

Synthesis of nickel nanoparticles with magnetic properties using the carbothermy method

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Abstract

The article discusses a method for producing nickel-based nano-microparticles with magnetic properties using the carbothermic reduction of precursors - nickel hydroxides. The possibility of a fairly simple and labor-intensive method for producing nickel (II) hydroxide by contact precipitation from a nickel-containing electrolyte on the surface of magnesium particles has been demonstrated. The method is based on the formation of local corrosive microgalvanic cells with predominant hydrogen depolarization. Both precursors and magnetic nickel powders were analyzed by SEM microscopy, X-ray diffraction and X-ray fluorescence analyses. Using the SEM microscopy method, it was found that, depending on the surface structure of the reducing agent particles, both lamellar structures of α/β -Ni(OH)₂ and three-dimensional (3D) micro-nanostructures flower-like of β -Ni(OH)₂ with varying degrees of crystallinity can be obtained. Thermal reduction annealing of α/β -Ni(OH)₂ using the carbothermic method makes it possible to obtain a non-pyrophoric highly dispersed magnetic nickel-containing powder in a composite with microparticles of porous carbon, stabilized by the kotoite phase.

Keywords: synthesis by contact precipitation, carbothermic reduction, nickel hydroxides, magnetic nickel-based nano-microparticles

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1. Introduction

Recently, interest in Ni-based nanomaterials that are stable in an oxidizing atmosphere has increased significantly, which is associated with the possibility of their use in modern technologies. For example, nickel flake micropowders can be used in coatings, catalysis, magnetic storage materials, electromagnetic shielding, and electromagnetic absorption. Wear-resistant, corrosion-resistant, precision powder nickel-based alloys, shape memory alloys and hydrogen storage alloys used in the aerospace, automotive, electronics and shipbuilding industries.

Nickel oxide, oxohydroxide and hydroxide nanoparticles demonstrate enhanced chemical stability, electro- and photocatalytic properties, magnetic properties, superconductivity and electron transfer ability, making them indispensable in nuclear energy, renewable energy technologies and catalysis. The photocatalytic properties of NiO nanoparticles found application in technologies for water purification from organic pollutants, in particular dyes [1, 2 3]. In addition, was found, that the nickel-based oxide nanoparticles exhibit antimicrobial activity against multidrug-resistant pathogenic bacteria. It finds application in the pharmaceutical industry [4]. Ni hydroxides have also attracted much attention as an electrode material in energy and power storage devices, especially supercapacitors, due to their unique physical and chemical properties such as high theoretical surface area and well-defined electrochemical redox behavior [5].

Nickel hydroxide has been used as an electrode material in battery technologies since the first half of the 20th century. In the late 1960s, a simple scheme was proposed to explain the electrochemical oxidation of α - and β -Ni(OH)₂ to nickel(III) oxohydroxides (β - and γ -NiOOH), followed by reduction back to nickel(II) hydroxide. Complex morphological forms of nickel-based particles, such as nanoflowers, nanoribbons, and nanoflakes also are one of the key elements in modern nanotechnology [6]. Three-dimensional (3D) Ni(OH)₂ nanostructures have received considerable attention due to their large surface area and unique properties, which enable their applications in catalysis and energy storage [5].

Currently, there are many known methods for the synthesis of materials based on nickel, its oxides and hydroxides, including new methods such as microwave synthesis and sonochemistry. Among them, chemical precipitation methods are of particular interest. For example, nickel oxoforms are obtained from aqueous solutions of nickel salts by reduction with or without the addition of alcohol or oil, micellar or emulsion route [7], using plant extracts (*Averrhoa carambola L.juice*) as a reducing agent. Nano-sized particles of high purity magnetic nickel were obtained by chemical reduction of nickel(II) chloride at a temperature of 80 °C using hydrazine hydrate as a reducing agent in aqueous solutions containing ethylene glycol and water with the addition of polyvinylpyrrolidone (PVP) as an anti-agglomeration

agent [8, 9]. Hypophosphites also used as a reducing agent in alkaline aqueous-alcoholic environment [10].

Depending on the environmental conditions (oxidizing; air or oxygen), neutral (nitrogen, helium) or restorative (hydrogen), the thermal decomposition of nickel oxalates under mild thermal conditions with the formation of CO₂/CO gas mixtures makes it possible to obtain either pure metal or Ni oxide. This method is preferred because of the possibility of obtaining a controlled structure of Ni-based particles. Chemical vapor precipitation (CVD) and electroprecipitation methods [11], hydrothermal synthesis, selective leaching from solid nickel-containing alloys Al-20Ni in 20 % NaOH solution also offered [9, 12, 13].

Thus, most methods for producing nano- and microparticles of nickel, its oxides and hydroxides based on chemical transformations, use a variety of reducing agents, and the reduction process itself can occur both in an aqueous environment and with the use of organic substances, which are often toxic to the environment. For the most part, these methods are energy-intensive, time-consuming and multi-stage, requiring complex equipment. Therefore, the development of a non-labor-intensive green process for the synthesis of nickel-containing particles, including those with magnetic properties, still remains one of the priority tasks.

2. Experiments and methods

2.1. Materials

The electrolyte was prepared by mixing the salts nickel(II) sulfate heptahydrate and nickel(II) chloride heptahydrate with boric acid, after which the resulting mixture was dissolved in deionized water. The composition of the aqueous electrolyte solution is presented in Table 1.

Table 1. Quantitative electrolyte composition.

Electrolyte composition	Molar mass, g/mol	Sample weight, g	Electrolyte volume, ml	Molar concentration of salt in solution, mol/l	Recovery process role
NiSO ₄ ·7H ₂ O	281	45	250	0,640	Precipitation of nickel(II) hydroxide
NiCl ₂ ·7H ₂ O	256	6		0,094	
H ₃ BO ₃	62	7.5		0,484	Buffer additive

Metallic magnesium in the form of MP-1 powder and shavings was used as a reducing agent; the magnesium content was 99.7%.

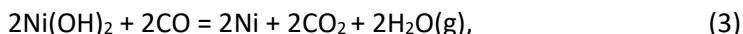
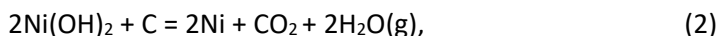
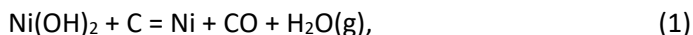
All chemical reagents used in the work were analytically pure and were not subjected to additional purification.

2.2. Synthesis of micro-nano nickel-content powders

The synthesis of magnetic nickel powder was carried out in two stages. At the first stage, nickel(II) hydroxide was precipitated from an aqueous solution of nickel-containing salts by contact precipitation on the surface of magnesium particles. The formation of nickel hydroxide proceeded differently depending on the surface morphology of magnesium particles —magnesium powder and magnesium shavings. In the first case, the solution temperature did not rise above 25 °C. When using magnesium shavings, the temperature spontaneously increased during the first 10 minutes of synthesis and remained within 45-48 °C during the entire process. In addition, different intensities of the release of gaseous substances were observed: a significant release of hydrogen was recorded when using magnesium shavings, while in the presence of magnesium powder the process was uniform, smooth and slow. As a result, after 24 hours, all Ni^{2+} was precipitated from the electrolytes in the form of nickel(II) hydroxide. Next, the precipitated nickel(II) hydroxide powders were washed with hot distilled water on a filter to wash away associated ions, filtered and dried at a temperature of 90 °C.

At the second stage, the carbothermic method was used to obtain magnetic nickel powders. The choice of this method was based on well-known studies showing that metal oxides can be reduced to pure metal in combustion mode. In this case, the combustion temperatures are lower than the melting temperatures of the corresponding metals, as a result of which the metal is obtained in powder form. In the case of nickel reduction from nickel oxide or hydroxide, for example, with polystyrene, combustion temperatures are quite high and amount to 900-1000 °C [14]. A similar process of thermal reduction, where hexamethylenetetramine (urotropin) was used as a reducing agent, is described in the work [15].

In this work, it was proposed to use only carbon as a reducing agent. According to the thermodynamic characteristics of all processes occurring during thermal burning of a mixture of nickel hydroxides with carbon (Fig. 1), at temperatures above 900 °C several parallel processes are possible – reduction of nickel hydroxide with coal and/or carbon(II) oxide, according to reaction equations 2 and 3:



as well as the combustion of carbon to form CO and the oxidation of CO to CO_2 . To increase the formation of CO, the process was carried out in a closed chamber of a SNOL 40/1100 muffle furnace under conditions of lack of oxygen. In addition, when preparing the carbon mixture, the mass of carbon was calculated so that its amount (mol) exceeded the amount of Ni(OH)_2 by 2-3 times.

Dried samples of $\text{Ni}(\text{OH})_2$ were mixed with carbon powder; the resulting mixture was transferred to a crucible. A layer of carbon was first poured onto the bottom of the crucible, followed by a layer of the mixture, and on top of the mixture, it was covered with a layer of carbon crumbs 2 cm thick. The crucible was tightly closed with a lid and placed in a muffle furnace. The temperature was raised at a rate of 10 degrees per minute.

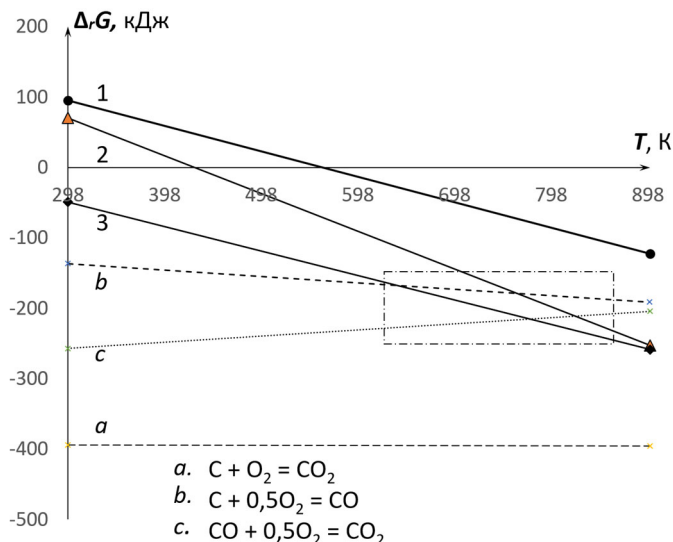


Fig. 1. Thermodynamic characteristics of the processes occurring during the reduction of nickel(II) hydroxide using the carbothermic method.

2.3. Equipment

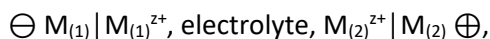
The diffractograms of nickel hydroxide and nickel magnetic powder (XRD) were obtained using an X'Pert Pro diffractometer in $\text{Cu K}\alpha$ radiation mode ($\lambda = 0.15406$ nm; 60 kV, 55 mA). Diffraction angle values were scanned from 5 to 120 (with a 2 θ step of 0.001). The surface morphology of purified saponite powders was characterized by scanning electron microscopy. SEM images were obtained using a Zeiss Evo-10 microscope (Carl Zeiss Microscopy, USA) operating at 20.0 kV.

The elemental composition of nickel hydroxide and nickel magnetic powder was determined using a precision analyzer "EXPERT 3L" (model U168), which is designed for direct operational non-destructive measurement of the mass fraction (%) of chemical elements in samples, as well as by the method of non-destructive energy dispersive X-ray fluorescence analysis (ED XRF – Energy Dispersive X-Ray Fluorescence) on the SEP-01 "Elvax-mini" X-ray spectrometer.

3. Results and discussion

According to the general scheme of the process of synthesis of nickel magnetic powder, at the first stage $\text{Ni}(\text{OH})_2$ was obtained, for which we used the method of contact precipitation at the interface of an aqueous solution of nickel salts – metallic magnesium. The nucleation and growth of nickel (II) hydroxide particles in an aqueous solution were carried out during nickel-hydrogen electrochemical competition for the process of removing nickel from an aqueous solution of its salt under the influence of magnesium.

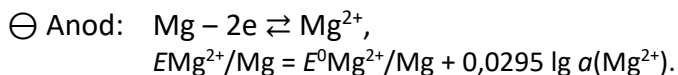
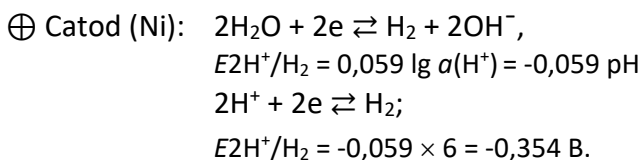
At the initial moment, upon contact of magnesium with the electrolyte, the cementation of nickel begins, which is precipitated on the surface of magnesium particles and forms local short-circuited microgalvanic elements at the phase boundary:



where the cathode is the precipitated less active metal ($\text{M}_{(2)}$ – nickel), and the anode is the more active metal ($\text{M}_{(1)}$ – magnesium). However, these processes are accompanied by a concomitant intense eduction of hydrogen, since nickel belongs to the group of metal elements for which electroreduction always occurs with a large overvoltage. During the immersion reduction of nickel with magnesium, the role of the main and accompanying processes changes places - the cathodic eduction of hydrogen becomes the main one under the conditions of the formation of a microgalvanic couple



which corresponds to a corrosion element that occurs in localized areas of the surface. This becomes possible when the hydrogen discharge potential is greater than the anodic destruction potential of magnesium:

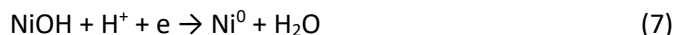
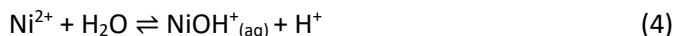


Indeed, in solutions of salts with $\text{pH} = 6$, the equilibrium potential of the hydrogen electrode is $E_{\text{H}^+/\text{H}_2} = -0.354 \text{ V}$ and the potential value is significantly greater than the standard potential of the magnesium electrode $E^0_{\text{Mg}^{2+}/\text{Mg}} = -2.372 \text{ V}$. Magnesium in local areas of the surface undergoes oxidation with the formation of

magnesium ions. The presence of chloride-ions in the electrolyte facilitates the occurrence of local electrochemical corrosion, since they can easily damage the surface coating due to their small diameter and high permeability.

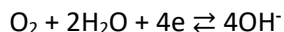
Cathode sites on the surface of magnesium are formed by thin layers of metallic nickel precipitated at the beginning of the process. The standard and equilibrium discharge potentials of Ni^{2+} ions: $E^0\text{Ni}^{2+}/\text{Ni} = -0.25 \text{ V}$ and $ENi^{2+}/\text{Ni} = -0.263 \text{ V}$, respectively, are greater than the value of the equilibrium potential of hydrogen discharge. This gives an advantage to the nickel crystallization process at the initial stage. However, due to the large overvoltage of electrocrystallization, nickel ions are practically not reduced to elemental nickel.

Crystallization of nickel on the surface of a metal reducing agent is described as a series of sequential reactions of hydrolysis of Ni^{2+} cations with the formation of nickel(II) hydroxylation (reaction 4), adsorption of the formed NiOH^+ on the surface (reaction 5) and its electric discharge with the formation of nickel(I) hydroxide (reaction 6) and then nickel (reaction 7):



The limiting stages of this overall process are, obviously, electrochemical reactions 6 and 7. Therefore, quite quickly the main process becomes cathodic reduction of hydrogen with the accompanying formation of OH^- -ions, the accumulation of which locally sharply alkalizes the surface. The formation of hydroxide anions on the cathode areas of the surface of magnesium particles is also possibly due to the fact that, according to [16] nickel precipitates on magnesium alloys mainly in the form of Laves phases MgNi_2 , which are cathodically active.

In addition, in the absence of an inert atmosphere, a local increase in pH may also be possible due to the cathodic reduction of oxygen dissolved in the electrolyte:



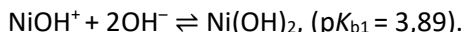
$$E(2\text{H}_2\text{O}, \text{O}_2/4\text{OH}^-) = 0,401 - 0,059 \lg a(\text{OH}^-).$$

$$E(2\text{H}_2\text{O}, \text{O}_2/4\text{OH}^-) = +0,755 \text{ B.}$$

The oxygen present in the solution can also block the progress of reaction 6 and cause re-oxidation of NiOH if it is formed.

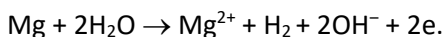
The accumulation of OH^- -ions does not lead to an increase in the pH of the electrolyte, since the formed anions interact with protons arising in hydrolysis processes, as well as with adsorbed NiOH^+ cations with the formation of nickel(II)

hydroxide according to the equation:

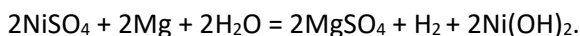


The preferential formation of nickel(II) hydroxide rather than magnesium hydroxide is due to the significantly lower dissociation constant of $\text{Ni}(\text{OH})_2$ ($\text{p}K_{\text{b}} \text{Mg}(\text{OH})_2 = 2.6$).

Anodic oxidation of magnesium leads to the formation of magnesium cations, which go into solution:



The total reaction of the process can be represented by reaction equations:



Thus, due to the formation of local corrosive galvanic elements on the surface of magnesium particles, nickel(II) hydroxide is formed instead of metallic nickel. Elemental analysis of $\text{Ni}(\text{OH})_2$ powders obtained on morphologically different magnesium particles is presented in Table. 2.

Table 2. Mass fraction of the element (%) in the precipitated $\text{Ni}(\text{OH})_2$

Morphology of magnesium particles		^{28}Ni	^8O	^{12}Mg	^{16}S	^{17}Cl
1	Magnesium powder	61.799 ± 0.382	32.051 ± 0.194	3.976 ± 0.591	3.174 ± 0.027	-
2	Magnesium shavings	46.41 ± 0.976	32.294 ± 1.341	11.187 ± 0.718	5.968 ± 0.127	4.139 ± 0.090

The color of the precipitated nickel hydroxides is different: in the first case, $\text{Ni}(\text{OH})_2$ was a dense powder of a bright light green color, and in the second, the powder was loose, pale light green. The obtained SEM images of these samples explain these differences, Fig. 2.

Particles $\text{Ni}(\text{OH})_2$ precipitated using magnesium powder are characterized by uniformity with sizes ranging from 1.235 to 2.93 μm . In terms of morphology, the particles are densely packed and homogeneous rounded plates. When magnesium shavings are added to the electrolyte, 3D flower-like nickel hydroxide are formed, which begin as spherical grains and gradually grow to form structures resembling flower petals. Such micro-nano three-dimensional (3D) structures are among the best building blocks as electrode materials in the field of supercapacitors. They have a high specific surface area and are able to provide more effective contact with the electrolyte [17]. The 3D flower-like morphology creates some porosity.

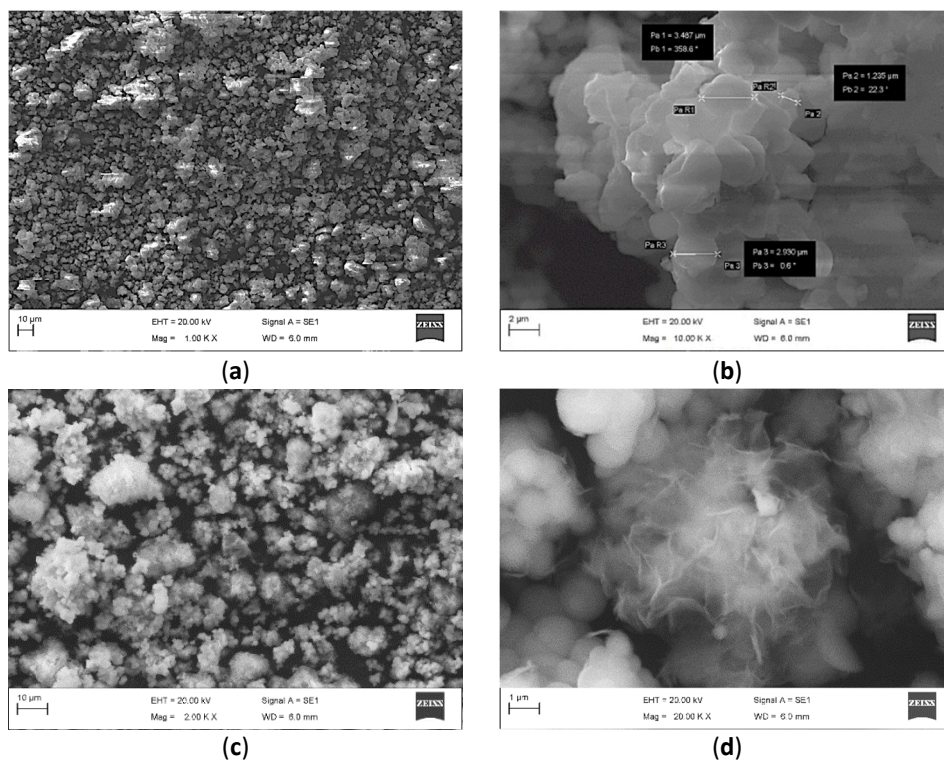


Fig. 2. SEM images of Ni(OH)_2 powders precipitated on magnesium powder (a, b) and shavings (c, d).

The established morphological differences in particles of precipitated nickel (II) hydroxide powders are obviously due to different conditions of the electrochemical process associated with the defective structure of magnesium powder particles and shavings, the effective contact area with the magnesium surface, the rate of ion discharge processes at the cathode and anodic areas of the surface, and also the growth rate of the formed crystal nucleus [19]. The surface of magnesium shavings is more cracked [20] and, possibly, at the interface between the magnesium shavings /electrolyte phases, processes occur that are accompanied by a large overvoltage of “dissolution” of magnesium, for example, as a result of the formation of a surface passivating film of magnesium hydroxide, which creates an energy barrier for charge transfer at the electrode - electrolyte interface. The external manifestation of passivation is a spontaneous solution temperature increase up to 40-45 °C and a more intense release of gaseous substances. This assumption is also confirmed by a smaller amount of nickel hydroxide and a larger mass fraction of magnesium in the samples obtained by precipitation of Ni(OH)_2 using magnesium shavings.

The specific surface area of magnesium powder particles is larger, therefore, the

active surface is larger and the corrosion process is carried out with less overvoltage. In this case, conditions are created for the uniform crystallization of Ni(OH)_2 particles, when the nucleation rates are comparable to the particle growth rate. The particles are in the form of flakes, layered to form columnar structures. Accordingly, the mass fraction of nickel in Ni(OH)_2 precipitated with magnesium powder is greater [20].

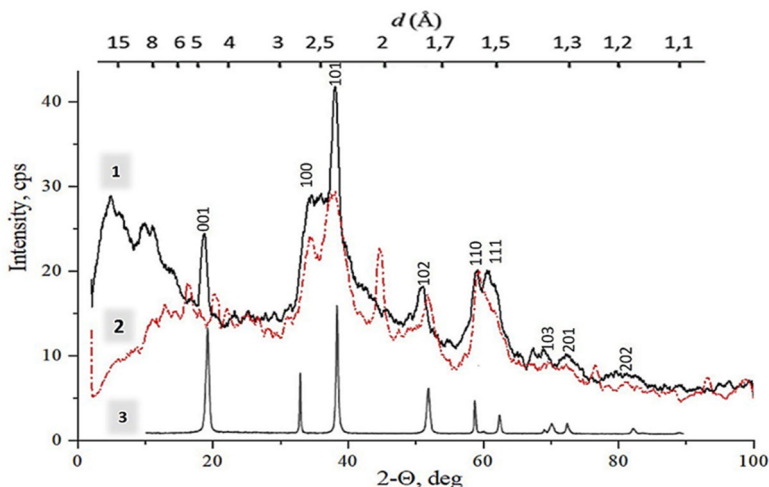


Fig. 3. X-ray diffraction patterns of nickel(II) hydroxide powders: 1) α/β -phase Ni(OH)_2 precipitated with magnesium powder; 2) β -phase Ni(OH)_2 precipitated with magnesium shavings; 3) standard $\beta\text{-Ni(OH)}_2$.

X-ray phase analysis of the obtained nickel hydroxides showed that sample No 1 (Table 2) is polymorphic and is represented by α - and β - Ni(OH)_2 with the main reflections of 2.405 Å and 2.0287 Å. Sample No 2 is $\beta\text{-Ni(OH)}_2$ with reflections of 2.364 Å and 1.512 Å, which correlates with the data of the work [21]. The broadening of the reflections in the diffraction patterns indicates a certain degree of disorder along the direction of the crystallographic c -axis, caused by defects in the crystal structure. A lower degree of crystallinity is characteristic of 3D flower-like.

The resulting Ni(OH)_2 powders were subjected to reduction firing at a temperature of 900 °C using carbon as a reducing agent, according to the above procedure. As a result, black nickel-containing powders were obtained, of which only sample No. 1 (see Table 2) with an initially higher nickel content had magnetic properties. SEM images of magnetic powder particles are shown in Fig. 4.

Morphological analysis shows that the powder contains both particles of apparently metallic nickel with a size of 0.4-2.0 μm , as well as porous particles of carbon formed under conditions of lack of oxygen at 900 °C. Considering that the entire

powder is magnetic, it can be assumed that the carbon particles are covered with a layer of solid molten carbon in the nickel phase, since carbon atoms are able to dissolve in the nickel lattice [22].

The diffraction patterns of the magnetic powder under study (Fig. 5) clearly show reflections of metallic nickel (the main reflection of 2.0299 Å) and the mineral kotoite [23]. The latter obviously crystallizes at the firing temperature, since an admixture of magnesium is detected in the powders. The kotoite structure can also be formed by nickel $\text{Ni}_3(\text{BO}_3)_2$.

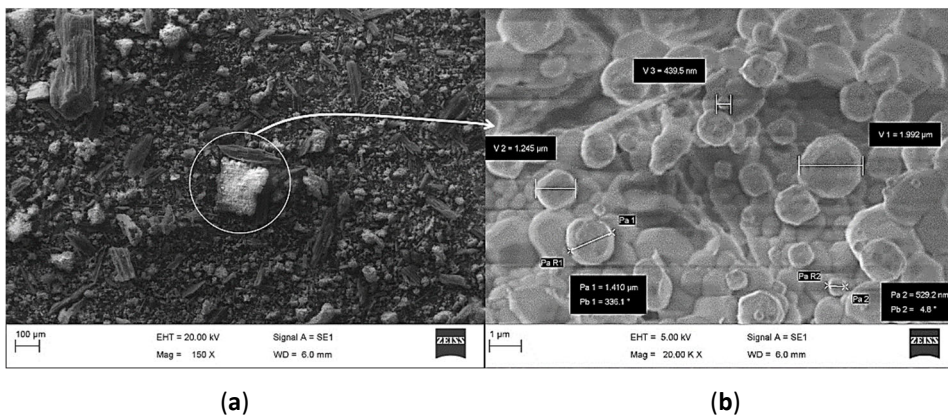


Fig. 4. SEM images of magnetic nickel-containing powder at various enlargements.

4. Conclusions

Magnetic nano-micro-sized nickel-containing powders can be obtained from a precursor - α/β -nickel (II) hydroxide under conditions of limited air access using the method of reduction roasting, where the reducing agent is carbon.

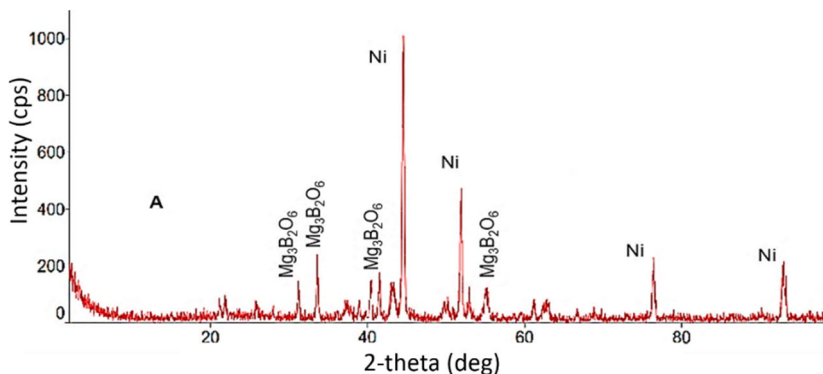


Fig. 5. X-ray diffraction patterns of Ni magnetic powder.

The synthesis of the nickel(II) hydroxide precursor can be carried out by an environmentally friendly, simple and labor-intensive method of contact precipitation from a nickel-containing electrolyte on the surface of magnesium particles. The method is based on the formation of local corrosive microgalvanic cells with predominant hydrogen depolarization. Depending on the surface structure of magnesium particles, both lamellar structures of α/β -Ni(OH)₂ and three-dimensional flower-like β -Ni(OH)₂ with varying degrees of crystallinity can be obtained.

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