INTRODUCTION TO MODERN TECHNOLOGIES IN BELARUS – PHYSICAL-TECHNICAL INSTITUTE

Part 2

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VACUUM INSTALLATION VU1000 «ALMAZ»

- Is intended for a wide range of coatings deposition and equipped with different plasma and ion sources.
- Specifications: conductive and dielectric coatings can be deposited
- Automatic control system
- Coating temperature and color monitoring are available
- Half-automatic loading system
- Oil-free pumps
- Chamber dimensions: 1000 × 1000 mm
The carbon plasma is formed by a short-time electrical arc discharge owing to thermal erosion of graphite cathode. The plasma is accelerated by a Hall-type plasma accelerator and directed towards the substrate. In the plasma source, the discharge ignition is realized by a discharge between special electrodes placed around cathode. The electrodes have a contact through thin carbon film that deposits periodically during previous discharge. Control thyristor is used to switch discharge of the ignition capacitor of 2 ... 10 \( \mu \)F. The pulse arc discharge releases owing to energy of main capacitors battery having a capacity of some thousand of microfarads. The ignition system has a localized electrical contact of thin film conductor, deposited on the surface of the insulator. The localised contact moves from pulse to pulse along ring-shaped ignition electrode around the cathode. Such technique provides a high discharge ignition probability and practically avoid the erosion of an insulator, providing a long service life of the ignition system.
TECHNICAL SPECIFICATIONS:

The graphite cathode diameter, mm                  33
Main discharge voltage, V 200...400
Auxiliary discharge voltage, V 200...400
Ignition discharge voltage, V 600
Discharge impulse repetition rate, Hz 1...10
Maximal power, W 1000
Maximal deposition rate, μm/min 0.12
Range of discharge impulse setting 1 to 99999
Weight, kg 15
Control of graphite cathode feeding
Equipment

Vacuum machine of UVNIIPA -1001 and its vacuum chamber containing arc DLC coated parts of a plastic mold
Vacuum machine VU-700DL for arc DLC films and coatings deposition equipped with laser ignition system and large graphite rotated cathode

<table>
<thead>
<tr>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter of vacuum chamber, mm</td>
<td>700</td>
</tr>
<tr>
<td>Height of vacuum chamber, mm</td>
<td>700</td>
</tr>
<tr>
<td>High-vacuum pumping, l/s</td>
<td>3200</td>
</tr>
<tr>
<td>Low-vacuum pumping, l/s</td>
<td>80</td>
</tr>
<tr>
<td>Time to achieve pressure $10^{-3}$ Pa, min</td>
<td>25</td>
</tr>
<tr>
<td>Speed of deposition DLC using double planetary rotation, nm/min</td>
<td>4</td>
</tr>
<tr>
<td>Area occupied by machine, m²</td>
<td>15</td>
</tr>
<tr>
<td>Height of machine, cm</td>
<td>240</td>
</tr>
<tr>
<td>Dimensions of graphite cathode, mm</td>
<td>Ø140x330</td>
</tr>
<tr>
<td>Hydraulic system</td>
<td>SMC, Japan</td>
</tr>
<tr>
<td>Pneumosystem</td>
<td>SMC, Japan</td>
</tr>
<tr>
<td>Control system</td>
<td>PLC, Omron</td>
</tr>
</tbody>
</table>
Vacuum Machine VU-700-001 for arc DLC films and coatings deposition equipped with two pulsed arc carbon plasma sources and magnetic separators

SPECIFICATIONS

Vacuum chamber dimensions, mm 500x600x650
Vacuum pump set: turbopump (1500 l/s), rotary vane (16 l/s)
Gas flow control system: 5 channels (Ar, N₂, O₂, CH₄, C₂H₆)
Rotary drive: planetary rotation with variable and monitoring rotary speed is up to 10 rpm.
Coating area, cm² 400
Discharge impulse repetition, Hz 1…20
Max deposition rate, μm/h 0.3
Process control: Fully automated process control by PLC (with color touch panel) having computer interface. Up to 20 coating programs for different applications can be stored.
Power connection: 400 V ±10%, zero, four wires, protective earth, 50 Hz, approx. 45 kW
Vacuum machine VDU-CI for pulsed arc DLC films and coatings deposition equipped with four arc carbon plasma sources and magnetic separators

**SPECIFICATIONS**

- Power consumption, kW: 60
- Vacuum chamber, mm: 900×900
- Number of carbon plasma sources: 4
- Number of gas ion sources: 2
- Graphite cathode diameter, mm: 29
- Number of metal plasma sources: 3
- Metal cathode diameter, mm: 80
- Pulse repetition, Hz: 10
SUPER HARD DIAMOND-LIKE CARBON COATINGS

DLC film characterization

AFM image of the DLC film on silicone wafer
Ra = 0.19 nm, Rq = 0.34 nm,
Rv-p = 15.96 nm

Raman shift
D-peak position: 1379.1 cm⁻¹
G-peak position: 1574.1 cm⁻¹
I_D/I_G = 0.15

Hardness & Young’s modulus vs nanoindentation depth for the 1200 nm DLC film on the WC substrate
HV = 8500 (85 GPa), E = 950 GPa

DLC film specifications

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density, g/cm³</td>
<td>2.8…3.0</td>
</tr>
<tr>
<td>Hardness, GPa</td>
<td>60…85</td>
</tr>
<tr>
<td>Young’s modulus, GPa</td>
<td>650…950</td>
</tr>
<tr>
<td>Thermal stability, °C</td>
<td>300…350</td>
</tr>
<tr>
<td>Thickness, nm</td>
<td>50…2500</td>
</tr>
<tr>
<td>Roughness Ra, nm</td>
<td>0.2…0.5</td>
</tr>
<tr>
<td>Friction coefficient</td>
<td>0.05…0.1</td>
</tr>
</tbody>
</table>
NANOSIZE STRUCTURE OF THE DLC FILM

TEM image of the DLC film
Advantages of method

◊ **High ionization rate of plasma plum up to 98%** ⇒ high nucleation density ⇒ continuous films at small thickness ⇒ smooth film surface is provided.

◊ **Quasi-neutrality of plasma flow on a substrate** ⇒ 1) dielectric substrates may be used, 2) no harmful micro discharges.

◊ **High energies of carbon ion up to 60...70 ev** ⇒ 1) high content of sp³ carbon bonds up to 85% ⇒ high density up 2.9...3.1 g/cm³ ⇒ excellent barrier against metal ion diffusion, 2) high hydrophoby ⇒ good biocompatibility, 3) good adhesion, 4) no bias voltage is needed ⇒ uniform coating on a substrate of a complex geometry.

◊ **A number of simultaneously existing cathode spots and their high velocity** ⇒ low contamination of plasma plum by the graphite macroparticles ⇒ high film surface quality ⇒ application of DLC films for precession technique and medicine using simple plasma filter set.

◊ **Possibility to use high discharge current to guide plasma through magnetic filter effective** ⇒ no need for any additional high power current sources.

◊ **Precession film thickness control by discharge impulse number set.**
DLC COATING PROPERTIES

- Thickness, nm: 10-1000
- Density, g/cm³: 2.8-3.1
- Young’s Modulus, GPa: 650-700
- Hardness, GPa: 60-70
- Friction coefficient: 0.1-0.15
- Wearing, μm³/N·m: (1-3)·10³
- Thermo-stability, °C: up to 400
- Thrombocyte test Index: > 4.5
- Endotelization period, days: 30

SUPER HARD DIAMOND-LIKE CARBON COATINGS

CUTTING TOOLS

MEASURING INSTRUMENT

MACHINE PARTS (a ball connection, plungers)

ARTIFICIAL HEART VALVES
APPLICATION OF THE DLC COATINGS IN MEDICINE

The DLC coatings reduce the extent of valve-related thrombogenesis by surface modification making the surfaces more adherent to endothelial cells and albumin and less adherent to platelets, thus improving. About two thousand articles have been manufactured and used in medicine by now.

Bodies of the artificial heard valves made of VT-16 Ti-alloy coated by the DLC thin films

The artificial heart valves assembled
APPLICATION OF THE DLC COATINGS IN MEDICINE

View of DLC coated implants: fixing damaged back bone section set (A), and rotor of the blood pump (B)
Arc DLC films deposed on very little watch pinion axis to decrease friction and to extend their survivability by several times as well as decorative coatings on watch bracelets which change interference color depending on their thicknesses.
INDUSTRIAL APPLICATION OF
THE DLC COATINGS - flexible polymer band


The sample of DLC flexible abrasive

Fields of using:
- precision polishing of superhard materials;
- silicon wafer polishing;
- treatment of magnetic disks and heads;
- finish treatment of optic lens and optic disks

Polishing machine equipped with DLC flexible abrasive (3M Corporation, USA)
An appreciable life time increase have been achieved for plungers of Diesel engines and ball pivots coated with DLC films.

Plungers for Diesel engines coated with DLC coating.

Ball pivots for MAZ trucks coated with DLC and TiN films as the best combination for friction pairs.
Super hard diamond-like carbon coatings on cutting tools increase their life time 1.5…2 times as much. These coatings are especially effective in a case of wood cutting tool due to corrosion resistance and anti-sticking resin effect of the DLC coatings. The good results of DLC coating application have been obtained for cutters assigned for nonferrous metal and reinforced plastics treatment.
Punches for chain production

The punches for chain fabrication coated with DLC films obtained using a new combined PVD/CVD arc technology revealed increasing of the life time by a factor of 2.5 - 3
Measuring tools

Wear resistant carbon DLC deposited on a work surface of measuring tools such as plain gages-plugs, thread gages, Johansson blocks, etc. repeatedly magnify their resistance to wear, longevity and calibration interval periods.

Measuring tools with DLC coatings have the following technical characteristics:

- increase of in term service > 3 times
- increase of recalibration interval 5…10 times
- friction coefficient with steel < 0.15
Arc DLC is a chemically inert material in respect of all plastics we know. The arc DLC coating prevents wear, corrosion and sticking of any plastic on work molds and dies surfaces. They improve plastic articles surface smoothness. The positive combined effect achieved if the coatings are applied for friction part of mold due to a low dry friction coefficient of arc DLC coatings. It was shown in short-run environment that arc DLC coatings extend survivability of molds by 3…5 times.
Fine abrasive material

Abrasive material is a flexible polymer band having special surface texture with deposited arc DLC

**Fields of Application**
- precise polishing of super hard materials
- polishing of silicon plates for microelectronics
- treatment of magnet disks and magnet heads
- finish treatment of optical disks and lenses

The arc DLC abrasive material has been patented all over the world
Technology description

Technology of Electrolyte-Plasma Polishing is based on physical-chemical processes taking place in electrolyte region closed to surface of electrodes immersed into solutions of mineral salts in the conditions of high electrical potential leading to micro arc plasma etching just of a treatment surface.

This technology excepts mechanical influences on articles and charging abrasive particles into a surface.

It can be used to treatment of metal articles manufactured from copper and its alloys (brass, bronze, German silver), low-carbon and stainless steels and other electroconductive materials.
The process realizes in electrolyte which constitutes a solution of ammonium salts (chloride, sulfate and nitrate) and a little domestic of the organic dissolvine Z. \((C_{10}H_{16}N_2O_8)\). Concentration of salts varies in a range 1\% to 10 \% to provide electrolyte density of 1.02 g/cm\(^3\). Before carrying out of polishing the electrolyte is heated up to temperatures of 60...90 °C.

The positive potential of 150...200 V is applied to any article treatment. The potential is higher for articles made of steel comparing with those of copper alloys. Processing time is 30 s for deflashing, 5..8 and 10...15 mins for copper- and steel polish, respectively.

As a result of the polishing process, there is a deflashing and blunting of sharp edges as well as pretreatment of article surface to be coated by galvanic or vacuum films. The surface roughness decreases up to \(Ra = 0.2...0.05 \mu m\).
EIP-10 machine was designed and manufactured to realize the technology. Specifications:
- Maximal consume power, kW: 10
- Maximal polishing surface, cm²: 250
- Specific electrical energy consumption, kW·h/m²: 30
- Anode current density, A/cm²: 0.2...0.6
- Efficiency, %: 85
- Time of continuous work, h: 8
- Sizes: length, cm: 1400
  - width, cm: 710
  - high, cm: 2100
- Wight, kg: 200
Samples of stainless steel (A), copper (C), before polishing and the same samples after polish (B) and (D), respectively

Wares: (E) knifes for vegetables before (top) and after (down) polishing, (F) brass bracelets polished for watches
CROSS - WEDGE ROLLING

Forming of various-shape parts is carried out within one rolling cycle. Tool is easy in design, manufacturing, adjustment and repair. Tool is replaced in several minutes.

All constructional steels, some instrumental steels and copper and titanium alloys can be cross-wedge rolled.

Metal loss is decreased by up to 40 %
MANUFACTURING OF FEW-LEAF SPRINGS

- The process productivity is increased by a factor of 4 to 5
- Power requirement is decreased by 35 to 40 %
- Spring life is increased by a factor of 1.5 to 2.0
- Spring weight is decreased by 25 to 30 kg

The cost of technological equipment is decreased by a factor of 4 to 5

16 patents are obtained in Belarus, Russia, the USA and Great Britain. The license is sold and production is mastered at “Eaton” Company (USA)
MAGNETIC PULSE STAMPING

Strength, thermal and electrophysical effects of interaction between magnetic fields and material

- Multi-functionality
- Flexibility
- High quality
- Minimum power requirements and material and labor consumption

Decrease of expenditures on
- Power consumption is up to 3 times
- Die tooling up to 10 times
- Period of preparation for production up to 5 times
OBTAINING of BULK NANOSTRUCTURED MATERIALS by ECAP

Equal-channel angular pressing (ECAP) was invented in 1972 at the Physical Technical Institute. Multi-pass ECAP processing of materials by plastic deformation to produce a uniform nanostructured materials down to the nanometer range. Any type of microstructure, from equiaxed to any degree of grain elongation can be formed. The capability to control grain orientation has great practical significance for creation of anisotropic or isotropic products with various functional designations: structural, magnetic, superconducting, etc.

Pure metals such as Al, Mg, Ti, Cu, Ni, Fe, Nb, etc. and for a number of their alloys
## MECHANICAL PROPERTIES OF NANOSTRUCTURED MATERIALS

<table>
<thead>
<tr>
<th>Material</th>
<th>Structure</th>
<th>Hardness HV</th>
<th>Ultimate tensile strength, MPa</th>
<th>Elongation δ, %</th>
<th>Reduction of area ψ, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure Fe</td>
<td>Nano polycristal</td>
<td>300 90</td>
<td>980 350</td>
<td>6 21</td>
<td>55 70</td>
</tr>
<tr>
<td>Pure Cu</td>
<td>Nano polycristal</td>
<td>155 55</td>
<td>480 240</td>
<td>11 19</td>
<td>67 87</td>
</tr>
<tr>
<td>Pure Ni</td>
<td>Nano polycristal</td>
<td>310 70</td>
<td>920 400</td>
<td>8 22</td>
<td>62 90</td>
</tr>
<tr>
<td>18-10 steel</td>
<td>Nano polycristal</td>
<td>640 200</td>
<td>1550 560</td>
<td>27 70</td>
<td>41 69</td>
</tr>
<tr>
<td>Invar N36</td>
<td>Nano polycristal</td>
<td>300 120</td>
<td>850 460</td>
<td>16 30</td>
<td>74 88</td>
</tr>
</tbody>
</table>
ITEMS FOR BIOMEDICAL USE
(PELVIS-FEMUR JOINT ENDOPROTHESIS)

Endoprothesis of the system SLPS (Self locking Porous Systems) are manufactured jointly with “Altimed” Company.

Standard requirements of ISO 5832/3 are observed.

Development of the technology for production of endoprothesis blanks of 12 sizes from the titanium VT6 and Co-Cr-Mo alloys using the method of hot volume forming. V-shape of shank ensures exclusive fixation and rotary stability in three planes.

Mechanical properties:

• Tensile strength is 960 to 1100 MPa
• Yield strength is 860 to 880 MPa

Economic efficiency is 1500 USD for one endoprothesis

X-ray photograph after implantation of endoprothesis
Thank you!

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