

DISPERSED IRON OBTAINING BY THE METHOD OF SOLID STATE HYDRIDE SYNTHESIS AND THE PROBLEM OF HYDROPHOBICITY OF METAL

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ABSTRACT

The process of obtaining of a dispersed product based on metallic iron, containing a protective carboxiloxane film on the surface, has been investigated and implemented. The structure and composition of the metal product are characterized by modern physical methods, including X-ray phase and X-ray fluorescence analysis, XPS spectroscopy. The influence of the synthesis mechanism, the role of the metal-silicon chemical interaction in the surface layer on the water-repellent and anticorrosion properties of the obtained samples is considered. It was found that the heat resistance of the samples in air and their hydrophobicity, depending on the type of the reduced metal, increase in the order: nickel - copper - iron. It was shown that highly hydrophobic iron-based samples can be obtained by reduction from iron (II) oxide and Olenegorsk superconcentrate. The prospects of the synthesized powders and the method of their preparation for the creation of superhydrophobic materials are shown. A method for obtaining a metal product has been theoretically and experimentally substantiated, where the formation of a metal structure and its protection from an aggressive environment are carried out within a given volume on the same technological unit with a flow-through reactor.

Introduction

The industrial processes of production of metals, including iron, are based not only on using solid reducing agents [1–6], but also on restoring the initial metal-containing raw materials with various gaseous reagents [7–15]. The mentioned processes are high-temperature (700–1400 °C) and high-energetic [2, 5, 13, 14]. The high temperature which is necessary to obtain the metal cause a range of problems: from the need for proper selection of heat-stable materials for the ore reduction (with carbon materials) furnace [1, 4] to the intense pollution of the metal with carbon black in the case of the reduction of oxides with hydrocarbon reagents [12, 15]. According to the authors [15], who studied the reduction of iron oxides in methane environment at the temperature in the range of 700–100 °C, the obtained metals contained more than 30 mass. % of carbon. Despite the fact that the reduction of iron by gases is widespread in industrial metallurgy in Russia [10] and in foreign countries [11, 12], another undesirable effect of the process may occur: the possibility of gas corrosion of the metal [16, 17]. Above 700 °C, methane decomposes into carbon and hydrogen, which initiates hydrogen corrosion of the metal, worsening the properties of the metal (hydrogen embrittlement) [17]. In the case of using converted natural gas in the reduction of metal from oxide raw materials, the reaction gas mixture additionally contains carbon monoxide (CO) impurities, which cause carbonyl corrosion of the metal [16].

In addition, iron-based metal powders are used in delicate areas, which often do not require large-scale industrial production. Such areas include molecular surgery (targeted drug delivery and treatment of the body using magnetic carriers - highly dispersed iron powder), catalysis and also the addition of nanostructured iron powder into organopolymer matrix to improve the properties of protective coatings, lubricants, and to create highly hydrophobic materials [18–22].

According to the authors' evaluation [21], in order to satisfy the need of the megalopolis for organosilicon enamels, household chemicals, and lubricants containing surface-modified dispersed iron additives, it is enough to synthesize 250 kg of the Fe-powder per year. This is due to the fact that the even relatively low content of Fe-powder (less than 1 mass. %) leads to significant increase in heat resistance, hydrophobicity and antifriction properties (in lubricants) of the modified materials. Thus, the production of 250 kg of Fe-powder ensures the production of 25 tons of household and industrial chemicals. The synthesis of 250 kg of dispersed iron per year is a feasible task even for university research laboratory. This was demonstrated in the elaboration of St. Petersburg Mining University [19, 21].

Problem Statement

A promising way of production of dispersed metals (Ni, Fe, Cu etc.) with regulated surface properties is the method of solid state hydride synthesis (SSHS) of metals

[22–24]. The method is based on the reduction of solid metal compounds in an open flow system by volatile hydrogen-element compounds ($E = N, C, Si$) in its thermal stability region [23]. Today the following compounds have been studied as hydride reducing agents: ammonia, methane, natural gas (after purification in a low-temperature ($-160\text{ }^{\circ}\text{C}$) zeolite trap), monosilane, methylchlorosilane, and vapor of hydrophobic silicon-organic liquids based on methyl- or ethyl hydride siloxane [21]. Even in the case of simple hydrides with the small sizes of molecules ($\text{NH}_3, \text{CH}_4, \text{SiH}_4$), the dissolution of gaseous-state hydrides in the metal lattice during SSHS is uncharacteristic [16, 17, 23] and thus the possibility of gas corrosion is minimized. Since the temperature of SSHS of metals is $60\text{--}100\text{ }^{\circ}\text{C}$ lower than the temperature of thermal decomposition of the corresponding hydride in the gas phase, hydrogen corrosion of the metal and hardly controlled processes of deposition of carbon or silicon on the metal from the reaction gas phase are almost excluded [19, 23, 24].

The treatment of metal oxide or metal halides at high temperatures by ammonia, methane or monosilane is used to synthesize nitrides (above $600\text{ }^{\circ}\text{C}$), carbides (above $700\text{ }^{\circ}\text{C}$) or silicides (above $500\text{ }^{\circ}\text{C}$) of iron, respectively [25–27]. It is easy to notice that the synthesis temperatures of these compounds significantly exceed the temperatures of the thermal decomposition of the corresponding hydride in the gas phase: $500\text{ }^{\circ}\text{C}$ for NH_3 , $700\text{ }^{\circ}\text{C}$ for CH_4 , and $400\text{ }^{\circ}\text{C}$ for SiH_4 [28]. The formation of carbon or silicon during thermal decomposition of methane or monosilane, respectively, promotes the formation of metal compounds (carbides or silicides), where the non-metal atom (C or Si) is negatively charged [21, 23]. Under SSHS temperature conditions, thermal decomposition of the reducing hydride does not occur; X-ray graphically identifiable phases of metal nitrides, carbides, or silicides are not detected [23]. There is chemisorption of hydride on the metal surface, reliably detected by XP-spectroscopy [23, 24]. By varying reducing hydrides, it is possible to change the nature of the metal surface from hydrophilic (in NH_3) to hydrophobic (in CH_4) and highly hydrophobic (treatment with organosilicon reagents and CH_4) [21, 22].

The most studied are the processes of obtaining samples based on nickel and copper under SSHS conditions [21–24]. According to thermodynamic calculations, the reduction of copper dichloride in ammonia, as well as copper (II) oxide in methane are favorable processes above 700 K ($\Delta G < 0$) even under equilibrium conditions (Fig. 1). However, under the same conditions, the reduction of Fe (II) oxide in methane is not a thermodynamically favorable process ($\Delta G > 0$). Above 700 K the reduction of iron dichloride is possible (Fig. 1), but in this temperature range, the metal chloride begins to sublime into the gas phase [26], and the process of reduction ceases proceeding in the solid phase. Since the SSHS process of metals is carried out under conditions far away from equilibrium [23, 24] (in a flow of reducing

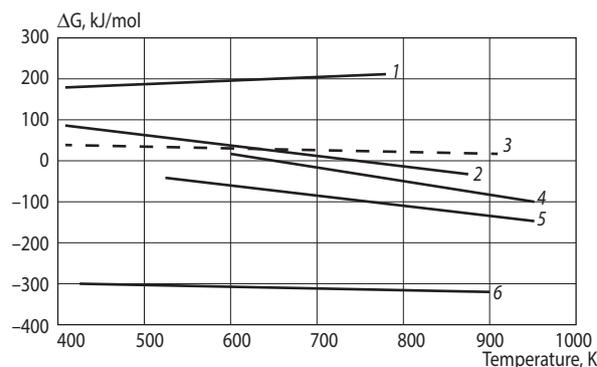
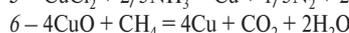
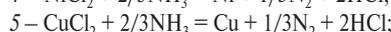
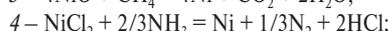
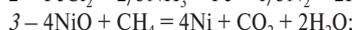
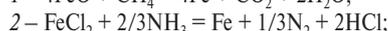
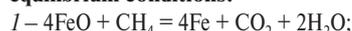


Fig. 1. Temperature dependence of the change of the standard isobaric potential of the reduction reactions of chlorides and metal oxides by hydrides under equilibrium conditions:



gas), it is theoretically possible, by varying of hydride reducing agents, to lower the temperature conditions and to obtain unique corrosion-resistant metal materials with hydrophobic surface.

The main purpose was to obtain dispersed iron modified in the surface layer by organosilicon compounds under SSHS conditions, to estimate its water-repellent properties, and to compare hydrophobicity with samples based on different metals obtained by various methods.

Experiments and Investigation

The production of surface-modified metals by SSHS method was carried out on the open flow installation, which includes systems for purification of reducing (methane) and noble-carrier gas (argon) from traces of oxygen and moisture [21, 24]. The synthesis temperature was monitored potentiometrically and kept constant in the isothermal zone (on the grid of the reactor) with an accuracy $\pm 5\text{ }^{\circ}\text{C}$. Vapor of the silicon hydride reductant-methylchlorosilane (MDCS) were fed into the reactor in argon flow at a flow rate of no more than $4.2 \times 10^{-6}\text{ m}^3/\text{s}$. Fractions of solid-phase metal-containing raw materials with a particle size of $0.5\text{--}0.8\text{ mm}$ were taken for sequential reduction in MDCS vapor and methane. As the initial raw materials powder of chemically pure iron (II) oxide and Olenegorsk superconcentrate (OSC), which is close in composition to Fe_3O_4 (99.5 %; GOST 52939-2008), were used. The mentioned iron-containing powders were dried to constant weight in argon flow at a temperature not lower than $340\text{ }^{\circ}\text{C}$ before filling the reactor with MDCS. Metal formation during SSHS was proved by X-ray diffraction analysis (DRF-2.0 instrument), X-ray photoelectron spectroscopy (VG Escalab iXL instrument) and chemical analysis. The composition of the obtained solid SSHS products was determined by the methods of X-ray fluorescence analysis (Bruker S4 Explorer), EDX spec-

Sample	M, wt. %		Si, wt. %		C, wt. %	Cl, wt. %		Si-C
	Chem. an.	RFA	Chem. an.	RFA	Chem. an.	Chem. an.	RFA	Chem. an.
NiCl ₂ +MDCS+CH ₄	96.0±0.1	—	2.3±0.2	—	1.6±0.1	0.1±0.001	—	3.4
NiO+ MDCS+CH ₄	95.8±0.3	95.6	2.4±0.1	2.0	1.7±0.1	0.05±0.03	—	3.4
CuO+ MDCS+CH ₄	95.3±0.1	94.9	2.3±0.7	—	1.4±0.2	0.09±0.01	0.084	3.3
FeO+ MDCS+CH ₄	94.5±0.5	—	0.8±0.4	—	1.4±0.2	0.06±0.03	—	1.5

*Chem. an. – Chemical analysis; XFA – X-ray fluorescence analysis; MDCS – methylchlorosilane.

troscopy (analytical attachment EDAX/TSL to electron microscope Nanolab). The determination of the adsorption of water vapor on the samples was carried out gravimetrically by the desiccator method at a temperature of 20±2 °C. Mathematical description of the results and spectral dependences was carried out using MathCAD and Origin 6.0 software.

Results and Discussion

Our earlier studies showed that reduction of Cu (II), Ni (II) and Fe (II) oxides to metal at SSHS temperatures in methane (500–650 °C) or in monosilane (320–340 °C) do not provide serious results [21, 23]. Possible reason is the absence of CH₄ adsorption on solid oxide due to energy and steric factors [23]. Chemisorption of CH₄, which precedes the reduction of oxide [2], is determined by the distance between the neighboring oxygen on the surface of the solid oxide and the size of the methyl group (about 0.3 nm) [29]. The similar distance between surface bonded oxygen atoms (0.26–0.3 nm) appears for Fe₂O₃.

However, due to thermodynamic instability of Fe (II) oxide below 580 °C the distance can be noticeably higher [30], which prevents dissociative chemisorption of CH₄ on solid surface [21, 29].

The problem of quantitative and deep reduction of Fe (II) oxide, as well as Ni and Cu oxides, is solved by using MDCS (alkyl-chlorine derivative of monosilane) as a hydride reagent. From the XR diffraction patterns (see Fig. 2, *d*) it follows that upon the reduction of Fe (II) oxide under SSHS conditions, a metallic Fe- α phase with $d = 2.03$, $d = 1.43$ and $d = 1.16$ is formed. Within a few percent broadening of the diffraction maxima is observed. It caused by the dispersion of the iron-based product (specific surface area of 1 m²/g, submicron size of particle aggregates). The final stage of the treatment of the sample in methane under heating is necessary to remove chlorine, which is adsorbed in the first stage of reduction in MDCS vapor.

During the reduction of iron, nickel and copper, the contribution of competing processes of siliconizing and carbidization of metal metal is minimized. In the XRD patterns (Fig. 2) there is no necessary set of analytical diffraction maxima, which is needed to identify the known crystalline phases of silicides or carbides of the metals. There are only single peaks of low intensity, which can be attributed to the lowest silicide of nickel (Fig. 2, *a*, *b*) or copper (Fig. 2, *c*). In addition to the analytical diffraction maxima of metallic iron in the XRD pattern the obtained iron product (Fig. 2, *d*) has a line $d = 2.15$ with several times lower intensity, which is most suitable for Fe₂C.

In terms of data in Table 1, only the surface siliconization of nickel and copper or carbonization of the iron surface could occur. The carbon content of Fe-product of SSHS, is 1.4–1.6 wt. %, and the carbon content of Fe₂C is about 10 %. The silicon content of the Fe-product (0.8 wt.%, see Table 1) is 16 times less than the silicon content of the lower silicide – Fe₃Si (13 wt.%).

In our research, the enhancement of the reducing properties of MDCS in comparison with monosilane during gas-phase treatment of Ni, Fe, and Cu oxides is associated with the intermediate stage of chlorination of the initial solid reagent [21, 23]. The fact of oxide chlorination during the reduction of Fe (II) oxide and iron oxide concentrate (OSC) was confirmed experimentally (X-ray structural and chemical analysis, X-ray photoelectron spectroscopy) [21]. Metal chlorides are more easily reduced to metal than oxides [26]. Therefore, the treatment of the solid oxide with the bifunctional reagent (MDCS), which has the reduction potential due to Si-H-groups and chlorinating effect, allows to destroy strong metal-oxide bonds and to obtain iron, nickel and copper in the metallic

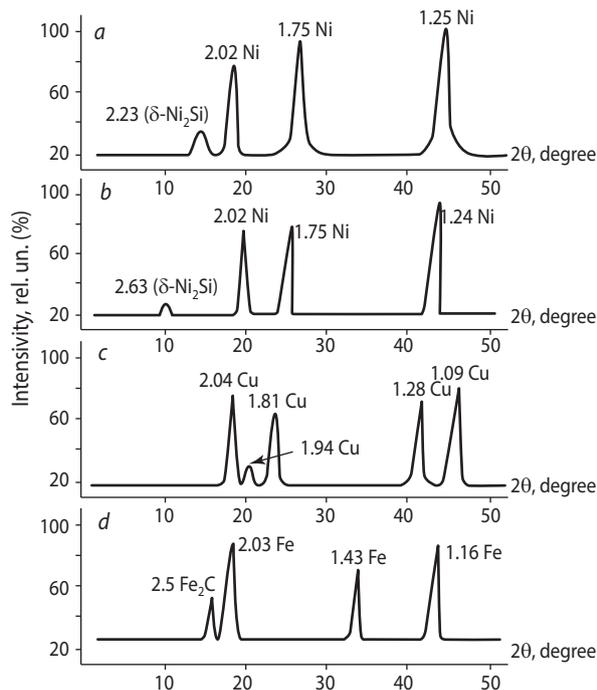
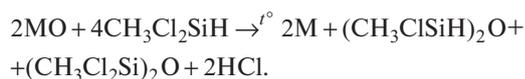


Fig. 2. The XR diffraction patterns of solid SSHS products obtained by sequential reduction of metal-containing raw materials in MDCS vapor (340 °C) and methane (600 °C), from:
a – Ni (II) chloride; *b* – Ni (II) oxide; *c* – Cu (II) oxide; *d* – Fe (II) oxide

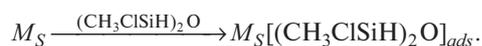
state. Under the SSHS conditions the prevalence of iron carbonization over surface “siliconization” of the metal (characteristic of nickel and copper) can be explained by the good affinity of iron for carbon. The absence of metal carbonization (carbide formation) in the case of nickel and copper is associated with the instability of their carbides at a temperature of 600 °C [27], at which the final stage of reduction in a flow of methane occurs [21].

Here are the gross-reactions of the main processes of the reduction of metal oxide (M = Fe, Ni, Cu) under SSHS conditions, which were discussed above.

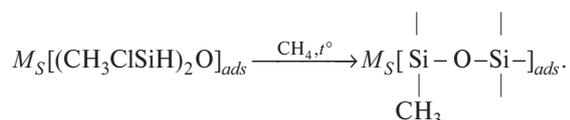
Reaction of the oxide reduction process in MDCS vapor:



Here is the scheme of chemisorption of MDCS oxidation product on metal, confirmed by XPS and IR spectroscopy [24, 31]:



Here is the scheme of reduction (dechlorination) of the solid product of the previous stage with a chemisorbed organosilicon compound in methane:



According to the data in **Table 2**, the reduction of the iron ore oxide concentrate OSC under SSHS conditions leads to a significant hydrophobic effect of the resulting solid product. Water adsorption drops by almost 30 times (5.4 and 0.19 wt.%). It is interesting that during the first day of sorption of saturated water vapor, the sample

(OSC + MDCS + CH₄) did not add, but lost (at the level of 0.9 wt. %) in mass. The sample is poorly wetted by water. The particles are capable of not settling on the bottom of a container filled with water for a long (Table 2).

The hydrophobicity of fresh iron-based samples obtained by the SSHS method is significantly higher than hydrophobicity of carbonyl nickel iron treated with the industrial organosilicon hydrophobizator HSL-94 (Ni/HSL), as well as powders of (P10), nickel (PNK-UTZ) and copper (PM-1), treated in various combinations of HSL-94 and reagents with hydrophobic organic groups - alkamone (A) and triamone (T). This is confirmed by the data in **Table 3**. The value of water vapor absorption by the sample obtained from iron (II) oxide under SSHS conditions is 16 μmol/m², which is two orders of magnitude less than the water absorption of metal powders modified with hydrophobizing agents. The water absorption of this Fe sample is at the corresponding level of highly hydrophobic organosilicon adsorbents synthesized at Kiev Scientific School (14–16 μmol/m²) [32].

Today, the problem of obtaining highly hydrophobic materials (including metal-based) is rapidly developing in Russia and abroad [33, 34]. Leading experts in this field recommend application of perfluorooxysilane, various fluorine-containing copolymers and polydimethylsiloxanes. The least toxic of these hydrophobic agents are polydimethylsiloxanes, which are silicones. Similar substances are polymerized in the processes of the syntheses (see schemes of gross SSHS processes above).

The feature of our synthesis is that, strong chemical interaction between the metal and the protective film occurs due to the combination in time (on one installation) of the processes of reduction and modification with organosilicon compounds (carbosiloxane structures). Heteroatomic electronic interaction M → Si is confirmed by X-ray photoelectron spectroscopy data from **Table 4**. The data (binding energy Si2p level and Δ – chemical shift of M2p_{3/2} level)

Table 2. Adsorption of water and hydrophobicity of Fe-based powder

Sample	Relative weight gain in water vapor at $p/p_s \rightarrow 1$, %			Weight loss after drying over P ₂ O ₅ , wt. %		Note
	1 day	2 days	5 days	1 day	5 days	
OSC	0.36	3.76	5.4	–0.11	–0.44	The sample is hydrophilic
OSC+MDCS+CH ₄	–0.09	0.15	0.19	–0.13	–0.16	Hydrophobic powder particles float for weeks on the surface of the water

Table 3. The value of water vapor absorption (a) by freshly prepared samples based on iron and other metals and obtained by different methods

Sample, method of obtaining	Fe(II) oxide + MDCS + CH ₄	Ni/HSL	Fe/T/HSL	Ni/T/A	Cu/A/HSL
a, mmol/m ²	0.016	2.2	2.0	2.3	1.0

Table 4. Comparison of the adsorption of water vapor, specific surface area, air corrosion (K) and the parameters of the XPS spectra of the samples

a, mmol/m ²	The compound from which the metal is reduced (M)	S _{SSA} , m ² /g	K, mg/cm ² (900 °C, 100 h)	E _{Si2p} , eV	Δ for M2p _{3/2} , eV
0.027	Ni (II) chloride	11	0.00051	104.8	2.4
0.030	Ni (II) oxide	57	0.00042	105.7	1.4
0.024	Cu (II) oxide	12	0.00033	103.8	2.5
0.016	Fe (II) oxide	1	0.00011	102.4	4.9

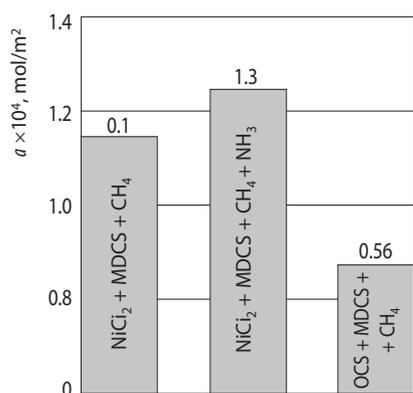


Fig. 3. Absorption of saturated water vapor ($p/p_s \rightarrow 1$) by samples based on nickel and iron synthesized by the SSHS method. Samples were stored for 30 years in buckets

shows the importance of chemisorption of carbosiloxane groups on chemically active metal centers, which provides high hydrophobicity of the samples and high heat stability in air with an increase in mass (K) at a level of 0.1–0.4 $\mu\text{g}/\text{cm}^2$. For high-quality nickel-chromium alloys, the heat resistance under the same conditions is about 7 $\mu\text{g}/\text{cm}^2$, which is 70 times worse than that of the Fe sample obtained under SSHS conditions (Table 4). Even after 30 years of storage (Fig. 3) the Fe sample obtained from OSC remains the leader and is almost 2 times more hydrophobic than Ni samples obtained under the same conditions (SSHS).

SSHS fulfills at least two important goals. It returns to the metal its “original” property inherent in the atomically pure metal surface - hydrophobicity [16, 35]. Base metals in a real air atmosphere are rapidly oxidized, and their surface becomes hydrophilic [16, 35, 36]. In addition, the demonstrated SSHS method allows to synthesize a set of chemically stable dispersed metal materials which are perspective for solving the problem of superhydrophobicity and corresponding applications [33, 34]. According to experts, in order to create superhydrophobic materials and coatings, it is preferable to use solid-state systems with bimodal particle size distribution, for example, substrate with micro- and macro-sized structural elements and hydrophobic substance nanofilm with adhesion to the substrate. The obtained Fe-, Ni-, and Cu-samples correspond perfectly with this model. The metal substrate consists of aggregates of micron-sized particles [31] with a chemically interacting carbosiloxane protective film (no more than 4–5 nm thick) [23, 24] (see Table 4). Chemical resistance and hydrophobicity of the synthesized powders enhances depending on the nature of the metal in the series Ni, Cu, Fe. These powders have been applied at the enterprises of the mineral resource complex in the Russian Federation and in the Republic of Belarus [21, 37], including PJSC “Belgorkhimprom”, PJSC “Soligorsk Institute of Resource Saving Problems with Pilot Production” (Belarus), Research Institute “Energostal”, company “GMC”, LLC “GSK-Shakhtproekt” (Russia). According to these organizations experts, the economic effect of using powders as components of protective coatings and lubricants makes on average 7–10 mln rubles/year [21, 31].

It is important to underline the possibility of using the Olenegorsk superconcentrate (OSC) in SSHS technology. Fields of raw materials for OSC in North-West Russia are estimated at millions of tons. OSC is a product of magnetite concentrate refinery. OSC is a finely dispersed black powder, which is easily, can be grind to particles of 30 microns and below. The specific surface area of OSC is about 10 m^2/g . The water content is at the level of 0.5–1.0 wt. %. The OSC price on the domestic market (May 25, 2020) is 66.3 USD per ton. Recalculated in rubles, the price for 1 kg of OSC is less than 5 rubles (4.95 rubles/kg). The use of such iron oxide raw materials is very beneficial, since its price is an order of magnitude lower than reactive purity iron oxide. Another advantage of OSC is that it is used in metallurgy in the production of iron powders of almost all grades in accordance with GOST 9849–74.

The obtained results supplement and develop the theory and practice of metallurgical processes of synthesis of new materials (based on iron and other metals) with improved properties, and also contribute to the modern research of the scientific school of metallurgists of Mining Institute (now the University) in St. Petersburg [38–44], at the origins of which was academician N.S. Kurnakov [21, 24, 45, 46].

Conclusions

The obtaining of dispersed iron containing a carbosiloxane protective film chemically bonded to the metal in the surface layer has been theoretically and experimentally substantiated. The metal content is 94.5 %, silicon is about 1 %, carbon is 1.4 %, oxygen is not more than 2 %. The specific surface area of the solid product is in the order of 1 m^2/g . The water absorption of the sample in saturated water vapor is at the level of 16 $\mu\text{mol}/\text{m}^2$ when tested for up to 200 h. Heat stability in air is about 0.1 $\mu\text{g}/\text{cm}^2$ at 900 °C for 100 h at atmospheric pressure and free air access.

The water-repelling properties and heat stability in air of samples based on iron, nickel, and copper obtained from oxides under SSHS conditions by their successive reduction with methyldichlorosilane vapor and methane are compared. It was found that, depending on the type of metal, the hydrophobicity and corrosion resistance of the samples increases in the following order: nickel - copper - iron.

It was found that the improved characteristics of the samples in terms of hydrophobicity and corrosion resistance correspond to the strongest chemical interaction between metal and silicon, which, according to XPS spectroscopy data, is found in the sample based on iron.

It has been shown that not only a chemically pure reactants of bivalent iron oxide, but also a much cheaper Russian-made Olenegorsk superconcentrate (OSC), can be used as the initial iron oxide raw material for obtaining surface-modified iron powders with improved properties.

The prospects for the use of synthesized metal powders for the creation of superhydrophobic materials are analyzed. Some of the obtained dispersed samples based on metallic iron and other metals have already found application at en-

terprises in Russia and Belarus for solving problems of equipment protection and lubrication.

A method for the synthesis of the metal product has been implemented, which is characterized by the complex production of a metal structure and its protection within a given volume on one technological unit (in a flow reactor). Modification of the metal surface with carbosiloxane structures containing thermo- and chemically resistant Si-C bonds occurs not during the application of modifiers to the prepared metal surface, but directly during the reduction of the initial solid metal compound (ore) with an organosilicon reagent. The metal, in our opinion, at the moment of its appearance, actively interacts with a reducing agent containing Si-H groups, with the formation of a chemical bond with the applied protective carbosiloxane film.

Acknowledgements and Dedication

The authors dedicate this article to blessed memory and to the 70th anniversary of the birth of Ilya Nikitich Beloglazov (1950-2011), a famous metallurgist, scientist-encyclopaedist. One of the authors of this article worked with him on interesting projects in 2003–2011, including those organized by the “Ore and Metals” Publishing House: writing one of the first Russian monographs on nanomaterials, preparing a special thematic issue “Nanostructured Metals and Materials” within “Tsvetnye metally” journal, including its English version “Non-ferrous metals” [20], writing articles for the journal “CIS Iron & Steel Review” (2008), as well as organizing and conducting the International Symposium “Nanophysics and Nanomaterials” (since 2003). It is important to note with gratitude that most of these projects, inspired by the energy of Professor I.N. Beloglazov, continue to function to this day, contributing, among other things, the strengthening of scientific cooperation of specialized universities of the Union State.

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