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ХАРАКТЕРИСТИКИ ПРОЦЕССОВ СИНТЕЗА НАНОПОРОШКОВ НА ОСНОВЕ КОБАЛЬТА ХИМИКО-МЕТАЛЛУРГИЧЕСКИМ МЕТОДОМ

Изучены характеристики процессов синтеза нанопорошков на основе кобальта ($Co(OH)_2$, Co_3O_4, Co) химико-металлургическим методом. Нанопорошок $Co(OH)_2$ получали методом химического осаждения из водных растворов нитрата кобальта $Co(NO_3)_2$ (10 мас. %) и щелочи NaOH (10 мас. %) в условиях непрерывного перемешивания, контроля температуры T = 25°C и водородного показателя pH = 9. Промывали синтезированный осадок $Co(OH)_2$ дистиллированной водой с помощью воронки Бюхнера до полной отмывки ионов растворенной соли с водородным показателем над осадком pH = 7. Нанопорошки Co_3O_4 и Co получены термическим разложением и водородным восстановлением гидроксида $Co(OH)_2$ в трубчатой печи СНОЛ 0,2/1250. Исследование кристаллической структуры и фазового состава образцов порошков выполняли методом рентгенофазового анализа. Удельную поверхность S образцов измеряли методом БЭТ по низкотемпературной адсорбции азота. Средний размер частиц D рассчитали по данным измерения величины удельной поверхности. Размерные характеристики и морфологию частиц порошков изучали методом сканирующей электронной микроскопии. Установлено, что оптимальные температуры для проведения процессов термического разложения и водородного восстановления равны 180 и 280 °C соответственно, время выдержки процессов примерно в течение 2 ч. Полученные наночастицы $Co(OH)_2$ и Co_3O_4 в основном обладают игольчатой формой, размером до десятков нанометров и длиной до 200–300 нм. Наночастицы Co главным образом имеют сферическую форму, размер – также до десятков нанометров, они находятся в спеченном состоянии, каждая из них соединена с несколькими соседними частицами перешейками.

Ключевые слова: нанопорошок, наночастицы, кобальт, оксид кобальта (II, III), гидроксид кобальта, химико-металлургический метод, химическое осаждение, термическое разложение, водородное восстановление, удельная поверхность.

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CHARACTERISTICS OF SYNTHESIZING PROCESSES OF COBALT-BASED NANOPOWDERS BY CHEMICAL-METALLURGY METHOD

In this work, the characteristics of synthesizing processes of cobalt-based nanopowders ($Co(OH)_2$, Co_3O_4, Co) by chemical-metallurgy method were studied. $Co(OH)_2$ nanopowder was synthesized by chemical precipitation from aqueous solutions of cobalt nitrate $Co(NO_3)_2$ (10 wt. %) and alkali NaOH (10 wt. %) under conditions of continuous stirring, temperature control T=25°C and pH=9. The synthesized $Co(OH)_2$ precipitate was washed with distilled water using a Buchner funnel until the dissolved salt ions with a pH value were completely washed out over the precipitate was 7. Co_3O_4 and Co nanopowders were obtained by thermal decomposition and hydrogen reduction of $Co(OH)_2$ hydroxide, respectively, in a tubular furnace “SNOL 0.2/1250”. The study of the crystal structure and phase composition of the powder samples was carried out by the method of XRD phase analysis. The specific surface area of the powder samples was determined using the BET method with adsorption of nitrogen in low temperature. The average particle size D was calculated from the measurement of the specific surface area. The size characteristics and morphology of the powder particles were studied by scanning electron microscopy. It has been established that the optimal temperatures for thermal decomposition and reduction processes are 180 and 280°C respectively, the holding time of the processes is about two hours. The obtained nanoparticles $Co(OH)_2$ and Co_3O_4 mainly have an acicular shape, with a diameter up to a few tens of nm and the length up to 200-300 nm. Co nanoparticles mainly have a spherical shape, the size of which is also up until several tens of nm. They are in a sintered state, each of them is connected to several neighboring particles by isthmuses.

Keywords: nanopowder, nanoparticles, cobalt, cobalt (II, III) oxide, cobalt hydroxide, chemical-metallurgy method, chemical precipitation, thermal decomposition, hydrogen reduction, specific surface area.

Introduction

At present, materials based on cobalt, in particular, nanomaterials, have found increasingly widespread practical applications in various fields of science, technology and industry. About 80 % of Co is consumed to obtain superhard, high-temperature, tool and wear-resistant alloys, which are used in mechanical engineering, especially in aviation and space technology, rocketry, electrical and nuclear industries [1-15]. Furthermore, Cobalt (II, III) oxide Co_3O_4 has found application as a catalyst in organic synthesis; as part of electrodes for organizing the electrolysis process in industrial plants and batteries; additive in the production of heat-resistant alloys; production of varnishes, paints, building enamels; adsorbent for hydrocyanic acid for filtering installations; in glass and ceramic production, etc. [16-18]. Of particular scientific and practical interest is the production and application of Co-based nanopowders (NP) with desired properties, which primarily are determined by the morphology and dispersion of particles [9, 10]. The production of NP ($Co(OH)_2$, Co_3O_4 and Co) is carried out in various ways, most of which are characterized by a number of disadvantages, such as reduced productivity, high energy consumption [19].

However, the chemical-metallurgy method, which includes the stages of chemical precipitation of oxygen-containing metal compounds, followed by thermal decomposition and hydrogen reduction, is a highly efficient method for saving energy, enhancing the possibility of utilizing industrial waste as raw materials and the ability to regulate the dimensional characteristics of metal nanoparticles (NPs) in the course of their receipt [20].

A big disadvantage of the chemical-metallurgy method for producing NP is low productivity due to a low rate of thermal decomposition and hydrogen reduction processes under low temperatures. During the processes, an excessive increase in the temperature is not recommended, because this leads to an intensive occurrence of aggregation and sintering of NPs as well as to the formation of particles with a size outside the nanometer range [21].

Therefore, the study of the physical characteristics of the processes for synthesizing of NP based on cobalt *via* chemical-metallurgy method to establish the optimal time-temperature parameters while guaranteeing the necessary properties of the products obtained is a scientific and practical task.

The aim of this work is to determine the main parameters of the processes of NP synthesis ($Co(OH)_2$, Co_3O_4 and Co) by chemical-metallurgy method to establish the optimal modes of the processes, as well as to study the properties of the products obtained.

Materials and methods of research

$Co(OH)_2$ hydroxide was obtained by chemical precipitation from aqueous solutions of cobalt nitrate $Co(NO_3)_2$ (10 wt. %) and alkali NaOH (10 wt. %) under conditions of continuous stirring, temperature control T = 25 °C and acidity pH = 9. The reaction of precipitation of $Co(OH)_2$ hydroxide is represented as follows:

$$Co(NO_3)_2 + 2NaOH = Co(OH)_2\downarrow + 2NaNO_3. \quad (1)$$

The acidity of the pH of the solution mixture was recorded using a pH meter with an accuracy of ±0.03. The synthesized $Co(OH)_2$ precipitate was washed with distilled water using a Buchner funnel. Complete purification of the precipitate from nitrate salt ions was achieved when the pH value of the precipitate was 7. After that, the resulting precipitate was dried at 40 °C for two days.

The dried $Co(OH)_2$ was ground in a "Fritsch Pulverisette 2" mortar mill, the obtained $Co(OH)_2$ NP was used for further research.

To select the temperature conditions for carrying out the processes of thermal decomposition and hydrogen reduction, the Co $(OH)_2$ NP was investigated by the thermogravimetric (TG) method on a "SDT Q600" device (USA) with a linear heating mode at a rate of 5 °C/min in the temperature range 25-700 °C in air atmosphere (for thermal decomposition). The reduction process is carried out in the temperature range 25-500 °C and in a hydrogen atmosphere.

The phase composition and structure of the samples were investigated by XRD phase analysis on a "Difrey 401" diffractometer (Russia).

The specific surface area (S) of the samples was determined by the BET method with adsorption of nitrogen in low temperature, on a "NOVA 1200e" analyzer (USA). The average particle size of powders D (m) was calculated by the formula:

$$D = \frac{6}{\rho S}, \quad (2)$$

where ρ is the density of the material, kg/m^3.

The shape and size of NPs of the samples were observed by a scanning electron microscope (SEM) TESCAN VEGA 3B (Czech Republic).

Results and discussion

The XRD and SEM results for the initial sample of $Co(OH)_2$ NP are shown in Fig. 1.

The result of XRD analysis (See Fig. 1, *a*) revealed that the sample under study is single-phase, containing only the hydroxide phase $Co(OH)_2$ and has a crystal structure. Analysis of the SEM image (See

Fig. 1, *b*) showed that $Co(OH)_2$ NPs are mainly acicular, with a length up to 300 nm, and they tend to form large aggregates of a spherical shape. The specific surface area of $Co(OH)_2$ NP is 31.7 m^2/g; accordingly, the average NPs size *D* is 53 nm.

In Fig. 2 shows the TG curves obtained in the course of thermal decomposition and hydrogen reduction of $Co(OH)_2$ NP.

According to the TG studies, it can be seen that the process of thermal decomposition of a $Co(OH)_2$ NP sample consists of three stages (See Fig. 2, *a*). In accordance with the calculations, in the first stage, the adsorbed water is removed in the temperature range 25-150 °C, the second and third stages are associated with the elimination of structural water by reaction (3).

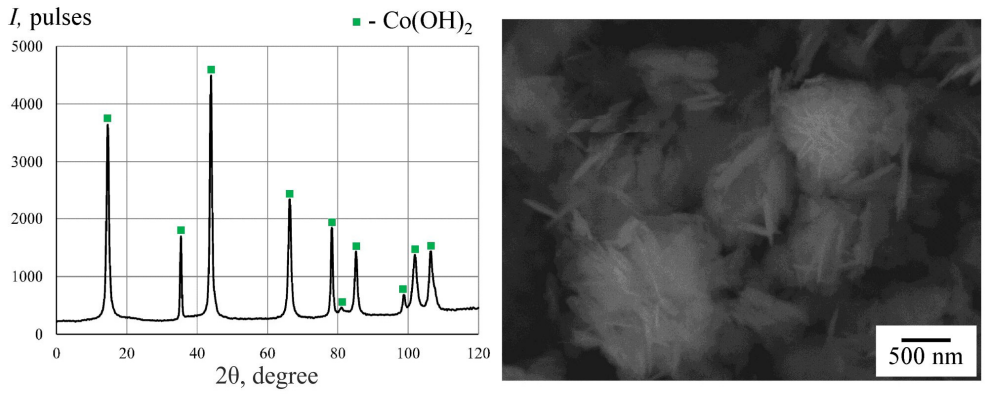
The presence of two peaks (I, III) of the elimination of structural water, which are recorded on the TG curve (See Fig. 2, *a*), can be explained by the fact that in all cases the particles have closed pores. As the temperature rises, pores are opened, and structural water is removed from them. In the temperature range 150-200 °C, the process of thermal decomposition takes place with a maximum specific rate of $11.5 \cdot 10^{-8}$ kg/s at 180 °C.

$$6Co(OH)_2 + O_2 \rightarrow 2Co_3O_4 + 6H_2O. \quad (3)$$

The TG curves of the hydrogen reduction process for the $Co(OH)_2$ NP sample (See Fig. 2, *b*) shows that the process proceeds in two stages. At the first stage, in the temperature range 160-265 °C, the process of removing structural water takes place and in the second stage, with the temperature ranging from 265 to 310 °C, the process of metallization to Co proceeds.

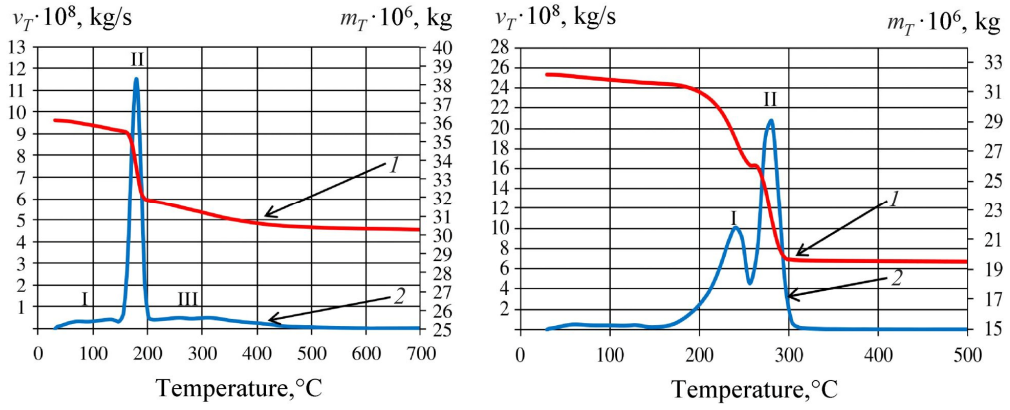
While the reaction (4) takes place, the maximum specific rate is achieved at a temperature 280 °C with a value of $20.6 \cdot 10^{-8}$ kg/s.

$$Co(OH)_2 + H_2 \rightarrow Co + 2H_2O. \quad (4)$$



a *b*

Fig. 1. XRD pattern (*a*) and SEM image (*b*) of the initial sample of $Co(OH)_2$ NP



a *b*

Fig. 2. TG curves of thermal decomposition (*a*) and hydrogen reduction (*b*) of $Co(OH)_2$ NP:
1 – mass change, *2* – rate of mass change

Analysis of the TG data makes it possible to choose the optimal temperatures which correspond to the values of the maximum specific rate in both thermal decomposition and hydrogen reduction of $Co(OH)_2$ NP. These temperatures are 180 and 280 °C for the synthesis of Co_3O_4 and Co NP, respectively. The processes of thermal decomposition and hydrogen reduction of α-FeOOH NP at optimal temperatures 180 and 280 °C, respectively were carried out in a tube furnace "SNOL 0.2/1250", the holding time was 2 hours. In this case, the chemical reactions were indicated by the formulas (3) and (4).

In Fig. 3 presents the results of XRD for the products of thermal decomposition and reduction of $Co(OH)_2$ NP.

The XRD analysis of the thermal decomposition product (See Fig. 3, *a*) shows that the sample contains only the crystalline Co_3O_4 phase, no other phases were found; the resulting product is NP of pure Co_3O_4; thermal decomposition of $Co(OH)_2$ NP at 180 °C was complete after 2 hours of exposure.

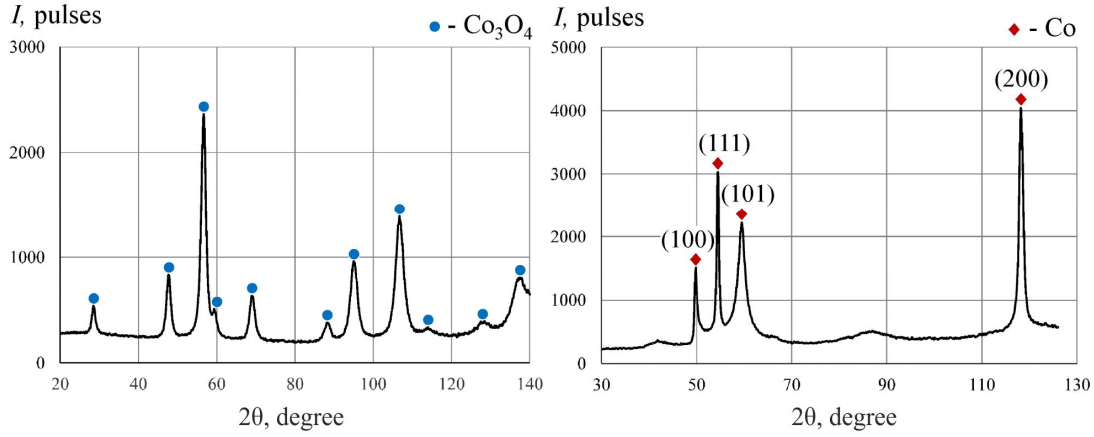
The result of the XRD analysis of the reduction product (See Fig. 3, *b*) reveals that the sample under study is single-phase, consisting only of Co NP particles with an HCP crystal lattice; the exposure time for the hydrogen reduction process was also 2 hours.

In Fig. 4 presents the results of SEM images of the obtained products of thermal decomposition and reduction of $Co(OH)_2$ NPs.

Fig. 4, *a*, it can be seen that Co_3O_4 NPs mainly consist of elongated ovoid and acicular aggregates with dimensions of tens of nm in diameter and up to 200 nm in length.

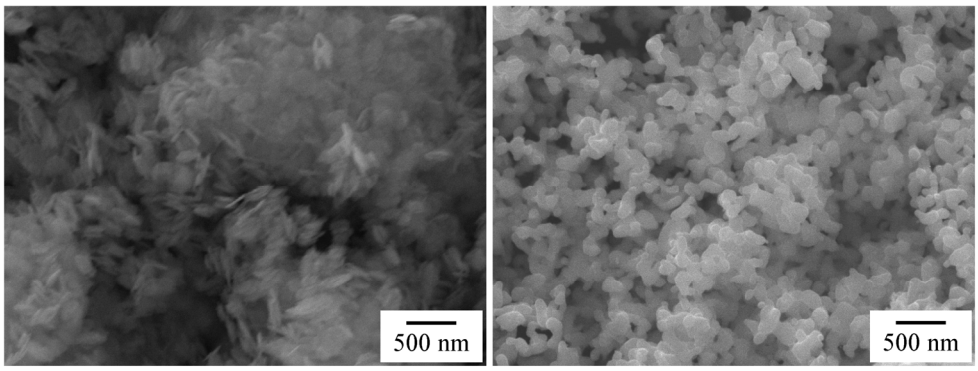
Analysis of the SEM image of the sample of the reduction product (See Fig. 4, *b*) shows that Co NPs are mainly spherical in shape with a nanometer size (on the order of tens of nm). In this case, the obtained Co NPs are in a sintered state; each of them is connected to several neighboring particles by isthmuses.

The result of measuring *S* and calculating the average particle size *D* of the obtained powder samples are given in the Table.



a *b*

Fig. 3. XRD pattern of the thermal decomposition (*a*) and hydrogen reduction (*b*) products of $Co(OH)_2$ NP



a *b*

Fig. 4. SEM images of Co_3O_4 (*a*) and Co (*b*) NP

The result of measuring *S* and *D* of particles of powder samples

No.	Nanopowder samples	Method for obtaining	*S*, m^2/g	*D*, nm
1	$Co(OH)_2$	Chemical precipitation	31.7	53
2	Co_3O_4	Thermal decomposition at 180 °C	28.2	47
3	Co	Hydrogen reduction at 280 °C	11.8	58

The result of measuring the specific surface area confirms that the processes of sintering and aggregation of the formed NPs during thermal decomposition and hydrogen reduction lead to a decrease in the specific surface area of the obtained products (Co_3O_4 and Co NP) compared to the initial sample of $Co(OH)_2$ NP. The calculation of the average particle size of powders *D* according to the *S* data by formula (2) shows that all the NP obtained have a nanometer size (less than one hundred nm), the results of this are in good agreement with the result of the study of the size of the samples ($Co(OH)_2$, Co_3O_4 and Co NP) by the SEM method.

Conclusion

The characteristics of synthesizing processes of cobalt-based nanopowders by chemical-metallurgy method were studied and series of nanopowders ($Co(OH)_2$, Co_3O_4 and Co) were obtained. Based on the analysis of TG data, it was found that the optimal temperatures for carrying out the processes of thermal decomposition and reduction are 180 and 280 °C, respectively. The products obtained represent NP of pure Co_3O_4 and Co upon reduction after 2 hours of exposure.

The size and shape of the starting material and the samples obtained are investigated. It has been shown that $Co(OH)_2$ NPs have an acicular shape, the length of which is up to 300 nm, and they tend to form large aggregates of a spherical shape. Co_3O_4 NPs mainly consist of elongated ovoid and acicular aggregates with a size of about a few tens of nm in diameter and up to 200 nm in length. Co NPs are mainly spherical with a nanometer size (on the order of tens of nm), they are in a sintered state, each of them is connected with several neighboring particles by isthmuses.

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