Investigation of the Structure of Hard Alloys That Have Undergone Bulk Pulsed Laser Hardening Using X-Ray Structural Analysis

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Abstract. The main principles of volumetric pulsed laser hardening method are presented in the article. The results of experiments based on tensometry with further processing using the LabVIEW system are shown. Obtained results confirm the appearance of the mechanical shock wave in materials due to the action of a single high-energy laser pulse. The received dependences were confirmed for hard alloys with different chemical compositions. Using of X-ray structural analysis allowed to establish that various hard alloys processed by pulsed laser hardening characterized by the third kind (nanostructure) changing the structure due to the accumulation of structural defects (sizes of mosaic blocks, crystal microscopic distortions, density of dislocations). It has been established that the third kind changing the structure increases the main service mechanical properties of hard alloys (microhardness, abrasive wear resistance, flexing strength). In addition, it was found that the change in the hard alloys properties is directly proportional to the change in the nanostructure of materials.

Current techniques and technologies require the application of new progressive materials with an improved complex of physical and mechanical properties. Such problems can be successfully solved by creating of new materials and modifying the structure of already known compounds using advanced hardening technologies. [1, 2]. The creation of highly concentrated energy flow sources (lasers, plasmatrons, etc.) at the end of the twentieth century allowed to significantly increase the quantity of hardening methods and raise them to the new higher level [3, 4]. A fairly large number of electrophysical hardening methods have appeared. These methods include ion-plasma, magnetic-pulse, radiation, laser and plasma cladding, gas discharge plasmas, electrospark, laser, etc [5, 6]. At the same time, most of the existing methods have their own application field, i.e. specialization, which can depend on the hardenable material grade or operating conditions, which may require the presence of any special properties of the product material (decorative appearance, corrosion resistance, surface heat or electrical conductivity, wear resistance, flexing strength and etc.) [7, 8]. In most cases, surface hardening methods are used depending on the operational product purpose. However, there are products groups that are widely used in various spheres of human activity, which can retain their performance with some allowable change

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in geometric parameters (wear, dulling, etc.). Such products include: cutting tools, drill bits, road cutters, operating devices of earth-moving and agricultural machines, track links of tracked vehicles, etc.). For these products groups it is advisable to apply hardening methods, in which the structure and properties modification extends to a certain local depth into the body material. It maintains the operability over the spectrum of permissible wear values. These methods include the method of volumetric pulsed laser hardening (VPLH), which was developed at the Mechanical Engineering and Technological Equipment Department of the North Caucasus Federal University and showed positive results in laboratory tests and in production conditions during the hardening carbide cutting tools process [9, 10]. This method allows modifying the structure and increasing the mechanical properties of materials to a depth of 30 - 40 millimeters. In this case, the action mechanism is a shock wave that is excited in the material under the single high-energy laser pulse treatment. The mechanism of volumetric pulsed laser hardening is shown on the Fig. 1.

![Diagram of the mechanism of volumetric pulsed laser hardening](image)

**Fig. 1.** The mechanism of volumetric pulsed laser hardening

Earlier in laboratory research it was determined that at the first stage of the VPLH the laser pulse energy is transformed into the thermal energy of the hardenable material [11]. In this case, the formation of a crater at the treated place (Fig. 1) and the emission of evaporated material (due to the high temperatures up to 80 000 °C, i.e. cold plasma is formed), the mass of which depends on the VPHL modes and its chemical composition, is observed. For example, the mass of the evaporated material ranges from 30 to 80 mg for hard alloys in the zone of VPHL optimal modes (laser radiation energy $E$, laser ray diameter $d_l$, laser pulse duration $t_p$).

The second stage consists in the redistribution of the pulse excess thermal energy into the shock wave mechanical energy, which spread deep into the material. The shock wave characteristics were studied using tensometry and the LabVIEW system (Fig. 2). For the tensometry the tension sensors with 1 mm base and 5 mm pitch were glued on the hard alloys samples in different directions. Samples from various groups and grades of hard alloys were studied: VK8, T5K10, TT7K12, KNT-16.
As seen from Fig. 2, the mechanical shock wave is decaying and obeys an exponential law. In this case, the dependences of the wave transmission law will depend on the VPHL modes (constants in formulas 1 and 2) and on the material chemical composition, which characterizes the coefficient of the medium resistance (the time of the wave transmission $t_p$). So, for example, on the basis of the studies carried out for the hard alloy T5K10, the following dependences were obtained. Experimentally for the hard alloy T5K10, the following dependencies were obtained:

\[
X = 0.036 \cdot e^{-1.2t} \cdot \cos(2.4 \cdot t + 12,4) \tag{1}
\]

\[
X = 0.032 \cdot e^{-1.6t} \cdot \cos(2.4 \cdot t + 12,4) \tag{2}
\]

Dependences 1 correspond to the following modes of VPHL: $E = 160 \, J$; $d_l = 2.0 \, mm$; $t_p = 0.8 \, ms$; dependencies 2: $E = 200 \, J$; $d_l = 2.2 \, mm$; $t_p = 1.0 \, ms$.

At the third stage of VPHL the hardenable material structure is modified and followed by its mechanical properties changing. This can be explained by the set of complex mechano-physicochemical processes that are initiated by the shock wave passage and spontaneously act on each other. This process by its physical nature is similar to the cutting
materials process and practically cannot be analyzed by mathematical processing and modeling [12, 13]. Material changes caused by the VPHL can be determined using laboratory mechanical tests or using X-ray structural analysis. The results of X-ray structural analysis will be presented in this work.

X-ray structural analysis was carried out using X-ray diffractometers «ARL Xtra», «Difrey-401» and «Minilab-6». Samples of different hard alloys groups were used in the experiment: VK6, VK8, T5K10, T15K6, TT7K12, KNT-16, Sandvik. The results of X-ray structural analysis showed the third kind changes of the materials structure in the studied samples. Increase in the defect structure degree of the samples after VPHL (diffraction line broadening, decreasing of interplanar distance, increasing of the reflected pulses intensity and the dislocations density) was observed (Fig. 3).

Fig. 3. X-ray diffraction pattern sections of the hard alloys VK8 (a) and T5K10 (b) tungsten carbide WC (211) samples: 1 - initial; 2 - after VPLH with an energy density 182 MJ/m2
Fig. 3 shows that VPLH leads to increasing of the WC reflections intensity. At the same time, the test report, obtained using the program for definition of the lattice parameters from the peaks list and user-assigned Miller's DDView indices, showed diffraction lines broadening and decreasing of the interplanar distance for all hard alloys samples treated by VPHL. This indicates increasing of the defectiveness degree in the hard alloys structure.

In this case, the values of the structure defectiveness degree indicators depend on almost all input factors of the "VPHL - sample" system (chemical composition and sample geometric shape, energy in the laser ray, laser ray diameter and distance from the irradiation site). The results of X-ray structural analysis are closely correlated with the data obtained under studying the shock wave characteristics using the LabVIEW system. The results allow determining the indices of the third kind structure of hard alloy samples treated by VPHL using the relationship between the physical broadening of the lines $B$, the position of the X-ray diffraction lines $Q$ gravity center and the mosaic blocks $L$ sizes and the crystal lattice $\Delta d/d$ microdistortions:

$$L = \frac{\lambda}{B \cdot \cos \theta}$$

$$\Delta d/d = \frac{B}{4 \cdot \sin \theta}$$

The dislocation density was determined from the obtained values of $Q$ and $\Delta d/d$, as well as the value of the Burgers vector for hard alloys $b = 0.301 \text{ nm}$:

$$\rho = \sqrt{\rho_2 \cdot \rho_L} = \sqrt{\frac{90}{b^2} \cdot \left(\frac{\Delta d/d}{d}\right) \cdot \frac{\pi}{L^2}}$$

Calculation data are presented in the Table 1, 2.

### Table 1. WC-phase structure parameters of the VK6 alloy

| VPHL modes (radiation energy density $P$, MJ/m$^2$; laser ray diameter $d_l$, mm) | Distance from the irradiation place $l$, mm | $L$, nm | $\Delta d/d$, $\times 10^{-3}$ | $\rho$, $\times 10^{11}$ cm$^{-2}$ |
|-----------------------------|---------------------------------|--------|-----------------|---------------------|
| $P = 172$, $d_l = 1,6$     | 8                              | 28/30  | 3,0/2,8        | 4,3/3,9             |
|                            | 14                             | 32/36  | 3,2/2,6        | 4,5/3,9             |
|                            | 20                             | 24/31  | 3,6/2,7        | 4,9/3,9             |
|                            | 26                             | 27/32  | 3,4/2,9        | 4,6/4,3             |
| $P = 264$, $d_l = 1,8$     | 8                              | 26/28  | 2,9/2,8        | 4,1/3,9             |
|                            | 14                             | 29/30  | 3,0/2,9        | 4,2/3,8             |
|                            | 20                             | 28/30  | 3,1/2,9        | 4,4/4,2             |
|                            | 26                             | 27/28  | 2,9/2,8        | 3,9/3,7             |
| $P = 178$, $d_l = 1,4$     | 8                              | 31/32  | 3,4/3,3        | 4,7/4,5             |
|                            | 14                             | 35/36  | 3,8/3,6        | 4,4/4,2             |
|                            | 20                             | 30/35  | 3,6/3,3        | 4,4/3,9             |
|                            | 26                             | 30/32  | 3,2/3,1        | 3,8/3,6             |

Note: below the line the values of $L$, $\Delta d/d$, $\rho$ for the WC-phase of hard alloy samples before VPHL are presented.
Table 2. WC phase structure parameters of the T15K6 alloy

| VPHL modes (radiation energy density $P$, MJ/m$^2$; laser ray diameter $d_l$, mm) | Distance from the irradiation place $l$, mm | $L$, nm | $\Delta d/d$, $\times 10^{-3}$ | $\rho$, $\times 10^{11}$ cm$^{-2}$ |
|---|---|---|---|---|
| $P = 172$, $d_l = 1,6$ | 8 | 44/46 | 3,9/3,7 | 4,2/3,9 |
| | 14 | 46/49 | 4,6/4,3 | 4,5/4,2 |
| | 20 | 38/47 | 3,6/2,7 | 4,5/3,9 |
| | 26 | 39/44 | 3,4/2,9 | 4,6/4,2 |
| $P = 264$, $d_l = 1,8$ | 8 | 49/51 | 3,9/3,8 | 4,4/4,2 |
| | 14 | 42/46 | 4,3/3,9 | 4,2/3,8 |
| | 20 | 39/44 | 4,4/3,6 | 4,6/3,8 |
| | 26 | 46/49 | 3,9/3,6 | 4,5/4,1 |
| $P = 178$, $d_l = 1,4$ | 8 | 49/51 | 4,4/4,3 | 4,8/4,7 |
| | 14 | 44/46 | 4,6/4,5 | 4,6/4,4 |
| | 20 | 42/46 | 4,6/4,2 | 4,9/4,6 |
| | 26 | 49/51 | 4,4/4,3 | 4,7/4,6 |

Note: below the line the values of $L$, $\Delta d/d$, $\rho$ for the WC-phase of hard alloy samples before VPHL are presented.

The presented results allowed to make following conclusions:
1. Volumetric hardening method of hard-alloy samples is considered in the article. Method allows modifying the structure and properties of materials to a depth of 40 millimeters from the place of laser ray exposure.
2. The wave nature of the VPHL method is determined using the tensometry method and the LabVIEW system.
3. Using X-ray structural analysis, it has been established that hard alloys samples treated by VPLH have structural changes of the third kind (nanostructure). This is observed in increasing of the reflections intensity, broadening of diffraction lines, and decreasing of interplanar distances.
4. Third kind structural changes of the hard alloy samples lead to changing of the main defectiveness indicators of the materials structure (sizes of mosaic blocks, microdistortions of the crystal lattice, dislocation density). This, according to the classical theory of plasticity, has a significant effect on their mechanical properties.

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