

PROPERTIES OF IRON AND NICKEL METAL-CARBON NANOCOMPOSITES SYNTHESIZED BY ARC DISCHARGE IN LIQUID

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Introduction

Many of unusual physical and chemical properties of nanoparticles are defined by high ratio of their surface area to volume. Possibilities of changing surface area and its chemical composition open far-reaching perspectives for synthesis of new magnetic materials.

Evaporation of metals in carbon-containing liquids or simultaneously with graphite allows the preparation of nanometallic particles in the carbon matrix which properties are scantily known.

In the present work, Me-C composites have been produced by the arc discharge method in toluene using a powder metal anode (ADIP); their structure, phase composition and magnetic properties have been studied.

Results and discussion

Crystalline structures and phase compositions of powders were determined using X-ray diffractometer DRON-3.0 in Cok_α irradiation; magnetic properties were measured on a ballistic magnetometer; extent of the coherent-scattering region (CSR) were calculated by broadening X-ray lines according to Selyakov-Sherrer formula.

Table 1 represents the results of studies on initial iron and nickel powders and the product prepared by arc discharge in the liquid phase using a powder anode (ADIP) in toluene. Also, the table gives the data on Fe(B-5-2) and Ni(B-2) powders before and after thermo-magnetic measurements. Figs. 1-6 and table 2 illustrate the results of measurements of magnetic properties: specific saturation magnetization, σ_s , coercive force, Hc, and residual induction, Br. Figs. 1-4 demonstrate field dependences of specific magnetization, and figs. 5-6 show temperature dependences.

Diffraction patterns of initial iron and nickel powders show only lines from *bcc* iron and *fcc* nickel, respectively. After ADIP treatment in toluene, the phase composition of synthesized powders has changed. The diffraction pattern of Fe(B-5-2) powder demonstrates two crystalline phases, α -Fe (~24%) and Fe_3C (~76%). In addition to the lines from pure nickel, the diffraction pattern of Ni(B-2) powder has the most intensive line from carbon solid solution in nickel. In magnetic thermograms, Curie temperatures, 400 and 760°C for iron powders (fig.5) and 225 and 360°C for nickel powder (fig.6) correspond to these phases. For

M-C composites, specific saturation magnetization of nickel powder is almost unchangeable (figs. 3 and 4, table 2); this is related to a low amount of Ni-C crystalline phase that is formed in powder by ADIP treatment. Specific saturation magnetization of Fe-C composites changes more significantly (figs. 1 and 2, table 2). This is conditioned by the considerable change in the phase composition of iron powders after ADIP treatment. In synthesis, initial α -Fe transforms into carbide, Fe_3C almost completely (table 1). For the products, changes in coercive force, Hc, and residual induction, Br, can be attributed to the corresponding changes in the phase composition and dispersion of the powders.

The results of changing the phase composition of the powders after heating in measuring the temperature dependence of specific saturation magnetization are also of interest. These results are shown in figs.5, 6 and table 1. As can be seen, the phase composition of iron and nickel in the synthesized powders has considerably changed on heating. After heating, the line from Ni-C solid solution has disappeared from the diffraction pattern of Ni(B-2) powder. After heating, intensities of the lines from Fe_3C carbide have reduced in the diffractogram of Fe(B-5-2) powder, intensities of lines from α -Fe increased, and the lines from crystalline phases, FeO and Fe_3O_4 appeared. According to the X-ray data, after heating and in subsequent cooling the knee at ~225°C (Ni-C solid solution) has disappeared from the magnetic thermogram of Ni(B-2) powder. In subsequent cooling, in addition to the knees at ~760°C (α -Fe) and ~400°C (Fe_3C), the knee at ~600°C corresponding to the Curie temperature for Fe_3O_4 oxide has also appeared in the thermogram of Fe(B-5-2) powder.

On the basis of TEM observations, one can note that the synthesized nanocomposites of both iron and nickel contain particles 1-400 nm in diameter. The major part of particles has a diameter ranging from 10 to 20 nm (figs.7-8).

The shell on the large nickel particles (fig.7) indicates that this particle is formed from the melt. Interaction of melted Ni and carbon vapor gives rise to Ni_3C carbide that decomposes during the alloy crystallization, liberating carbon. The carbon forms graphite-like nanostructures on the surface of a particle.

Table 1.
Phase composition and extent of CSR for iron and nickel powders.

Sample	Phase composition	Content, %	D, nm
Fe powder, initial state	α - Fe	100	270
Fe powder (B-5-2)	α - Fe Fe ₃ C	24 76	- 24
Ni powder, initial state	Ni	100	150
Ni powder (B-2)	Ni Ni- C	96 4	150
Fe powder (B-5-2), after heating	α - Fe Fe ₃ C Fe ₃ O ₄ FeO	73 11 16	110 40 100
Ni powder (B-2), after heating	Ni	100	130

Table 2.
Magnetic properties of iron and nickel powders.

Material	σ_{ss} A·m ² /kg		Oe, Э	Br, Gs
	20	-196		
Ni, init.	56	58.5	50	1434
Fe, init.	219	222	6	102.2
Ni(B-2)	56	58	50	860
Fe(B-5-2)	84.3	95.8	20	108.7

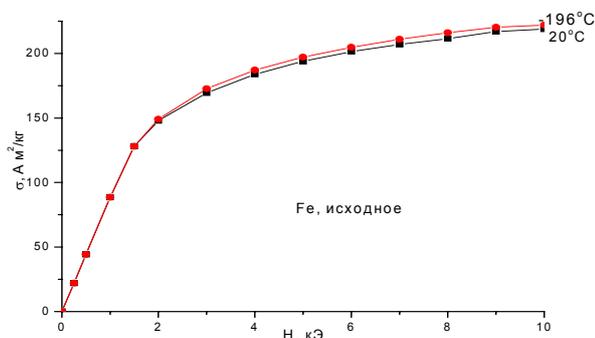


Fig. 1. Field dependence of initial iron powder at temperatures -196 and 20°C.

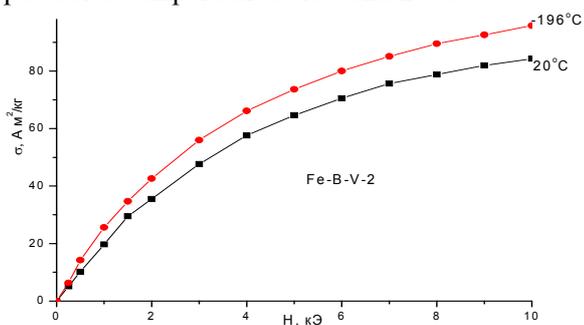


Fig. 2. Field dependence of Fe-C nanocomposite at temperatures -196 and 20°C.

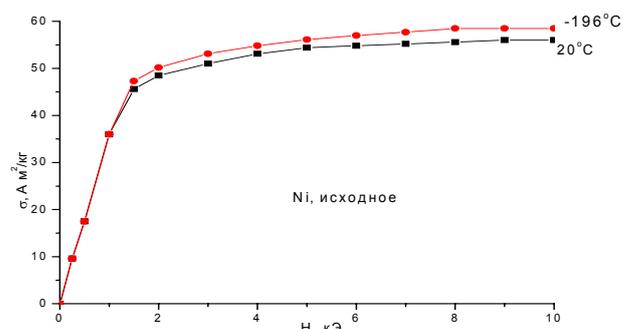


Fig. 3. Field dependence of initial nickel powder at temperatures -196 and 20°C.

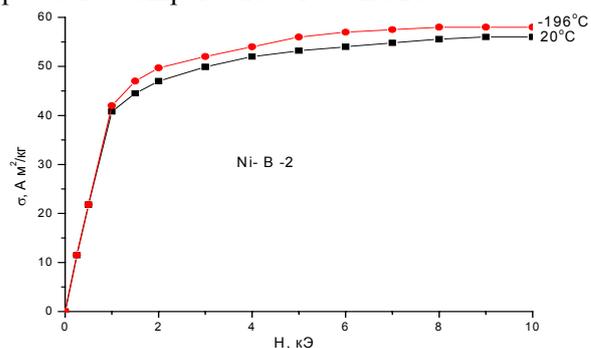


Fig. 4. Field dependence of Ni-C nanocomposite at temperatures -196 and 20°C.

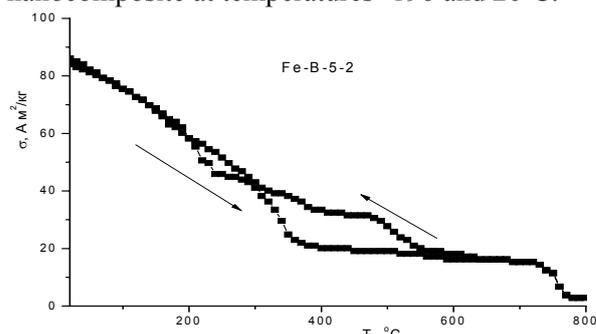


Fig. 5. Temperature dependence of specific magnetization of Fe-C nanocomposite before and after heating to 800°C.

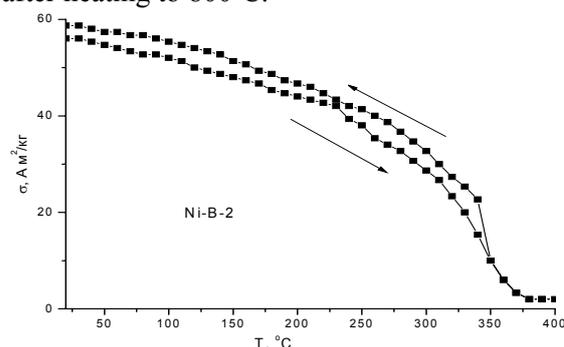


Fig. 6. Temperature dependence of specific magnetization of Fe-C nanocomposite before and after heating to 800°C.

Large iron particles (~ 150 nm) do not have such prominent boundaries, although all particles of 10-30 nm fractions are wrapped into carbon shells.

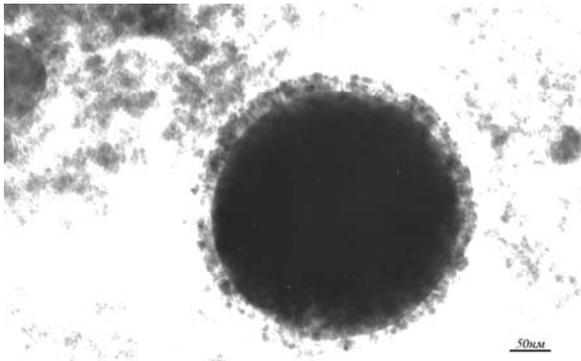


Fig. 7.

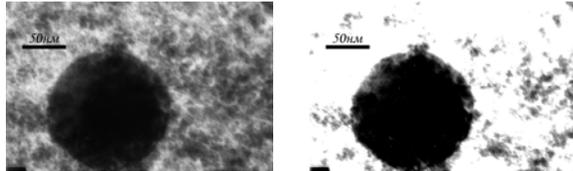
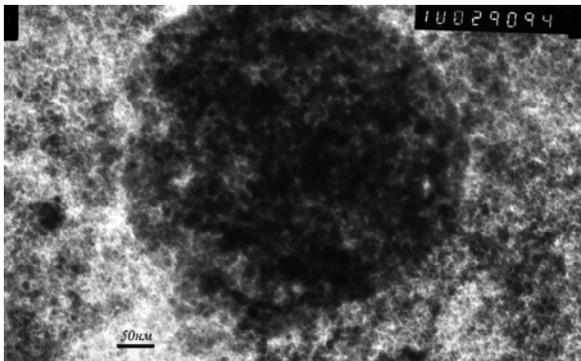
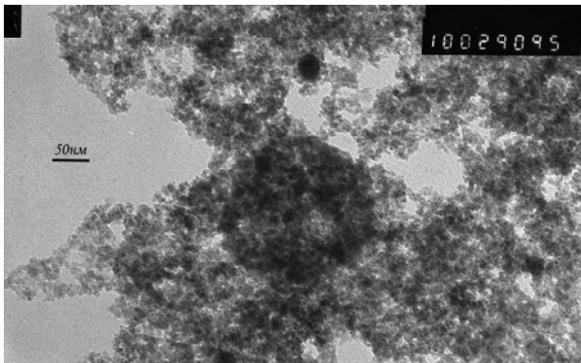


Fig. 8.



a)



b)

Fig. 9.

In addition, iron nanoparticles formed by the arc magnetic field exhibit sufficient magnetization. This causes the nanoparticles to agglomerate in spherical clusters up to 1 μm in diameter (fig. 9).

Conclusions

In the present work Me-C nanocomposites have been produced by the ADIP method. Their structure, phase composition and magnetic properties have been studied.

The performed studies have shown that Me-C composites have a significantly changed phase composition. α -Fe powder transforms into Fe_3C carbide almost completely, and solid carbon solution in nickel, Ni-C forms in the Ni powder.

Heating the synthesized nanocomposites also leads to the change in their phase composition: after heating, a crystalline phase, Ni-C disappears in the Ni(B-2) powder, and oxides, FeO and Fe_3O_4 appear in the powder Fe(B-5-2) as well as the ratio of crystalline phases, α -Fe and Fe_3C changes.

Acknowledgment

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