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REGULAR ARTICLE

Preparation of Fe-Based Composite Materials with Controlled Cu and Au Doping by a Three Source EB-PVD Technique

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The paper presents a technological approach to modifying the iron vapor flow during electron beam physical vapor deposition (EB-PVD) by introducing a directed atomic flow of 1A subgroup alloying elements (Cu, Au). A technological scheme was proposed, consisting of three types of substrates: a vertical stationary substrate designed to analyze the vertical distribution of components in the vapor stream depending on the angle of inclination of the evaporator (5°, 15°, 45° and 65°); a horizontal stationary substrate designed to control the basic parameters of the deposition process without rotation; a cylindrical substrate, rotating, the main working substrate for obtaining composites with a uniform distribution of components. The proposed method of deposition of spatial overlap vapor streams in a vacuum with the formation of homogeneous composites in the NaCl matrix without layering of the initial components. It has been revealed that the angle of inclination of the peripheral evaporator determines the degree of overlap of the flows, which controls the concentration of dopant impurities in the films up to 1.9 mas. % Cu and 9.4 mas. % Au. The obtained films are characterized by a dense crystalline structure with columnar grain sizes of 0.8…1.4 μm, which is typical for non-equilibrium condensation conditions at temperatures lower than 0.3 of the melting point of the main components. It has been shown that the use of a rotating cylindrical substrate ensures a more uniform distribution of components and promotes the formation of homogeneous compositions. The results obtained confirm the prospects of using co-deposition from multiple evaporation sources EB-PVD technique with controlled doping to form metal-salt composites with potential applications of their active components in the fields of catalysis, biomedical coatings and magnetic structures.

Keywords: EB-PVD, Evaporator, Vapor flow, Nanoparticle, Composite materials, Structure.

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1. INTRODUCTION

The improvement of physicochemical methods for obtaining metal nanoparticles (NPs) is driven not only by the wide range of practical applications of such materials, but also by scientific and practical interest in improving the technology for their production [1-4].

Existing methods of chemical deposition, thermal decomposition, and hydrothermal synthesis do not fully meet the requirements for obtaining chemically pure metal NPs without the impurities. The main disadvantage of the chemical deposition method is the difficulty of predicting the size and morphology of particles. The disadvantage of thermal decomposition is the use of toxic and rather expensive precursors.

The hydrothermal method is quite versatile, but the relatively slow reaction kinetics limit even small-scale production. EB-PVD is one of the methods for obtaining iron-based composite materials [5].

Fe-NaCl composites obtained from two independent sources on rotating horizontal substrates have a layered structure that depends on the vapor flows (VF) placement and is regulated by the rotation rate (V_s) of the substrate [6].

The formation and size of the Fe-NaCl composite structure is also determined by the elemental composition of the VF and the deposition rate of the vapor phase. A technological scheme has been proposed with the introduction of an additional Au (Cu) vapor flow by using an additional W(Mo) evaporator with a corresponding ingot. This gave us the means to mix Fe and Cu (Au) VFs and to deposit the modified VF directly onto the substrate without the layering of the evaporated materials. Thus Fe-Cu-NaCl and Fe-Au-NaCl composites obtained on a rotating cylindrical substrate were selected as research objects.

The aim of the work was to evaluate the effectiveness of doping/modifying the base iron VF with a directed VF of copper or gold for the directed deposition of Fe-Cu-NaCl and Fe-Au-NaCl composites using EB-PVD on a rotating cylindrical substrate.

2. MATERIALS AND METHODS

Gold, copper, iron, and sodium chloride were used as source materials for evaporation. NaCl ingots were formed by cold pressing from vacuum-evaporated salt powder, Fe and Cu ingots were obtained by electron-

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beam remelting, and Au additives was processed from a bank ingot. Data on the purity of the source materials used are presented in Table 1.

Table 1 – Source materials, were used for evaporations

Material	Form/ preparing	Purity, mas.% (no less)
Au	bank's ingot	≥ 99.9
Cu	after EB-melting	≥ 99.85
Fe	after EB-melting	≥ 99.85
NaCl	cold pressure	≥ 99.9

The physical and chemical properties of evaporation materials are presented in Table 2.

The amount of material evaporated during the experiment was controlled by comparing the mass of the Fe, NaC and Au(Cu) ingot weights between experiments. NaCl through a graphite channeled plate tightly fixed on the crucible, and the Au and Cu weights with W and Mo crucibles.

Table 2 – Physical-chemical properties of sourced materials [7, 8]

Material	T_L, K	Density, g/cm^3	Vapor pressure at the T_L, Pa	V_{vap} at the T_L, g/(cm^2·s)	Atom mass, 1/12 ^{12}C	Atomic orbit R, nm
Au	1336	19.3	$2.11 \cdot 10^{-3}$	$36.43 \cdot 10^{-8}$	186.97	0.1187
Cu	1356	8.96	$5.86 \cdot 10^{-2}$	$57.36 \cdot 10^{-7}$	63.55	0.1191
Fe	1812	7.87	33.34	$26.45 \cdot 10^{-5}$	55.85	0.1227
NaCl	1074	2.17	–	–	–	–

The deposition process was carried out in a vacuum of $6.7 \cdot 10^{-3}$ Pa. An iron ingot (diameter of 25 mm) was evaporated using an electron-beam projector (EBP) at a current of 0.11...0.12 A with a rate of 0.0049...0.0092 g·s^{-1}. The NaCl vapor was directed through channels in a graphite plate fixed to a 50 mm diameter crucible onto a rotating cylindrical substrate. EBP current of 0.05 A ensured the evaporation of NaCl at a rate of 10.42...29.17 g·s^{-1}, maintaining a uniform metal phase distribution across the substrate.

Based on the experimentally established evaporation rates of Au and Cu at 0.0015...0.0025 g·s^{-1} at a current of EBP of 0.08 A for Mo and W evaporators, the optimal portions were calculated to be 0.20 g for the gold and 0.18 g for the copper.

The research was conducted on film samples separated from the substrate (Fig. 1). The surface morphology, chemical and phase composition, structure of composites were studied on samples.

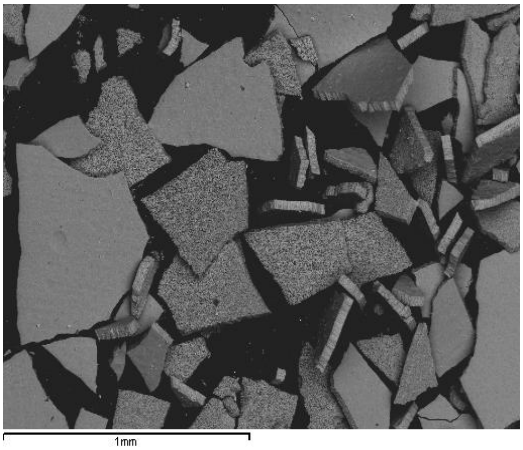
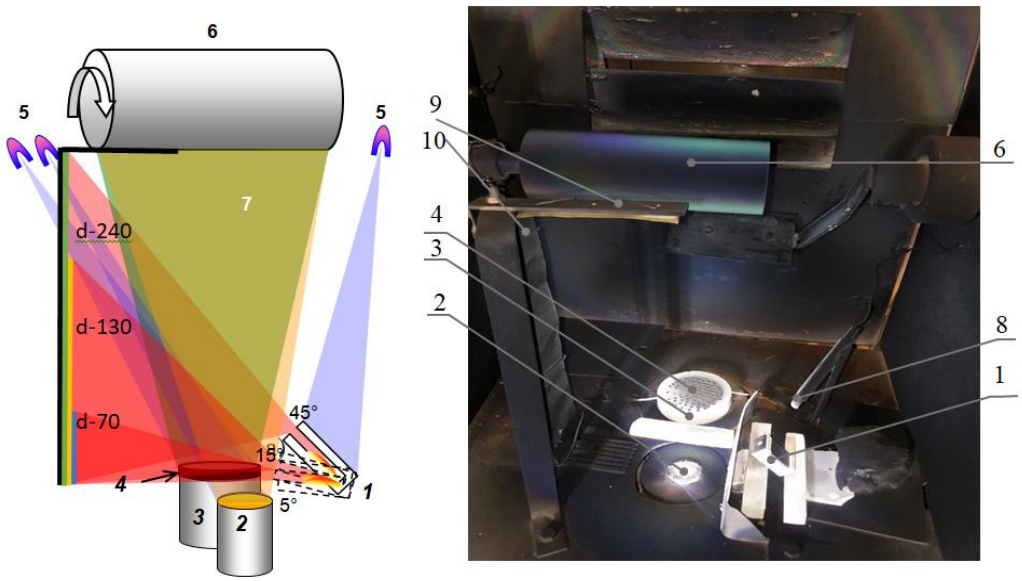


Fig. 1 – Typical view of samples of obtained composite material

Scanning electron microscopy studies (SEM) of the surface morphology and structure of cross-sectional samples were carried out on a Camscan 4S in back-scattered electron mode at magnifications ranging from ×300 to ×3000.



a b

Fig. 2 – Technological scheme of Fe vapor steam modification (a) and technological chamber EB-facility UE-142 (b) 1 – Mo- vapor source of Cu or W- vapor source of Au; 2 – Fe- ingot; 3- NaCl – tablet, 4 – reactor-evaporator; 5 – EB-projector; 6 – cylindrical substrate; 7 – modified vapor flow; 8 – thermo-couple; 9 – test stationary (horizontal) substrate; 10 – test stationary (vertical) substrate

The experiments were carried out on an electron beam facility UE-142 (Fig. 2b). To modify the main Fe vapor flow with a directed Cu/Au flow, a technological scheme with three evaporation sources was developed and applied (Fig. 2a): Fe – crucible diameter of 25 mm: NaCl – crucible diameter of 50 mm and Mo reactor-evaporator with a vapor's pipe diameter of 3.2 mm.

The adjacent Mo(W) evaporator serves to direct the copper (or gold) vapor flow into the iron vapor flow by means of an angle of inclination to obtain unified atomic-molecular flow of Fe-Cu-NaCl and Fe-Au-NaCl evaporated material.

The pressure above the liquid iron bath at a melting point of 1812 K is 33.34 Pa, and the evaporation rate is $26.45 \cdot 10^{-5}$ g·(cm^2·s)$^{-1}$. The vapor pressure of copper and gold at their melting point are $5.86 \cdot 10^{-2}$ Pa and $2.11 \cdot 10^{-3}$ Pa, respectively. The evaporation rate is given in a Table 2.

Refractory high-temperature metals were used to make the evaporator: molybdenum (Mo, melting point – 2893 K) and tungsten (W, melting point – 3693 K). These metals do not interact at high temperatures with the metals being evaporated: copper and gold. The temperature of the Cu/Au evaporator was controlled by a chromel-alumel thermocouple positioned 20 mm from the W evaporator.

A steel cylinder with a diameter of 55 mm and a length of 145 mm was used as a substrate. It was fixed on a water-cooled rod, which rotates at a given rate (V_s = 10 rpm) by an electric motor. Geometrically, the substrate is located in the main zone of spreading of the iron and NaCl vapor flows.

The adjacent Mo evaporator introduces directed copper vapor into the main zone of the iron vapor flow for modification, thus obtaining a common atomic-molecular flow of Fe-Cu-NaCl.

3. RESULTS AND DISCUSSION

When evaporating from a single crucible, the vapor flow of the material spreads according to the cosine law (cos φ). To estimate the modification of the Fe vapor flow by the directed Cu flow, experiments were conducted to measure distribution of the unified Fe-Cu vapor flow on vertical, stationary, and rotating cylindrical (V_s = 10 rpm) substrates depending on the angle of 5°, 15°, and 45° inclination of the Mo evaporator with a Cu overhang. The deposition time was 600 s, at a power of the electric heating device: P_{Fe} = 2.42 ± 0.02 kW, $P_{Mo/Cu}$ = 1.62±0.02 kW, P_{NaCl} = 1.±0.02 kW (Tables 3, 4, 5).

Table 3 – Technological parameters of evaporating of initial materials (Fe-Cu-NaCl)

Experiment No, / angle of vapor source incline axis	Technological parameters:			
	Vacuum, Pa	P_{Fe}, kW	$P_{Mo(Cu)}$, kW	P_{NaCl}, kW
#15 Fe-Cu-NaCl / 5°	$1.0 \cdot 10^{-3}$	2.45	1.63	1.02
#16 Fe-Cu-NaCl / 15°	$1.3 \cdot 10^{-3}$	2.42	1.62	1.01
#17 Fe-Cu-NaCl / 45°	$1.2 \cdot 10^{-3}$	2.44	1.62	1.02

Under the specified technological parameters for the experiments, the evaporation rate of the starting materials varied: Fe – 1.1...1.8 $\cdot 10^{-3}$ g·s^{-1}, Cu – 1.7...3.3 $\cdot 10^{-5}$ g·s^{-1}, NaCl – 2..1...4.9 $\cdot 10^{-3}$ g·s^{-1}.

Analysis of the elemental distribution in the upper part of the vertical substrate, in the vicinity of the rotating cylinder, revealed a decrease in iron content from 10.0 to 4.0 mas. % accompanied by an increase in copper content from 0.1 to 0.4 mas. % as the inclination angle of the peripheral evaporator increased from 5° to 45°.

This behavior is attributed to the change in the spatial orientation and angular distribution of the Cu vapor flow relative to the Fe-containing flow. As the inclination angle increases, the overlap between the Cu and Fe vapor flows on the substrate surface becomes more pronounced, leading to an increase in the local contribution of copper to the total deposition flow.

Table 4 – Distribution of elements of modified Fe-Cu-NaCl vapor flow on a vertical substrate depending on the angle of inclination of the evaporator (Fig. 2a)

Elements of vapor steam	Inclines angel of the axis additional vapor source / Chemical composition, mas. %								
	5°			15°			45°		
	downstaris	middle part	upper part	downstaris	middle part	upper part	downstaris	middle part	upper part
Fe	74.0	13.9	10.0	32.3	34.0	30.0	29.0	5.3	4.6
Cu	1.0	0.1	0.1	1.7	3.0	2.0	1.6	0.7	0.4
NaCl	25.0	86.0	89.9	66.0	63.0	68.0	69.4	94.0	95.0

Table 5 – Distribution of elements of unified Fe-Cu-NaCl vapor flow on a horizontal stationary and cylindrical rotating substrate depending on the angle of inclination of the evaporator (Fig. 2a)

Substrate	Inclines angel of the axis additional vapor source / Chemical composition, mas. %								
	5°			15°			45°		
	Fe	Cu	NaCl	Fe	Cu	NaCl	Fe	Cu	NaCl
stationary	13.6	0.1	86.3	12.2	2.3	85.5	4.9	0.4	94.7
rotating	19.6	0.1	80.3	18.1	0.7	81.2	16.2	1.9	81.9

The Cu vapor flow generated from the Mo evaporator follows an angular distribution close to the cosine law (cos φ). With increasing evaporator inclination, the effective deposition profile of the Cu flow expands and shifts relative to the substrate, resulting in a broader deposition area and enhanced spatial overlap with the Fe flow.

The observed changes in composition should be interpreted in terms of spatial superposition of independent vapor flows rather than the formation of a unified mixed stream. Under high-vacuum conditions, the mean free path of evaporated species is sufficiently large, and interparticle collisions are limited; therefore,

Fe and Cu atoms are transported to the substrate largely independently. The local composition of the deposited film is determined by the relative contributions of individual flows at a given point on the substrate surface, which are controlled by the geometry of evaporation sources and their angular flow distributions. Under these conditions, a composite with a composition of approximately 4.6 mas. % Fe, 0.4 mas. % Cu, and 95 mas. % NaCl is formed.

On a stationary substrate, the concentrations of the corresponding elements are slightly lower compared to those obtained on a rotating substrate, which can be attributed to the larger effective distance between the substrate and the evaporator and the absence of flow averaging.

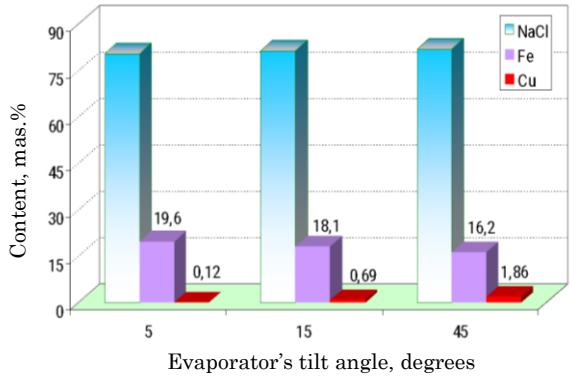


Fig. 3 – Histograms of the distribution of system components 16.2 mas. % Fe-1.9 mas. % Cu-81.9 mas. % NaCl depending on the angle of inclination of the Mo reactor-evaporator on a cylindrical substrate

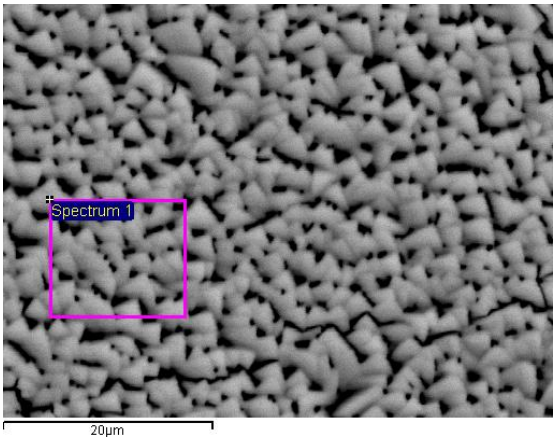
The distribution of elements in films deposited from Fe-Cu vapor flow on a rotating cylindrical substrate shows a slight decrease in iron content from 19.6 to 16.2 mas. % and a simultaneous increase in copper content from 0.1 to 1.9 mas. % as the inclination angle of the Cu-loaded evaporator increases from 5° to 45°. This behavior is associated with changes in the spatial distribution and angular orientation of the Cu vapor flow relative to the substrate. As the inclination angle increases, the spatial overlap between the Fe and Cu vapor flows becomes more significant, leading to an increase in the local contribution of copper to the total deposition flow.

Thus, the observed compositional variation is governed by the superposition of individual vapor flows rather than their physical spatial overlap, resulting in a composite with a composition of approximately 16.2 mas. % Fe, 1.9 mas. % Cu, and 81.9 mas. % NaCl (Fig. 3).

After deposition, Fe-Cu-NaCl composites were heat-treated in a technological chamber at a temperature of 373 K for 2400 s under a vacuum of $6.7 \cdot 10^{-3}$ Pa. The results of elemental distribution analysis in films deposited from modified Fe-Cu-NaCl vapor flow confirm the feasibility of controlled doping of the Fe vapor flow and the formation of compositionally uniform Fe-Cu-NaCl composites directly on a rotating substrate. The observed compositional uniformity is attributed to the spatial superposition of individual vapor flows combined with the averaging effect provided by substrate rotation. Based on the experimental results on the introduction of dopants with relatively high atomic mass into the Fe vapor flow and on the analysis of the elemental distribution in Fe-Cu systems as a function of the evaporator inclination angle, the applicability of other dopant elements, including those of subgroup 1 A of the periodic table, for modifying the Fe vapor flow was further explored. At the next stage, composite films were deposited using a gold-modified Fe-Au vapor flow. Due to the specific geometrical configuration of the experimental setup and the deposition conditions, a larger inclination angle of the W evaporator (up to 65°) was required to ensure sufficient spatial overlap between the Au and Fe vapor flows and to achieve a measurable contribution of Au to the total deposition flow. SEM analysis of the surface and cross-sectional fractures of Fe-Au-NaCl composites obtained on both stationary and rotating cylindrical substrates (Fig. 4) indicates a relatively uniform and dense microstructure without pronounced macroscopic separation of the source components. At deposition temperatures of $T_d = 353..383$ K, corresponding to $T_d < 0.3T_L$ (where T_L is the absolute temperature of melting of components),

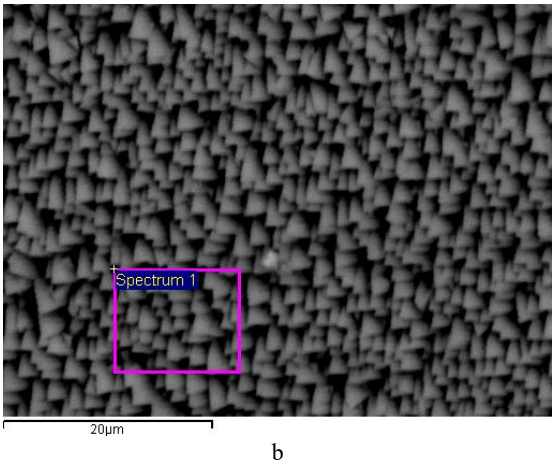
Surface of the samples

Stationary substrate



a

Cylindrical rotating substrate

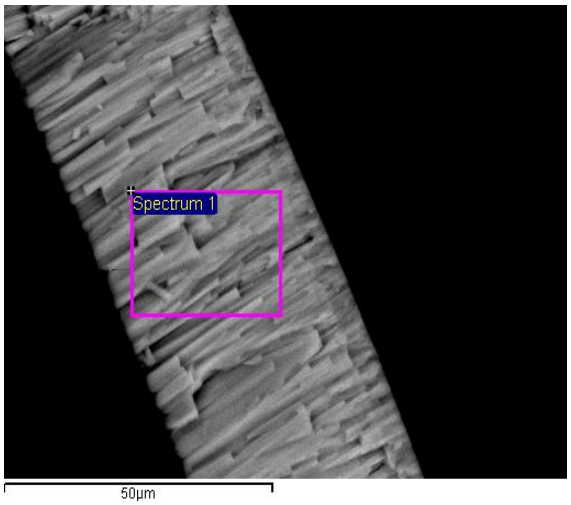


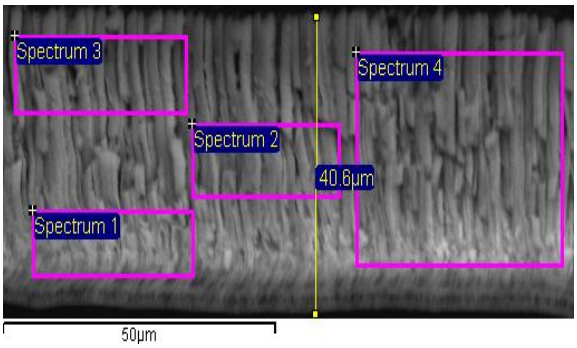
b

Chemical composition, mas. %	Na	Cl	Fe
Spectrum 1	21.61	75.82	2.58

Chemical composition, mas. %	O	Na	Cl	Fe
Spectrum 1	2.35	21.41	7.56	8.67

Cross-section spall





Chemical composition, mas. %	Na	Cl	Fe	Au
Spectrum 1	21.33	73.82	3.97	0.88

Chemical composition, mas. %	O	Na	Cl	Fe	Au
Spectrum 1	11.13	17.98	25.83	38.02	7.04
Spectrum 2	8.86	15.1	20.09	46.23	9.72
Spectrum 3	9.07	15.16	28.15	38.22	9.4
Spectrum 4	16.3	19.54	27.84	30.11	6.21

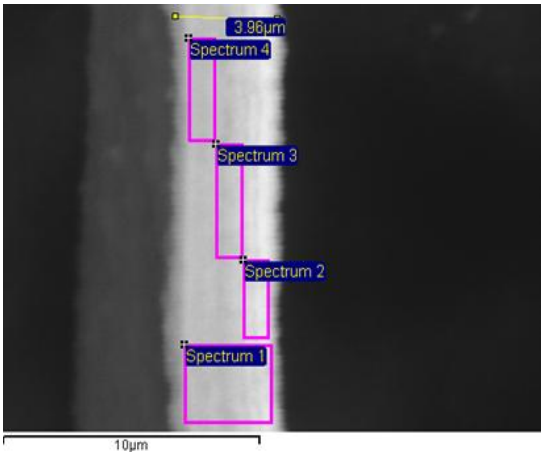
a b

Fig. 4 – Surface structure and elemental composition of cross fracture of Fe-Au-NaCl composites obtained on a stationary (a) and cylindrical (b) substrate

the film growth conditions can be associated with the Zone 1 of the Movchan-Demchishin Structure-Zone Model (SZM) [9]. Under these conditions, limited surface diffusion of atoms leads to the formation of fine-grained or columnar microstructures rather than well-developed equilibrium grains. The observed changes in surface topography of composites obtained on stationary and cylindrical substrates are consistent with the corresponding variations in cross-sectional microstructure.

The morphology and characteristic size of structural features in 8.7 mas. % Fe-1.4 mas. % Au-NaCl composites depend on the amount of Au introduced into the Fe vapor flow during modification. SEM analysis of cross-sectional fractures of the composites deposited at a rate of $V_d = 0.07$ μnm·s^{-1} revealed the formation of a columnar microstructure with column widths of approximately 1.2-1.4 μm for a stationary substrate and 0.8-1.0 μm for a rotating cylindrical substrate. Such a non-equilibrium columnar structure is associated with low substrate temperature during condensation and limited surface diffusion of atoms, which restricts lateral growth and promotes competitive columnar evolution.

The finer column size observed for the rotating substrate can be attributed to the dynamic change in the local incidence angle and temporal averaging of the deposition flow, which affects growth conditions and suppresses excessive column coarsening. Modification of the Fe vapor flow with a higher Au dopant content (6.2…9 mas. %) was carried out at the substrate temperature of $T_d = 333..363$ K and at reduced deposition rate of $V_c = 0.005$ μm·s^{-1} (Fig. 5).



Chemical composition, % mas.	O	Na	Cl	Fe	Au
Spectrum 1	14.59	22.62	51.27	10.28	1.23
Spectrum 2	9.59	21.82	59.12	8.05	1.42
Spectrum 3	5.76	20.55	63.79	9.34	0.56
Spectrum 4	11.46	20.99	57.34	8.87	1.35

Fig. 5 – Structure (a) and elemental composition (b) of cross fractures of Fe-Au-NaCl composites obtained on a rotating cylindrical substrate

Under these conditions, the resulting composites exhibit a more uniform and compact cross-sectional structure. The observed densification is primarily attributed to the decrease in deposition rate, which allows increased surface diffusion of atoms and promotes structural relaxation during film growth. These observations are consistent with the behavior of composites containing lower Au concentrations.

4. CONCLUSIONS

1. A technological approach for modifying the Fe vapor flow by co-deposition from multiple evaporation sources using EB-PVD has been proposed and experimentally implemented, enabling controlled introduction of Cu and Au dopants during film growth.

2. It has been established that the inclination angle of the peripheral evaporator determines the spatial overlap of individual vapor flows and, consequently, controls the local contribution of dopants to the total deposition flow. This provides a geometrical mechanism for regulating the film composition, allowing the formation of Fe-Cu-NaCl and Fe-Au-NaCl composites with dopant contents of up to 1.9 mas. % Cu and 9.4 mas. % Au without pronounced macroscopic separation of components.

3. The structure of the obtained composites is characterized by a columnar morphology with characteristic feature sizes of 0.8-1.4 μm, which is typical for non-equilibrium film growth at low homologous temperatures ($T_d < 0.3T_L$) and limited surface diffusion of atoms.

4. The use of a rotating cylindrical substrate promotes a more uniform distribution of components in the film and reduces compositional gradients. The obtained results demonstrate that geometrically controlled co-deposition in EB-PVD is an effective approach for tailoring the composition and structure of metal-salt composite systems, which are of interest for applications including catalysis, biomedical coatings, and functional magnetic materials.

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Отримання композитних матеріалів на основі заліза з контрольованим легуванням Cu та Au способом EB-PVD із трьома випарниками

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У роботі представлено технологічний підхід до модифікування парового потоку заліза під час електронно-променевого осадження (EB-PVD) шляхом введення направленого атомно-молекулярного потоку легуючих елементів 1A підгрупи (Cu, Au). Запропоновано технологічну схему, що складається з трьох типів підкладок: вертикальна стаціонарна підкладка, призначена для аналізу вертикального розподілу компонентів у потоці пари залежно від кута нахилу випарника (5°, 15°, 45° та 65°); горизонтальна стаціонарна підкладка, призначена для керування основними параметрами процесу осадження без обертання; головна циліндрична горизонтальна підкладка, що обертається - для отримання композитів з рівномірним розподілом компонентів.Запропоновано технологічну схему, яка дозволяє здійснювати геометрично контрольоване змішування парових потоків у вакуумі з формуванням однорідних нанокомпозитів у матриці NaCl без шаруватості вихідних компонентів у отриманому композиційному матеріалі. Встановлено, що кут нахилу периферійного випарника визначає ступінь перекриття потоків, що контролює концентрацію легуючих домішок у конденсаті до 1,9 мас. % Cu та 9,4 мас. % Au. Отримані конденсати характеризуються щільною нанокристалічною або стовпчастою структурою з розмірами зерен 0,8-1,4 мкм, що характерно для неріноважних умов конденсації при температурах < 0,3 від температури плавлення основних компонентів. Показано, що застосування циліндричної підкладки, що обертається, забезпечує більш рівномірний розподіл компонентів та сприяє формуванню гомогенних композицій. Отримані результати свідчать про перспективність застосування методики багатопотокового EB-PVD з керованим легуванням для створення метало-сольових нанокомпозитів із потенційним їх застосуванням у якості активних компонентів у галузях каталізу, біомедичних покриттів і магнітних наноструктур.

Ключові слова: EB-PVD, Випарник, Паровий потік, Наночастинки, Композитні матеріали, Структура.