Justification of a rational processing scheme for ilmenite concentrate with production of soft iron and titanium dioxide concentrate

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The results of theoretical analysis and experimental studies of selective solid-phase Fe reduction from an ilmenite concentrate by carbon or hydrogen are presented. It is shown that hydrogen reduces Fe with a higher rate at a relatively low (900 °C) temperature, what allows to obtain soft iron and titanium dioxide concentrate, which are easily separated by melting and are commercial products. Hydrogen is practically not dissolved in solid iron, and is easily removed from the unit due to the gaseous state, what eliminates titanium reduction and formation of its lower oxides when the temperature rises at the stage of separation of the reduction products. Fe reduction by carbon at an acceptable rate and at a higher (above 1100 °C) temperature is accompanied by reduction of titanium to lower oxides, while energy consumption for reduction increases by 2.25 times. The scheme is proposed for processing of ilmenite concentrate in a non-agglomerated form – selective reduction of iron with hydrogen in a multi-hearth furnace, with consequent separation of reduction products into soft iron and titanium dioxide concentrate in a plasma furnace.

Key words: ilmenite, reduction by hydrogen, reduction by carbon, selective solid-phase reduction, soft iron, titanium dioxide concentrate, rutile, iron dititanate, anosovite.
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Introduction

Titanium-containing iron ore raw materials are considered as a valuable source of such strategic elements as Fe and Ti. At present time Russia meets lack of raw materials for manufacture of pigment titanium dioxide. Based on the data of 2019, import of pigment titanium dioxide was 53.6 thousand t, what made 67.5 % of consumed import [1]. At the same time, Russia occupies one of the leading places worldwide for reserves of titanium-containing iron ore raw materials; its complex processing with obtaining metallic iron and titanium dioxide can allow to organize reliable raw material base for domestic production of titanium dioxide with long-term prospect.

Depending on correlation between iron and titanium dioxide amounts, titanium-containing iron ore raw materials are separated to low-titanium raw materials (with Fe/TiO_2 correlation exceeding 8), iron-vanadium raw materials (with Fe/TiO_2 correlation from 2 to 8) and high-titanium raw materials (with Fe/TiO_2 correlation lower than 2) [2]. Most part of titanium-containing iron ore raw materials is presented by complex oxides in the form of ilmenite minerals $FeO \cdot TiO_2$ and titanium magnetite $[Fe,Ti]_3O_4$. As a result of mineral processing, not only gangue separation occur, but also extraction of two concentrates – ilmenite and titanium magnetite ones. Concentration possibility is determined by grain size of ore minerals, by impregnation value of ore minerals ilmenite and titanium magnetite (mutual germination of minerals). In this connection, it is the tendency to achieve size of particles less than 0.074 mm during comminution, in order to provide better separation of minerals. As a result, concentrates of titanium-containing iron ore raw materials are obtained in fine-dispersed form and require agglomeration for consequent production stage according to the existing technologies.

Low-titanium (titanium magnetite) concentrates are used at the integrated metallurgical works in the technological route "blast furnace – basic oxygen converter" with use of iron and vanadium. Titanium magnetite processing in a blast furnace practice, with titanium dioxide content 2.5 %, is realized at present time at Nizhny Tagil Iron and Steel Works. TiO_2 content in blast furnace slag achieves 11 % in this case, while use of the ore concentrate from Gusevogorskoe deposit (TiO_2 content in this concentrate is 3.41 %) leads to increase of TiO_2 content in blast furnace slag to 14.8 %, what makes definite difficulties for a blast furnace practice [3]. At the same time, in China this process is carried out even with more high TiO_2 content in blast furnace slag (25 %) and TiO_2 content in the concentrate about 13 %) [4, 5].

Ore thermal electric melting of "Sorel" process is another pyrometallurgical method for processing of titanium-containing iron ore raw material. This method is used for processing of ilmenite concentrates with obtaining vanadium cast iron and high-titanium slag with TiO_2 content 70–85 % [6]. As a result of electric melting, 96–97 % of Fe and 45–48 % of V transit in cast iron [7]. Separation of titanium and iron is based on their essential difference affinity to oxygen; however, partial titanium reduction up to lower oxides and even to metal occurs during melting. Titanium transition in metallic phase can make in this case up to 2 % from its amount in the concentrate [8]. In Russia, high-titanium slags, which were obtained via melting in an ore thermal furnace, are used for manufacture of titanium sponge or metallic titanium. This method requires larger electric power consumption due to forming of high-melting-point slags, thereby it is used only in the regions with cheap electric power [9]. Additionally, partial titanium reduction takes place inevitably.

Lowering of electric power consumption can be achieved via transfer of reduction processes in a separate unit for conduction of solid phase metallization with use of cheap reducing agents and fuel, while separation of metallization products can be realized in this case in an arc furnace. Studies on possibility assessment of metallized charge use during melting in an ore thermal furnace show that use of metallized charge (with metallization degree about 70 %) allows to decrease electric power consumption approximately by 35 % for 1 t of high-titanium slag, and by 20 % more with introduction of physical heat [10]. Based on the results of these studies, the testing of pyrometallurgical processing of the concentrates obtained from ores of Chineyskoe deposit, were carried out. As a result, the dual-stage processing technology with Fe, Ti and V extraction was suggested [2, 11]. This technology includes preliminary Fe reduction by carbon in a rotating tubular furnace with achieving Fe metallization degree 90–93 % and separation of hot metallized concentrate via melting in an ore thermal furnace with obtaining of vanadium iron and titanium slag. This technology was also recommended for other medium- and high-titanium concentrates. Other variants of the dual-stage processing technology are proposed, when shaft furnaces are used as reducing units instead of tubular furnaces [12]. However, despite lower temperature in reducing tubular and shaft furnaces in comparison with ore thermal melting, titanium is also reducing in these furnaces up to lower oxides or even to the metal with consequent forming of carbides and oxy-carbides in addition to iron and vanadium. It will complicate the technological separation process of reduction products [13].

Hydrogen can be an alternative to usual carbon as a Fe reducing agent [14]. Hydrogen has several advantages in comparison with carbon-containing reducing agents, which are especially important for Fe reduction from complex ores and ores that hardly processed via conventional technologies. In this case Fe selective reduction in complex ores in order to obtain soft iron and concentrate of titanium, magnesium, manganese etc. oxides is considered as a prospective direction of hydrogen use. It is shown [15] that Fe hydrogen reduction is economically expedient just at present time for selective reduction in sideroplesite, ferromanganese, titanium magnetite and ilmenite ores with obtaining titanium, manganese or magnesium concentrates.

Fe reduction by hydrogen, carbon monoxide and gas mixtures on their base as well as solid carbon was examined in the researches [16–18]. The obtained results display that reduction by hydrogen is characterized by essentially larger speed at relatively low temperatures (< 1000 °C) in comparison with carbon monoxide and carbon. Fe reduction by pure hydrogen from ilmenite concentrate was studied in the works [19–23]. However, these experimental researches were carried out at the small (< 0.5 l/min) hydrogen consumption, what has significant effect on reduction speed. Comparison of the reduction results with the existing carbothermal processes is also absent in these works.

Substantiation of the expedience of hydrogen use for Fe selective reduction from ilmenite concentrates with obtaining the required products (soft iron and titanium dioxide concentrate) is the aim of this research.

Materials and methods

When carrying out the experiments, ilmenite concentrate with the following composition (%, mass.) was used:

O	Mg	Al	Si	Ti	V	Mn	Fe
42.6	0.4	0.3	0.7	24.0	0.3	0.4	31.2

Equilibrium composition of the system components and the temperature sequence of transformation of the system components during reduction by carbon or hydrogen was determined via thermodynamic simulation using “TERRA” software complex [24, 25]. The amount of reducing agent was accepted equal to the required amount according to stoichiometry for Fe reduction, or with excess which was 10, 20, 30 and 100 % (mass.) for carbon and which exceeded the stoichiometric amount by 10, 100 and 1000 times for hydrogen.

When conducting the experiments, ilmenite concentrate with the following composition was used: $FeTiO_3$ – 92 %, Fe_2O_3 – 5.5 % and TiO_2 – 2.5 %, and with fraction less than 0.4 mm. The briquettes with mass about 3 g were fabricated via extrusion with the force 222.93 MPa, using paraffin as a binder. Paraffin was evaporated after briquette fabrication within the temperature range 200–300 °C during 1 hour.

The experiments on concentrate metallization using solid carbon as a reducing agent were conducted in a hermetic resistance furnace with graphite heater. A briquetted sample with mass about 3 g was placed in a corundum crucible and filled up with ground graphite from heater wastes (with fraction less than 0.4 mm), to provide the contact between graphite and total surface of a briquetted sample. The furnace was heated up to the temperatures 900, 1000, 1100 or 1300 °C an held during 60 min. The temperature in the working zone was measured by a thermocouple BP5/20 with measuring error ±10 °C.

The experiment with hydrogen use was carried out in the multi-functional resistance furnace “RB Automa-zione” MM 6000, in the reactor with flowing atmosphere at the temperature 900 °C. When heating a sample from the room temperature to 900, 1000, 1100 °C, the reactor was blown up by argon in order to provide system rinsing with air removal, with consumption 0.5 l/min. High-grade argon (99.993 %) according to the GOST 10157-2016 was used in this experiment. Then hydrogen was injected in the system (with consumption 5 l/min) and was held during 30 min. Hydrogen purity was 99.99 % according to the GOST 3022-80. Then, after holding, hydrogen injection was switched off and the system was subjected to rinsing by argon with consumption 0.5 l/min.

Obtained metallized samples were examined via the methods of X-ray phase analysis as well as optical and electron microscopes. X-ray phase analysis was conducted using powder diffractometer Rigaku Ultima IV, equipped with

X-ray pipe with a copper anode (wave length 0.154 nm) and high-speed detector D-teX Ultra. Voltage and current of an X-ray pipe was 40 kV and 30 mA respectively. Capturing was carried out with the speed 5 grad per min within the angle range 2theta from 5 to 90 grad. Decoding of diffraction patterns was conducted using the PDXL program, using the PDF2 base of crystallographic data of 2009. Elementary phase composition was detected via micro-X-ray spectral method using energy and dispersion detector Oxford INCA X-max 80 with a scanning electron microscope JSM-6460LV of JEOL company.

Products obtained after reducing roasting of ilmenite concentrate at the temperature 900 °C with holding during 30 min, with use of hydrogen as a reducing agent, were separated via melting to metal and slag in and open resistance furnace. In this process, graphite crucible was used, with placing a molybdenum plate bent in a pipe in this furnace, in order to eliminate the contact between oxide phase and crucible carbon.

Experimental results and discussion

The results of thermodynamic simulation testify that Fe selective reduction by carbon is possible only at the temperature below 750 °C. However, equilibrium degree of Fe metallization at the temperature 750 °C makes only 6.74 % (**Fig. 1**). Temperature rise leads to also vanadium and silicon reduction, in addition to Fe reduction. The equilibrium degree of Fe metallization achieves 99.29 % at the temperature 1150 °C, while equilibrium degrees of vanadium, silicon and titanium metallization are equal to 100, 96,03 and 0.37 % respectively.

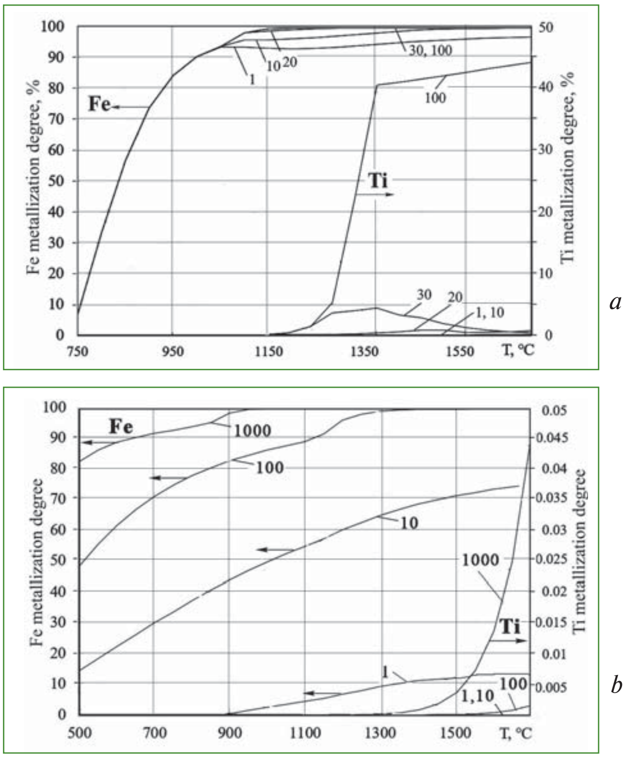

Fig. 1. Influence of amount of a reducing agent on Fe and Ti metallization degree by carbon (*a*) and by hydrogen (*b*). Figures near the curves mean excess of a reducing agent

Selective Fe reduction by hydrogen, with achieving its metallization degree about 90 %, is possible at the temperature below 700 °C and 1000-fold hydrogen excess. Silicon and vanadium are also reduced within the temperature range 700–1100 °C with large hydrogen excess. At the same time, titanium is not reduced by hydrogen at the temperature below 1400 °C even with 1000-fold hydrogen excess.

Examination of metallized samples using a microscope displayed that extraction of metallic phase in ilmenite grains occurs through the whole volume of samples. The aimed products (iron and titanium dioxide) are forming at the temperature 900 °C as a result of Fe reduction by carbon; however, the speed of this process is low and ilmenite is observed in the samples even with holding during 300 min. At the same time, the iron dititanate phase $FeO{\cdot}2TiO_2$ is observed in metallization products at the temperatures 1000 and 1100 °C, in addition to Fe and TiO_2. In these conditions, in the same way as at the temperature 900 °C, complete Fe reduction is not achieved even with the minimal holding time owing to the low process speed, while Fe reduction by hydrogen at the temperature 900 °C finished during 30 min. Temperature rise up to 1200 and 1300 °C promotes not only Fe reduction by hydrogen, but also Ti reduction to lower oxides (**Fig. 2, Table 1**).

The results of X-ray phase analysis testify that Fe and titanium dioxide are forming as a result of reduction by both carbon and hydrogen at the relatively low temperatures (900 and 1000 °C) (**Fig. 3,** *a*, *b*). However, the speed of reduction be carbon in this case is essentially lower than in the case of reduction by hydrogen: Fe metallization degree close to 100 % with hydrogen use and at the temperature 900 °C is achieved during 30 min, while Fe reduction by carbon at the temperature 1100 °C does not finish even during 60 min (Fig. 3, *c*). Temperature rise up to 1100 °C and higher leads to interaction between titanium dioxide (product of reduction reaction) and ilmenite and to forming of the iron dititanate phase $FeO{\cdot}2TiO_2$; for the case of reduction by carbon, it leads also to titanium reduction to its lower oxides.

The mechanism of selective reduction and extraction of a metallic phase inside a volume of complex ore via transition of anionic vacancies charged by electrons was described earlier [26] and substantiated in details in the manual [27]. Regarding to Fe selective reduction in ilmenite, it can be imagined as formation of "free" electrons in anionic vacancies (V2e) of oxide phase on oxide surface, due to transition of oxygen atoms in a reducing medium, transition of charged vacancies in the ilmenite crystal lattice until they will meet easily reducible Fe cations with consequent interaction between electrons and cations.

The following reactions take place in the gaseous phase:

$$C^0 + O^0 = (C^{2+}, O^{2-}) \text{ or } 2H^0 + O^0 = (2H^+, O^{2-}) \qquad (1 \text{ or } 1a)$$

in the oxide phase at the temperature below 1100 °C on the surface:

$$O^{2-} = \uparrow O^0 + (V2e) \qquad (2)$$

in the ilmenite volume:

$$(Fe^{2+}, O^{2-}){\cdot}TiO_2 + (V2e) = (VFe^0) + TiO_2 \qquad (3)$$

As a result of these reactions, reduced metal is extracted in oxide volume, in an emptiness of flowing anionic

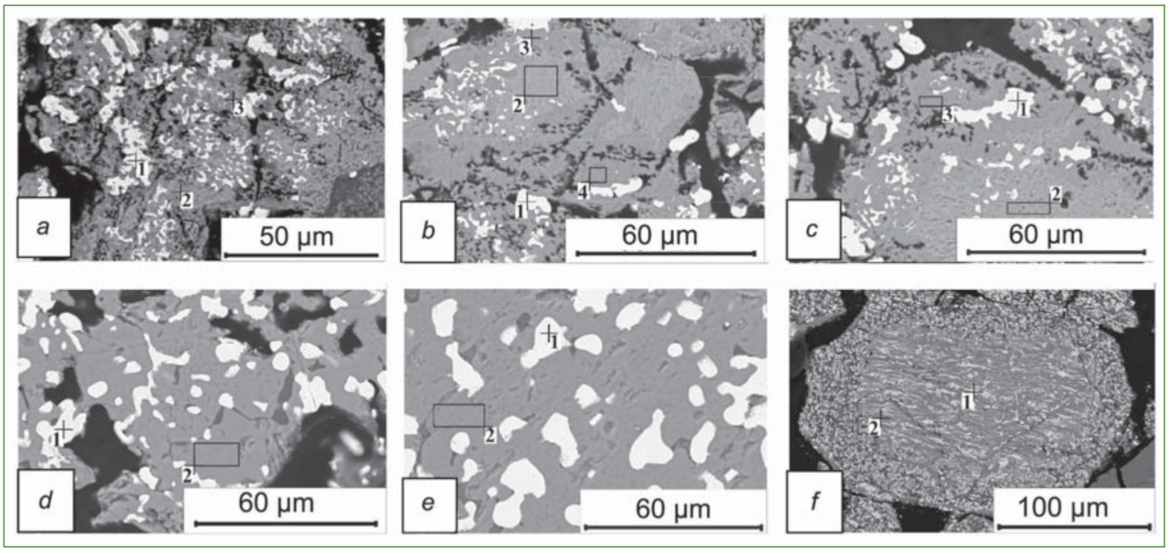

Fig. 2. Products of the sample reducing roasting in the contact with carbon (*a* – temperature 900 °C, holding 60 min; *b* – temperature 1000 °C, holding 180 min; *c* – temperature 1100 °C, holding 180 min; *d* – temperature 1200 °C, holding 180 min; *e* – temperature 1300 °C, holding 60 min) and in the hydrogen atmosphere (*f* – temperature 900 °C, holding 30 min)

Table 1. The results of analysis of composition of reduction products, at. %

Fig. 2	Area of analysis	O	Ti	V	Fe	Phase
a	1	–	–	–	100	Fe
	2	72.8	14.5	0.1	12.6	$FeO \cdot TiO_2$
	3	74.8	22.5	0.3	2.4	TiO_2
b	1	–	–	–	100	Fe
	2	69.3	16.6	0.2	14.0	$FeO \cdot TiO_2$
	3	71.6	25.4	0.5	2.6	TiO_2
	4	66.1	22.6	0.4	10.9	$FeO \cdot 2TiO_2$
c	1	–	–	–	100	Fe
	2	70.0	16.6	0.3	13.1	$FeO \cdot TiO_2$
	3	69.9	21.9	0.4	7.8	$FeO \cdot 2TiO_2$
d	1	–	–	0.1	99.9	Fe
	2	69.3	26.3	0.4	4.0	Ti_3O_5
e	1	–	–	0.1	99.9	Fe
	2	71.3	26.7	0.5	1.5	Ti_3O_5
f	1	22.1	6.0	0.1	71.8	Fe
	2	73.3	23.4	0.3	3.0	TiO_2

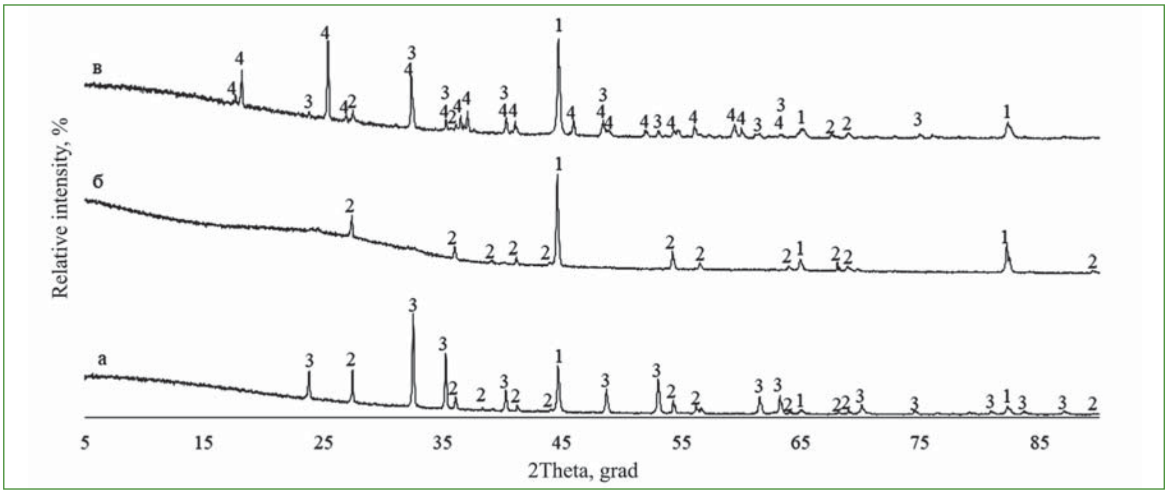

Fig. 3. The results of phase analysis of the samples after reducing roasting: *a* – at the temperature 900 °C with holding during 60 min, reduction by carbon; *b* – at the temperature 900 °C with holding during 30 min, reduction by hydrogen; *c* – at the temperature 1100 °C with holding during 60 min, reduction by carbon. (1 – Fe; 2 – TiO_2; 3 – $FeO \cdot TiO_2$; 4 – Ti_3O_5)

vacancies, which move finally on oxide surface owing to thermal motion of ions, and then these vacancies disappear.

If we shall summarize these reduction reactions, they can be expressed as follows:

$$(Fe^{2+},O^{2-})\cdot TiO_2 + C^0 = \mathbf{Fe^0} + \mathbf{TiO_2} + (C^{2+}, O^{2-}) \quad (4)$$

$$(Fe^{2+},O^{2-})\cdot TiO_2 + 2H^0 = \mathbf{Fe^0} + \mathbf{TiO_2} + (2H^+, O^{2-}) \quad (4a)$$

When the temperature is higher (1100 °C and above), the same products (iron and titanium dioxide) are forming as a result of reduction; however, the forming titanium dioxide is solving in ilmenite (not reacting yet) with forming of Fe dititanate:

$$FeO\cdot TiO_2 + TiO_2 = FeO\cdot 2TiO_2 \quad (5)$$

Consequent Fe reduction from dititanate is accompanied by titanium reduction with forming of anosovite Ti_3O_5. Forming of iron and anosovite at the high temperature takes place via the following reactions:

$$3[(Fe^{2+}, O^{2-})\cdot 2(Ti^{4+}, 2O^{2-})] + 5C^0 = 3\mathbf{Fe^0} + 2[(\mathbf{Ti^{2+}}, 2Ti^{4+}, 5O^{2-})] + 5(C^{2+},O^{2-}) \quad (6)$$

$$3[(Fe^{2+}, O^{2-})\cdot 2(Ti^{4+}, 2O^{2-})] + 10H^0 = 3\mathbf{Fe^0} + 2[(\mathbf{Ti^{2+}}, 2Ti^{4+}, 5O^{2-})] + 5(2H^+,O^2) \quad (6a)$$

Thus, Fe reduction by hydrogen or carbon in ilmenite, with forming of metallic iron and titanium oxides, can be presented as the following scheme (**Fig. 4**).

Comparison of the results of Fe reduction by solid carbon or hydrogen at different temperature values and various holding time testify on substantial energetic advantages of the hydrogen reduction process. Quick Fe reduction by carbon at the relatively low temperature does not allow forming of Fe dititanate, what decreases significantly process duration and energy consumption. Required energy for Fe reduction by hydrogen at the temperature 900 °C is lower by 1.58 times than expenses for reduction by carbon at the same temperature and lower by 2.25 times than required energy for reduction by carbon at the temperature 1300 °C [28].

Metal and slag composition after separation of semiproduct, which was metallized at the temperature 900 °C with holding during 30 min with hydrogen use, are presented in the **Table 2**. The metal obtained after separation does not contain carbon, while slag contains mainly (90 % mass.) of titanium dioxide with impurities of Fe oxides, what is supposedly stipulated by conduction of separating melting in the air atmosphere. Slight molybdenum content is revealed in metal, its presence is caused by use of a molybdenum pipe during melting in order to exclude contact between slag and graphite of a melting crucible.

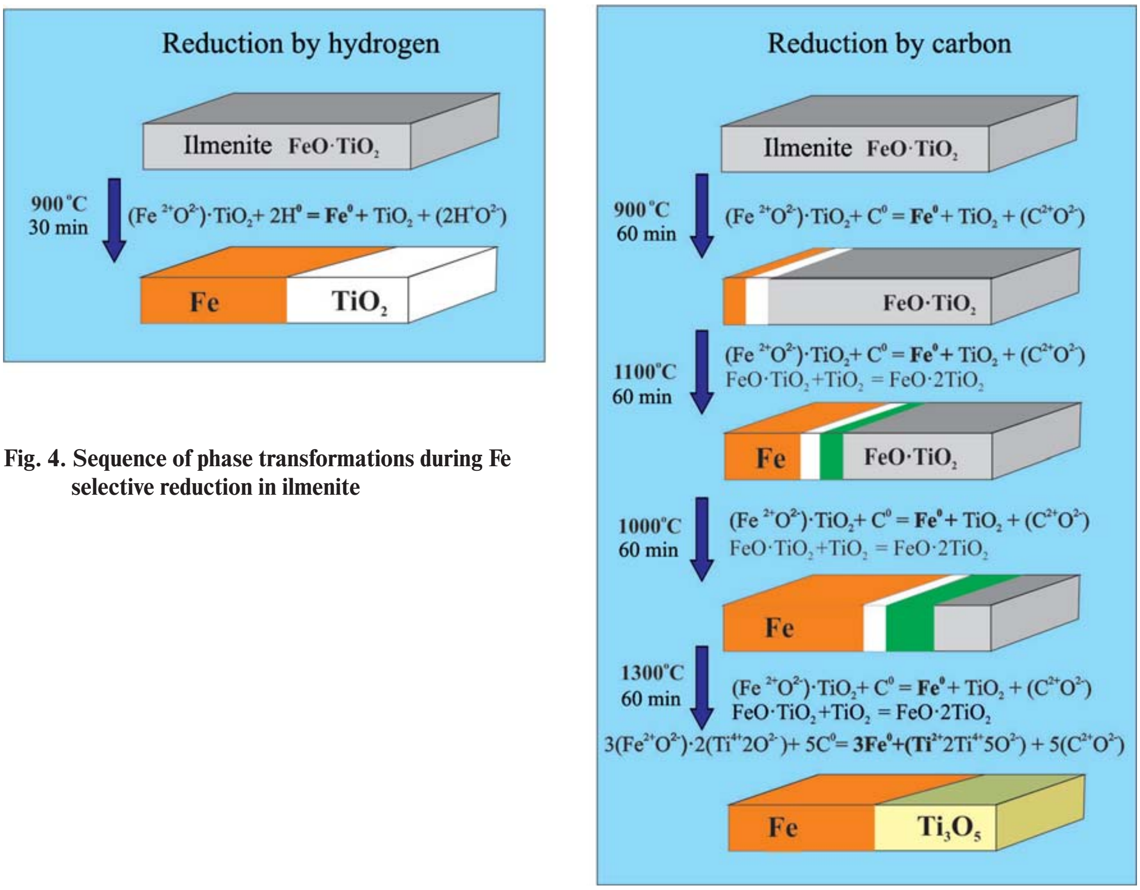

Fig. 4. Sequence of phase transformations during Fe selective reduction in ilmenite

Table 2. Metal and slag composition after separating melting of semiproducts with reduction by hydrogen

Metal							Slag					
O	Si	Ti	V	Fe	Mo	Total	Al_2O_3	SiO_2	TiO_2	V_2O_5	FeO	Total
0.4	0.0	0.0	0.0	99.4	0.2	100	1.2	1.6	90.5	0.5	6.2	100

Practical recommendations for processing of ilmenite concentrate

The composite material, which was obtained during reducing roasting, contains of iron and titanium oxides concentrate. Composition of products is determined by parameters of reduction process. As can be concluded from the experiments, Fe selective reduction from ilmenite concentrate is realized successfully when using both carbon and hydrogen. However, carbon use needs higher temperature values, when titanium reduction to lower oxides becomes also inevitable. Hydrogen use allows to carry out reduction with more energy efficiency at lower temperature (~ 900 °C) and with sufficiently high speed, finalizing in obtaining more valuable products.

Dispersed ilmenite concentrate, which was obtained as a result of concentration of titanium-containing raw materials, can be subjected to reducing roasting in multi-hearth furnaces in a bulk form with hydrogen use; it will allow to cut the expenses for agglomeration and to increase reacting surface for reduction by hydrogen. It is expedient to conduct the reduction process in hydrogen flow, where waste gases can be dried and returned to the technological cycle.

It is not recommended to use electric arc furnaces for separating melting of products of solid phase reduction due to possible metal contamination by carbon of electrodes, partial titanium reduction and forming of hard-melting titanium oxycarbonitrides [9, 29]. It was noted in the researches [30, 31] that high electronic conductivity of titanium slags is an obstacle for dipping of electrodes in a slag melt and leads to n arc melting procedure, when slag heating is hampered. Plasma furnaces can be used for operating with such slags; it will allow to conduct the technological process in the protective atmosphere of plasma-forming gas without contact with electrodes and with high efficiency of slag heating [32].

Thus, it is expedient to realize Fe reduction from ilmenite concentrate and obtaining of the required products (soft carbon-free iron and titanium dioxide concentrate) via the technological route entitled as “selective solid-phase iron reduction in a multi-hearth furnace and separation of reduction products in a plasma furnace” (**Fig. 5**).

It is expedient to use soft iron, which is obtained via such route, for manufacture of the most expensive metal products, e.g. iron powder for 3D printing of the components via layered laser sintering, or thin sheet via ISP production technology in the thin slab casting and rolling unit or two-roll stand according the technology of Krupp Thyssen Nirosta GmbH.Titanium dioxide concentrate is recommended to be processed via the chloride technology with obtaining of pigment titanium dioxide or metallic titanium.

Conclusions

1. The results of thermodynamic calculations and experiments confirm expedience of hydrogen use as a reducing agent in processing of complex ores, in order to obtain Fe and concentrates of oxides of the second metal.

2. Selective solid phase Fe reduction by hydrogen from ilmenite occurs significantly faster and at lower temperature in comparison with reduction by solid carbon. Reducing roasting of ilmenite concentrate at the relatively low (~ 900 °C) temperature and with short (~30 min) holding in hydrogen flow provides obtaining of two required products – soft iron and titanium dioxide concentrate.

3. Fe reduction from ilmenite both by carbon and hydrogen at the temperature above 1100 °C is accompanied by titanium reduction to lower oxides, what substantially (by 2.25 times) increases energy consumption expenses for reduction.

4. It is expedient to conduct selective solid phase Fe reduction by hydrogen in non-agglomerated ilmenite concentrate with consequent separation of reduction products

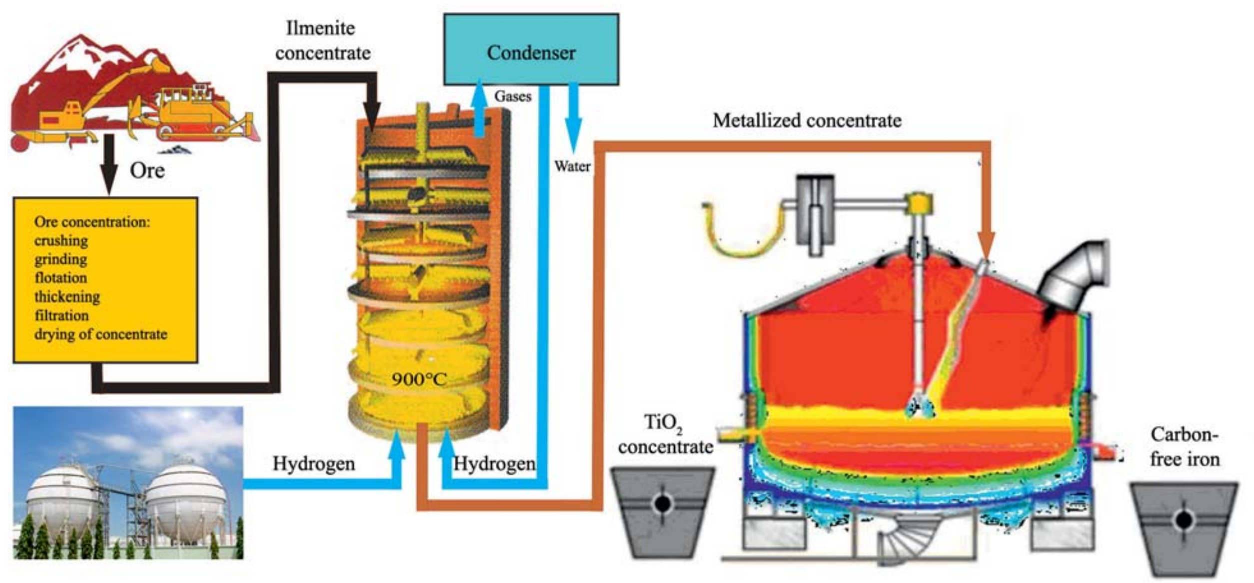

Fig. 5. Rational route for processing of ilmenite concentrates

to soft carbon-free iron and titanium dioxide concentrate. This process is carried out in a plasma furnace, while reduction is conducted in a multi-hearth furnace in hydrogen flow, with drying of waste gases and their returning in a reduction unit.

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