. Ytterbium Fiber Laser Systems (YLS) are used for different purposes where small focal spot and high energy density are required, such as cutting high reflectivity metals and foils, micro-machining (micro-trimming, micro-scribing, micro-milling, micro-drilling etc.), surface treatment (precision surface finishing, coating removal, texturing, ablation), single-mode and remote welding. For instance, pulsed Yb: fiber laser is used to improve adhesive properties of Al alloys [2]. Laser ablation is applied on the surface of different Al alloy substrates: sheet, cast, and extrusion. This kind of treatment improves the joint strength for Al alloys through an increase in bonding area of the rough laser-ablated surfaces.

Welding of titanium alloys by means of laser with an Yb: YAG Disk Source is reported in work [3]. They offer to employ laser welding of titanium joint from CP Ti and Ti-6Al-4V alloy by creating full penetration welds with a specific microstructure. Tensile test shows a similar or slightly higher joint strength for the full penetration welds, compared with the base metal.
Another example of YLS application is nanoparticles synthesis [4]. Laser ablation is utilized for Fe₃O₄ nano oxide formation on the surface of carbon steel in gases (oxygen) or liquids (water and ethanol). The authors report about synthesized magnetite of 18 nm in oxygen and 30 nm in liquids.

High-power pulsed Nd: YAG laser have found application in surface alloying [5]. Powders of hexagonal boron nitride (h-BN) or boron are used for surface modification of the X30Cr13 stainless steel. h-BN-α-Fe(Cr) and Fe₂B-α-Fe(Cr) coatings with the hardness of 1000 and 1450 HV, respectively, are obtained. The coatings show lower friction coefficient and much better wear resistance compared to untreated stainless steel.

Boron-based diffusion layers and coatings have found wide application due to their high performance. These layers and coatings can be produced by different methods. For instance, common boriding in furnace (pack or case method) leads to saw- or tooth-like structure formation, which is quite brittle. A number of methods are designed to reduce brittleness of borided layers. One them is using highly concentrated energy fluxes (HCEF), such as laser and electron beam. The surface modification by means of HCEF can be divided in four processes: remelting, alloying, cladding and dispersing [6, 7]. The latter process implies incomplete melting of the surface layer under electron beam or laser heating, where the base material surface is heated up above the melting point and the alloying or additional material is treated below its melting point. As a result, the alloying materials are dispersed in the form of particles in the melt bath (figure 1).

In present study the possibility of boron and aluminum coatings formation by means of pulsed Yb: fiber laser will be investigated. It is intended to produce B/Al coatings with high performance on the surface of hot-work steel.

![Figure 1. Scheme of laser dispersing: a – laser treatment; b – the result structure.](image)

2. Materials and methods

3Kh2V8F steel (the analogue of AISI H21 steel) was used as a tested material. It is a high-quality hot work tool steel, alloyed with W, Cr, V. The full chemical composition of the sample steel is given in table 1. It is used for manufacturing heavy-duty pressing tools (small inserts of final forming stream, dies and extrusion punches, etc.) during hot deformation of the alloyed structural steels and high-temperature alloys, molds for injecting molding copper alloys [8].

YLP-V2-1-100-100-100 Ytterbium Pulsed Fiber Laser or pulsed Yb: fiber laser was used for present study (figure 2). This type of lasers is used for engraving, marking, microprocessing, precision cutting etc. The radiation is emitted through an optical fiber cable up to 6 meters long. The output collimator is equipped with an optical isolator for protection from back reflection. The central lasing line lies in the range of 1060-1070 nm. The pulsed lasers of this series are powered by 24 V DC and are air-cooled with built-in fans.

| Table 1. The chemical composition of 3Kh2V8F steel (wt.%) | C | Si | Mn | P | S | Cr | W | V | Fe |
|---|---|---|---|---|---|---|---|---|---|
| 0.26– | 0.15– | 0.15– | up to | up to | 3.0– | 8.5– | 0.3– | 84.33– |
| 0.36 | 0.50 | 0.40 | 0.03 | 0.03 | 3.75 | 10.0 | 0.6 | 87.58 |
Processing parameters for laser surface modification were taken as radiation wavelength of 1070 nm, 100 W of output power, 1 mJ of radiation pulse energy, 100 ns of pulse duration and pulse frequency range from 50 kHz to 90 kHz. The treatment was carried out on 3Kh2V8F steel samples processed by preliminary mixed 78% B₄C + 18% Al + 4% NaF (wt.) paste with BF-6 glue. The thickness of the paste was 1-3 mm. Two rectangular samples with the size of 20×15×20 mm (sample 1) and 10×12×20 mm (sample 2) were treated. Two laser positions (higher and lower) and two treatment periods (4 minutes and 10-20 s) modes were tested.

Optical microscopy, SEM, EDS and XRD analyses were used to establish structural state, elemental and phase composition of the obtained laser tracks. For these purposes Olympus BX43 Optical Microscope, JEOL JSM-6510LV Scanning Electron Microscope with an INCA Energy 350 microanalysis system and Bruker Phaser 2D X-ray Diffractometer were used.

![Figure 2. YLP-V2-1-100-100-100 Ytterbium Pulsed Fiber Laser.](image)

3. Results and discussion
Several types of laser tracks were obtained depending on the velocity and laser position. Three laser tracks, the light broad track and two dark narrow ones, were observed on sample 1 after 4 min treatment period (figure 3). A lower laser position produced the first track of about 2 mm width where intense remelting processes on the surface of steel with preplaced B₄C/Al paste took place. A higher laser positioning resulted in much narrower tracks. The laser ablation process was prevalent on laser tracks 2 and 3. The base material was evaporated forming continuous pits along laser movement.

Figure 4. Laser tracks on sample 2 (a) with magnified pictures of each track (b, c, d, e).

Figure 5. Cross-section microstructure of the broad track on sample 1 at ×100 (a) and ×400 (b).

The shorter treatment duration of 10-20 s on sample 2 resulted in two narrow light tracks and two dark broad ones depending on the laser position (figure 4). Two middle broad tracks of about 2 mm width were obtained after lower laser positioning. On the contrary, the higher laser positioning led to the formation of two narrow tracks on the edges. The shorter treatment period of 10-20 s resulted in
high porosity of broad tracks due to insufficient heat transfer of the defocused laser. When narrow tracks were being created laser velocity was just enough to remelt the surface of sample 2 without evaporation.

Cross-section of sample 1 have shown that track thickness varies significantly form several micrometers to 25-30 µm (figure 5). At the same time the base metal microstructure revealed significant grain coarsening in heat affected zone (HAZ). As a result of laser processing, the micrelief of the surface has been changed. In addition, micro defects such as porosity, inclusions, cracks are visible in the track microstructure (figure 5 (b)).

EDS analysis revealed areas of high boron concentration up to 42.51 wt.% (figure 6 (a), table 2). The laser temperature was probably insufficient for B₄C particles fusion due to its high melting point of 2,763°C. At the same time aluminum concentration did not exceed 0.34 wt.% which was deficient to create aluminum-based compounds. Boron concentration was even higher on the magnified area with undissolved an B₄C particle in the center (figure 6 (b), table 3). Carbon wasn’t detected automatically by EDS software partially due to carbon peak overlapping by boron peak. It is known that their kα radiation values are close each other, for boron it is 0.183 kV and for carbon it is 0.277 kV. Aluminum concentration was slightly higher (up to 0.79 wt.%) compared to the results from table 2. It should be noted that Al was detected only in the area of high boron concentration. Spectra 1 and 2 also showed boron presence with 8.94 wt.% and 12.66 wt.% respectively. This concentration corresponds to Fe₂B iron boride and even exceeds it [9]. This means that diffusion processes occured in the vicinity of B₄C particles which resulted in additional compound formation.

**Table 2.** Concentration of the elements on the surface of laser track shown on figure 6 (a) (wt.%).

| No spectrum | B   | C   | Al  | V  | Cr  | Fe  | W   | Total |
|-------------|-----|-----|-----|----|-----|-----|-----|-------|
| Spectrum 1  | 5.03| 0.21| 0.40| 2.23| 79.37| 12.76| 100.00|
| Spectrum 2  | 14.03| 0.25| 0.45| 2.13| 73.85| 9.30| 100.00|
| Spectrum 3  | 28.39| 12.73| 0.15| 0.41| 1.78| 52.06| 4.48| 100.00|
| Spectrum 4  | 42.51| 10.02| 0.34| 0.19| 1.72| 42.42| 2.81| 100.00|
| Spectrum 5  | 15.45| 4.16| 0.08| 0.51| 1.88| 65.68| 12.24| 100.00|
| Spectrum 6  | 6.52| 0.08| 0.58| 1.93| 75.92| 14.96| 100.00|
| Spectrum 7  | 16.42| 0.11| 0.49| 2.02| 68.22| 12.74| 100.00|
Table 3. Concentration of the elements on the surface of laser track shown on figure 6 (b) (wt.%).

| No spectrum | B   | C   | Al  | V   | Cr  | Fe  | W   | Total |
|-------------|-----|-----|-----|-----|-----|-----|-----|-------|
| Spectrum 1  | 8.94| 6.02| 0.44| 2.20| 73.46| 8.94|     | 100.00|
| Spectrum 2  | 12.66| 6.21| 0.38| 2.08| 69.38| 9.30|     | 100.00|
| Spectrum 3  | 9.85 |     | 0.51| 1.97| 78.28|     |     | 9.39  |
| Spectrum 4  | 7.00 |     | 0.51| 1.87| 79.56|     |     | 11.06 |
| Spectrum 5  |     |     |     | 0.49| 2.57| 83.86|     | 13.07 |
| Spectrum 6  | 73.27|     | 0.79|     | 0.90| 22.54|     | 2.50  |
| Spectrum 7  | 76.44|     | 0.51|     | 0.83| 20.28|     | 1.95  |
| Spectrum 8  | 71.42| 0.57| 0.28| 1.04|     | 24.22|     | 2.47  |

Besides, surface morphology revealed cracks confirming optical microscopy data, which are probably due to compressive residual stresses in the laser track.

Figure 7. SEM pictures of laser track 2 (a) and 3 (b) on sample 1.

Table 4. Concentration of the elements on the surface of laser track 2 shown on figure 7 (a) (wt.%).

| No spectrum | B   | C   | Na  | Al  | Si  | V   | Cr  | Fe  | W   | Total |
|-------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-------|
| Spectrum 1  | 4.41| 29.37| 0.81| 0.33| 1.87| 63.21|     |     |     | 100.00|
| Spectrum 2  | 5.02| 37.13| 0.49| 0.65| 52.62| 4.09|     |     |     | 100.00|
| Spectrum 3  | 5.06| 30.38| 1.01|     | 0.98| 62.57|     |     |     | 100.00|
| Spectrum 4  | 60.59|     | 2.87| 1.67| 10.76| 24.12|     |     |     | 100.00|
| Spectrum 5  | 4.51| 2.39 |     | 0.87| 4.48| 79.10| 8.64|     |     | 100.00|
| Spectrum 6  | 56.47| 39.17| 3.35| 0.04|     |     |     | 0.96|     | 100.00|
| Spectrum 7  | 75.05| 20.54| 0.89|     | 0.61| 2.91|     |     |     | 100.00|
| Spectrum 8  | 78.50| 10.87|     | 1.11| 9.52|     |     |     |     | 100.00|
| Spectrum 9  | 16.97| 50.58| 3.47| 14.72| 0.91| 1.03| 12.30|     |     | 100.00|
EDS analysis showed that both narrow laser tracks on sample 1 were severely oxidized (figure 7, tables 4, 5). The track centers mostly consisted of iron oxides due to high concentration of both in spectra 1-3. Spectra 7-9 (track 2) and 5-7 (track 3) showed elevated Al concentration, which indicates aluminum oxide presence on the laser track edges. Behind aluminum oxide zone on the track edges, B$_4$C particles were outbounded with melted oxides and base metal. Sodium from the treatment paste was also detected in the same zone with carbides.

**Table 5.** Concentration of the elements on the surface of laser track 3 shown on figure 7 (b) (wt.%).

| No spectrum | B   | C   | O   | Na  | Al  | Si  | V   | Cr  | Fe  | W   | Total  |
|-------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|--------|
| Spectrum 1  | 5.74| 32.91| 2.12 |     |     |     | 2.37 | 56.86|     |     | 100.00 |
| Spectrum 2  | 4.27| 37.93| 1.90 | 0.29| 0.80| 49.75| 5.06 |     |     |     |        |
| Spectrum 3  | 30.46|     | 2.06 |     |     |     | 3.19 | 64.29|     |     |        |
| Spectrum 4  | 7.46| 25.05| 0.80 | 0.47| 0.66| 2.76 | 60.65| 2.16 |     |     | 100.00 |
| Spectrum 5  | 21.37| 56.85| 2.22 | 10.62| 0.52| 0.88 | 0.48 | 5.99 | 1.08 |     |        |
| Spectrum 6  | 25.33| 64.68| 4.98 | 3.64 | 0.32|     |     |     |     |     | 100.00 |
| Spectrum 7  | 21.65| 65.82| 0.51 | 8.66 | 0.39| 0.91 | 2.06 |     |     |     |        |
| Spectrum 8  | 44.11| 18.28| 32.08| 0.62| 0.56| 0.08 | 0.09 | 3.46 | 0.72 |     |        |
| Spectrum 9  | 6.89 | 5.35 | 11.48|     |     | 0.40 | 2.15 | 68.61| 5.12 |     | 100.00 |
| Spectrum 10 | 2.24 | 1.65 |     |     |     | 1.42 | 11.50| 78.82| 4.37 |     |        |
| Spectrum 11 | 14.71| 63.42| 12.38| 5.16|     |     |     |     |     | 4.33 |        |

XRD analysis of sample 1 in track 1 confirmed presence of previously described Fe$_2$B, Al and Fe. (figure 8). In addition, W$_3$C and CrC carbides were revealed, which are normal impurities of this type of steel.

**Figure 8.** XRD-pattern of laser track 1 on sample 1.
4. Conclusion
The research confirmed boron- and aluminum-based coating formation on the surface of 3Kh2V8F steel sample by pulsed Yb: fiber laser. Proper laser parameters allowed to control the width of laser tracks and implement one of the four types of surface modification, such as laser dispersing process. It is planned to perform track overlapping in order to create different coatings for further investigation of mechanical properties (microhardness and wear resistance). It is recommended to use more powerful laser systems for medium and large size machine tools and units.

5. Acknowledgments
The work was carried out within the framework of State Task to conduct research No. 0270-2021-0001.

The authors are grateful to Dorzho Dasheev, researcher of the Laboratory of Physical Materials Science, Institute of Physical Materials Science SB RAS, for assistance in XRD analysis.

References
[1] Ann R and Jahan M 2019 Encyclopedia of Aluminum and Its Alloys (Boca Raton: CRC Press)
[2] Zhu C, Wan H, Min J, Mei Y, Lin J, Carlson B E and Maddela S 2019 Opt. Laser. Eng. 119 65–76
[3] Beguin J D, Gazagne V, Balcaen Y, Alexis J and Andrieu E 2018 Mater. Sci. Forum 941 845–50
[4] Khartaeva E Ch, Nomoev A V, Bardakhanov S P, Sholohov E S, Batoroev A S, Syzrantsev V V, Zhalsanov B G and Lygdenov V T 2018 Vestnik BGU. Khimiya, Fizika (BSU bulletin. Chemistry. Physics) 2-3 3–14 [in Russia]
[5] Avril L, Courant B and Hantzpergue J J 2006 Wear 260 351–60
[6] Schneider M F 1998 Laser Cladding With Powder (Enschede: Universiteit Twente)
[7] Wang Q J and Chung Y W 2013 Encyclopedia of Tribology (Boston: Springer)
[8] Roberts G A, Kraus G and Kennedy R L 1998 Tool Steels (Ohio: ASM International)
[9] Krukovich M G, Prusakov B A and Sizov I G 2016 Plasticity of Boronized Layers (Switzerland: Springer)