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The Synthesis of Nanodispersed Magnetite Using Electrochemical Method

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The purpose of the work was to obtain nanodispersed magnetite (Fe_3O_4) with fewer impurities comparing to its natural form. For this the electrochemical method of dissolution of steel sheets or shavings in the heated electrolyte was employed. In order to optimize the technological parameters of this process the structural and magnetic properties of the obtained materials were studied using powder X-ray diffraction and vibrational magnetometry techniques correspondingly.

Keywords: Magnetite, Electrochemical deposition, Heated electrolyte, Powder XRD, Magnetization.

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1. INTRODUCTION

Magnetite is one of the most important ferrimagnetic materials and is quite common in nature. However, due to intensive use for the industrial purposes the reserves of natural magnetite in igneous rocks decrease rapidly [1-4]. Thus, finding and optimization of alternative ways allowing to obtain this material is an important task. The promising approach is to synthesize it from iron containing industrial waste.

In the case when magnetite is used as the redox agent for iron separation, the presence of the natural impurities does not noticeably affect this process [5]. But in the case when the magnetic properties of the material become crucial, i.e. when it is used in transformer cores, inductors, filters, and many other technical devices [6], the highest possible purity is strongly desired [7].

Magnetite synthesized by electrochemical dissolution of steel St3 (sheets, shavings) can possibly meet these requirements. The impurities content of these materials does not exceed the following values (%): carbon – 0.14-0.15; sulfur – up to 0.055; phosphorus – up to 0.045; arsenic – 0,015. In previous experiments using the electrochemical method we have synthesized samples of nanodispersed magnetite with the value of magnetization equal to (282 ± 10) kA/m [8]. The aim of the present work along with generalization of the previous data is to study the impact of synthesis conditions on the phase composition of obtained materials in details.

2. RESULTS AND DISCUSSION

For electrochemical dissolution the sheets of steel St3 with the thickness of 2 mm were used. The electric current with the density of 1 A/dm² was maintained in the 0.2 % solution of NaCl at the constant temperature T_e which for different preparation cycles had different values from 20 °C to 100 °C with the step of 10 °C (the boiling point of the solution is 103 °C). As the result of this process the amount of magnetite sufficient for the further measurements precipitated in about 5 minutes with the increase of the pH of the solution to the value of 8. Dried at the temperature of 105 °C to the constant mass the precipitate was studied by means of powder X-ray diffraction. Measurements were done using powder X-ray diffractometer DRON-UM-1 with CoK-α radiation (λ = 0,17902 nm). As the result for the obtained materials the crystalline phases were identified and the average size of the particles was calculated. For the samples synthesized by electrochemical method under various conditions the structural analysis using X-ray diffractometer ARL X'TRA was also done (see Fig. 1).

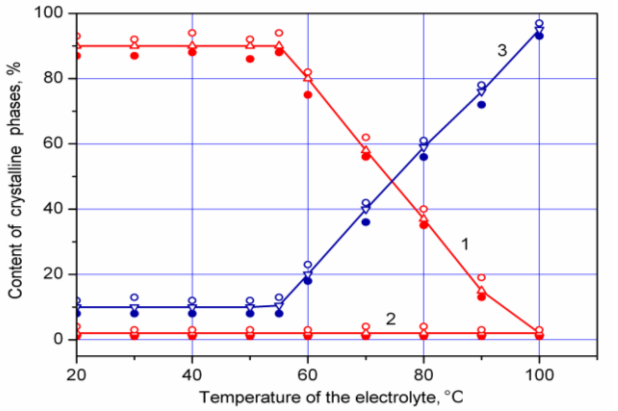

Fig. 1 – The concentration of the crystalline phases in synthesized materials depending on the temperature of electrolyte

Fig. 1 reflects the influence of the electrolyte temperature T_e on the composition of the resulting material. Analyzing X-ray diffraction patterns one can see that along with magnetite (Fe_3O_4) the samples contain lepidocrocite (γ-FeOOH) and goethite (α-FeOOH); the concentration of goethite (Fig. 1, curve 2) is negligible while the concentration of lepidocrocite (Fig. 1, curve 1) decreases with the increase of the temperature T_e resulting in the almost 100 % concentration of magnetite in the samples synthesized at the temperature T_e = 100 °C (Fig. 1, curve 3). From the figure it is clear that for the samples prepared at the temperatures be-

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low 50-55 °C γ-FeOOH is the dominant phase while the concentration of the rest two phases reaches only about 10 %. When the value of T_e exceeds 50-55 °C the concentrations of Fe_3O_4 and γ-FeOOH drastically change increasing and decreasing correspondingly while there is no sign of changes in the concentration of α-FeOOH. Thus in the process of electrochemical dissolution of St3 steel in the electrolyte heated up to 100 °C the precipitate consists almost entirely of magnetite (Fe_3O_4).

These results inspired further research with an ambition of optimization of the technological parameters for the high temperature synthesis of magnetite and evaluation of the resulting product quality.

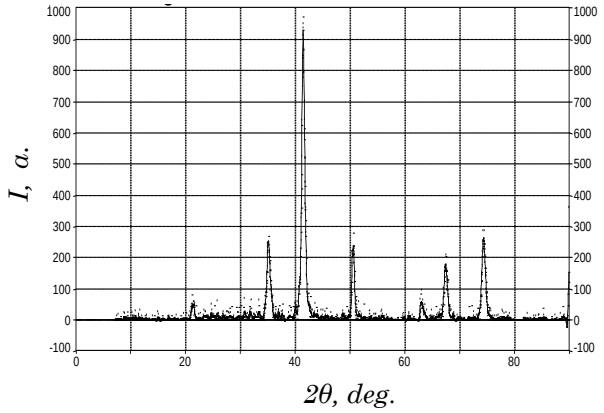

Fig. 2 – X-ray diffraction pattern of the sample containing magnetite nanoparticles with the average size of 24 nm electrochemically synthesized at 90 °C

The synthesis was done in the electrochemical cell filled with 0.2 % NaCl solution and with anode and cathode made of St3 steel shavings and sheet correspondingly. The distance between the electrodes varied between 50 and 60 mm. The cell was thermally stabilized at the temperature (100 ± 1) °C. For the process of anode shavings dissolution the electric current density was maintained at the value of 5 A/dm^2. The energy consumption of the described process was (2.30 ± 0.20) W·h/g.

Nanodispersed magnetite synthesized under specified conditions had the high purity (the concentration of impurities did not exceed 5 % according to X-ray diffraction data) and high values of saturation magnetization reaching (305 ± 10) kA/m. The studied samples turned into paramagnetic state upon heating to the temperatures higher than 580 °C [9], which corresponds to the Curie temperature of magnetite.

Fig. 2 and 3 show the results of X-ray and Mossbauer studies of the sample synthesized by electrochemical method with the average particle size of 24 nm according to X-ray diffraction data. Parameters of the Mossbauer spectrum calculated using «Spectr» software correspond to nanodispersed Fe_3O_4 being in agreement with the results of X-ray diffraction pattern analysis.

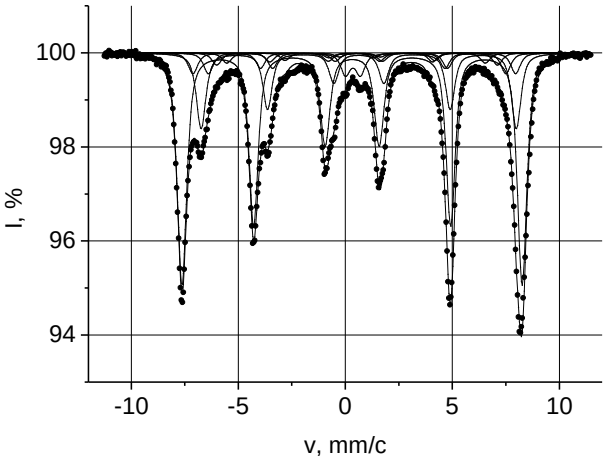

Fig. 3 – Mossbauer spectrum of the sample containing magnetite nanoparticles with the average size of 24 nm electrochemically synthesized at 90 °C

3. CONCLUSIONS

Thus, it is possible to make a conclusion that the technological process described above allows to synthesize magnetite comparable favorably with known analogs in terms of purity and magnetic properties.

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