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MORPHOLOGY, CHEMICAL COMPOSITION AND MAGNETIZATION OF ARC DISCHARGE FE-C SOOT

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ABSTRACT

Composite Fe-C anode sputtering in a low pressure arc discharge has been used to produce Fe-C soot. The chemical composition and size distribution function of iron containing nanoparticles have been measured. The dependency of magnetic susceptibility at different frequencies and magnetization up to saturation were obtained. It was shown synthesized material is superparamagnetic.

Keywords: arc discharge soot, superparamagnetic, nanoparticles.

1. INTRODUCTION

The studies of the plasma-arc method for the synthesis of magnetic nanoparticles, encapsulated into the carbon coating, started from the paper [1] on the fullerene synthesis. The discovery of a possibility to encapsulate the atoms and nanocrystals by the fullerene structures [2] allowed the start of a study on the magnetic properties of encapsulated atoms at the example of gadolinium carbide [3]. The plasma-arc synthesis and systematic studies of the magnetic properties of nanoparticles obtained by spraying the composite "graphite-transition metal" electrodes, were performed in the work of [4]. The works on the synthesis of magnetic nanoparticles in a carbon coating by the electric arc method are continued. This method uses the DC electric carbon arc with a hot cathode in the atmosphere of inert gas of the reduced pressure. Metal precursors are usually put into a cavity drilled in a graphite electrode, and then they are sprayed together with graphite. Under these conditions, the discharge is maintained by thermal emission of electrons from the cathode. The high temperatures in the zone of arc glow lead to thermal spraying of the anode material. The flow of high-temperature atomic spray products into the buffer gas medium occurs. Diffusion and turbulent mixing of outflowing products with the buffer gas results in cooling, processes of heterogeneous condensation and chemical reactions of the spray products. As a result, the metal particles "packed" into the carbon material are formed. The determining parameters of the synthesis are the pressure and type of the buffer gas, current and discharge voltage, electrode geometry and composition, molar content of the precursor in the sprayed electrode. Varying these parameters, we can control the morphology and chemical composition of the synthesized particles. The pressure of the buffer gas is one of the most important parameters that determine the rate of cooling and kinetics of condensation processes and chemical reactions, which allows the control of an average size of nanoparticles [5]. Usually, the synthesis of nanoparticles is implemented in the inert gas atmosphere. The presence of oxygen in the reactor leads to undesirable oxidation of the graphite electrode. Therefore, the synthesis of metal oxide nanoparticles is performed in two stages. At the first stage, the metal nanoparticles are synthesized on the carbon matrix and at the second stage; synthesized material is annealed in the oxygen atmosphere. The second stage allows oxidation of the metal particles and removal of the carbon material in the form of carbon oxides [6]. The disadvantages of this method are low productivity, broad function of nanoparticles distribution by sizes, and heterogeneity of the carbon coating thickness. Moreover, as usual, the mixture various forms of carbon is synthesized and it is difficult to separate the product from the impurities [7]. Typically, the composite material consisting of graphite and pure metal is used as the sprayed anode, but the plasma-arc method synthesizes nanoparticles from the complex chemical compounds at their spraying and pyrolysis. This ability is demonstrated in experiments [8] on the synthesis of bimetallic magnetic nanoparticles with the use of double salts as the precursors.

The interest in preparation and study of magnetic nanoparticles covered by an inert shell [9, 10] is connected with both the possibility of preventing the coagulation and oxidation of magnetic nanoparticles and the need to ensure biocompatibility in medical applications [11, 12].

In the present study, we investigated morphology, chemical composition, and magnetization of the iron - carbon soot synthesized by the plasma-arc method.

2. EXPERIMENTAL

The experiments were carried out in a direct current electric arc, which had a current of 100 A, in a buffer gas (helium) at 50 Torr. The spray electrode (anode) was a graphite rod 70 mm in length and 7 mm in diameter. A hole (with a diameter of 4 mm) was drilled in the center of the electrode to be filled with graphite–iron mixture powder. The Fe/C weight ratio was 2/1. Monatomic spray products were diffused in the buffer gas from the hot zone of the arc, which resulted in cooling and heterogeneous condensation of the spray products. The composite material was precipitated on a cooled shield located 5 cm from the arc discharge area. The synthesized material consisted of iron-containing nanoparticles on a carbon matrix.

High-resolution TEM images were obtained using a JEM-2010 electron microscope (JEOL, Japan) with lattice-fringe resolution of 0.14 nm and accelerating

voltage of 200 kV. The high-resolution images of periodic structures were analyzed by the Fourier method. Local energy-dispersive X-ray analysis (EDXA) was carried out using an EDX spectrometer (EDAX Co.), which was fitted with an Si (Li) detector, at a resolution of 130 eV. The samples for the HRTEM study were prepared on a perforated carbon film mounted on a copper grid. XRD analysis was carried out using a Bruker D8 Advance diffractometer, which was equipped with a Lynxeye (1D) linear detector, over the angular range of 10-75º at 2Θ = 0.05º with a storage time of 1 s for each point. Monochromatic CuK-radiation (1.5418˚A) was applied in these experiments. Magnetic properties were measured by magnetometer SM-150L ZH Instrument, and SQUID magnetometer MPMSXL (Quantum Design).

3. RESULTS

Electron microscopy of the synthesized material indicated that it consisted of iron-containing nanoparticles of 5-10 nm (see Figure-1) embedded in amorphous carbon matrix.

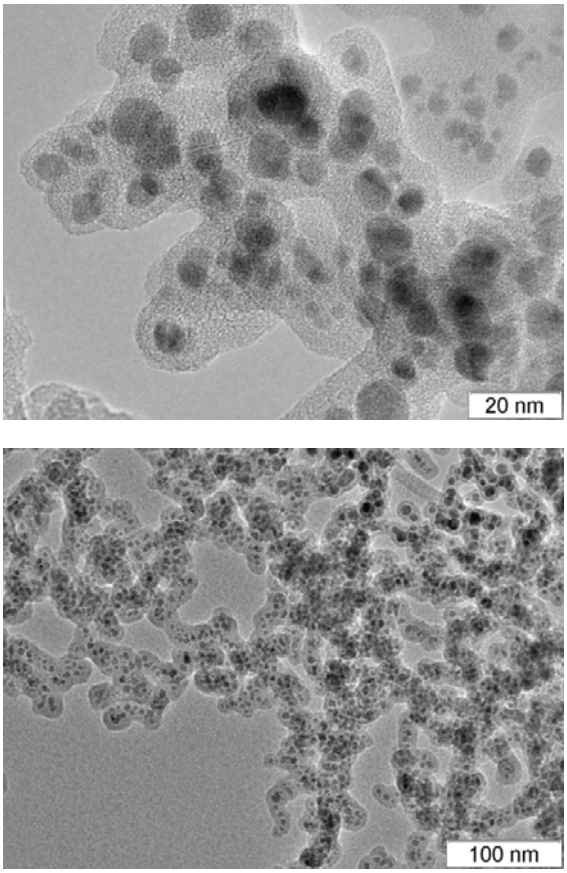

Figure-1. The morphology of the synthesized material.

The function of size distribution of nanoparticles is shown in Figure-2. The solid line is the logarithmically normal approximation.

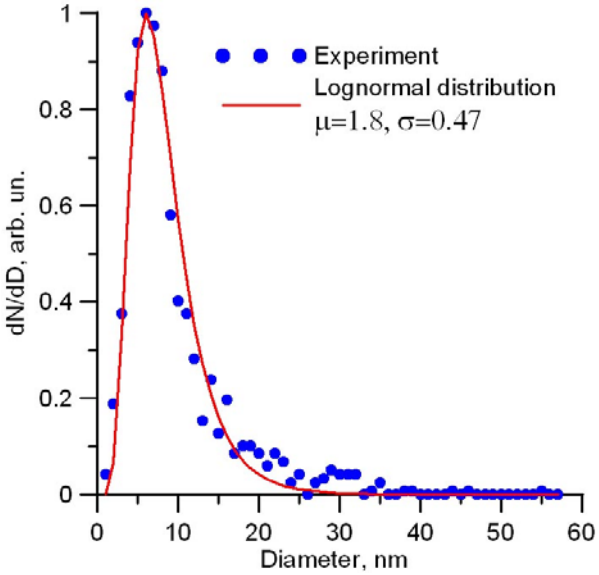

Figure-2. Size distribution function.

XRD spectroscopy shows that Fe-C soot consists of graphite, iron, and iron carbide (see Figure-3).

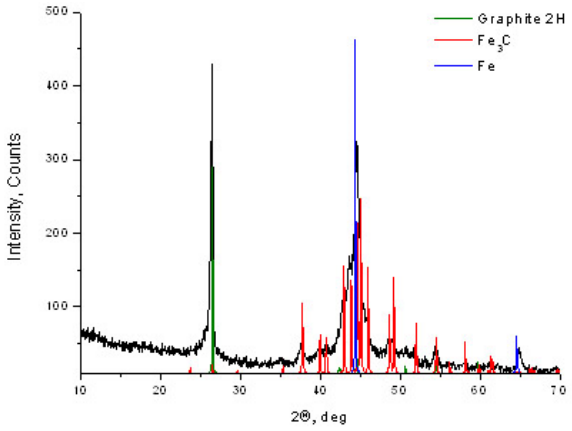

Figure-3. XRD spectrum of synthesized material.

Magnetic susceptibility was measured in wide range of frequencies (63 Hz - 16 KHz). Figure-4 shows logarithmic decreasing of magnetic susceptibility with frequency. Magnetization curve is shown on Figure-5. Saturation value of about 50 G cm^3/g is reached at magnetic field of about 10^4 Oersted. No hysteresis has been observed at room temperature.

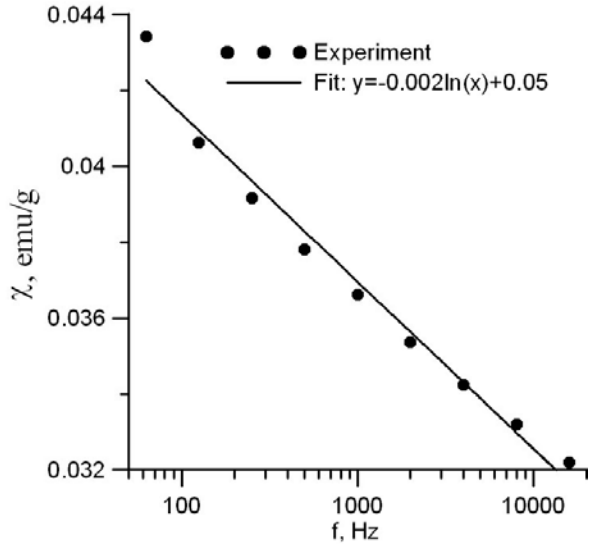

Figure-4. Magnetic susceptibility at different frequencies.

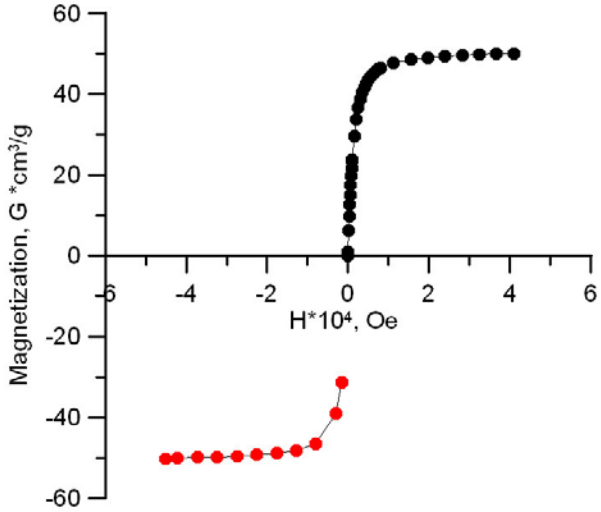

Figure-5. Magnetization of Fe-C soot.

4. CONCLUSIONS

Composite Fe-C anode sputtering in a low pressure arc discharge has been used to produce Fe-containing nanoparticles on a carbon matrix. The chemical composition of the synthesized material consists of carbon soot, graphite, iron, and iron carbide. Size distribution function of iron containing nanoparticles is lognormal with maximum at about 7 nm. Relaxation processes of magnetization resulted in logarithmic decreasing of magnetic susceptibility with frequency. The dependency of magnetization on magnetic field showed no hysteresis. It means that synthesized material is superparamagnetic.

ACKNOWLEDGEMENTS

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