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PRODUCING Fe-BASED NANOPARTICLES IN NaCl MATRIX BY THE METHOD OF EB-PVD ON A ROTATING SUBSTRATE

Yu.A. Kurapov, V.O. Osokin, G.G. Didikin, L.A. Krushynska, S.E. Lytvyn, V.V. Boretskyi

E.O. Paton Electric Welding Institute of the NASU

11 Kazymyr Malevych Str., 03150, Kyiv, Ukraine

ABSTRACT

The paper gives the results on the features of formation of a microlayered structure of NaCl–Fe composite and the possibility of producing iron nanoparticles of different size on a rotating substrate by EB-PVD method with peripheral position of evaporation material sources relative to the substrate rotation axis. It is shown that the layer thickness is determined by the substrate rotation speed. It is found that Fe nanoparticles are present in NaCl matrix in the form of Fe_3O_4 oxide.

KEY WORDS: electron beam evaporation, condensation, EB-PVD, composite, nanoparticles, iron oxide, phase composition

INTRODUCTION

Investigations of the conditions of formation of composite materials based on iron nanoparticles, having a set of medicobiological characteristics, are required for development of nanostructured medicinal substances with the purpose of modification of the existing medical drugs by active nanoparticles. The nanoparticles have a well-developed active surface and, consequently, high sorption capacity. Due to their size (less than 15 nm) the iron nanoparticles can interact and bind with biological objects, the size of which is equal to 10–100 nm in the case of cells, 920–450 nm for viruses, 2 nm wide, 10–100 nm long for DNA [1]. Formation of nanoparticles in water-soluble matrices allows storing them while preserving their size for a long time.

Experimental data accumulated so far, confirm the possibility of obtaining NaCl–Fe composite materials with nanoparticles of iron oxide (Fe_3O_4 , Fe_2O_3) on stationary substrates. The geometry of stationary substrate location relative to vapour flows of initial materials leads to formation of films with composition gradient up to almost 40 %, increases the inhomogeneity of NaCl–Fe materials and limits the technological process productivity [2–5].

EB-PVD process combines the possibilities of intensive controlled evaporation of various materials in vacuum with their subsequent condensation on a cooled rotating substrate, and it opens up broad possibilities of variation of application of diverse control schemes and automation of the parameters of this technological process performance [6–8]. Therefore, development of scientifically based electron beam technology of producing iron nanoparticles in NaCl matrix on a rotating substrate is an urgent task.

The work deals with the features of application of a technological procedure of conducting the EB-PVD process with the position of evaporation material sources peripheral relative to the substrate rotation axis to produce NaCl–Fe composites [9], and presents the results of experimental studies of the structure and some properties of the produced micro- and nanoscale materials.

INVESTIGATION MATERIALS AND PROCEDURES

Sodium chloride and Armco-iron were used as initial materials for evaporation. Material purity was 99.5 %. Sodium chloride cylinders of 49.8 mm diameter and 60–80 mm height were made by cold pressing. Iron ingots were produced by electron beam remelting of 60 mm diameter rods into a 52 mm crucible. Produced ingots were ground to finish size of 49.5 mm diameter and 150–200 mm length, which prevented their jamming in the crucible at vertical feed as they evaporated.

After complete depressurizing of the vacuum chamber, NaCl–Fe composite was separated from the substrate and stored in the initial state in air and in alcohol. In connection with the problem of making sections by a standard procedure from NaCl–Fe composites (NaCl matrix dissolves in H_2O), investigations were conducted on self-standing brittle chips of composites.

MICROSTRUCTURE

was studied on transverse (in the condensation direction) chips of the samples, using scanning electron microscopy Cam Scan 4D.

ELEMENTAL COMPOSITION

of the composites was determined using an X-ray spectral microanalyser — EDX attachment to Cam Scan 4D microscope. At application of INCA-200 Energy program for result processing, the measurement error was equal to 0.3 %.

X-RAY PHASE ANALYSIS (XPA)

Diffraction patterns of the samples were recorded in DRON-UM1 diffractometer in filtered cobalt (CoK_α) radiation in Bragg-Brentano filming geometry in the angle range of $10\text{--}85^\circ$ with 0.05 step and exposure in 2 s point. They were saved in the digital form in a file of $2\theta(\text{degr.})\text{--}I(\text{intensity, s}^{-1})$ format. Phase identification was conducted with application of X-ray data base PDF-2. Average crystallite size was calculated by Scherrer equation. Semi-quantitative phase analysis was performed using Match program.

STUDYING THE SIZE OF IRON POWDER

PARTICLES OF $\text{Fe}_2\text{O}_3\text{--NaCl--H}_2\text{O}$

COLLOID SYSTEMS (CS)

Deionizer water was used to prepare $\text{Fe}_3\text{O}_4\text{--NaCl--H}_2\text{OCS}$. Not less than three measurements were taken in each sample to control the repeatability of the results. Higher degree of particle dispersion in the studied samples was achieved by heating the samples up to 80°C temperature in a quartz cuvette. In DLS-method (Dynamic Light Scattering) the main parameters characterizing the particle size distribution are the average hydrodynamic diameter of particles (Z-average) and polydispersity index (PdI) — a dimensionless value characterizing the distribution width (this parameter is highly sensitive to the presence of aggregates in the sample). For monodispersed samples PdI parameter is not higher than 0.1.

PARTICLE SIZE DISTRIBUTION IN THE COLLOID SYSTEMS

was studied by the method of laser correlation spectroscopy (LCS) in laser correlation spectrometer “Zeta

Sizer-3” (Malvern Instruments, Great Britain) with “Multi 8 computing correlator type 7032 ce” correlator. The instrument is fitted with helium-neon (He-Ne) laser with 633 nm wave length and 25 mW power. Instrument measurement range is from 1 nm to $20\text{ }\mu\text{m}$. The function of size distribution of the nanoparticles was computed from experimentally derived autocorrelation function using PCS Size Mode v.1.61 program. Particles being in a suspended state in the liquid, at their irradiation by the laser beam scatter the light, which is focused at a certain angle and recorded by the photodetector. Scattered light registration angle was 90° . Particle size distribution was converted from intensity units into quantitative ones using Mie theory (Mie 1908).

KINETICS OF OXIDATION OF MICROLAYERED NaCl-Fe COMPOSITES IN AIR

was studied in thermogravimetric analyzer (TGA) TGA-7 of Perkin Elmer Company (USA) at a stable fixed heating/cooling rate of $10^\circ\text{C min}^{-1}$ in the range of $20\text{--}650^\circ\text{C}$. A sample of NaCl-Fe composite of up to 100 mg weight was placed on a platinum plate, which was hung on sensitive balance (up to $0.1\text{ }\mu\text{g}$) and was heated or cooled, fixing all the weight changes in dynamics.

EXPERIMENTAL TECHNIQUE

Technological procedure of producing NaCl-Fe composites on a rotating substrate is shown in Figure 1. Evaporation materials 5, 6 are placed into copper water-cooled crucibles 4, 7 and they are transformed into vapour by electron beam guns 1, 2, and the mixed vapour flow is condensed on the rotating substrate 3 positioned above the crucibles.

Initial material evaporation was performed from crucibles 4, 7 with inner diameter of 50 mm by electron beam guns 1, 2 of 60 kW power. The gun accelerating voltage was 20 kV . Application of washer 8 with a system of inclined vapour channels at NaCl evaporation ensured uniform distribution of sodium chloride layer thickness in the produced composites along the rotating substrate radius.

Vacuum in the process chamber during evaporation was $(1.2\text{--}2.4)\cdot 10^{-2}\text{ Pa}$. The total vapour flow was condensed on a rotating water-cooled copper substrate 3 of 400 mm diameter with pre-applied anti-adhesion layer of NaCl. Substrate temperature was equal to approximately $70 \pm 10^\circ\text{C}$. The distance from the crucibles to the substrate was equal to 230 mm , and the distance between the crucible axes was 115 mm . One composite layer (composite) — H which consisted of two NaCl- and Fe-enriched layers, h_{NaCl} and h_{Fe} , respectively, was produced in one cycle of substrate ro-

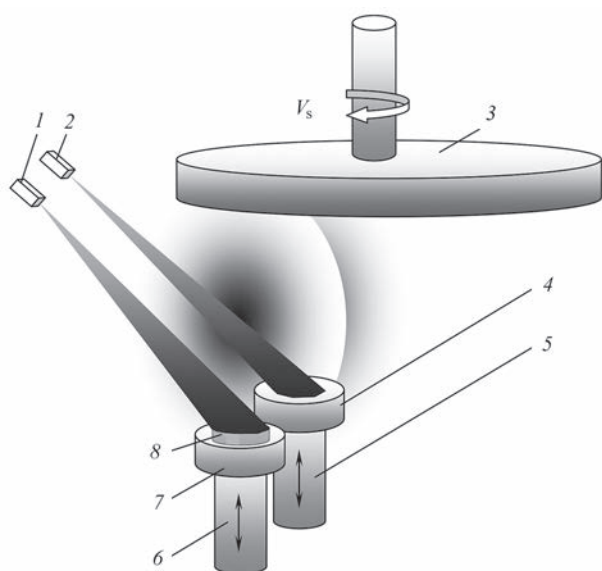


Figure 1. Diagram of producing NaCl-Fe composites on a rotating substrate: 1, 2 — electron beam guns; 3 — rotating substrate; 4, 7 — water-cooled crucibles; 5 — Fe ingot; 6 — NaCl cylinder; 8 — graphite washer

Таблица 1. Characteristics of typical microlayered NaCl–Fe composites produced at different speeds of substrate rotation (V_s)

V_s , rpm·min ⁻¹	t_{ex} , min	Number of layers	H , μm	h_{NaCl} , μm	h_{Fe} , μm	$h_{\text{Fe}}/h_{\text{NaCl}}$
2	11	22	3.89	3.25	0.64	0.2
20	12	240	0.68	0.56	0.12	0.2
25	20	500	0.55	0.46	0.09	0.2

Note. H — total thickness of an isolated NaCl composite; h_{NaCl} — thickness of NaCl-enriched layer in an isolated NaCl–Fe composite. h_{Fe} — thickness of the layer enriched in Fe in an isolated NaCl–Fe composite

tation on the path over the total vapour flow of ion-sodium chloride. The thickness of the composite layers and iron concentration in the composite was regulated by substrate rotation speed and intensity of initial material evaporation. The deposition rate of the total vapour flow (condensation rate V_c) was 6–9 $\mu\text{m}\cdot\text{min}^{-1}$. The produced condensate thickness was 80–240 μm .

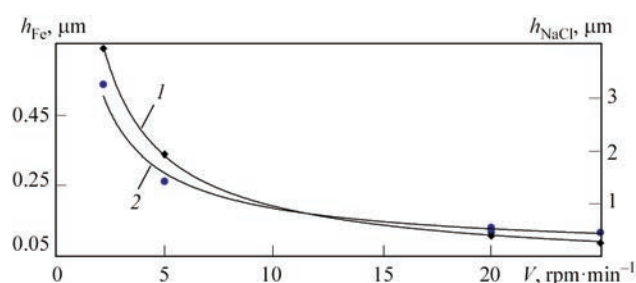
EXPERIMENTAL RESULTS

A feature of the produced NaCl–Fe composites at evaporation from two independent crucibles and deposition of mixed vapour flows on the rotating substrate is their layering. It is logical to assume that the main causes for appearance of layering are variations of the condensation flows and features of crystal growth. Generalized comparative characteristics of the studied NaCl composites produced on a rotating substrate, are given in Table 1.

As one can see from the presented data, a reduction of the total thickness of an individual NaCl layer by 14 % is noted with increase of the substrate rotation speed from 2 to 25 rpm·min⁻¹, here the ratio of layers enriched in iron and sodium chloride $h_{\text{Fe}}/h_{\text{NaCl}}$ remains constant and equal to approximately 0.2.

Influence of the rotating substrate speed on thickness of the layers, enriched in Fe and NaCl in NaCl–Fe composite, is shown in Figure 2.

Thicknesses of the layers enriched in NaCl and Fe in NaCl–Fe composite depend on the rotating substrate speed (Figure 2). At the speeds of 25 rpm·min⁻¹ the thicknesses of the layers enriched in NaCl and Fe, are equal to 0.46 and 0.09 μm , respectively and are by 14 % lower, compared to speed $V = 2.0$ rpm·min⁻¹.


Figure 2. Dependence of the thickness of h_{Fe} — (1) and h_{NaCl} (2) layers of NaCl–Fe composite on the substrate rotation speed

STRUCTURE OF CHIPS OF INITIAL NaCl–Fe COMPOSITE

Figure 3 shows the microstructure of the produced NaCl–Fe composites at condensation temperature of approximately 70 °C with different speed of substrate rotation of 2 and 20 rpm·min⁻¹. The composites consist of layers located parallel to the rotating substrate plane (Figure 3, a). The layers are made up of crystallites directed along the condensation flow, and have a weakly pronounced columnar structure across the condensate thickness. Columnar crystallites are oriented in the direction, normal to the rotating substrate surface and have NaCl and Fe-enriched alternating layers of different thickness (Figure 3, b). Such a growth pattern corresponds to the earlier established regularity, according to which columnar crystallites form in the range of the ratio of substrate temperature to metal melting temperature of 0.3–0.5 [10]. The maximum size of the crystallites calculated by Sherrer equation in NaCl-enriched layers, is close to 45 nm. Maximum size of crystallites in Fe-enriched layers, is

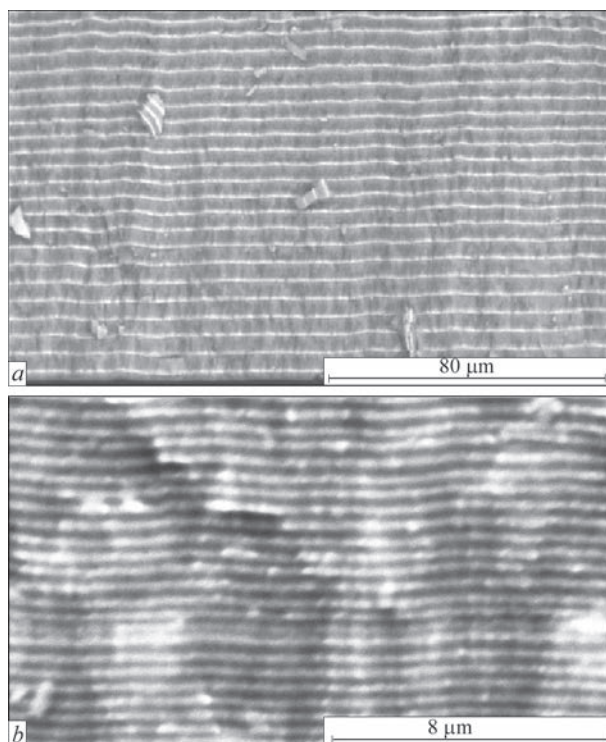
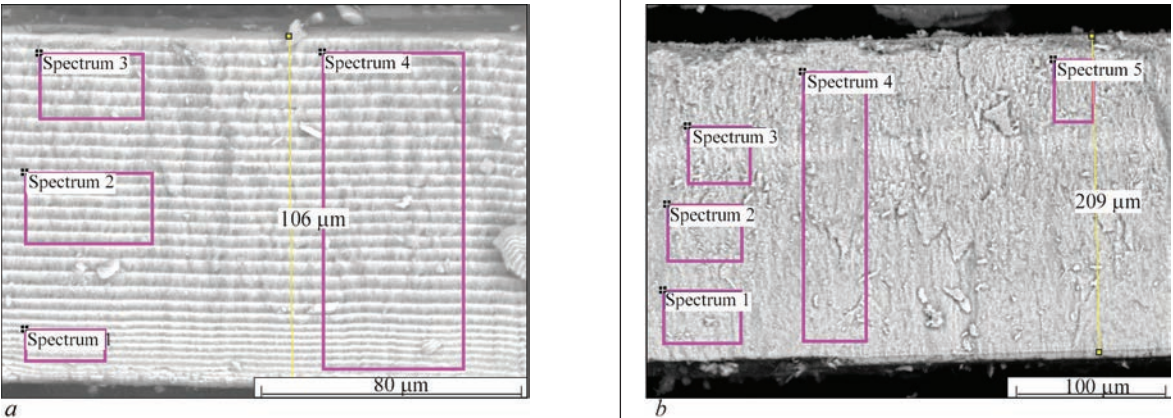

Figure 3. Typical microstructure of the transverse chip of NaCl–Fe composite samples, produced on a rotating substrate: a — 2; b — 20 rpm·min⁻¹

Table 2. Elemental composition of transverse chip of samples of NaCl–Fe composites, produced at different speed of substrate rotation, wt. %



Spectrum	Fe	O	Na	Cl
Substrate rotation speed $V_s \sim 2.0 \text{ rpm}\cdot\text{min}^{-1}$ (a)				
1	27.85	13.96	28.05	30.14
2	16.46	18.00	30.39	35.15
3	14.22	17.36	30.68	37.73
4	18.38	15.43	29.49	36.69
Substrate rotation speed, $V_s \sim 25.0 \text{ rpm}\cdot\text{min}^{-1}$ (b)				
1	19.75	5.64	29.40	45.21
2	18.06	6.28	28.79	46.87
3	19.93	7.22	28.74	44.12
4	23.43	4.80	25.18	46.60
5	16.75	8.78	27.48	46.99

within 8–10 nm. Certain differences in the crystalline structure of alternating layers (sodium chloride and iron) are due to the condensation features and are in accordance with the main regularities of direct electron beam evaporation of inorganic substances with subsequent condensation of the vapour flow at substrate temperature $T_s < 0.3T_m$ (T_m is the melting temperature), where condensates have an amorphous or nanosized structure [11].

So, at $V_s = 20 \text{ rpm}\cdot\text{min}^{-1}$ (Figure 3, b) the composite layering is preserved, but the intensity of reduction of the thickness of iron-enriched metal layers is more pronounced, compared with thicknesses of NaCl-enriched layers. More over, in metal layers h_{Fe} in the area of crossing the joint crystallite boundaries additional opening of the channels along intercrystalline

boundaries is observed across the composite thickness. Presence of porosity in h_{Fe} layers stimulates this mechanism. At increase of substrate rotation speed, as a result of thinning of iron-enriched layers, the intensity of channel formation across the layer thickness is increased, leading to their fracture.

Investigations of element content on the transverse chip of NaCl–Fe composite showed a large amount of oxygen, adsorbed by Fe nanoparticles from the air, after depressurizing the vacuum chamber (Table 2). Here, the average percentage of oxygen decreases from 16.2 to 6.5 wt.% at increase of substrate rotation speed from 2 to 25 $\text{rpm}\cdot\text{min}^{-1}$.

The value of the ratio of atomic fraction of oxygen to atomic fraction of iron depends on the quantity of iron, and decreases with increase of its content in NaCl–Fe composite (Figure 4). Lowering of the ratio of atomic percentage of oxygen to that of iron, depending on increase of the rotating substrate speed is attributable to reduction of average percent of oxygen in NaCl–Fe composites.

Indeed, the high adsorption capacity for oxygen is inherent in smaller nanoparticles, and the size of iron particles decreases with increase of iron content in the composite, leading to increase of the total area of nanoparticle surface, which results in decrease of the value of the ratio of atomic fraction of oxygen to that of iron.

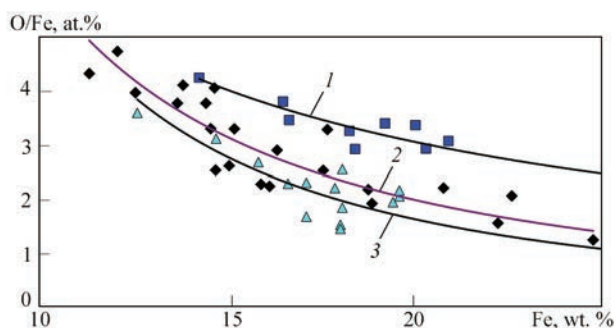


Figure 4. O/Fe ratio in NaCl–Fe composites produced on rotating substrate at V_s , $\text{rpm}\cdot\text{min}^{-1}$: 1 — 2; 2 — 20; 3 — 25 depending on Fe content

Table 3. Influence of Fe content on phase characteristics of initial NaCl–Fe composites, stored in air and placed into containers with alcohol

Fe, wt. %	Air				Alcohol			
	Fe ₃ O ₄		NaCl		Fe ₃ O ₄		NaCl	
	<i>D</i> _{av. cr.} , nm	<i>F</i> _{cr.} , wt. %	<i>D</i> _{av. cr.} , nm	<i>F</i> _{cr.} , wt. %	<i>D</i> _{av. cr.} , nm	<i>F</i> _{cr.} , wt. %	<i>D</i> _{av. cr.} , nm	<i>F</i> _{cr.} , wt. %
14	–	~ 1	30	99	15	5	95	95
15	7	3	27	97	14	12	50	88
17	8	4	40	96	13	6	58	94
19	»	»	30	»	–	–	–	–
20	»	5	45	94	13	12	60	88
23	5	»	30	95	»	10	55	90
24	6	8	»	92	14	12	50	88
25	»	6	28	94	»	7	65	93

Note. *D*_{av. cr.} — average crystallite size, *F*_{cr.} — crystalline phase content.

Metal phase content in NaCl–Fe composites was varied from 16 up to 25 wt.%. X-ray phase analysis (Table 3) showed that iron nanoparticles are present in the form of Fe₃O₄ in sodium chloride matrix. Oxidation of iron nanoparticles to Fe₃O₄ occurs at depressurizing of the vacuum chamber during composite separation from the rotating substrate, resulting in violation of the composite integrity and acceleration of the reaction of oxidation of the open surface of iron nanoparticles by oxygen in porous NaCl matrix. At interaction of iron nanoparticles with oxygen from the air an exothermal reaction with heat evolution takes place. In some cases, the temperature of condensate plates separated from the substrate exceeds 100 °C. Therefore, in order to preserve a stable state of the composite at separation from the substrate, fractures of NaCl–Fe composites were directly placed into a container with alcohol.

Phase composition and average crystallite size of (Sherrer equations) of initial NaCl–Fe composites, stored in air and in alcohol, are given in Table 3.

In samples of NaCl–14 wt.%Fe composite, stored in air, XRD records reflexes only from NaCl. It is obvious that the dimension of iron oxide phase particles (~ 1 wt.% Fe content in the composite) is beyond the

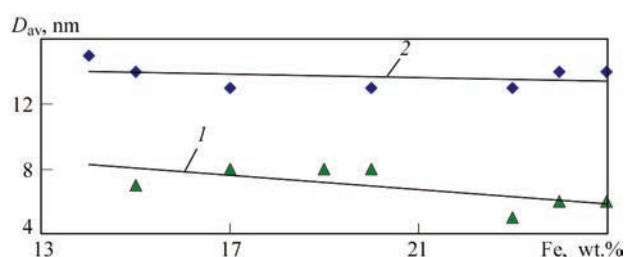


Figure 5. Dependence of average size of Fe₃O₄ crystallites on Fe content in NaCl–Fe composites, produced in air (1) and in alcohol (2) limits of sensitivity of this method. Increase of the amount of iron leads to appearance of Fe₃O₄ oxide with average crystallite size (*D*_{av}) equal to 5–9 nm. Here, with increase of the content of crystalline phase *F*_{cr} the average size of crystallites (*D*_{av}) becomes smaller (Figure 5). Reduction of particle size leads to an increase in the role of surface energy and points to a high adsorption ability of small nanoparticles [11].

At formation of Fe₃O₄ nanoparticles in alcohol, their size practically does not change (*D*_{av} = 13 ± 1 nm) depending on Fe content from 14 to 25 wt.% (Figure 5). Increase of average size of Fe₃O₄ nanoparticles in alcohol is probably related to better protecting properties of alcohol at the stage of oxygen sorption, but this requires further studies.

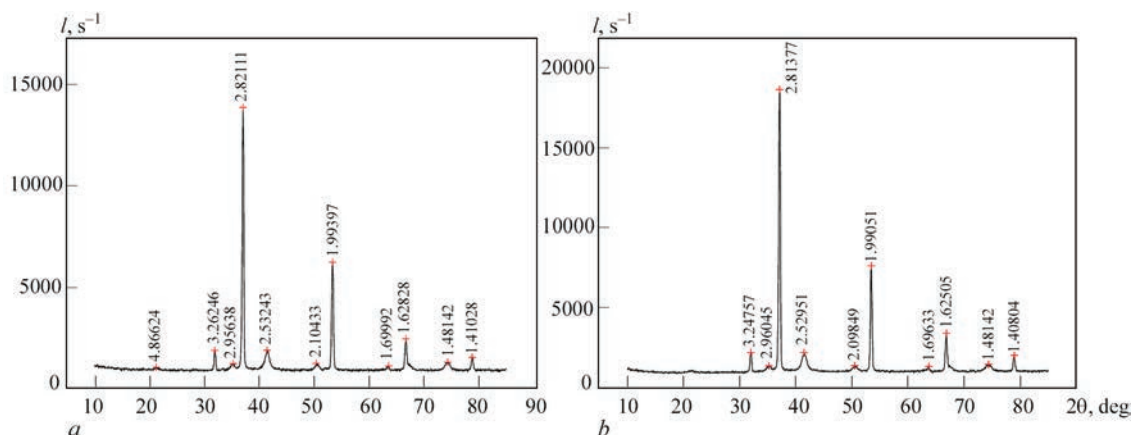


Figure 6. X-Ray diffraction patterns of NaCl–Fe composite powders: *a* — 25; *b* — 17 wt.% Fe

Table 4. Phase composition of NaCl–Fe composites

Sample	Fe, wt.%	Phase composition	Average crystallite size, nm	Crystalline phase content, wt.%
1	17	NaCl JCPDS # 75-306	68	94
		Fe ₃ O ₄ JCPDS # 88-315	13	6
2	25	NaCl JCPDS # 75-306	65	93
		Fe ₃ O ₄ JCPDS # 88-315	14	7

Samples of Fe₃O₄–NaCl–H₂O colloid systems (CS) with iron content of 17 to 25 wt.% were studied in the work. In keeping with X-ray data, the samples were single-phase Fe₃O₄. Material dispersion was determined by X-ray diffraction methods (XRD) and dynamic light scattering (DLS). Figure 6 and Table 4 show the results of analysis, performed by X-ray diffraction method.

In keeping with the calculations, the size of Fe₃O₄ oxide crystallites in the studied powders was in the range of 13–14 nm (Table 4).

Data presented in Figure 7 and in Table 5 show that heating of the studied samples up to the temperature of 80 °C leads to stabilization of powder dispersion. For each of the samples, a monomodal distribution with a maximum in the range of 106.3–115.6 nm (for different compositions) and 60.5 nm for a sample washed from salt (not given in the paper) is in place. A large value of distribution width (PdI ≥ 0.252) is indicative of presence of agglomerates of particles of different size in the dispersion. Maximum part of the

particles is represented by 90 and 170 nm fractions, and the size of the smallest particles is close to 40 nm.

In the studied CS the value, corresponding to the distribution maximum (Table 5) can be regarded as characteristic average value of particle size. These values are higher than the average size of the crystallites determined by X-ray diffraction method. It means that the studied samples contained aggregates, which do not break down at heating to 80 °C.

Data given in Table 6 show that for dispersions of CS samples at temperatures of 25 and 80 °C the parameters of average particle size (Z-average) and distribution width (PdI) had high values and scatter that characterizes the samples as polydispersed with presence of aggregates.

NaCl–20 wt.% Fe composite, produced at substrate rotation with maximum speed of 25 rpm·min⁻¹, was selected to study the kinetic characteristics, as the condensate with maximum possible uniform distribution of iron across its thickness and optimum iron content (up to 20 wt.%) [8]. X-ray phase analysis of

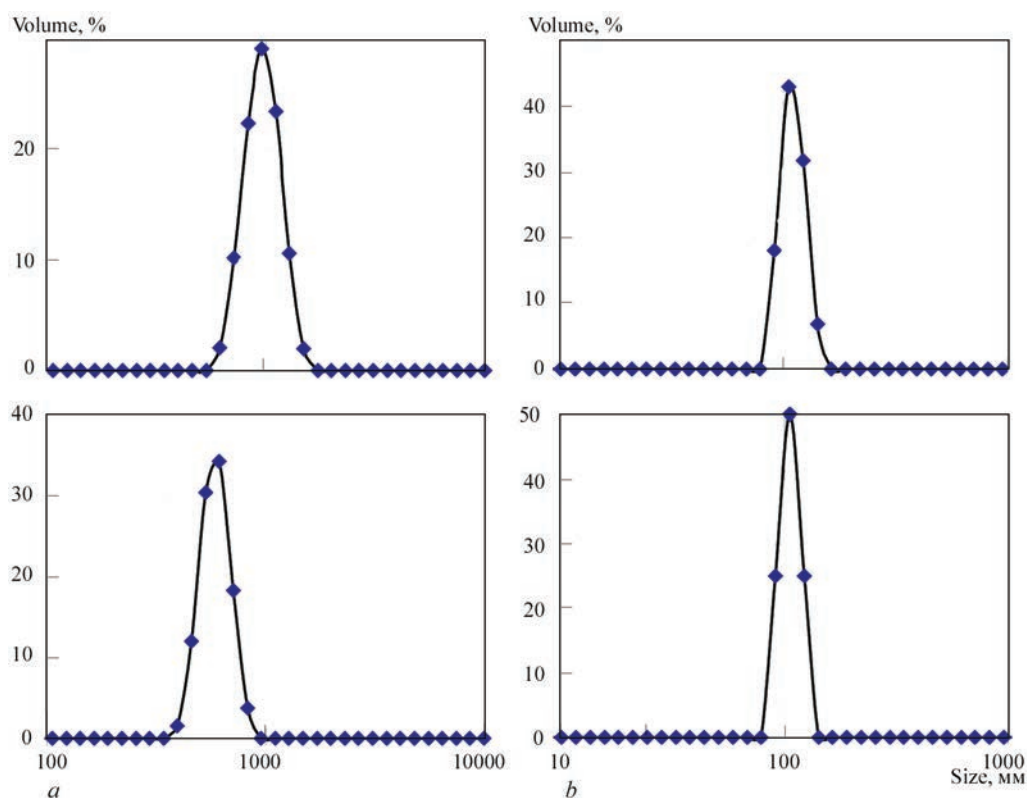


Figure 7. Particle size distribution (abscissa is given by the logarithmic scale) of NaCl–Fe composite powders at measurement temperature, °C: a — 25; b — 80, samples 1 and 2

Table 5. Average size of CSA* measured by XRD method, and of particles measured by DLS method, for NaCl–Fe powders

Sample	Fe, wt.%	Method of measuring particles size		
		DLS (maximum of particle size distribution, nm)		XRD (CSA size, nm)
		$T, ^\circ\text{C}$		
		25	80	
1	17	796.5	110.9	13
2	25	593.2	106.3	14

*Coherent scatter area.

Table 6. Estimated values of the quality of dispersion of NaCl–Fe powders

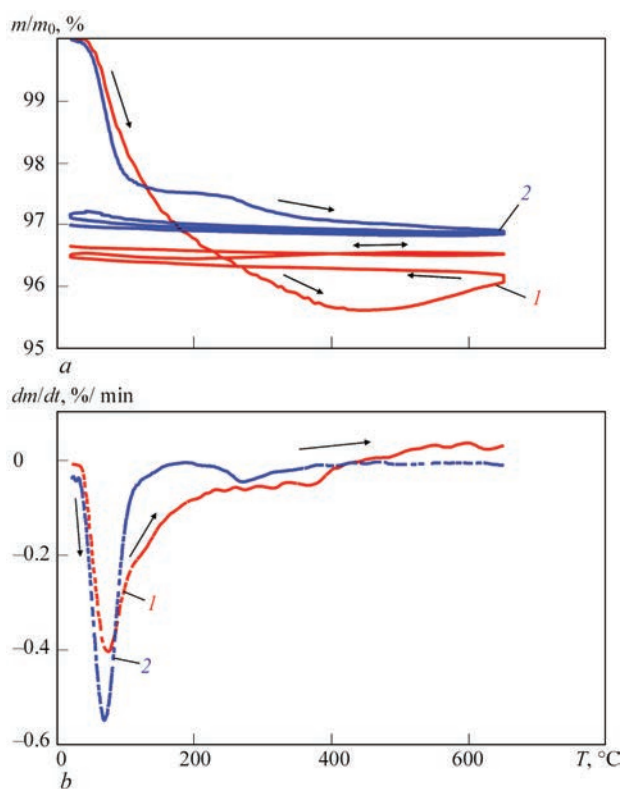
Sample	Fe, wt.%	$T, ^\circ\text{C}$			
		25		80	
		Z-average	PdI	Z-average	PdI
1	17	894.1	0.889	2893	1.000
2	25	1182	0.663	2578	»

Table 7. X-Ray phase analysis of NaCl–20 wt.% composites after storing in air and in alcohol for 30 days

Phase	Fe_3O_4		NaCl	
	nm	%	nm	%
Air	10	3	30	97
Alcohol	13	12	60	88

the initial NaCl–20 wt.% Fe composites after storage in air and in alcohol for 3-days is indicative of the presence of Fe_3O_4 phase with 8 and 13 nm nanoparticles in them (Table 7).

Figure 8, *a* shows kinetic curves of the initial sample, stored in air for a day (curve 1) and in alcohol (curve 2). Sample, stored in air (Figure 8, *a*, curve 1) is characterized by 4.5 % reduction in mass at heating to 430 °C [12], that is attributable to desorption of adsorbed moisture (differential curve 1, Figure 8, *b*). Further increase of temperature to 650 °C is accompanied by increase of sample mass due to additional oxidation of Fe_3O_4 phase to Fe_2O_3 [13]. The sample stored in alcohol (Figure 8, *a*, curve 2) is characterized by 3 % reduction of mass at heating to 650 °C, that is also attributable to desorption of adsorbed moisture (differential curve 2, Figure 8, *b*). However, a smaller percentage of sample mass decrease can be accounted for by better protective properties of alcohol at the stage of moisture sorption. It is possible that storing in alcohol prevented the start of additional oxidation of Fe_3O_4 to Fe_2O_3 (12 % of Fe_3O_4 phase instead of 3 %, Table 7), and formation of a more stable Fe_3O_4 , although it still requires additional studies.


Figure 8. Thermogravimetric analysis (*a*) and differential thermogravimetric (*b*) analysis in air of NaCl–20wt.% Fe composites after storing for 24 h in air (1) and in alcohol (2)

Assessment of magnetic characteristics showed that the sample stored in alcohol (with greater mass of Fe_3O_4), has better magnetic properties.

CONCLUSIONS

1. NaCl–Fe composites, produced by the procedure with arrangement of evaporation material sources peripheral relative to the substrate rotation axis, have a layered structure.

2. The total thickness of the layers depends on substrate rotation speed and is equal to 3.89 to 0.55 μm for $V_s = 2$ and 25 $\text{rpm}\cdot\text{min}^{-1}$, respectively.

3. The ratio of the thickness of layers enriched in iron and sodium chloride, remains constant and is equal to approximately 0.2, irrespective of the substrate rotation speed; thickness of Fe- and NaCl-enriched layers in an isolated composite for $V_s = 25$ $\text{rpm}\cdot\text{min}^{-1}$ is equal to $h_{\text{Fe}} = 0.09$ and $h_{\text{NaCl}} = 0.46$ μm , respectively.

4. It was found that oxidation of iron nanoparticles to Fe_3O_4 oxide occurs at depressurizing of the vacuum chamber during the condensate separation from the substrate.

5. Average size of Fe_3O_4 crystallites, depending on Fe content in NaCl–Fe condensate, is determined by the method of its storage: in air or in alcohol. In 14 to 25 wt.% range of iron concentration the average size

of Fe_3O_4 particles, stored in air is 5–10, and in alcohol — 13 ± 1 nm.

6. It was found that nanoparticles, produced in NaCl–17–24 % Fe composites, have a monomodal distribution with a maximum in the range of 106.3–115.6 nm. Minimum size of the nanoparticles is equal to 40 nm.

7. Investigations on the transverse chip of the composite confirmed the presence of oxygen, adsorbed by Fe nanoparticles from the air. Depending on substrate rotation speed, the average percentage of oxygen decreases from 16.2 to 6.5 wt.% at V_s increase from 2 to 25 rpm·min⁻¹, respectively.

8. Value of the ratio of atomic fraction of oxygen to atomic fraction of iron depends on the amount of iron, decreases with increase of its content in NaCl–Fe composite and increase of substrate rotation speed.

9. Storing NaCl–Fe composites in alcohol protects them from saturation by moisture and iron oxidation, and, consequently, leads to more stable physical properties in time.

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ORCID

Yu.A. Kurapov: 0000-0003-3460-1712,

V.O. Osokin: 0000-0002-0632-7739,

S.E. Lytvyn: 0000-0001-6113-5038

CONFLICT OF INTEREST

The Authors declare no conflict of interest

CORRESPONDING AUTHOR

V.O. Osokin

E.O. Paton Electric Welding Institute of the NASU

11 Kazymyr Malevych Str., 03150, Kyiv, Ukraine

E-mail: 4osokin@gmail.com

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