Increasing the production efficiency of metal nanoparticles using electron beam deposition in a vacuum

Gornostai Oleksii Volodymyrovych

1 Ph.D., science researcher; E.O. Paton Electric Welding Institute of the National Academy of Sciences of Ukraine, Ukraine

Abstract. Approaches to increase the efficiency of the use of materials that evaporate with subsequent deposition in a vacuum have been analyzed. The high efficiency and advantage of deposition from an evaporator with a directed vapor flow in a vacuum compared to a classical crucible evaporation scheme are shown. The optimal technological modes of deposition from the evaporator to achieve homogeneity of the directional steam flow were determined. The dependence between the temperature of the target, the efficiency of the evaporation scheme and the distance of the evaporator from the target was determined experimentally.

Keywords: electron-beam technology EB-PVD nanoparticles of silver and copper
The use of methods of condensation from the vapor phase in a vacuum (CVP) to obtain functional coatings allows you to give products properties that cannot be achieved by other methods. One of the areas of development of functional coatings is metal nanoparticles (NPs). Their unique characteristics, which are inherent to them due to the dimensional effect, make it possible to improve the mechanical, thermal, electrical, magnetic and optical properties of coatings and expand the range of their applications [1-3]. Today, the above-mentioned materials are increasingly used in the medical, paint, and textile industries [4-7]. At the same time, as a rule, we are talking about the use of NPs made of expensive materials (Cu, Zn, Ag, Au, Pt). So the question of increasing the efficiency of the use of deposited materials, especially considering the fact that the CVP methods have low efficiency [8], therefore obviously arises the search for possible approaches to increase the efficiency of the use of materials is relevant.

Analysis of literary sources. One of the possible ways to improve the efficiency of the use of vaporized/deposited materials is the direction of the steam flow: the use of steam pipes, the flow of inert gas to direct the steam flow to the target. Taking into account that among the methods of CVP, the electron beam surpasses all other sources of heating in terms of specific energy capacity, ease of control, efficiency and locality of heating, a decision was made using the example of the method of electron beam evaporation with subsequent deposition in a vacuum to implement a method of directed deposition of a vapor flow in a vacuum. Works [9, 10] present an approach consisting in directed deposition from the vapor phase (Directed vapor deposition DVD) - this is an electron beam evaporation technology designed to improve the efficiency of creating thick and thin film coatings on surfaces of a small area (usually 100 cm$^2$ or Less). It consists in the fact that the electron beam DVD system generates a stream of vapor from one or more crucible sources; a flowing carrier gas interacts with this stream and directs its stream to the surface of the coating. Instead of allowing the vapor stream to diverge in the standard EB-PVD manner, the DVD carrier gas stream generates gas atom/vapor atom collisions.
that keep the vapor stream focused and directed at the coating surface as a dense beam [9]. The first results of research on the new technology show that electron-beam evaporation of DVDs in combination with carrier gas transport have very defective, even porous structures [9, 11]. To solve this problem in the second generation of DVD technology, additional technical components plasma activation and electrical displacement of the coating surface are used. At the same time, there still remains a complex set of gas-/vapor-atom-surface/carrier-coating interactions, which are only now beginning to be investigated [11, 12].

Works [13, 14] demonstrated the possibility of using electron beam evaporation followed by deposition in a vacuum using an evaporator and a steam pipe, which makes it possible to direct the steam flow directly to the target at an angle of 45°. The use of an evaporator with a vapor flow direction at an angle of 90° can increase the efficiency of the evaporation scheme and increase the reproducibility of the NP synthesis process. The works [15, 16] demonstrated the possibility of using the EB-PVD method with a directed vapor flow for the synthesis of Ag and Cu nanoparticles in the size range of 20...40 nm in liquid matrices based on monomers, polyurethane precursors, fatty and synthetic oils. The possibility of obtaining discrete Ag and Cu nanometer-sized coatings on powders and granules of different dispersion [17] and medical bandages [18] is also presented.

The purpose of this scientific work is to increase the efficiency of the EB-PVD method due to the formation of a directed steam flow using a reactor-type evaporator, which will lead to optimization of material consumption and more efficient synthesis of nanoparticles.

Materials, equipment and research methods

Experiments were conducted on the UE-142 electron beam unit to determine the efficiency of the technological scheme of using vaporizers with a directed vapor flow and the homogeneity of the distribution of the vapor flow on the surface of the target. An electron gun with an accelerating voltage of 20 kV was used to heat the materials and convert them into the vapor phase. The working chamber had a vacuum level of 5*10^{-3} Pa. Schemes of angular and vertical evaporation
with directed vapor flow from top to bottom were developed and applied (Fig. 1). The steam flow was directed to the horizontal plane at 45° and 90° at a negative angle. A steam stream was formed in the form of a cone over the target. Increasing the area (surface) of the target and decreasing the distance from the steam pipe to the target allows the target to collect as much steam flow as possible.

In this case, the distance between the steam pipe and the target affects the efficiency and heating of the target due to heat transfer. The values of efficiency, thermal effect, and uniformity of distribution of the metal vapor flow over the surface of the target were used to determine the distance from the reactor steam line to the surface of the dispersed liquid. The length of the steam pipe was chosen to prevent condensation of metal from the steam stream on the inner walls of the molybdenum tube.

Deposition was carried out on the surface of a flat copper disk with a diameter of 90 mm. The size of the disc corresponded to the inner diameter of the copper water cooling bowl in which the target material (liquid or powder) was placed. Before deposition, the disk surface was cleaned and degreased with technical alcohol. The disk was placed on top of the bowl so that the axis of the steam pipe coincided with the center of the copper disk. The distance between the evaporator and the disc corresponded to the distance between the evaporator and the surface of the liquid, powdery target. Weighed the copper disc after placing it in the vacuum
chamber. After the deposition, the weight of the disk was fixed again. The weight of the silver sample before and after deposition was also recorded. Knowing the amount of evaporated silver and the increase in the weight of the copper disc, the proportional efficiency was determined. During the deposition process, the temperature of the copper disc was fixed using a chromel-alumel thermocouple.

At the next stage of experimental work, it was determined how the steam flow is distributed on the surface of the target. For this purpose, witnesses were made - flat copper squares measuring 10 by 10 millimeters. To determine the distribution of the steam flow on the surface of the copper disk, the witnesses were placed on its surface with a step of 10 mm along and across the horizontal and vertical diameters of the copper disk. The weight of the witnesses was recorded before and after deposition, just as for the copper disk. To determine the amount of evaporated silver, the silver samples that were used for the experiment were first weighed. Proportions were then used to determine the percentage weight gain of each witness compared to the total weight gain of all witnesses. Based on the data, diagrams of the distribution of steam flow on the surface of the target were created for both types of evaporators (Fig. 2). A set of computer programs known as "Statgraphics" was used to process statistical data.

**Research results and their discussion.**

Experiments conducted to determine the distribution of the steam flow over the surface of the target showed that the results show that the distribution of the steam flow for the corner evaporator is not uniform (Fig. 2, a). Vapor flow concentration gradients of 12–16% are observed for the left and right sides of the target. The left side of the target accounts for 67% of the deposited material, and the right side for 33%. In the vertical evaporator (Fig. 2, b), there is a concentration gradient of the vapor flow from the center to the edge of the target, which indicates a distribution interval of the vapor flow from 16 to 10% on both the left and right sides of the target. This is explained by the coincidence of the axis of the steam pipe with the center of the target, which indicates the relative homogeneity (symmetry) of the distribution of steam molasses for the left
and right parts of the target. To establish the distribution of the steam flow, a cover glass with the same area was used. The mass of the glass was compared before deposition and after deposition.

Experiments were carried out with vertical and corner evaporators. The efficiency of the vaporizer with the angular orientation of the steam pipe was 16 to 18% depending on the distance to the target, and the efficiency of the vaporizer with the vertical orientation was 36–40%.

![Figure 2](image)

The uniformity of the steam flow distribution on the surface of the test copper disk depending on the location of the graphite evaporator, %: a— the angular scheme of the steam flow deposition; b is vertical, where 1 is the direction of the steam flow.

A vertical reactor arrangement was chosen for further investigation because this arrangement has a more uniform distribution of steam flow and has an efficiency 2.2 times greater than the angular one. Table 1 shows the experimental data obtained regarding the dependence of the efficiency and temperature of the copper disc-target depending on the distance to the target.

<table>
<thead>
<tr>
<th>The distance from the tube to the surface of the copper disc, mm</th>
<th>Efficiency of the evaporation scheme, %</th>
<th>Surface temperature of the copper disk, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>25</td>
<td>61</td>
<td>116</td>
</tr>
<tr>
<td>45</td>
<td>56</td>
<td>72</td>
</tr>
<tr>
<td>65</td>
<td>40</td>
<td>47</td>
</tr>
<tr>
<td>85</td>
<td>33</td>
<td>41</td>
</tr>
<tr>
<td>115</td>
<td>22</td>
<td>36</td>
</tr>
</tbody>
</table>
Conclusions

1. The proposed version of evaporators with the possibility of forming in a given direction an intense steam flow of the evaporating material (Ag, Cu) makes it possible to reduce its costs by 6...10 times compared to the traditional scheme.

2. It was established that the efficiency of the evaporator with the angular orientation of the steam pipe is 16...18%, and with the vertical one - 22...61%. An evaporator with a vertical orientation of the steam pipe has a higher homogeneity of the distribution of the steam flow on the surface of the target in comparison with an angular evaporator, which gives a higher value of the reproducibility of the process of synthesis of nanoparticles in the volume of liquid matrices - carriers and on the surface of powders, granules, fabrics. The optimal distance is 70 mm, since at such values the limit temperature is not exceeded and the efficiency of the evaporation scheme is quite high.

References:


MODELING AND NANOTECHNOLOGY


This work is distributed under the terms of the Creative Commons Attribution-ShareAlike 4.0 International License (https://creativecommons.org/licenses/by-sa/4.0/).