Application of the dusty plasma technology for diamond ceramics production

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One of the most widespread industrial methods for producing diamond compacts and two-layer plates (a diamond layer on a substrate from a hard alloy WC-Co) is sintering the diamond in the presence of an activating additive – cobalt. Cobalt is a solvent for carbon and a catalyst in the transformation of graphite to diamond, which stimulates the sintering of diamond and the formation of a hard diamond frame (matrix). However, the presence of cobalt in the final product has a rather negative effect upon the diamond thermo-stability in the processes of production and use of the instrument. The difference in a thermal expansion of the matrix and its inclusions results in a formation of cracks and, finally, in a short life or even a failure of the operation part of the instrument. Cobalt is usually incorporated into a diamond powder either by being infiltrated or by previously mixing reagents. However, this technique does not allow homogeneous distribution of cobalt in the sintered mixture when the cobalt density becomes less than 5 in volume percent, especially if the size of sintered diamond particles is less than 5 µm. Therefore deposition of thin Co layers upon the surface of micron diamond particles with content 1–3 vol. (2–6 mass. percentage) in the charge mixture may become a promising approach to sintering of thermo-resistant diamond ceramics.

To deposit cobalt nanolayers upon individual diamond particles we used a plasma-dusty method. The idea of such method for depositing metal coatings upon the surface of microparticles having the size of about 1–10 µm was proposed in [1, 2]. The method for depositing coatings is based on particles’ levitating in the particular area of plasma, that is, in plasma-dusty traps. A coating is deposited upon the surface of levitating particles by an atom flow created by a magnetron sputtering system. The particles in plasma acquire great negative charges, which prevents the particles’ agglomeration. This method was practically used in [2], where the performance of a powder with a nano-sized coating production was several cm³/hour.

In Fig. 1 there is a scheme of the reactor, which was used in present work, for plasma-dusty deposition of cobalt coatings upon diamond microparticles. A reactor was placed in a vacuum chamber. Inside the reactor there is RF plasma in which there forms a plasma-dusty trap.
containing a dusty cloud with the particle density up to $10^6 \text{ cm}^{-3}$.

Fig. 1. Scheme of the reactor: 1 – the RF electrode, 2 – the reactor wall, 3 – the plasma-dusty cloud, 4 – the system for dispersion and collection of the powder, 5 – flow of Co atoms from a magnetron sputter. The arrows in the lower area of the figure point out to the direction of the reactor vibration in a powder dispersion process.

Powder particles from the inertial dispersion system in the lower part of the reactor enter the plasma area where they are coated by a flow of atoms from the magnetron sputtering system through the hole in the upper part of the reactor. Levitating particles are exposed to a cobalt atom flow from the sputtering system, after which they enter the dispersion system again. Such scheme decreases the probability for the formation of particle agglomerates in the final product in case of an incomplete dispersion.

Powders were produced with various (1–3) mass. percentage of cobalt which were then used for fabrication of compacts (with the thickness of 3 mm and the diameter of 4 mm) by sintering the diamond powder with the cobalt coating under the pressure of 8 GPa in high pressure chambers of the “toroid” type. Heating the reaction area up to the temperature of 2000–2100 K (it is higher than the melting temperature of Co under the pressure of 8 GPa) was performed under the pressure of 8 GPa with the rate of 50–100 °C/s. On being exposed to the constant P and T for 5–120 seconds, the specimens were cooled under a constant pressure.

In Fig. 2 there is a microstructure of diamond particles with a cobalt coating under the mean cobalt density in the powder about 3 mass percent. It is seen that cobalt is relatively homogeneous over the surface of diamond grains. Most interesting results are for the diamond powder in which the maximal cobalt density about 3 mass percent was achieved. In the Table there are physical properties of the produced compacts. For comparison there are literature data for the properties of the best diamond compacts produced according to the traditional methods of a binder injection.
Fig. 2. Microstructure of the diamond powders with a cobalt coating. X-ray point images corresponding to carbon (a) and cobalt (b) demonstrate relatively homogeneous distribution of cobalt on diamond particles.

Table 1. Physical properties of diamond compacts ($\rho$ is the density, $V_L$ and $V_T$ are the longitudinal and transverse velocities of sound, $K_s$ is the volume modulus of elasticity, $E$ is the modulus of elasticity, $\mu$ is the Poisson coefficient). The sintering temperature is 2050 K.

<table>
<thead>
<tr>
<th>N of specimen</th>
<th>$T_{\text{anneal}}$ (K)</th>
<th>Pressure and sintering duration</th>
<th>$\rho$ (g/cm$^3$)</th>
<th>$V_L$ (km/s)</th>
<th>$V_T$ (km/s)</th>
<th>$E$ (GPa)</th>
<th>$K_s$ (GPa)</th>
<th>$\mu$</th>
</tr>
</thead>
<tbody>
<tr>
<td>37-2</td>
<td>900</td>
<td>8–9 (GPa), 5(s)</td>
<td>3.6</td>
<td>16.612</td>
<td>10.25/9</td>
<td>917</td>
<td>495</td>
<td>0.19</td>
</tr>
<tr>
<td>37-3</td>
<td>970</td>
<td>7–8 (GPa), 20(s)</td>
<td>3.6</td>
<td>14.470</td>
<td>9.291</td>
<td>717</td>
<td>340</td>
<td>0.15</td>
</tr>
<tr>
<td>37-12</td>
<td>870</td>
<td>7–8 (GPa), 10(s)</td>
<td>3.6</td>
<td>13.968</td>
<td>7.958</td>
<td>575</td>
<td>399</td>
<td>0.26</td>
</tr>
</tbody>
</table>


It is seen that mechanical properties of the specimens produced in the first set of experiments are of extreme values in comparison with the corresponding literature data. Studying a microstructure specimens’ shows relatively homogeneous distribution of cobalt over the specimen (Fig. 2 b) under the mean cobalt density of about 3 mass percent, this agrees with the cobalt percentage in the sintered powder. On the whole, the close intergrowth of diamond grains is typical for specimens (Fig. 3), which provides unique properties of compacts in spite of the presence of submicron-sized metal particles in the sintered material.
Fig. 3 demonstrates diamond grains intergrowth (a) and rather homogenous distribution of cobalt among diamond grains (b).

Fig. 3. Microstructure of diamond compact N 37-2 in the secondary (a) or reflected (b) electrons.

Conclusions
A method for magnetron deposition of nano-sized cobalt coatings upon diamond particles confined in the plasma-dusty trap was developed. A diamond powder with the particle size of 3–7 μm with a cobalt coating was sintered under the temperatures within 2000–2100 K and the pressure of 8 GPa. The produced compacts had high values of the Young's modulus 917 GPa and the modulus of dilatation 495 GPa, which indicated the formation of the solid bond among diamond particles.

References: