

ИССЛЕДОВАНИЕ ПРОЦЕССОВ ПОЛУЧЕНИЯ НАНОПОРОШКА ЖЕЛЕЗА ПРИ НЕИЗОТЕРМИЧЕСКИХ УСЛОВИЯХ

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Проведено исследование кинетических параметров процесса получения нанопорошка железа водородным восстановлением гидроксида α -FeOOH при неизотермических условиях. Показано, что восстановление нанопорошка α -FeOOH протекает в интервале температур 180–550 °С с максимальной удельной скоростью при температуре 500 °С. Величина энергии активации процесса восстановления нанопорошка α -FeOOH составила ~43 кДж/моль, что говорит о смешанном режиме реагирования. Установлено, что восстановление α -FeOOH при температуре 500 °С обеспечивает получение продукта с необходимыми свойствами. Полученные наночастицы Fe, размер которых – от 70 до 100 нм, имеют округлую форму.

Ключевые слова: кинетика; нанопорошок железа; неизотермическое условие; дифференциально-разностный метод; энергия активации.

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PREPARATION PROCEDURE TO OBTAIN
IRON NANOPOWDER UNDER NON-ISOTHERMAL CONDITIONST. H. NGUYEN^{a, b}, V. M. NGUYEN^c, M. H. NGUYEN^b, V. N. DANCHUK^a^aNational University of Science and Technology «MISiS»,
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The kinetics for the procedure of preparing iron nanopowder from α -FeOOH by hydrogen reduction under non-isothermal conditions were studied. The reduction of α -FeOOH nanopowder was shown to occur within the temperature range from 180 to 550 °C, with a maximum specific rate value attained at 500 °C. The activation energy for the reduction process α -FeOOH nanopowder was measured to be ~43 kJ/mol, evidencing a mixed reaction mode. Performing the reduction of α -FeOOH at 500 °C accelerated the process while ensuring the required properties of the product obtained. The Fe nanoparticles thus prepared were of rounded shape, the size ranging from 70 to 100 nm.

Keywords: kinetics; iron nanopowder; non-isothermal condition; differential-difference method; activation energy.

Introduction

Iron nanopowders (NPs) have actually found wide application in various fields of science, technology and medicine [1; 2]. In particular, Fe NPs are often used as active adsorbents having magnetic properties to remove toxic contaminants from soil and wastewater [3; 4]. Magnetic properties of the adsorbents containing Fe NPs enables one to avoid laborious operations of removing the purifying agent from the treated material, since this removal can be achieved by using magnetic field. The NPs of metallic Fe were shown to have large specific surface area and high adsorbing capacity, and hence they are excellent adsorbents for heavy metals. Moreover, Fe nanowires have unique magnetic properties that can be controlled by magnetic field [5; 6].

Preparation of Fe NPs may be carried out by various procedures most of which have a number of disadvantages, such as poor productivity and (or) high energy consumption [7–12]. The chemical-metallurgical method, which includes the stages of chemical deposition of oxygen-containing metal compounds with subsequent hydrogen reduction of the oxides, is a highly efficient procedure as regards energy saving, utilising production waste and the ability to control the dimensional characteristics of the metal NPs during their formation [13; 14].

A major disadvantage of the chemical-metallurgical method for obtaining Fe NPs is relatively not grade productivity due to the low rate of the hydrogen reduction process under low temperature conditions. Increasing the reduction temperature is not recommended, since it would entail intensifying aggregation and sintering processes, as well as formation of large particles or particles outside the nanometer range [11; 13].

Investigation of kinetics of the processes leading to formation metal NPs in order to determine optimal time-temperature parameters that would ensure obtaining the product with the desired properties is an important practical task.

The aim of this work was investigation of kinetics of the processes leading to formation of Fe NP from its precursor hydroxide compound under non-isothermal conditions, as well as examination of properties of the products obtained.

Materials and methods

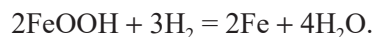
Fe NP was prepared from α -FeOOH NP by direct reduction with hydrogen. The starting α -FeOOH was obtained from aqueous solutions of iron(III) nitrate (10 wt. %) and NaOH (10 wt. %) by chemical precipitation of the product at 25 °C under continuous stirring:



The pH value was maintained at 11 ± 0.03 , monitored with an pH meter Expert-001 (*Econix-Expert Ltd.*, Russia).

The precipitate thus obtained was washed on a Buchner funnel with distilled water until the flushing water pH was 7. The precipitate was then dried at 40 °C for 48 h. Thereafter, the dried precipitate was ground in a laboratory mill Pulverisette 2 (*Fritsch*, Germany) to obtain α -FeOOH NP which was used for further research work.

The procedure of preparing Fe NP using hydrogen reduction of α -FeOOH under non-isothermal conditions was performed on a thermal analyser SDT Q600 (*TA Instruments*, USA) in a linear heating mode at a rate of 10 °C/min in the temperature range 25–700 °C. The reduction proceeded according to the equation



The value of the degree of conversion α (i. e. the ratio of the mass of the reacting sample to the mass of the initial sample) was calculated according to the formula

$$\alpha = \frac{m_o - m_t}{m_o} \frac{M_{\text{FeOOH}}}{M_{\text{O}_2\text{H}^{3-}}} = 2.69 \frac{m_o - m_t}{m_o}, \quad (1)$$

where m_o and m_t are the initial sample mass and the mass of the sample after the reaction time t respectively.

Calculation of kinetic parameters for the reduction of α -FeOOH under non-isothermal condition was performed by the differential-difference method using the data obtained from thermogravimetric (TG) analysis, as well as the equation of non-isothermal kinetics [15].

The differential-difference for this case may be written as follows

$$\frac{\Delta \ln \left(b \frac{d\alpha}{dT} \right)}{\Delta \ln(1-\alpha)} = n - \frac{E_a}{R} \frac{\Delta \frac{1}{T}}{\Delta \ln(1-\alpha)}, \quad (2)$$

$$y = \frac{\Delta \ln \left(b \frac{d\alpha}{dT} \right)}{\Delta \ln(1-\alpha)}, \quad x = \frac{\Delta \frac{1}{T}}{\Delta \ln(1-\alpha)}, \quad (3)$$

where α is the degree of conversion; T – temperature, K; b – heating rate, K/s¹; E_a – activation energy, J/mol; R – universal gas constant, J/(mol · K); n – order of the reaction.

The phase composition of the samples was investigated by XRD phase analysis on a diffractometer Difractometer 401 (*Scientific Instruments JSC*, Russia) using CrK_α radiation. The specific surface area (S) of the powders was determined by the BET (Brunauer – Emmett – Teller) method with a low temperature adsorption of nitrogen on the NOVA 1200 analyser (*Quantachrome*, USA). The average particle size $D(m)$ was calculated by formula (4) using the S data:

$$D = \frac{6}{\rho S}, \quad (4)$$

where ρ – the density of the material, kg/m³.

The morphology and dimensional characteristics of NP samples were studied by scanning electron microscopy (SEM) on the microscope VEGA 3B (*TESCAN*, Czech Republic).

Results and discussion

The XRD pattern and SEM image obtained from the initial α -FeOOH sample are shown in fig. 1. The XRD pattern (see fig. 1, *a*) reveals that the sample under study is a monophase one, containing the α -FeOOH phase only. Examination of the SEM image (see fig. 1, *b*) evidences dense location of α -FeOOH NPs having small needle-like shape. Specific surface area of α -FeOOH NPs was found to be 39.5 m²/g, with an average diameter of the particles amounting to 44 nm.

Analysis of the TG data obtained (fig. 2) indicates that the reduction process of FeOOH is realised in four stages.

The stage 1 occurs in the temperature range from 25 to 180 °C, when the α -FeOOH sample loses its adsorbed moisture. The stage 2 corresponds to the process of thermal decomposition of α -FeOOH according to scheme (5) shown below. This reaction is realised in the temperature range from 180 to 300 °C, the maximum specific rate being achieved at 240 °C. The stage 3 occurring between 300 and 375 °C corresponds to reduction of α -Fe₂O₃ to α -Fe₃O₄ according to scheme (6), with the maximum specific rate measured at 340 °C. In the stage 4, reduction of magnetite α -Fe₃O₄ to metallic Fe takes place according to scheme (7). This process is realised in the temperature range from 375 to 550 °C. The maximum specific rate value amounted to $1.079 \cdot 10^{-8}$ kg/s at 500 °C.



¹ $b = 10 \text{ °C/min} = 0.197 \text{ K/s}$.

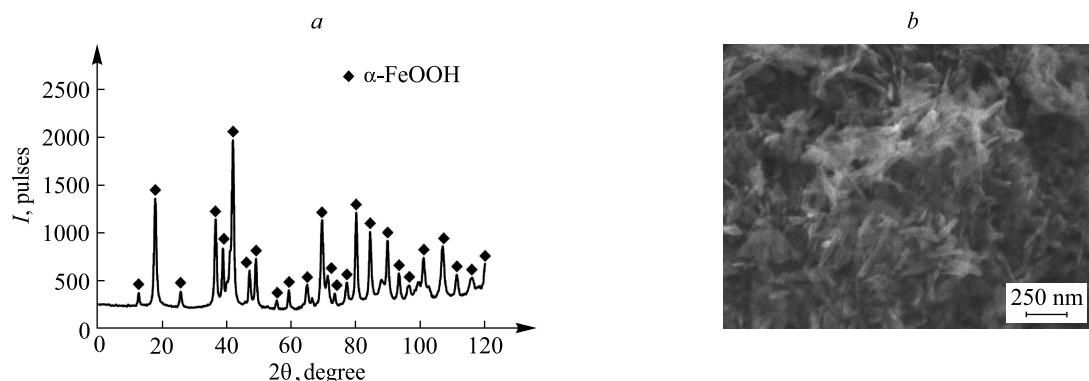


Fig. 1. XRD pattern (a) and SEM image (b) of the sample of α -FeOOH NP

Applying formula (1) to the TG data obtained makes it possible to calculate the values of α at different reduction temperatures and to plot the α values as function of T in the temperature range for each stage of the process of hydrogen reduction of α -FeOOH NP (fig. 3).

Using the least squares method, one can find the best matching mathematical functions $\alpha(T)$ to describe the curves shown in fig. 3. The derivative $\frac{d\alpha}{dT}$ is an important parameter for non-isothermal kinetics (equation (2)).

The results of calculations performed to obtain the parameters of non-isothermal kinetics of FeOOH reduction with hydrogen are shown in the table below.

**Determination of the parameters of the kinetic equation (2)
for the hydrogen reduction α -FeOOH NP**

Stage	$T, ^\circ\text{C}$	$\frac{d\alpha}{dT} 10^3$	$\ln\left(b \frac{d\alpha}{dT}\right)$	$\ln(1-\alpha)$	$\frac{10^3}{T}, K^{-1}$	$x \cdot 10^3$	y
<i>Function $\alpha(T): y = 0.0191 \exp(0.0108x)$</i>							
2	180	27.49	-5.22	-0.16	2.21	4.99	-12.42
	195	32.33	-5.05	-0.18	2.14	3.06	-8.48
	210	38.01	-4.86	-0.20	2.07	1.64	-5.25
	225	44.70	-4.66	-0.24	2.01	0.88	-3.43
	240	52.56	-4.44	-0.30	1.95	0.90	-3.67
	265	68.85	-4.06	-0.40	1.86	0.52	-2.66
	280	80.95	-3.80	-0.50	1.81	—	—
<i>Function $\alpha(T): y = 0.2042 \exp(0.0024x)$</i>							
3	300	19.39	-7.49	-0.55	1.75	4.84	-4.91
	315	20.10	-7.44	-0.56	1.70	1.61	-2.37
	330	20.83	-7.38	-0.58	1.66	1.17	-2.04
	340	21.34	-7.36	-0.61	1.63	1.12	-2.03
	355	22.12	-7.26	-0.64	1.59	1.48	-2.44
	375	23.21	-7.18	-0.67	1.54	—	—
<i>Function $\alpha(T): y = 0.0682 \exp(0.0051x)$</i>							
4	375	94.76	-5.78	-0.67	1.54	0.77	-2.71
	400	107.64	-5.57	-0.75	1.49	0.39	-1.95
	425	122.28	-5.31	-0.88	1.43	0.22	-1.57
	450	138.91	-4.96	-1.11	1.38	0.12	-1.34
	475	157.80	-4.46	-1.48	1.34	0.06	-1.17
	500	179.26	-3.60	-2.21	1.29	0.02	-1.06
	520	198.51	-1.71	-4.01	1.26	—	—

To determine the activation energy E_a for the reduction process under non-isothermal conditions, the data from the table were used to plot y against x according to equations (3) for each stage. The respective graphs are presented in fig. 4.

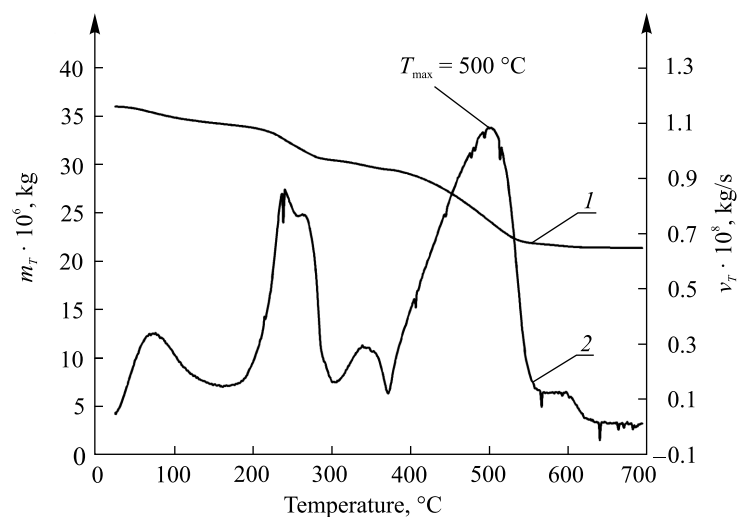


Fig. 2. TG curves of hydrogen reduction of α -FeOOH NP:
1 – mass change; 2 – the rate of mass change

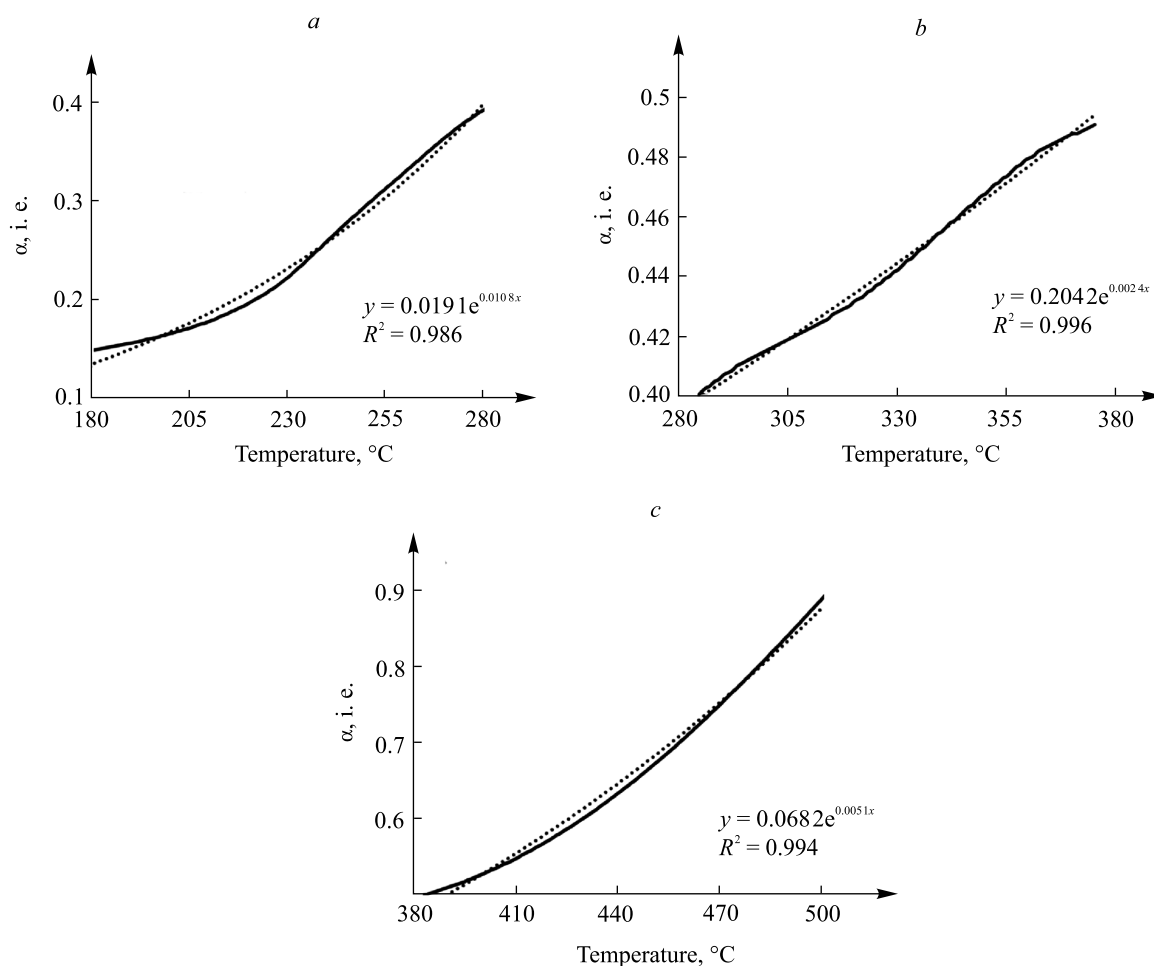


Fig. 3. Degree of conversion as function of temperature
for the hydrogen reduction of FeOOH NP
in different stages of the process:
a – stage 2; b – stage 3; c – stage 4

In accordance with equation (2), the values of E_a for each stage were found as tangent of the straight line passing through the experimental points, giving the following results (in kJ/mol): $E_{a2} = 18.2$; $E_{a3} = 6.4$; $E_{a4} = 18.1$. The total value of E_a for the hydrogen reduction of FeOOH NP is the sum of all activation energy values for each stage: $E_a = E_{a2} + E_{a3} + E_{a4}$ ($E_a \sim 43$ kJ/mol).

Confronting the calculated E_a values with the limiting values in the literature [16] corroborates the statement that the reduction of FeOOH NP proceeds in a mixed reaction mode. In this case, the kinetics of the overall reduction process of FeOOH NP is limited by both the kinetics of the chemical reaction and the kinetics of diffusion. Hence an expedient way to accelerate the process is to increase the temperature or eliminate the diffusion layer around the reacting substance by intensive stirring.

As noted above, the temperature to obtain maximum rate of the reduction process of FeOOH NP to yield Fe NP was 500 °C. However, carrying out the reduction at this temperature may result in intense aggregation and sintering of the metallic iron particles being formed, which degrades the quality of the final product.

The following task of the work was investigation of the properties of Fe NP prepared at the temperature 500 °C, when maximum reaction rate of the hydrogen reduction was reached. The XRD pattern and SEM image of a Fe NP sample prepared under these conditions are shown in fig. 5 (the exposure time was 30 min).

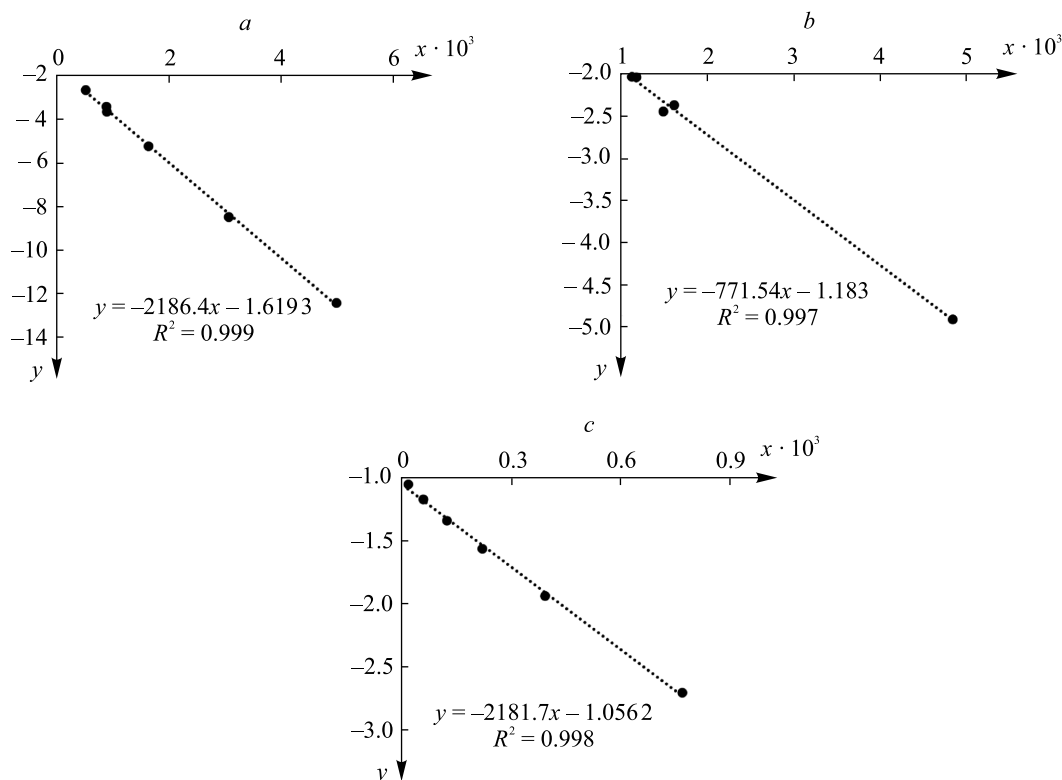


Fig. 4. Determination of the activation energy for the reduction of FeOOH NP:
a – stage 2; b – stage 3; c – stage 4

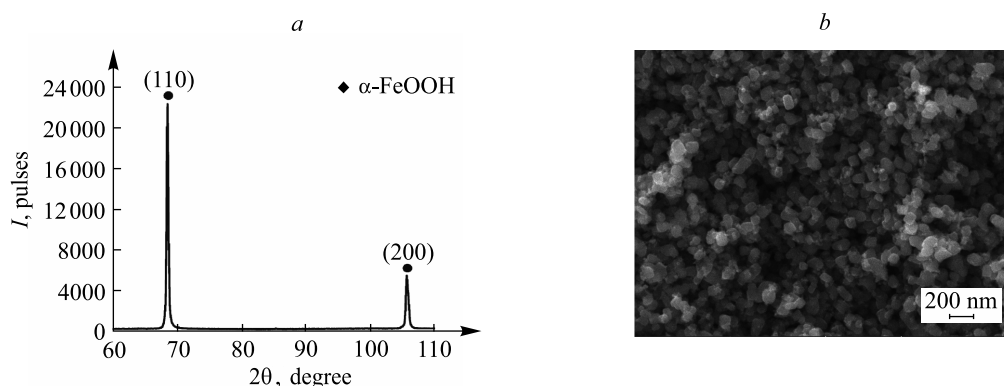


Fig. 5. XRD pattern (a) and SEM image (b) of a Fe NP sample prepared at 500 °C

The XRD (see fig. 5, *a*) demonstrates that the sample is a monophase one, consisting only of α -Fe with a body-centered cubic crystal lattice. Examination of the SEM image of the sample (see fig. 5, *b*) shows that Fe NP are mostly of a rounded shape, with sizes ranging from 70 to 100 nm. Measurements of the specific surface area for the sample gave an average value of 9.6 m²/g, which is in good agreement with the result obtained from micrographs, where the average size of Fe NP was evaluated to be 79 nm.

Thus, based on the analysis of kinetic data and investigation of properties of the reaction product, it may be concluded that the reduction of α -FeOOH NP at 500 °C both accelerates the process and ensures obtaining the desired properties of Fe NP.

Conclusions

The optimal temperature for the preparation of Fe NP by hydrogen reduction of α -FeOOH NP was found to be 500 °C. Calculations of the activation energies of the preparation process in its various stages were performed. Based on these data, an expedient way to carry out this procedure to obtain Fe NP with the desired properties is proposed: increasing the temperature and elimination of the diffusion layer around the reacting substance under intensive stirring.

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