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Modern iron nanoparticles production methods for steel modification

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INTRODUCTION & AIM

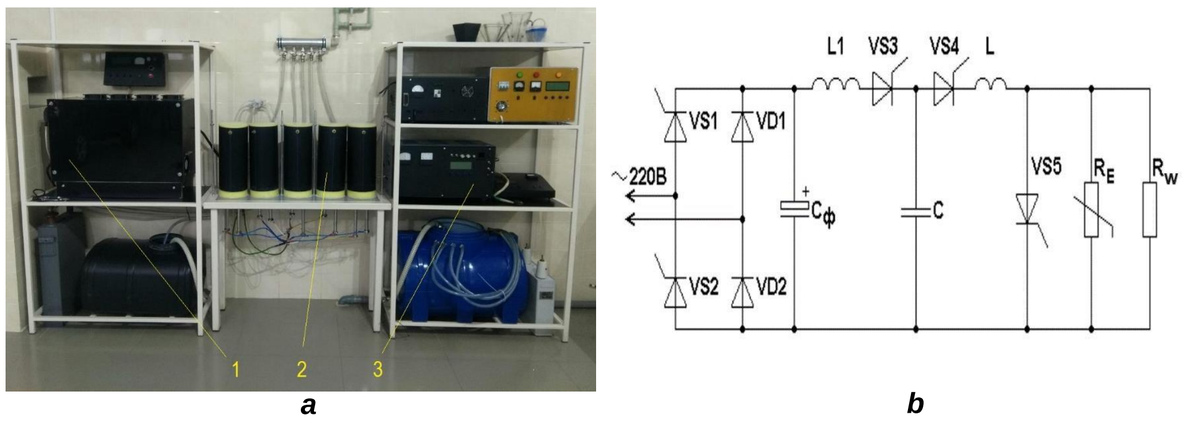
The development of physical methods of high-energy impact on the material makes obtaining a substance in a nano-dispersed state possible. The difference between nanoparticles obtained by the electrospark method, when condensation of the metal vapor phase occurs in a dispersion medium (water), the temperature of which does not exceed 30-40 degrees C, is a relatively narrow distribution of particles of the dispersed phase (20 - 100 nm). The field of application of such substances is not limited to agrobiological purposes. The authors investigated the electrospark metal granular dispersion method in an aqueous medium. The particles obtained by the above method have a size of 20-100 nm and several competitive advantages compared with similar methods of synthesis of metal nanoparticles.

The research presents the study results of the chemical composition, structure, and surface condition of nanoparticles obtained in the electric spark processing of iron. It was found that alpha, gamma, and iron oxides can be obtained during the processing of iron sparks. The efficiency of using iron nanoparticles as modifiers of the steel structure is shown. The specific properties of metals in an ultra-dispersed state open up vast opportunities for creating new effective materials and their use in engineering, medicine, and agriculture.

Physical methods of obtaining nanoparticles - grinding materials mechanically in mills or processing the material with plasma, laser, electric arc, or explosion - consist of intense thermal and force action on the original material, which is accompanied by an increase in the number of defects in the crystalline structure of the processed material to a level sufficient for destruction and crushing, melting or evaporation. Obtaining nanoparticles by electrical processing of materials is the most preferable since it combines a complex temperature-deformation effect on the material with the high technological efficiency of the method. The electro-spark processing of metal in liquid consists of the fact that an electric field sufficient for a breakdown of the liquid dielectric is created between the cathode and the anode. In this case, a flow of electrons moves from the cathode to the anode, which evaporates the liquid. When the electron flow approaches the anode, gas and plasma are in the space between the electrons and the liquid. In this case, forces directed away from the discharge axis arise for liquid and gas and towards the plasma and electron beam discharge axis. A shock wave arises and propagates in the liquid in a plane perpendicular to the discharge axis. When the electron flow reaches the anode, a conductivity channel is formed, through which the electrical system pulses the energy it has accumulated.

METHOD

The object of the study was nanoparticles obtained in the process of electric spark machining of iron granules containing (mass fraction %) 0.025C, 0.25Mn, 0.015P, 0.10S, 0.08Si, 0.10Cu, in water. By the methods, water (2) was poured into the discharge chamber (1) of the device for obtaining colloidal solutions of ultrafine metal powders (Fig. 1, a), iron granules (3) were loaded, a discharge pulse generator was turned on, and electrical pulses were applied to the electrodes (made of the same material as the granules), through which they were transmitted to the granules. As a result of the occurrence of electrical sparks (4), destruction, melting, and evaporation of the surface occurred at the points of contact of the granules, which was accompanied by saturation of water with electrical erosion products and the formation of an aqueous colloidal solution of iron in water (Fig. 1, b). The particles were isolated from the obtained aqueous colloidal solutions by drying the solution drops on crystalline quartz plates, aluminum foil, and carbon replicas. The particles' size, shape, and chemical composition were determined using Jeol – 6490LV and JEOL JSM6360 scanning electron microscopes, as well as an EEM-200 electron microscope.



a ***b***

Fig. 1. *Technology of obtaining (a): 1- electric spark generator, 2-bit cameras, 3- discharge pulse control unit; Power circuit of a thyristor generator with a discharge circuit (b)*

The grain boundaries, subgrains, and crystal structure defects were studied on a JEM-3010 JEOL transmission electron microscope with a GATAN Orius SC200D multiscanning camera.The phase composition, crystal lattice parameters of the nanoparticles, and the size of subgrains (blocks, coherent scattering regions (CSR)) were studied by X-ray diffraction in monochromatic CuKα radiation on a DRON-UM1 diffractometer.X-ray photoelectron spectroscopy (XPS) on an EC-2401 spectrometer studied the chemical composition of the nanoparticle surface. The surface analysis depth was <= 10 atomic layers. The charge state of iron and oxygen atoms was determined by the binding energies of Fe2p3/2 and O1s electrons with atoms. The regularities of phase transformations were studied on a thermal analyzer "STA 449 F1 Jupiter" using the method of synchronous thermal analysis, in which the thermal effects of phase transformations (DSC) and the change in mass (TG) of nanoparticles were simultaneously measured during heating and cooling in argon at a rate of 20 oC/min. The temperature measurement accuracy was +/- 0.25%, enthalpy was +/- 3%, and mass was +/- 2 μg. To assess the efficiency of nanoparticles obtained by electric spark machining of iron, their effect on the microstructure of 45L steel in the cast state and after annealing at 860 oC was studied.

RESULTS & DISCUSSION

The results of the studies show that in the process of electric spark machining of iron, agglomerates of nanoparticles are formed with a length of 13 to 174 nm, a width of 13 to 94 nm and a shape factor (length to width ratio) of 1 to 5.2. The average values of the nanoparticle length are 37.3 nm, the width is 30.5 nm, and the shape factor is 1.2. Analysis of the chemical composition of the nanoparticles using electron spectroscopy showed that they consist of oxides of MnO, Fe2O3, CuO, SiO2, and solid solutions of Si, Mn, and Cu in iron. In this case, oxygen is adsorbed on their surface when the nanoparticles consist only of oxides. When pure metals are present in the composition of the nanoparticles, there is no oxygen on the surface of the nanoparticles. X-ray structural analysis showed that the content of nanoparticles in the form of oxides in the total mass does not exceed the error of the method (5%) and is not recorded by X-ray structural analysis.

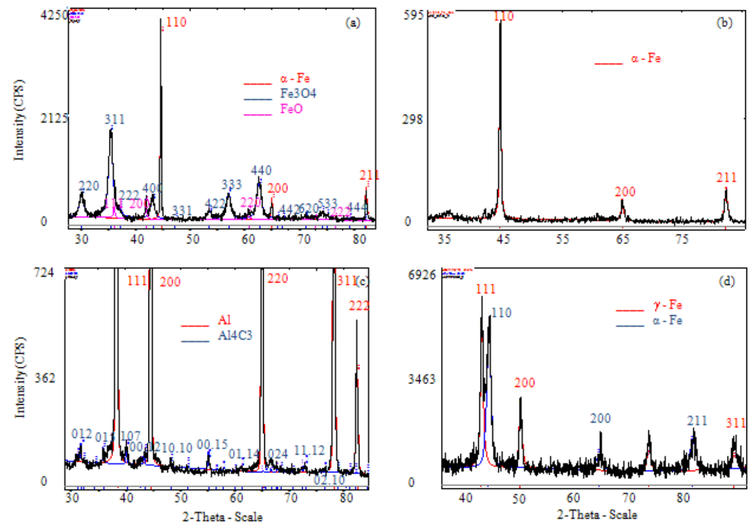


Fig 2 *X-ray diffraction patterns of the NPs of* ***a*** *(α-Fe&Fe_3O_4&FeO)$_w$,* ***b*** *α-Fe$_w$,* ***c*** *(Al&Al_4C_3)$_{COH}$,* ***d*** *(α-&γ-Fe)$_{COH}$.*
Index w for NP received in water, COH for NP received in liquid, contenting C, O, H

The modifying effect of NPs as cast can be explained by changes in patterns of interaction between particles and melt, the crystallization conditions and thermokinetic parameters of phase transformations during cooling of solid metal, as compared with bulk metals. By introduction of ordinary iron powders in metallic melt, supercooling of melt in the places of contact with the particles occurs. It is caused by particles heating and phase transformations: α-Fe → γ-Fe, γ-Fe → δ-Fe, δ-Fe → liquid. When metallic melts were treated by NPs Fe_{COH} patterns of their interaction with melt change. Fig. 3 shows sequence of phase transformations during heating of NPs Fe_{COH} up to 1300 ºC.

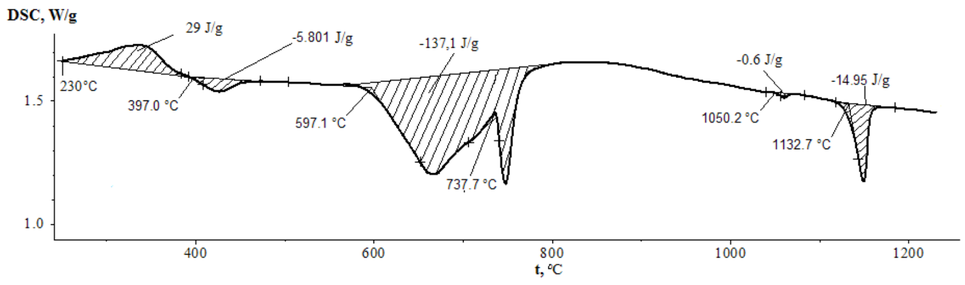
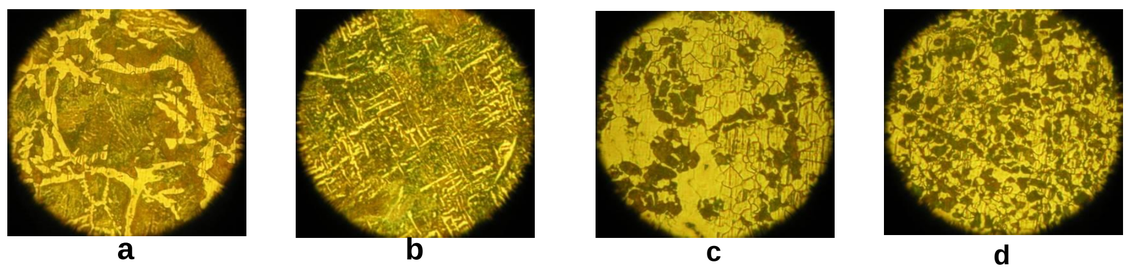


Fig 3 *Sequence of phase transformations during heating of NPs Fe_{COH} up to 1300 ºC*

To assess the effectiveness of nanoparticles obtained by electrospark machining of iron, their influence on the microstructure of 45L steel in the cast state and after annealing at 860 oC was studied. The steel was smelted in an induction furnace, deoxidized with aluminum, and poured into casting sand molds on liquid glass. The microstructure of 45L steel was studied in the initial state (without nanoparticle additives) and after melt treatment with nanoparticles. Photographs of the microstructures are shown in Fig. 4.



a **b** **c** **d**

Fig. 4. The effect of nanoparticles (b, g) obtained during electric spark processing of iron on the microstructure of steel 45 (a, c): a, b - cast condition; c, d - annealing at 860 oC. Magnification X 100.

CONCLUSION

The results of the conducted studies show that the phase and chemical composition of nanoparticles obtained in the process of electro spark processing of iron in water depends significantly on the modes of their production. Electrospark processing of metals in water makes it possible to obtain nanosized iron particles that effectively affect the process of formation and modification of the steel structure.

The model of the structure of nanoparticles depending on the ratio of their sizes and areas of coherent scattering is theoretically substantiated and experimentally confirmed. As a result of processing the 45L steel melt with iron-containing nanoparticles, effective modification of the structure was established. In this case, dispersion of ferrite grains in the cast state occurs by 2.3-2.4 times and an increase in the homogeneity of the structure by 1.7-2.4. After annealing, the size of ferrite grains decreases by 1.2-2.4 times and pearlite - by 1.2-1.5 times, while the homogeneity of the structure increases by 1.2-1.8 times.