Nanoparticle Formation in a Mixture of Fe, C, O$_2$ in Low-temperature Plasma in a Magnetic Field

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Abstract - **The paper presents the results of researching a magnetic field influence on the formation of dispersed particles from the mixture of Fe+C+N$_2$+Ar+O$_2$ at the temperature of more than 4000K. To optimize the composition of the plasma-forming gas, thermodynamic modeling was performed. The research establishes that an external magnetic field has a significant effect on the formation of a dispersed phase in the mixture of carbon and iron vapor. For example, in a powder obtained without a magnetic field, X-ray diffraction shows up to 95% C. In a powder obtained in the magnetic field of 15 mT, C (up to 50%), Fe$_3$O$_4$ (up to 45%), Fe$_2$O$_3$ (up to 15%), and FeO (less than 5%) are recorded. The observed results are explained by the coagulation of nanoparticles in the magnetic field.**

Keywords — vapor, nucleation, particle, magnetic field, X-ray diffraction, carbon, iron

I. INTRODUCTION

Isotopes are widely used in various spheres of human activity: in medicine (diagnosis and treatment of diseases), in the study of biological processes, nuclear energy, and for doping semiconductors. Under the conditions of certain stable isotope ratios in a solid body, the best physical and chemical parameters are achieved. However, the high cost of isotope materials prevents the expansion of their applications. Therefore, research into new isotope effects is topical. With developing efficient methods for separating isotopes, the applications will steadily increase.

Low-temperature plasma contains a large number of chemically active radicals. In a non-equilibrium plasma, the input energy is used more efficiently than in a high-temperature gas. Plasma technologies are thus widely used in etching or cleaning surfaces, applying refractory coatings and diamond-like films, and obtaining nanostructures.

It is known that an external magnetic field affects the dynamics of the spin orientation of the unpaired (valence) electrons in radicals. Therefore, chemical reactions in the liquid phase taking place in a weak constant magnetic field are isotope selective due to the fact that the chemical reactions of isotopes with different nuclei spins occur at different rates [1, 2]. Under magnetic influence, the growth rate of inorganic salt crystals from an aqueous solution is also greater. It was established that during crystallization of NaCl from an aqueous solution in a magnetic field, the appearance of the first crystals begins at a lower value of solution supersaturation.

We previously showed that chemical processes between radicals in low-temperature plasmas are also isotope selective [3]. With long contact of chemical compounds formed in plasma processes and initial reagents, the isotope effect of plasma processes is significantly reduced due to isotope exchange. To reduce the dilution of isotopes, it is necessary to separate isotope-enriched compounds formed in plasma processes from the initial reagents in a short time. One of the methods for separating the initial and final compounds of a chemical reaction is to separate them into different phase states [4, 5]. For example, compounds made in the gas phase form solid particles or liquid droplets. Therefore, the aim of our research is to study the effect of a magnetic field on the transition of plasma processes and their isotope-enriched products into the condensed phase while cooling a low-temperature plasma.

II. MODELING OF THE EQUILIBRIUM COMPOSITION

It is obvious that the composition of the initial mixture affects both the physicochemical processes and the composition of the chemical compounds formed during the plasma chemical processes [6]. Computer simulation allows us to reduce the number of necessary experimental studies aimed at increasing the number of the target compound during plasma chemical processes [7]. The Terra software package is widely used to calculate compounds formed in a state of thermodynamic equilibrium at given pressures, temperature, and initial composition of the mixture [8, 9]. We conducted a simulation of the equilibrium composition of a vapor-gas mixture in the temperature range of 1000–5000K at atmospheric pressure. Ar, O$_2$ and N$_2$ were set as the plasma-forming gas mixture, containing a small amount of Fe, C, O$_2$.

At room temperature, argon does not form chemical compounds. The results of the calculation of the equilibrium composition show the following regularities. In a nitrogen atmosphere (at temperatures above 2000 K) NO is formed. Carbon dioxide (CO$_2$) is formed only with an excess of oxygen relative to the stoichiometric value with respect to Fe and C. Carbon dioxide decomposes into CO and O in the temperature range of 2400-3400K. Carbon monoxide (CO) is

formed over the entire temperature range with an oxygen deficiency. At low temperatures, the dispersed phase is exhibited for the compounds Fe_3C, FeO, Fe_3O_4, Fe_2O_3.

Figure 1a shows the calculated values of the equilibrium composition in the temperature range of 1000–5000K for a mixture containing Fe – 1, C – 1, O_2 –2, Ar – 100, N_2 – 50 mol/kg of the mixture. The graphs of the concentration of nitrogen and argon contained in much larger quantities than other components are not shown in Fig. 1a.
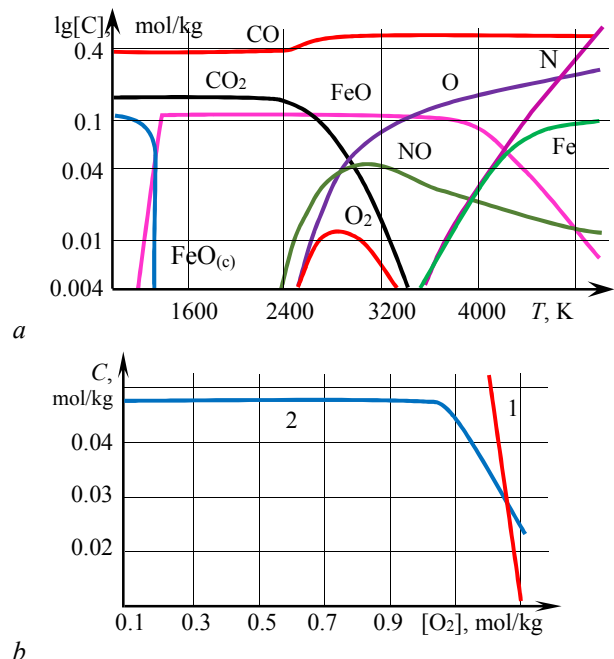

a

b

Fig. 1. *a* - Equilibrium composition of plasma containing Fe, C, N_2, O_2, Ar at different temperatures.

b – Dependence of highest concentration of (mol/kg) carbon (1) iron carbide (2) in plasma-forming gas Ar+O_2 and Ar+O_2+N_2 in the range of 1000–3100 K on oxygen concentration (mol/kg)

The calculated graphs showing the content of various compounds in equilibrium conditions may have maxima. The position of such maxima on the temperature scale depends on the composition of the plasma-forming mixture. The concentration graphs of dispersed carbon, Fe_3C and iron oxides versus oxygen concentration in plasma-forming mixtures (Ar-100, Fe-1, C-1) and (Ar-70, Fe-1, C-1, N_2-30) are shown in Fig. 1b. Under thermodynamic equilibrium conditions, the content of the condensed phase decreases with increasing oxygen concentration. Dispersed carbon is formed only when the oxygen content of the plasma-forming mixtures is less than 1.3 mol/kg, and Fe_3C is formed when the content of O_2 is less than 1.5 mol/kg. The content of Fe_3C with a low oxygen content is 0.058 mol/kg, and dispersed carbon is 1 mol/kg. The presence of nitrogen in the plasma-forming mixture has almost no effect on the content of the dispersed phase formed during cooling below the boiling point of the compounds under consideration. Iron oxide can simultaneously be present in the form of vapor (FeO) and dispersed phase (FeO_c).

The graphs in Fig. 2 show the maximum content dependence of various chemical compounds on the oxygen concentration in the plasma-forming mixture. The graphs in Fig. 2a are valid in the temperature range of 1000–2000 K (a condensed phase forms), while 2b is in the range of 3500–

5000K (vapor phase). An increase in the amount of CO and CO_2 in the gas phase is observed with an increase in the concentration of oxygen in the mixture. The amount of dispersed carbon formed decreases with increasing oxygen content in the plasma-forming mixture. At temperatures above 3780K, excess carbon is present in the gas phase as vapor. The concentration of CN is significantly reduced with increasing oxygen content. Nitrogen has almost no effect on the amount of dispersed carbon formed in the temperature range 1000-5000K with the minimum oxygen content in the plasma-forming mixture.
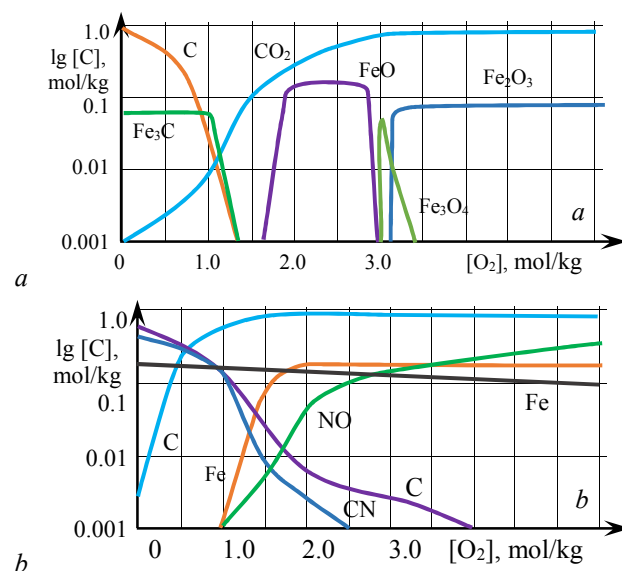

a

b

Fig. 2. Dependence of greatest concentration of various compounds in the products of plasma processes on oxygen content in the initial mixture at temperatures: a) 1000–1600 K, b) 3500–5000 K

Iron oxide (FeO) is in the form of steam in concentrations of 0.04-0.17 mol/kg in plasma-forming mixtures (Ar - 100, Fe - 1, C - 1 mol/kg) and (Ar - 100, Fe - 1, C - 1, N_2 - 30) provided that the temperature is above 1600K, and the oxygen concentration is more than 1.4 mol/kg. Iron oxides can be in the form of a condensed phase regardless of the presence of nitrogen. This is possible with the oxygen content for: FeO - 1.6-3.0 mol/kg; Fe_3O_4 - 3.0-3.3 mol / kg; Fe_2O_3 - more than 3.1 mol/kg. The highest concentration of oxides in the condensed phase is: FeO — 0.172 mol/kg; Fe_3O_4 - 0.052 mol/kg; Fe_2O_3 - 0.085 mol/kg.

III. EXPERIMENTAL METHOD

Studies on plasma gasification of graphite by capacitive RF discharge show that with slow cooling of plasma, all oxygen is consumed for the oxidation of carbon to form CO_2 or CO. Therefore, under these conditions, the formation of a dispersed phase is impossible with a stoichiometric content of oxygen and carbon atoms [3, 4].

To study the rapid formation of a condensed phase in an external magnetic field, a plasma-chemical installation was used (Fig. 3). To reach the temperatures at which graphite evaporates efficiently, an arc discharge plasma can be used. The evaporation temperature of iron is 3134K, and of graphite is 3780K. However, iron melts at 1812K. Therefore, to preserve the shape, the iron electrode was made in the form of a thick plate and was located under the graphite electrode.

The body of the plasma-chemical reactor is made of

aluminum. An electric arc is formed with the aid of a constant voltage source. The electrodes were placed in the center of the lower part of the plasma-chemical reactor. The cathode of the power source was connected to a solid steel plate, oriented horizontally. The anode of the power source is electrically connected to a graphite rod installed vertically. The specified polarity of connection to the electrodes allows the evaporation rates of iron and graphite to be leveled. The massive anode was heated to the boiling point only in the region of contact with the high-temperature electric arc channel.
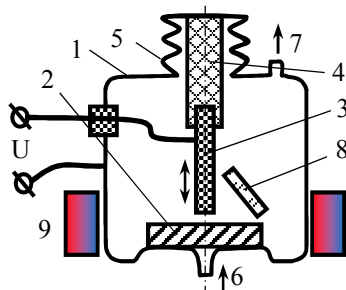

Fig. 3. Schematic diagram of the plasma chemical installation: 1 - body, 2 - steel cathode, 3 - graphite anode, 4 - insulator, 5 - bellows, 6 - plasma-forming gas mixture, 7 - spent gas, 8 - substrate, 9 - magnet

A magnetic field in the field of electric arc formation was created using two permanent magnets with a residual magnetization of 1.1 T (measuring 3×5 cm) or 0.2 T (measuring 9×12 cm). The magnets were installed perpendicular to the plasma channel by opposite poles to each other from the plasma-chemical reactor. When using magnets of 0.2 T, the arc was in a magnetic field of 15 mT, and when using magnets of 1.1 T, it was 27 mT. The magnetic field at the site of the arc being formed was measured with an ATE-8702 magnetic induction meter with the graphite anode removed.

The gas mixture Ar - 3%, O_2 - 18%, N_2 - 79% by volume was supplied at a total flow rate of 0.6 l/min through a nozzle located in the center of the plasma reactor bottom. After turning on the power supply, the electrodes were mechanically closed, which subsequently independently moved apart at a distance of 5-6 mm under the action of a spring. In this case, the electric arc current was 45 A at a voltage between the electrodes of 25 V. Under the action of the electric arc, the graphite and steel electrodes were heated and after 5–10 seconds the flow of electric current began to evaporate. The dispersed powder formed during the evaporation of the electrodes rose up and deposited on the inner walls of a 6.3-liter plasma-chemical reactor (diameter — 18 cm, height — 25 cm). Therefore, splashes of electrode materials, inevitable for an arc discharge, did not mix with the nano-dispersed powder formed during the cooling of the iron and carbon vapor. The accumulation time of the dispersed material was 5-6 minutes. The powder deposited on the walls of the plasma-chemical reactor was scraped off using a wooden spatula, which prevented the addition of aluminum particles to the powder from the walls of the reactor.

IV.   EXPERIMENTAL RESULTS

To determine the phase composition of brown powder samples formed as a result of the flow of physicochemical processes and deposited on the reactor walls, the X-ray diffractometry (XRD) method was used. A typical X-ray

diffraction pattern from the dispersed powder that forms in the reactor is shown in Figure 4. The intensities of the diffraction peaks are proportional to the content of the corresponding crystalline phase in the powder.
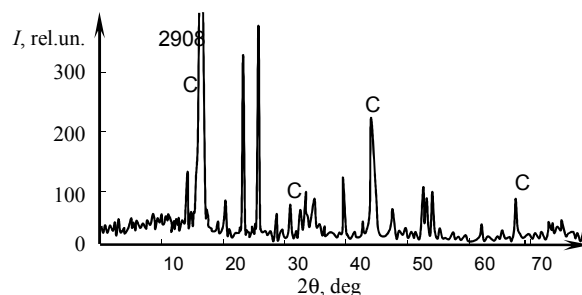

Fig. 4. X-ray diffraction pattern of a powder formed without a magnetic field

By weighing the plasma-chemical nanopowder taken from the walls, it was established that the external magnetic field has practically no effect on the total amount of powder obtained. X-ray diffraction analysis showed a carbon content up to 95% in the powder formed without a magnetic field in the plasma-forming mixture containing 18% oxygen. Iron oxides were recorded in a small amount. In the powder formed in an external magnetic field of 15 mT in the same plasma-forming mixture, iron oxides were observed in a significant amount. The data of X-ray diffraction show that the content of microcrystals C, FeO, Fe_2O_3, Fe_3O_4 in a weak magnetic field depends on the magnitude of the magnetic field. When using magnets with a residual magnetization of up to 0.2 T to create a magnetic field of 15 mT in the plasma channel region, the content of microcrystals decreases as follows: C (to 50%), Fe_3O_4 (to 45%), Fe_2O_3 (to 15%), FeO (less than 5%). A similar content of crystalline phases, according to X-ray phase analysis (XRD), was observed when using a magnet with a residual magnetization of 1.1 T. Under all conditions, iron carbide was not found in the dispersed phase. On the surface of the graphite electrode, after a long action of the arc discharge, a coating of iron oxide and a shiny film are visually observed. It is obvious that the experimentally detected composition of the dispersed phase does not coincide with the calculated data. Perhaps this is due to the fact that the calculations were carried out for equilibrium conditions that cannot be achieved under the conditions of the experiment.

The powder deposited on the glass plates installed in the plasma-chemical reactor at a distance of 15 mm from the discharge channel was also analyzed. Figure 5 shows the SEM images of the powder formed in the plasma channel of the arc discharge, obtained using a JEOL JSM-7500FA scanning electron microscope. Micrographs of the powder formed without an external magnetic field show conglomerates of nanodispersed particles, as well as a small number of spherical particles with sizes less than 5 microns. These conglomerates have sizes less than 15 microns and an arbitrary shape. Spherical particles with sizes less than 5 microns are likely to appear due to the splashing of the steel electrode. There are practically no spherical particles in the magnetic field, the dispersed particles whose sizes do not exceed 0.3 microns have sharp outlines.

V.   DISCUSSION

The significant difference in the ratio of graphite and iron oxides in the resulting powder, according to X-ray phase

analysis, cannot be explained by the influence of the magnetic field on the relative evaporation rate of iron and graphite. A slight oxidation of carbon, even with a high oxygen content, may be due to the isolation of dispersed carbon from oxygen by a layer of iron oxides. Let us consider the processes affecting the dynamics of the formation of dispersed particles when the plasma is cooled, which is a supersaturated vapor-gas mixture. Iron oxide is formed in a chemical reaction:

$$Fe + O_2 \rightarrow FeO + O,$$
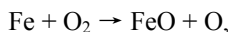
where the chemical reaction constant

$$k(T) = 1.6 \times 10^{-9} (T/298)^{-0.02} e^{-93322/RT} \quad cm^3/(molecule\ s)$$
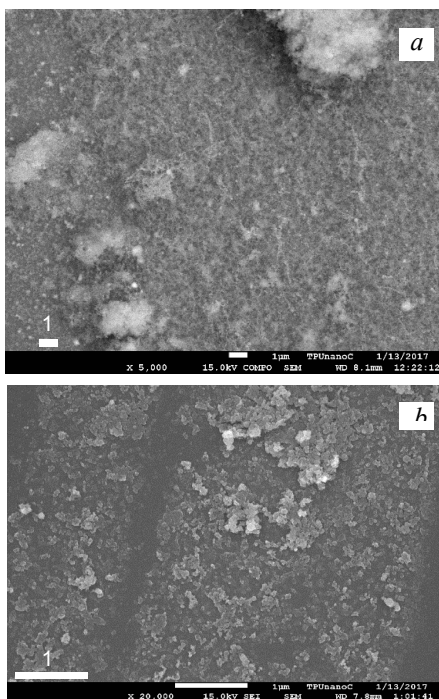$(T = 200\text{-}2500K)$ [11].

Fig. 5. SEM images of powder from arc plasma in a magnetic field: a - 0, b - 15 mT

With an excess of oxygen, the reaction of oxidation of FeO is possible:

$$FeO + O_2 \rightarrow FeO_2 + O,$$

where the chemical reaction constant

$$k(T) = 1.02 \times 10^{-11} (T/298)^{0.40} e^{-68180/RT} \quad (cm^3/molecule\ s)$$
$(T = 1000\text{-}3000K)$ [12].

The formation of nuclei and the formation of dispersed particles in a multicomponent system begins with one component, under given conditions, having had less pressure of saturated steam. The evaporation temperature of graphite is 3780K, and that of iron is 3134K. The vapor pressure of iron at a temperature of 1900K is 13.3 Pa, and that of carbon at 2030K is 1.33 Pa. In the gas phase, iron is oxidized. Iron oxide (Fe_2O_3) (hematite) melts at 1912K, and decomposes with increasing temperature. Iron oxide (Fe_3O_4) also decomposes at temperatures above 1800K. Therefore, at temperatures above 1912 K, only FeO can be formed.

The growth rate of the primary carbon particles is limited by their heating during the phase transition to the sublimation temperature (the specific heat of vaporization of graphite is 50,000 kJ / kg). In the future, slower coagulation of several cooled carbon particles will be possible. The release of energy during the formation of an agglomerate contributes to the formation of a particle with a single crystalline structure. The rate constants of coagulation of dispersed carbon at various processes in the gas stream, in the temperature range of 470-530°C, are given in the work by V.M. Shopin [13].

The nucleation theory for the formation of liquid droplets is well developed. It is known that the energy of formation of a neutral cluster, in which the surface can be clearly distinguished, is [14, 15]:

$$\Delta G_n = n(\mu_c - \mu_v) + 4\pi\,r^2(n)\Sigma,$$

where n is the number of atoms in the cluster, μ_c, μ_v are the chemical potentials of atoms in the condensed and the gas phases, respectively, $r(n)$ is the radius of the cluster consisting of n atoms, and Σ is the surface tension of the cluster.

From equation (1) it follows that the spontaneous growth of small clusters is impossible due to the increase in their Gibbs free energy. On the contrary, the growth of liquid droplets with a diameter above a certain critical value is accompanied by a decrease in energy, which contributes to their growth. Although the most stable form for liquids is a sphere, for solid particles it is a microcrystal, but the thermo-dynamic bases for the formation of liquid droplets and solid particles are the same.

Vapors of electrode material diffuse to the periphery of the discharge channel. In this case, in the peripheral region of the discharge, the pair is rapidly cooled due to the small size of the diameter of the arc discharge channel. Therefore, steam containing FeO and carbon becomes supersaturated. The conditions for the condensation of carbon are formed at temperatures below 3780K, and the conditions for FeO are formed at 3687K. It should be noted that during rapid cooling, the temperature of the beginning of condensed particle formation (nucleation) is lower than the phase transition temperature. Therefore, it is obvious that with such a small difference in the phase transition temperatures for FeO and C, the rapid cooling of the vapor leads to the simultaneous condensation of both components. We should thus expect a mixing of FeO and C with the growth of dispersed particles.

The frequency of fluctuation nucleation of condensation, depending on the temperature, is set at 10^{-9} s [16]. From the theory of fluctuations, it follows that the rate of formation of embryos of a new phase in a unit volume per unit time [17]:

$$\frac{dN}{d\tau} = \frac{\gamma}{\rho}\sqrt{\frac{2m\sigma}{\pi}}\left(\frac{P}{kT}\right)^2 \exp\left(-\frac{\Delta G}{kT}\right),$$

where N is the concentration of droplets in cm^{-3}; τ is time in sec, $k_B = 1.38\ 10\text{-}23$ J/K is the Boltzmann constant; γ is the coefficient of desublimation; m is the mass of the vapor molecule in kg; T is the temperature of the mixture in K; P is the total pressure of the vapor-gas mixture in mm Hg, ρ is the density of the sublimate, and σ is the force of surface tension of the fluid.

The value of γ expresses the fraction of vapor molecules remaining as a result of collisions on a solid surface.

The surface energy σ of a solid dispersed particle, such as carbon, can be determined from the equation [18]:

$$\Sigma = \frac{\Delta H - RT}{V^0}\frac{\Delta n}{n}h,$$

where ΔH is the change in enthalpy during evaporation in kJ/mol; $R = 8.314$ J/(mol.K) is the universal gas constant; n is the coordination number; Δn is the number of vacancies for atoms on the surface of the crystal lattice; h is the thickness of the monolayer of molecules in m; $V_0 = M: \rho$ is the molar volume of the solid substance in cm^3/mol, and M is the molar mass of the desublimate in g/mol.

The presence of charges on condensation nuclei facilitates the conditions for the formation of a new phase and contributes to the further growth of microcrystals. Therefore, in the low-temperature plasma, the size of critical nuclei is significantly reduced [19].

One should expect the appearance of Fe_3O_4 and Fe_2O_3 at temperatures below 1870K from condensed FeO due to its oxidation. At a temperature of 1693K, residual FeO in the liquid state crystallizes.

In the temperature range 1870K-5000K, iron has a single stable oxide, FeO, which does not have ferromagnetic properties. The oxides Fe_3O_4 and Fe_2O_3 exhibit ferromagnetism, which contributes to the coagulation of dispersed nanoparticles at temperatures below the Curie temperature.

Apparently, microcrystals of iron oxides formed without a magnetic field are smaller in size than the X-ray diffraction method can detect. For example, the size of the formed iron oxide microcrystals is smaller than the X-ray wavelength. The X-ray diffractometers used for phase analysis contain the lines $K_{Cu\alpha1}$ and $K_{Cu\alpha2}$ ($\lambda = 1.54$ A). It can be assumed that iron oxides form a barrier to oxygen from the gas phase, preventing graphite from oxidation. Therefore, carbon is slightly oxidized even at an oxygen concentration of 18%. In this case, nanoparticles containing Fe_xO_y, are formed at temperatures less than 1900K. Molecules of Fe_xO_y oxide having a magnetic moment, in a magnetic field, bond more quickly to each other. It also promotes the formation of larger nanoparticles containing Fe_xO_y. Thus in the powder formed in the magnetic field both graphite and iron oxides are recorded.

It should also be taken into account that carbon is represented by two isotopes: ^{12}C, ^{13}C (content 1.1%, spin 1/2). The natural mixture of iron isotopes includes: ^{54}Fe (5.8%), ^{56}Fe (91.7%), ^{57}Fe (2.1%), and ^{58}Fe (0.3%). The ^{57}Fe of iron isotope has a nuclear spin of 1/2. In a magnetic field, one should expect changes in both the relative oxidation rates and the nucleation of these isotopes with different g-factors, similar to the results of work [3, 20] on plasma oxidation of carbon isotopes in a magnetic field. This can increase the number of the ferromagnetic oxides Fe_xO_y having large sizes. However, for the analysis of radical processes in the gas phase, the patterns of the dynamics of pairs of radicals in liquids are unacceptable [2].

VI. Conclusion

The experiment showed that an external magnetic field has a significant effect on the formation of a dispersed phase when a low-temperature plasma is cooled in a mixture of carbon and iron vapor in a nitrogen-oxygen plasma.

1. The simultaneous nucleation of carbon and FeO, which have similar boiling points, prevents the oxidation of carbon even when the plasma-forming mixture of oxygen contains 18% by volume.

2. It is shown that in the external constant magnetic field the obtained particles of iron oxides are larger and have sharp contours than without a field.

3. With simultaneous nucleation of carbon and FeO, isotope effects are possible during carbon condensation due to different rates of carbon oxidation with magnetic and non-magnetic nuclei in the condensed phase.

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