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Nanodispersed Powders of Fe-Ni Particles with Carbon Shell

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The initial carbon condensate containing nickel-iron nanoparticles with a graphite conversion was synthesized in the high-frequency carbon-helium arc plasma at ambient pressure. The nickel-iron particles were extracted by the treatment of the carbon condensate with nitric and hydrochloric acids. Undissolved residue and dissolved sample were received. Undissolved residue has both the structure and magnetic properties of (MR/Ms = 0.30, Hc = 270 Oe). Lack of hysteresis loops for the dissolved sample shown that metals are in the paramagnetic state.

Keywords: nanoparticles, carbon, nickel, iron.

Introduction

Understanding the magnetic properties of nanometer scale particles is a central issue in magnetic materials. Magnetic nanoparticles themselves are used as the active component of ferrofluids, recording tape, flexible disk recording media, as well as biomedical materials and

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catalysts [1]. Carbon encapsulated metal nanoparticles have received considerable attention because of their high chemical and thermal stabilities [2]. Due to the high surface to volume ratio unshielded metal nanoparticles are easily oxidized in air atmosphere. For practical application it is necessary to coat metal nanoparticles with air-stable materials. Carbon is one of the best solution for encapsulation because of its biocompatibility and chemical stability [3].

In the last decades, much research has been devoted to the synthesis of magnetic nanoparticles. Especially during the last few years, publications have described sputtering of electrodes in a dc arc in the helium atmosphere at the pressure 10–26 kPa as the main synthesis of nanoparticles [4]. The drawback of this method is impossibility of controlling the synthesis parameters. Overly large or small currents and pressure (outside the mentioned range) usually lead to significant decrease nanoparticles content. The method of the synthesis in HF-arc plasma allows effective obtainment of nanostructures in wider synthesis parameter ranges [5].

1.   Experimental Details

In this work, we extracted and investigated the components of the condensate produced by cooling the plasma consisting of carbon, nickel, iron and helium. In this setup, which was described earlier by us [6], plasma was formed in a high frequency arc at atmospheric pressure. That setup can be used for obtaining carbon condensate with different dispersion and structure, as well as containing core-shell nanostructures (nanoparticles and endohedral metallofullerenes). The arc was ignited between two graphite rods 6 mm in diameter. Mixture of nickel and iron powders with a particle size of 0.25 μm and graphite powder was placed in an axial hole of the graphite rod electrodes. The ratio of the powder introduced into the plasma was 45 wt% Ni, 50 wt% Fe and 5 wt% C.

Synthesis was carried out under 66.5 kPa helium pressure at chamber. The arc was fed by an electric current of 240 A at a frequency of 66 kHz, which made it possible to transform almost all electrode material (98 %) into a condensate formed on the walls of the water cooled chamber. From the condensate collected off the chamber walls was extracted fullerene at Soxhlet apparatus using toluene as solvent. It was boiling at a temperature of 90 °C in a strong nitric acid for 3.5 h. The precipitate filtered through an FS-III paper filter was insoluble in the acid and amounted to 76 wt% of the initial condensate. It was washed in distilled water to remove the acid and salt products and then was dried under normal conditions (sample 1).

The solution, which was obtained upon boiling carbon black in the acid, was evaporated; then, the residue was washed in distilled water at a temperature of 60 °C until the solution reached the pH value of 5. The precipitate thus obtained was dissolved in a dilute hydrochloric acid at a temperature of 90 °C and then was filtered off. The undissolved residue again was washed in distilled water and dried (sample 2) [7]. This method may be also use for fictionalization of fullerene and endohedral metal fullerenes [8].

The structure and composition of the samples were investigated using X-ray powder diffraction (DRON-4 diffractometer), magnetometric measurements (VSM magnetometer), and TEM images (Hitachi HT-7700).

2.   Results and Discussion

The X-ray fluorescence analysis of samples has demonstrated that the samples contain nickel, iron, carbon and oxygen 21.62, 22.06, 14.72 and 41.6 wt% for sample 1, 14.24, 15.85, 51.54 and

18.37 wt% for sample 2, accordingly.

The X-ray powder diffraction pattern of sample 1 do not show the presence of crystal structure at the sample. The X-ray powder diffraction pattern of sample 2 contain reflections from graphite (26.4°, 42.2°, 44.4° and etc.), iron (43.7°, 63.4° and 80.1°), iron nickel (43.5°, 50.7° and 74.6°) and nickel oxide (37.3°, 43.3°, 62.9°, 75.4° and 79.4°) (Fig. 1). Phase of FeNi was formed by coagulation of the metal atoms in the plasma, wherein part of Ni oxide forms in consequence of residual oxygen present in the chamber.
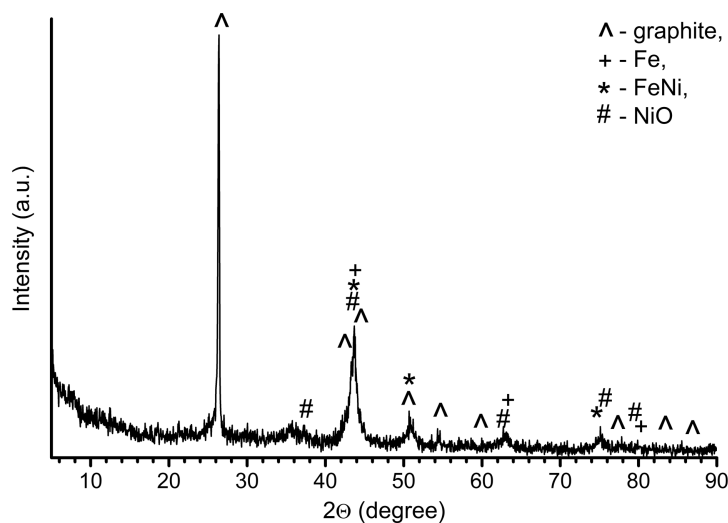

Fig. 1. X-ray diffraction pattern of sample 2

Examination of the samples by transmission electron microscopy showed that the sample 2 is in the metal particles with a size of several tens of nanometers with a multilayer coating carbon and part of carbon is graphene layers (Fig. 2b). As for of sample 1 particles have irregular shape with size of 3-5 nm (Fig. 2a). Sample image does not contain particles with a clear structure, which confirms by data of X-ray powder diffraction.

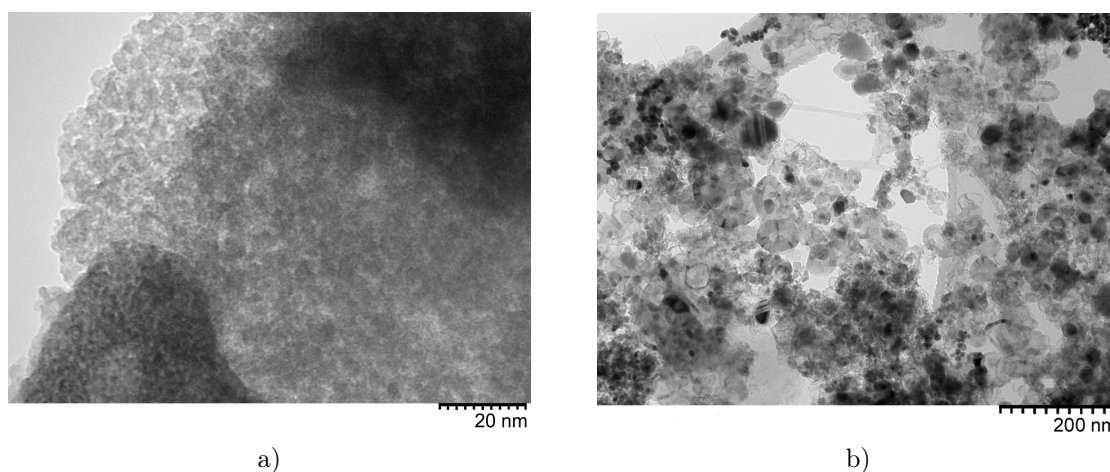a)   b)

Fig. 2. TEM images of sample 1 (a) and sample 2 (b)

The size effect was very significant. Sample 2 has both the structure and magnetic properties of (MR / Ms = 0.30, Hc = 270 Oe). Lack of hysteresis loops for the sample 1 shows that metals are in the paramagnetic state. Also, on the based of the presence of a large amount of oxygen (40 wt%) it may suggest that it is fine dispersed particles of oxides and carbides.

Analysis of the results of sample 2 investigation showed that a large coercivity (270 Oe) can not occur due to the shape anisotropy or inhomogeneity of chemical composition. Indeed, virtually all of the particles have a spheroidal shape, Fig. 3, and the hysteresis loop corresponds monophasic material. The coercive force of nano-dispersed particles exceeds by more than an order of magnitude coercivity force of massive alloys. Most probably this effect can be explained by nanoparticles stresses that arises as a result of rapid cooling and stores under the influence of the surface layer formed during the heat treatment.
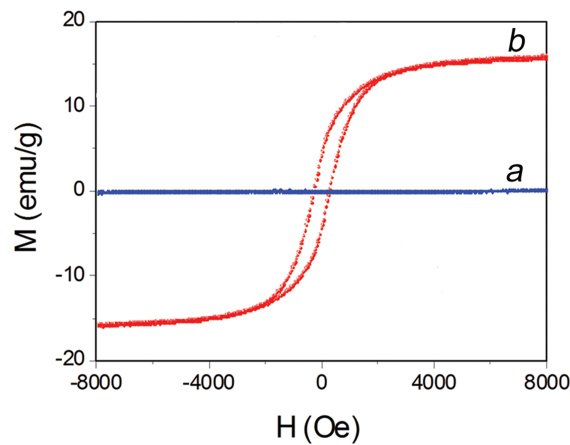

Fig. 3. The magnetization curve of sample 1 (a), sample 2 (b). The curves dimension on the automated vibrating magnetometer with Puzey electromagnet at room temperature for insoluble and soluble fractions

3.   Conclusion

Thus, from a fundamental point of view, not all the properties of nano-dispersed iron - nickel particles uniquely interpreted. As for applied problems, the paper shows that for the case where the required magnetic properties of the obtained iron, nickel particles may be useful. Hardware, including 3-d metal has a maximum saturation induction, but has a high reactivity and poor compatibility with gold. We plan to use them as the core of the synthesis of particles with the structure of the magnetic core–shell (a noble metal).

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Нанодисперсные порошки Fe-Ni частиц с углеродной оболочкой

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Исходный углеродный конденсат, содержащий железо-никелевые наночастицы с углеродной оболочкой был синтезирован в углеродно-гелиевом дуговом ВЧ разряде при атмосферном давлении. Железо-никелевые частицы были выделены обработкой углеродного конденсата азотной и гидрохлоридной кислотами. В результате были получены. Нерастворимый образец имеет структуру, а магнитные характеристики (MR / Ms = 0.30, Hc = 270 Oe). Растворимый образец продемонстрировал отсутствие петли гистререзиса, что характерно для металлов в парамагнитном состоянии.

Ключевые слова: наночастицы, углерод, никель, железо.