A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
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# THE WAY OF HARDENING THE HIGH PRESSURE APPARATUS MATRICES

# METODA UTWARDZANIA MATRYC WYSOKOCIŚNIENIOWEJ APARATURY

This paper presents the solution of the problem of the lifetime prolongation for the high pressure apparatus matrices, used for the artificial diamonds synthesis by means of a catalytic method, at working mixture squeezed up to the pressure of 4 GPa and heated up to the temperature 1700 K. Our investigation has shown that a considerable lifetime prolongation for the high pressure apparatus matrices can be achieved by increasing the mechanical hardness of their effective area and by their structure improvement. The matrices were handled in high vacuum conditions in the Ar+ plasma of a low energy ionic source. Simultaneously, a graphite target in the ionic source was sprayed with laser radiation. This processing led to the formation of the solid replacement solution and to the creation of the strengthened surface layer containing carbide. It also led to the elimination of the micro cracks with the i-carbon.

Praca prezentuje rozwiązanie problemu przedłużania trwałości matryc aparatury wysokociśnieniowej, stosowanej do syntezy sztucznych diamentów, przy wykorzystaniu metody katalitycznej, gdzie mieszanina robocza ściskana była do ciśnienia 4 GPa i podgrzewana do temperatury 1700 K. Nasze badania wykazały, iż znaczne przedłużenie trwałości matryc aparatury wysokociśnieniowej można uzyskać poprzez zwiększenie mechanicznej twardości ich obszaru efektywnego oraz ulepszenie strukturalne. Matryce umieszczono w warunkach wysokopróżniowych w plazmie Ar+ o niskoenergetycznym źródle jonowym. Równocześnie matryca grafitowa w źródle jonowym została poddana działaniu promieniowania laserowego. Taka obróbka doprowadziła do powstania trwałego zamiennika oraz do utworzenia wzmocnionej warstwy powierzchniowej zawierającej węglik. Wyeliminowało to także mikroszczeliny za pomocą i-węgla.

## 1. Introduction

Recently, the range of materials used in the industry has been constantly extending. It is necessary to emphasize that, on the one hand, the increase of strength, wear hardness and hardness of the used materials causes the difficulties in their handling, and on the other hand, there is a constant rise of requirements for the labor productivity and for the lowering of the energy consumption. Such state of production determines the wide application of instruments made of super solid materials and, in particular, the use of diamonds.

The industrial application of natural diamonds is restricted by their rarity and high cost. That‘s why the production of synthetic diamonds is extremely important. It allows to obtain crystals with the necessary heat and electro-physical properties, hardness, wear hardness and strength. The electro discharge sintering is one of the main methods of producing synthetic diamonds. Its essence is in squeezing the working mixture with the pressure up to dozens of Mpa and with its subsequent heating by means of electric current. The operation of the equipment for the synthesis, which takes place in extreme conditions, leads to the rapid wear of costly parts made of hard alloys. Thus, for the economic efficiency and the lowering of energy and resource consumption it is crucial to prolong the durability of the part of the equipment and to increase the productivity of synthetic diamonds synthesis.

Since the cost of the high pressure apparatus matrices make up to 40% of all the expenses of the synthetic diamonds production, the raise of the high pressure matrices durability is one of the possible ways of lowering the general cost of the process.

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## 2. Discussion of the method of hardening the high pressure apparatus matrixes

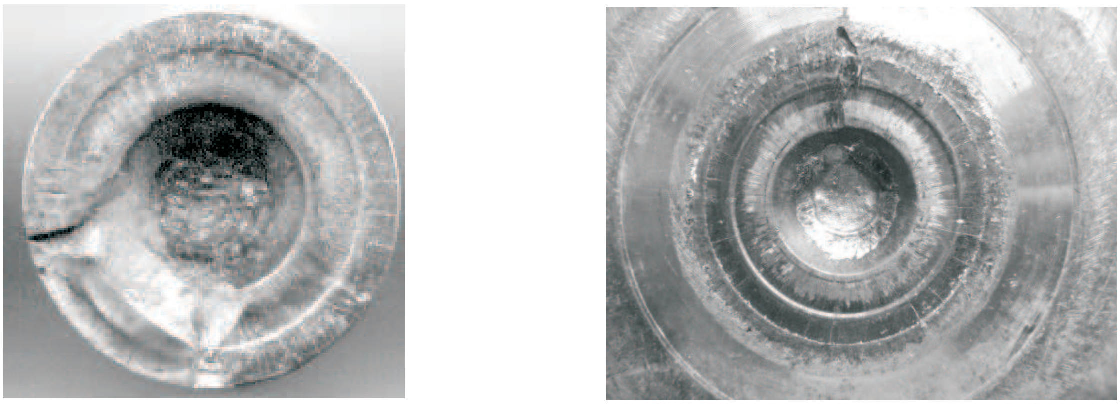
The analysis of the reasons for the failure of the matrices made of the WC6 alloy, which are used at the ”Kristall” enterprise (sited in Gomel), has shown that the main reason for this is the fragile destruction of the matrices, especially in the regions of the edges restricting the dimple in them (Fig.1).

Fig. 1. The matrix of high pressure apparatus. General view

The prolongation of the durability of high pressure apparatus matrices by changing the hard alloy type of which the matrix is made is not economically expedient because of the high cost of the more stable alloys.

One of the most rampant reasons for the high pressure apparatus matrices failure is the fatigue destruction due to the cyclic strain of its material. The analysis of the destruction of the matrices made of alloy WC6 under the maintenance has shown that the destruction of the useful area is specified by the presence of the obvious attributes of the corrosive cracking in it. Microcracks appear in the field of the matrix adjoining the dimple, already after several cycles of synthesis. Further, small erosive damages appear in the dimple at high temperature. Sufficient crack’s branching can serve development of other aspects of destruction, in particular, fragile chopped off. Therefore the surface of a working matrix dimple is covered with traces small-sized and larger having chopped off. As a rule, destruction of matrixes occurs on orthogonally related planes by distribution of radial and axial cracks.

Crack appearance in the matrix not at once leads to the high pressure apparatus matrixes failure. In the case of with the availability of side support from the part of the fastening ring block, the radial cracks are not the reason of high pressure apparatus matrixes failure, even in the case of their large number. Axial cracks are the cause of the high pressure apparatus matrixes failure, provided that they go into the work area surface of the matrix or their value exceeds the critical meaning values for these conditions.



a) b)

Fig. 2. Typical damages of the high pressure apparatus matrixes under the maintenance a – axial breaks a working edge of a matrix b – matrix in the block of bindinging rings

The methods of the ionic and laser implantation (when used both separately and together) allow to create various non-equilibrium surface alloys - even those that can‘t be obtained by the conventional methods of metal working. Practically, there are no restrictions for the thermodynamic and heat character of the forming of surface structures with the help of these methods.

Thus, it appeared to be expedient to modify the effective area of the high pressure apparatus matrix by means of concentrated streams of energy that enabled to create a piece with combined mechanical properties. On the one hand, the core of the matrix retains the optimum indices of the hardness, the strength under bending and the plasticity, but the thin layer of the effective area becomes several times harder and stronger, as the structure of the surface layer of the matrix is being improved under the low energy ionic bombardment. On the other hand, the impact of the carbon ions with a wide range of energies creates the solid solutions of replacement in the surface layer and forms the hardened surface layer containing carbides. Due to the designed technological modes, the carbon diamond-like film is formed on the surface of the matrices. It leads to the elimination of the micro cracks on the effective area of the matrices and to the flattening of the temperature and mechanical loading because of the high thermal conduction and the mechanical hardness of the formed coasts.

## 3. Experimental investigations

Owing to the implemented plan of handling hard alloy matrices of high pressure apparatuses, the diamond-like film on the matrix surface is formed by the mechanism of the combined ion-beam and laser method. The essence of the method is that the graphite target is situated in the core of the low energy ionic source made as the butt Hall accelerator with open anode layer that allows to form a bundle of ions within the range of energies of 40 – 200 eV and having the angle of ion scattering of 140 – 160°.

The laser erosive plume (LEP) starts to be formed in the ionic source. In such combination, the range of the LEP high-energy part remains practically unchanged, as it considerably exceeds the energy of the particles formed in the ionic source. In comparison, the energy of the LEP high-energy part is 340 eV, and the highest energy of the ions formed in the ion source at the anode voltage 150 V is 150 – 250 eV. However, the bulk of the low-energy (up to 10 eV) neutral part of the LEP is ionized and accelerated to the energies ~ 60 – 100 eV. As the lifetime of the LEP is limited and makes ~ 1.5 – 1.8 ms the neutral Ar gas is blown into the ionic source to maintain the discharge in it. The ionic source also causes definite changes in the geometry of the LEP that is formed in the ionic source. As the source forms a big angle of ion scattering it causes the LEP, being under the influence of the source field, to enlarge its geometrical dimensions. The fact is well confirmed by the experimental data. The size of the spot of the erosion yields of the LEP in the plane of handling is twice as big as the size of the spot obtained with the help of the laser of the similar capacity, only without an ionic source.

Thus, the use of the combined method allows to increase the energy of the LEP low-energy part and to ionize the neutral part of the LEP particles, which in its turn leads to the increase of the quality of the diamond-like film.

The process of strengthening the hard-alloy matrices of high pressure apparatuses is implemented on the basis of the “Bulat” vacuum installation for drawing coats supplemented with the technological laser and the “AIDA” low-energy ion source. The basic diagram of the installation is in Fig.3.

The installation consists of the following main parts:

1) laser;
2) rotary system;
3) focalizing lens with a protective glass;
4) work pieces (high pressure apparatus matrix);
5) mounting point of work pieces;
6) graphite target;
7) rod of a rotating apparatus;
8) ion-beam radiant;
9) electromagnetic clutch;
10) evacuated chamber.

Laser emission 1, being reflected from rotary mirror 2, gets at the system of focusing laser radiation 3, containing optical system for shaping laser radiation in a bundle of a given geometry and ensuring the high homogeneity of radiation. Then the focused laser radiation is guided on the surface of the graphite target 6, set in ion-beam radiant 8. The stream of the ions shaped by the focused laser radiation and the ion-beam radiant affects the work pieces.

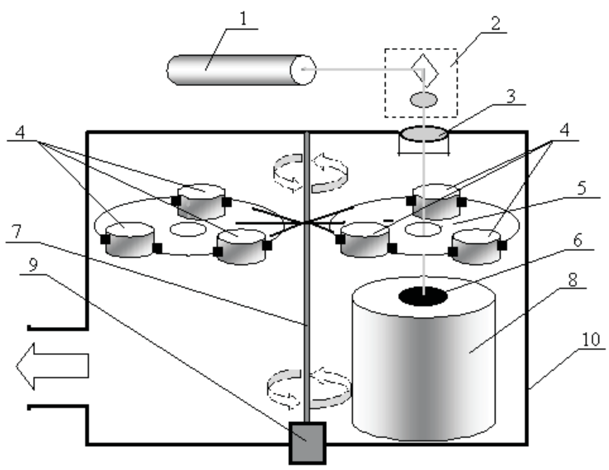



Fig. 3. The installation for hardening the high pressure apparatus matrices

We used the GOS-1001 laser (the energy in the pulse 1000J, the duration of the pulse 800μs, the divergence angle 20‘, pulse-repetition cycle – no more that 1 for 5 min.) and the GOI16-1 laser (the mean power of laser radiation 16 W, energy divergence of laser radiation –

$3.6^{*}10^{-3}$ rad., pulse repetition rate of laser radiation 8 – 50 kHz.

The trials of the matrices hardened by the combined ion-beam and laser method for the operational stability have took lace at the "Kristal" enterprise (sited in Gomel, Belarus).

TABLE 1

The results of the operational trials of the high pressure apparatus matrices

| Mode of handling | With the GOS – 1001 Laser | | | With the GOI – 16 laser | | | Without laser | | |
|---|---|---|---|---|---|---|---|---|---|
| Time of handling, min | 30 | 50 | 60 | 30 | 50 | 60 | 30 | 50 | 60 |
| The number of loadings before failure | 501 | 573 | 573 | 600 | 632 | 635 | 512 | 563 | 565 |
| Relative stability*, % | 103 | 117 | 117 | 123 | 130 | 130 | 105 | 115 | 116 |

*The stability of the matrices as compared to the uhandled matrices of the same batch.

The matrices were operated under the following conditions: outside pressure – $(240 - 250)^{*}10^{5}$Pa; the period under pressure – 18 sec. The data on the modes of handling and operational stability are given in table .

## 4. Conclusions

The trials have shown the efficiency of handling the hard-alloy matrices of high pressure apparatuses in low-energy argon plasma with the simultaneous spraying of the i-carbon film. There is a certain critical period of time after which it is not expedient to continue the process of handling. It can be explained by both the saturation of the thin surface layer of the piece of work with the $Ar^{+}$ and $C^{+}$ ions and the reaching of some critical width of the i-carbon film. In the experiment, which lasted for about 60 minutes, the width of the i-carbon film on the surface of the work piece was about 1μm. With the further growth, the mechanical properties of the i-carbon film have deteriorated because of its irregular structure and the big difference in the macroscopic properties of the materials of the film and the piece of work.

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