Study of possibility of manufacture of the complex titanium-containing ferroalloy via single-stage carbothermal method

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The object of research is the melting technology of a complex titanium-containing ferroalloy by single-stage slag-free carbothermal method using high-ash coals as a reducing agent. The ilmenite concentrate (Shokash deposit, Aktobe region, Kazakhstan), rich titanium slag, which was obtained by the scientists of the Chemical and Metallurgical Institute named after Zh. Abishev (Karaganda, Kazakhstan), as well as high-ash coal as a reducing agent were used as charge materials. Several physical-chemical characteristics of used charge materials were previously studied to carry out large-scale labour tests on the complex titanium-containing ferroalloy melting via single-stage slag-free carbothermal method. The chemical and phase composition of materials was established and two optimal compositions of the charge mixture for alloy melting were selected on their basis. The first composition contains: coal – 67.35 %; rich titanium slag – 26.12 %; quartzite – 6.5 %. The second composition includes^ coal – 66.95 %; rich titanium slag – 23.1 %; ilmenite – 3.3 %; quartzite – 6.61 %. Tests for melting of a new complex titanium-containing ferroalloy were conducted in the ore-smelting furnace with 0.2 MVA transformer power. As a result of the tests, a batch of new complex titanium-containing ferroalloy was obtained, with the following average chemical composition: Ti - 18-21 %; Si - 35-45 %; Al - 10-20 %; P is no more than 0.08 %, the rest is Fe. Also metallographic studies of the alloy sample were carried out in the work. The obtained alloy according corresponds in its titanium content to the brand FeTi25 (GOST 4761-91). The test results showed the principal possibility of obtaining a new complex titanium-containing ferroalloy from rich titanium slag and ilmenite concentrate using high-ash coal as a reducing agent.

Keywords: titanium-containing ferroalloy, complex ferroalloy, high-ash coal, rich titanium slag, ferrotitanium, carbothermal melting, ilmenite concentrate.
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Introduction

Titanium is very important material from the metallurgical point of view [1]. The main material which is used today for alloying, deoxidation and degassing of steel by titanium is ferrotitanium. More than 70 % of ferrotitanium global production is used for alloying of stainless and high-temperature steels. Titanium in steel is an active carbide-forming agent and can be added in small amount (0.02-0.5 %) mainly for carbon bonding in manganese, chromium, chromium-molybdenum and chromium-nickel stainless steels, what ensures elimination of intercrystalline corrosion, carbides and structure refining in steel castings [2]. Titanium addition in chromium cast iron leads to improvement of its wear resistance [3, 4]. Additionally, titanium compounds in steel plays three important functions: degassing, preventing grain growth during heating before rolling and forming of so-called strong frame for rise of metal strength properties [5-7]. It mainly predetermines both general titanium consumption and its production volumes. Low-alloyed steels containing titanium recently find the most wide application for manufacture of automotive parts via stamping. So, Severstal JSC, Novolipetsk Steel JSC and Magnitogorsk Iron and Steel Works JSC developed production of high-strength steels of JF grade, which are micro-alloyed by titanium, for fabrication of automotive bodies.

In Russian Federation ferrotitanium production is presented by such large inductrial enterprises as UralSpetsSplav JSC (Chelyabinsk), Volgovyatskvtortsvetmet JSC (Nizhniy Novgorod), PKF Okean (Berezovskiy), NPO Vtorproresurs (Moscow), VSMPO-AVISMA (Verkhnyaya Salda) and Klyuchevsky works of ferroalloys JSC (Dvurechensk). These companies cover maximal part of high-percentage ferrotitanium in Russia and other CIS countries. The key input in ferrotitanium export from Russia in natural expression is provided by VSMPO-AVISMA corporation (61.8 %) [8].

In Kazakhstan Republic, ferrotitanium is not manufactured. However, production of titanium containing concentrates (ilmenite, rutile) was started recently in Kazakhstan from local titanium ores. Mining and concentration of titanium-zirconium ores from Shokash deposit, (Aktobe region) is provided by Ekspoinzhiniring JSC. Manufactured products are bought mainly by VSMPO-AVISMA Corporation and «Roche Commodities Ltd» (China). Satpaevskoe mining and concentration plant (SGOP) realizes mining and processing of ilmenite sands at Satpaevskoe deposit (East Kazakhstan region) [5, 9]. Ore processing at Obukhovskoe titanium-zirconium deposit is provided at present time by Tiolain JSC. The most part of raw materials is supplied for production titanium sponge and slabs for aerospace and shipbuilding industries as well as for abroad deliveries.

Despite the wide raw materials base, UK TMK is the only manufacturer of titanium-containing products in Kazakhstan. This plant provides export of titanium sponge, titanium ingots and slabs in large volumes. But there is no works in Kazakhstan at present time for production of titanium-bearing ferroalloys, such as high- and low-percentage ferrotitanium silicotitanium etc. [9].

Kazakh enterprises, in particular ArcelorMittal Temirtau JSC, are forced to use alternative kinds of ferroalloys for manufacture of low- and medium-alloyed steels, owing to absence of domestic ferrotitanium in Kazakhstan; it does not allow to eliminate completely special steel defects. In this connection, dimension and grade range of high-strength steels reduces or is absent at all. The above-described situation causes necessity to realize the complex of scientific and research works for improvement of technological processes for production of titanium-containing ferroalloys with increase of their production volumes and import substitution for raw materials.

The scientists of the Chemical and Metallurgical Institute named after Zh. Abishev have previously developed the technology for production of titanium-rich slag (TRS) from Kazakh raw materials and proved possibility of its use in melting of ferrotitanium [10-12]. However, additional research works in this direction were not realized due to their multi-stage character and high cost of this technology. The authors suggested the technology for manufacture of titanium-containing ferroalloy from ilmenite concentrates and TRS via carbothermal method, using high-ash coal as a reducing agent. Possibility of use of high-ash Kazakh coal as a reducing agent was proved in several researches made by the Chemical and Metallurgical Institute specialists, and the complex alloys, such as alumosilicomanganese, ferrosilicoaluminium, ferroalumosilicocalcium, ferrochromium and ferroalumosilicochromium were obtained [13, 14].

Thereby, the aim of this work is conducting of the complex of scientific and research works in order to examine principal possibility of melting of complex titanium-containing ferroalloys from raw materials at Kazakhstan deposits via single-stage carbothermal method.

Methods and materials

Ilmenite concentrate of Shokash deposit was used in this work. Shokash is located in Martuk district of Aktobe region, Kazakhstan. Ore sands are characterized by leucoxene-rutile-zircon-ilmenite-quartzite composition [15, 16]. Domestic ilmenite concentrates can be raw materials for melting of saleable semiproduct – titanium-rich slag. In this connection, TRS which was molten via the technology developed in the Chemical and Metallurgical Institute named after Zh. Abishev [10, 11], was also used for melting of the new complex titanium-containing ferroalloy.

High-ash coal, which was used as a reducing agent, has the following technical composition: solid carbon 40-45 %; ash content more than 45 %; volatile substances: 10-14 %. Chemical composition of coal ash was as follows: SiO_2 – 60-64 %; Al_2O_3 – 25-35 %; Fe_2O_3 – 2-3 %; CaO – 8-10 %. Specific coal combustion heat was within the range 7.5-8.5 mln. kcal/kg.

Chemical composition of ilmenite concentrate from Shokash deposit and TRS were examined via spectral analytical method using vacuum wavelength-dispersive X-ray fluorescence spectrometer MAKS-GVM. Phase composition of materials was investigated via X-ray phase analysis at X-ray diffractometer Empyrean Malvern Panalytical, which is equipped with Cu-tube ($K_{\alpha 1}$ = 1.541874 Å). X-ray patterns were processed and decoded using Match!3 program and FullProf-2021 program database.

Experimental melting for manufacture of the new complex titanium-containing ferroalloy was conducted in the ore-smelting furnace with 0.2 MVA transformer power. This furnace is supplied with four stages for adjusting of secondary voltage – from 18 to 49 V. The furnace hearth is manufactured from electric conducting bottom mass, which is thoroughly compacted. The furnace lining was replaced by fireclay refractory bricks before the experiment, with filling of seams by fireclay powder. The temperature in a recreating area was provided by graphite electrode, via arc discharge.

To examine quality of the obtained new complex alloy, several investigations were conducted; they include analysis of chemical composition, analysis of phase composition using X-ray diffractometer Empyrean, as well as metallographic examinations of the alloy structure using scanning electron microscope JEOL-JSM7001F. This microscope is a unique device, because it calculates automatically chemical composition from a spectrum and makes it possible to determine a phase using chemical composition of the preset point. Chemical composition of the phases was determined via energy-dispersive spectrometer Oxford INCA X-max 80, which is mounted at the scanning microscope.

Obtained results and their analysis

The results of X-ray analysis of ilmenite concentrate are shown on the **Fig. 1** and in the **Table 1**.

The results of analysis testified that titanium is presented in concentrate by pseudo-rutile and ilmenite, while chromium is presented by a mineral of spinel group, i.e. by chromite. The basic iron-titanium-containing mineral (ilmenite) is mainly replaced by pseudo-rutile as a result of secondary processes. The sum of ore minerals made 95-96 %; where about 1.5-2.0 % are presented by zircon and other part – by non-ore minerals.

The results of TRS examinations via X-ray phase analysis are displayed on the **Fig. 2** and in the **Table 2**. These data testify that TRS can be related to spinel-anosovite type. Presence of anosovite solution means over-reduction state

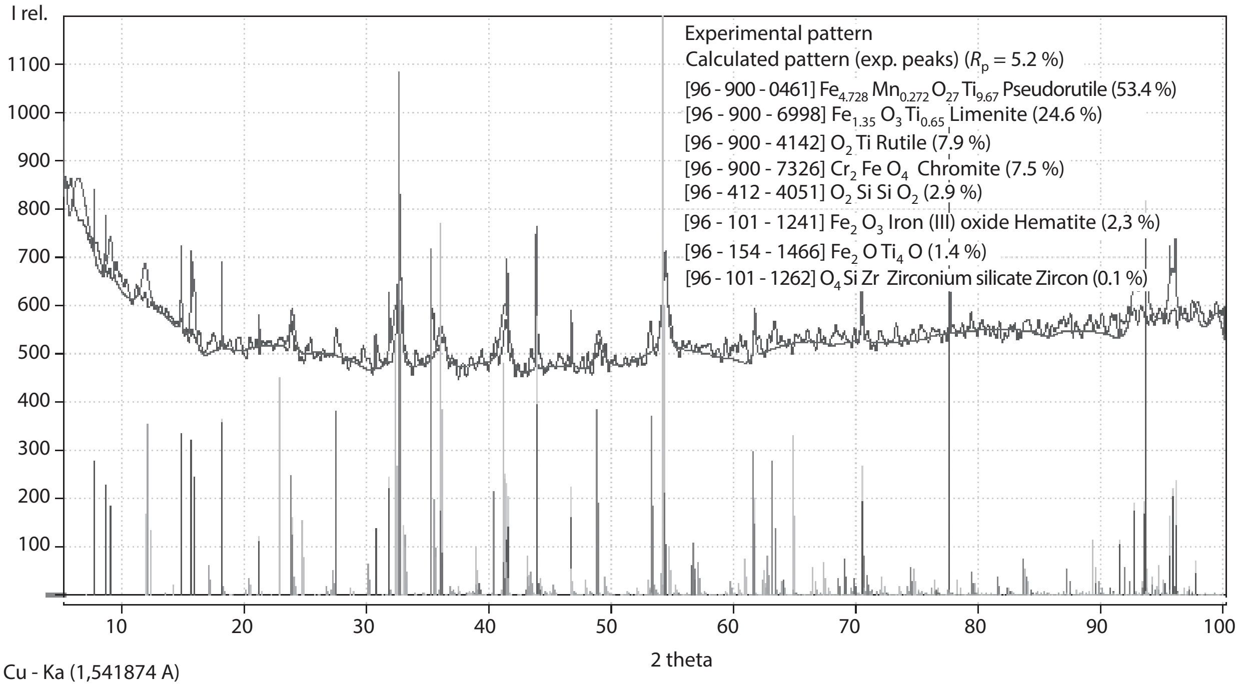


Fig. 1. Diffraction pattern of ilmenite concentrate

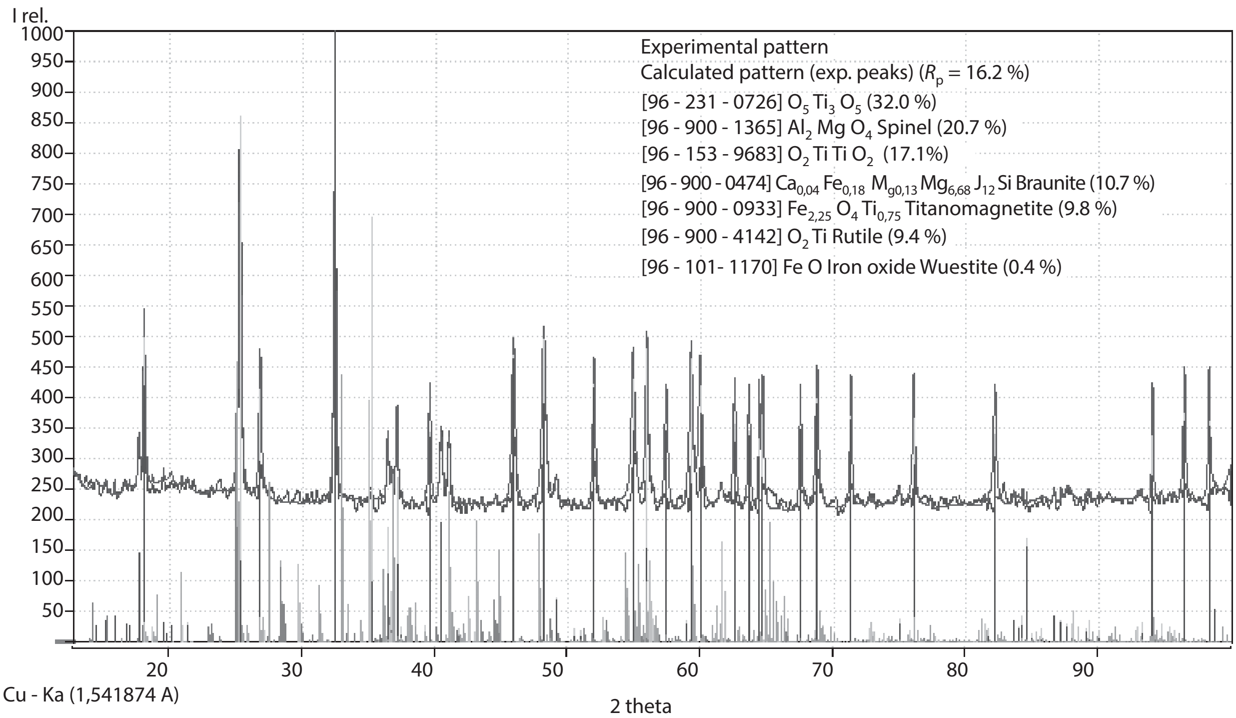


Fig. 2. Diffraction pattern of titanium-rich slag (TRS)

Table 1. **The results of X-ray phase analysis of ilmenite concentrate**

Mineral name	Formula	Mass part	Identification number in the database
Pseudo-rutile	$Fe_2Ti_3O_9$	53.4	96-900-0461
Ilmenite	$FeTiO_3$	24.6	96-900-6998
Rutile	TiO_2	7.9	96-900-4142
Chromite	$FeCr_2O_4$	7.5	96-900-7326
Quartzite	SiO_2	2.9	96-412-4051
Iron trioxide (hematite)	Fe_2O_3	2.3	96-101-1241
Ulvospinel	Fe_2TiO_4	1.4	96-154-1466
Zircon	$ZrSiO_4$	0.1	96-101-1262

Table 2. **The results of X-ray phase analysis of titanium-rich slag (TRS)**

Mineral name	Formula	Mass part	Identification number in the database
Anosovite	Ti_3O_5	32.0	96-231-0726
Spinel	$MgAl_2O_4$	20 7	96-900-1365
Anatase	TiO_2	17 1	96-500-0224
Brounite	$CaMn_{14}+3SiO_{24}$	10 7	96-900-0474
Armacolite	$Fe_{0,5}Mg_{0,5}Ti_2O_5$	9 8	96-900-0933
Rutile	TiO_2	9 4	96-900-4142
Wustite	FeO	0.4	96-101-1170

Table 3. **Chemical composition of the materials in the charge mix**

No. of composition	Material	Part of materials in composition of charge mix, %	Chemical composition of materials, %									
			TiO_2	Al_2O_3	SiO_2	Fe_2O_3	Cr_2O_3	MnO	P	V_2O_5	MgO	CaO
1	Coal	67.35	1	26.85	60.32	2.31	0.2		0.15	-	0,1	8.99
	RTS	26.12	59.09	4.41	4.14	14.33	1.42	3.59	0.01	9,73	2.89	0.28
	Quartzite	6.5	-	-	98	-	-	-	0.11	-	-	-
2	Coal	66.95	1	26.85	60.32	2.31	0.2	0	0.15	-	0.1	8.99
	RTS	23.13	59.09	4.41	4.14	14.33	1.42	3.59	0.01	9,73	2.89	0.28
	Concentrate	3.3	58.8	4.52	3.34	28.38	3.33	1.37	0.09	-	-	-
	Quartzite	6.61	-	-	98	-	-	-	0.11	-	-	-

of TRS; remained titanium is presented by spinel, armacolite, brounite, rutile and wustite, as it can be seen from the X-ray pattern.

The results of spectral chemical analysis of materials are presented in the **Table 3**. Composition of the charge mix was then calculated. It is known that titanium is a strong carbide-forming material, thereby it is necessary to have fluid alloy on the base of silicon and iron for normal operation of an ore-smelting furnace during melting of titanium-containing alloys. As a result, two optimal compositions of charge mixes, using ilmenite concentrate, rich titanium slag and quartzite, as well as high-ash coal as a reducing agent, were selected for melting of the new complex titanium-containing ferroalloy:

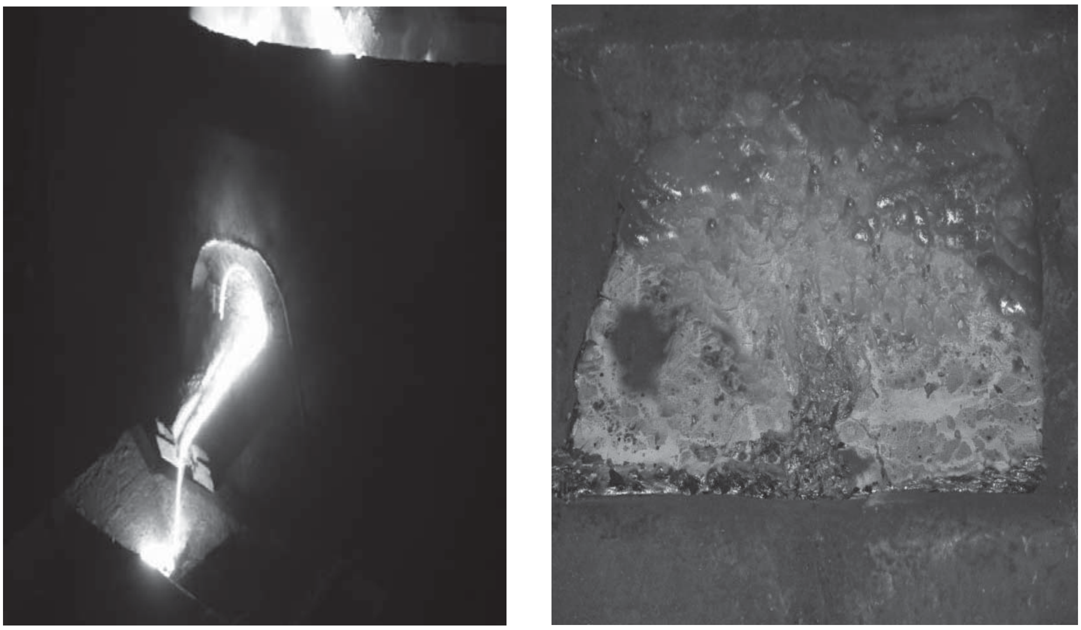
- carbonaceous reducing agent – high-ash coal with ash content 45 %, its part in the charge mix makes 67.35 %; TRS, its part in the charge mix makes 26.12 %; quartzite, its part in the charge mix makes 6.5 %. Decrease of quartzite (SiO_2) part in general charge mix (less than by 6.5 %) can lead to forming of too previous titanium carbide, what will violate seriously melting practice in an ore-smelting furnace. Thereby quartzite part in the ore mix should be not less than 20 %;

- carbonaceous reducing agent – high-ash coal with ash content 45 %, its part in the charge mix makes 66.95 %; TRS, its part in the charge mix makes 23.13 %; ilmenite concentrate, its part in the charge mix makes 3.3 %; quartzite, its part in the charge mix makes 6.61 %. According to the GOST 4761-91, phosphorus content in titanium-containing ferroalloy of FeTi25 grade should not exceed 0.08 %. Ilmenite concentrate is the main source of phosphorus appearance (P_2O_5 part in the ore mix makes 82 %), that's why increase of the concentrate part (more than by 10 % in the ore mix) can finalize in alloy production with high phosphorus content, i.e. to metal reject.

Testing for melting of the complex titanium-containing ferroalloy was conducted in the ore-smelting furnace with 0.2 MVA transformer power. Charge of the 1st composition was introduced in heated furnace bath by small batches. Furnace top throat operation was characterized by homogeneous gas emission from complete surface without knot holes. The notch was opened via electric burning. Metal was poured every two hours in iron moulds. Furnace operation on the charge with 1st composition was uniform and steady, and characterized by deep location of electrodes. Transition to the charge with 2nd composition was realized with addition of ilmenite concentrate in the charge mix. Operating voltage was elevated up to 36 V. Transition to the charge with 2nd composition was not accompanied by any dramatic deviations and titanium carbide forming was not observed. The consequent process was stable. Metal pouring was conducted according to the regular plan, every two hours, in iron moulds (**Fig. 3**).

After cooling the metal was mixed. Then metal samples were taken to determine its chemical composition, which is presented (for different batches) in the **Table 4**.

The results of X-ray phase analysis of the obtained alloy are displayed on the **Fig. 4** and in the **Table 5**. Examination of phase composition showed, that



a *b*

Fig. 3. Pouring (*a*) and cooling (*b*) of the ingot of titanium-containing alloy

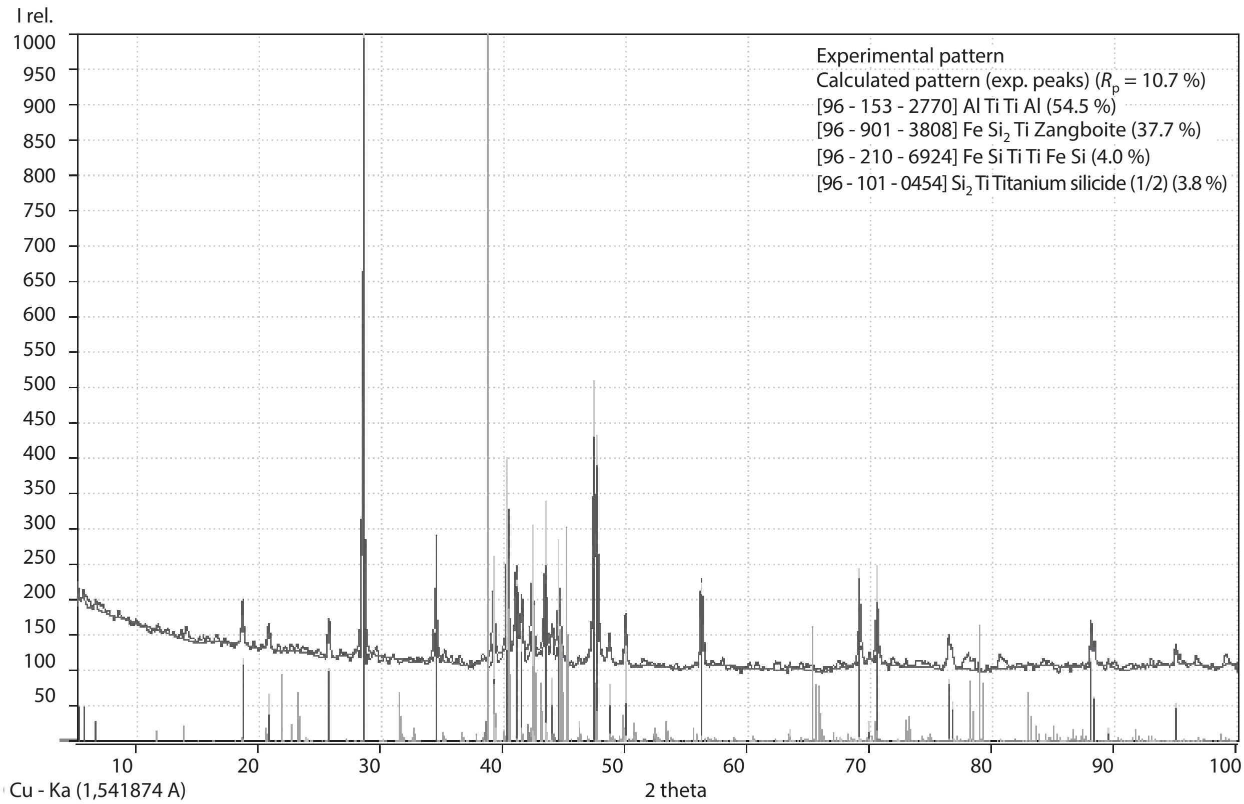


Fig. 4. Diffraction pattern of the obtained alloy

the active elements in the alloy (Si, Al, Fe and Ti) are presented by complicated intermetallics, such as TiAl, $TiFeSi_2$, FeSiTi, $TiSi_2$.

Structure of the ferroalloy sample was subjected to metallographic examination, and composition of spectra of the examined pilot complex alloy was revealed. Microstructure of titanium-containing ferroalloy, which was researched on scanning electron microscope JEOL-JSM7001F, is displayed on the **Fig. 5**.

Table 6 presents the results of spectra chemical analysis in weight and atomic percents.

Table 4. **Chemical composition of the obtained metal**

Pouring No.	Metall, %					
	Al	Fe	Si	C	Ti	Ba
1	9.57	13.56	46.2	1.12	16.6	0.14
2	10.58	40.03	37.06	0.6	17.84	0.09
3	10.42	33.94	34.54	1.39	18.9	0.26
4	10.22	24.66	34.67	3.62	16.29	0.29
5	9.25	15.3	44.7	0.51	21.54	0.16
6	8.33	15.44	53.82	0.35	20.99	0.14
7	9.88	19.53	44.51	0.97	18.45	0.27

Table 5. **The results of X-ray phase analysis for obtained complex titanium-containing ferroalloy**

Formula	Mass part	Identification number in the database
TiAl	54,5	96-153-2770
$TiFeSi_2$	37,7	96-901-3808
FeSiTi	4,0	96-210-6924
$TiSi_2$	3,8	96-101-0454

Metallographic analysis displays that the obtained complex titanium-containing ferroalloy is presented by three main structural components, which are characterized by different tones: white as impregnation of points, grey occupies the main form square, dark grey is a matrix.

Spectra 76, 88 and 89 are presented by dark-grey colour, these phases are characterized by presence of pure silicon (99.7-100 %), what is identified as structurally pure silicon (Si). The phases which occupy the main square are presented by spectra 75, 78, 80 and 83 with grey colour. Chemical composition of these spectra is characterized by four-component compound of Si, Ti, Al and Fe. The phases which are noted by spectra 77, 79, 81, 84, 85 and 87 are presented by white impregnations and are decoded as intermetallics Fe, Si and Al. Microstructure of the alloy also displayed presence of dual-component compounds Ti and Si – TiSi and $TiSi_2$.

Conclusions

Possibility of manufacture of the new complex titanium-containing ferroalloy via carbothermal method from Kazakhstan raw materials was proved on the base of experimental and research works in the large-scale laboratorial conditions. Two optimal compositions of charge mix were selected for conducting of pilot testing: titanium-rich slag (TRS), ilmenite concentrate and high-ash coal. The first composition includes coal (67.35 %), TRS (26.12 %) and quartzite (6.5 %). The second composition includes coal (66.95 %), TRS (23.1 %), ilmenite (3.3 %) and quartzite (6.61 %). The pilot batch of the new complex titanium-containing ferroalloy was obtained during the tests, with the following average chemical composition: Ti 18-21 %; Si 35-45 %; Al 10-20 %; P not more than 0.08 %, the rest is Fe. This alloy corresponds

Table 6. **The results of spectra chemical analysis in weight and atomic percents**

Spectrum	Content of elements, weight %					Content of elements, atomic %				
	Al	Si	Ti	Mn	Fe	Al	Si	Ti	Mn	Fe
75	17.5	46.8	20.7	1.7	13.3	21.5	55.2	14.3	1.0	7.9
76	-	99.7	-	-	0.3	-	99.9	-	-	0.1
77	19.2	75.9	-	-	4.0	20.3	77.0	-	-	2.0
78	10.2	34.3	23.2	3.6	28.7	14.2	45.8	18.2	2.5	19.3
79	20.0	78.5	-	-	1.5	20.8	78.4			0.8
80	4.0	34.1	28.2	3.7	30.0	5.8	47.5	23.0	2.7	21.0
81	37.0	34.6	-	2.4	26.1	44.0	39.6	-	1,4	15.0
82	0.5	54.6	45.0	-		0.6	67.0	32.4	-	-
83	16.1	29.0	22.6	1.3	31.0	22.3	38.5	17.6	0.9	20.7
84	19.4	79.3	-	-	1.3	20.2	79.1	-	-	0.6
85	36.6	34.8	-	2.2	26.4	43.6	39.8	-	1.3	15.2
86	0.4	54.5	44.9	-	0.3	0.5	67.0	32.4	-	0.2
87	34.4	39.6	-	3.1	22.9	40.5	44.7	-	1.8	13.0
88	-	100.0	-	-	-	-	100.0	-	-	-
89	-	100.0	-	-	-	-	100.0	-	-	-
Max	37.0	100.0	45.0	3.7	31.0	44.0	100.0	32.4	2.7	21.0
Min	0.4	29.0	20.7	1.3	0.3	0.5	38.5	14.3	0.9	0.1

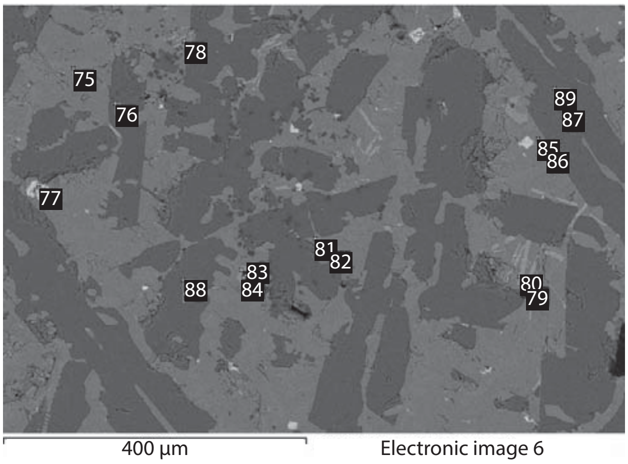


Fig. 5. Structure No. 1 of three obtained ferroalloy, magnification x100

in its composition to the grade FeTi25 (GOST 4761-91). Metallographic tests showed that phase composition of a pilot sample is presented by complicated intermetallics, such as TiAl, $TiFeSi_2$, FeSiTi, FeSiAl и $TiSi_2$. The results of tests displayed principal possibility to obtain the new complex titanium-containing ferroalloy from TRS and ilmenite concentrate using high-ash coal as a reducing agent. Necessity of mandatory inclusion of quartzite in charge composition for the case of use of this sort of high-ash coal was revealed for different composition of charge materials.

Therefore, the obtained data make it possible to use ilmenite concentrate, rich titanium slag and high-ash coal (as a reducing agent) in the process of melting od complex titanium-containing ferroalloys as the main charge materials. It can be concluded on the base of analysis of operating results, that development of complex and resource-saving melting technology for analogues of standard ferroalloys grades has started.

The obtained data also allow to make preliminary conclusion about possible economical and technological expedience for manufacture of titanium-containing ferroalloys using non-conventional reducing agents, such as high-ash coals, instead of expensive aluminium. Carrying out of additional technical and economical investigations and putting into practice this technology for melting of high-quality steels are required for more exact establishment of its efficiency.

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