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Composition and Structural Characteristics of Rare Metal Ores of the Chuktukon Deposit

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Abstract

Rare metal ores of the Chuktukon deposit (Krasnoyarsk Territory) were explored. It was found that the ores were complex, mainly of iron-manganese composition (iron oxides content of 50 % or above), with high contents of lanthanides, yttrium, niobium, vanadium, zinc, and phosphorus. The main mineral forms of rare earth metals are monazite and florencite, of niobium – barium-strontium pyrochlore. As demonstrated by granulometric composition study, above 50 % of ore mass is presented by a particle size of mainly 1–2 μm and $\sim 12 \mu\text{m}$. As a rule, elements are almost evenly distributed according to all size grades. According to microscopic study results, the ore is mainly formed by ultrafine micrometre-sized particles (the first micrometres) that, in turn, are nanoscale particle aggregates. The findings explain the complexity of excreting concentrates of rare earth metals and niobium from the ore using enrichment methods.

Keywords: Chuktukon deposit, rare metal ores; rare earth metals, niobium.

INTRODUCTION

According to the content of valuable metals and their reserves, the Chuktukon deposit of niobium-rare-metal ores is one of the most important objects for modernizing the rare-metal industry of Russia [1–3]. The deposit is located 110 km north to the Boguchanskaya hydroelectric power station (Boguchansky District, Krasnoyarsk Territory). It is timed to the cognominal dome of the Chadobets uplift of the Siberian platform and belongs to the ore-forming type of rare-metal crusts of weathering carbonatites associated with small bodies of alkaline ultrabasic rocks. The ore type is fine pyrochlore-monazite ores in laterite weathering crusts. The area of carbonatite weathering crust is relatively small and is 16 km², its power reaches

300 m naturally increasing to the vault of the dome structure. The lower and upper limits of the weathering crust are uneven.

In 2007, Russia's State Commission on Mineral Reserves approved reserves of balance rare earth ores in the C₂ category in the amount of 6.6 million t, with an average Nb₂O₅ content of 0.60 %, rare earth metal oxides (REMO) of 7.32 %, MnO₂ of 15.5 %, Fe_{tot} of 34.3 %. The inferred resources of ores at least by an order of magnitude exceed the approved reserves. It is expected that already by 2020, they will increase to 33.7 million t with REMO content of 6.14 % [4]. In 2015–2017, the company Rosgeologiya continued the works on additional exploration of the Chuktukon deposit. Their accomplishment will soon allow beginning the practical development of this unique Russian object.

As demonstrated by earlier studies [4–10], the main problem of processing Chuktukon deposit ores is related to the impossibility of their enrichment by traditional methods that, in our opinion, is due to fine dispersion of ore and close concrescence of its mineral particles. At the same time, new methods of enrichment and separation of such ores, in particular, using centrifugal devices, are currently being developed.

In this regard, the exploration of the composition and structural characteristics of Chuktukon deposit ores, and also the principal possibility of their enrichment are relevant in case of the appearance of new methods for separation of ultrafine materials.

EXPERIMENTAL

The work used chemically pure mineral acids (hydrochloric and nitric) and sodium hydroxide. The initial process sample of ore (TP-6) was made of counterparts of core samples of wells and assays of short wells (1999, Krasnoyarsk Research Institute of Geology and Minerals, G. V. Lomaev). Table 1 gives the chemical composition of averaged samples of ore of the Chuktukon deposit; prior to analysis, the sample was dried at 105–110 °C.

Particle size distribution for a size below 40 μm was explored using LA-300 laser diffraction particle analyser (Horiba). The particle size was determined using dynamic light scattering spectroscopy, simultaneously with measuring the Zeta potential using the

Zetasizer Nano ZS instrument. Ore disperse composition was defined in aqueous and slightly alkaline (pH ~ 8) suspensions. Electron-microscopic studies were carried out using S5500 and TM-3000 scanning electron microscopes (Hitachi) equipped with built-in energy dispersive spectrometers for electron microprobe analysis. To determine the composition of the main morphological types of particles, image elemental mapping was performed using Noran NSS-7 (ThermoFisher Scientific, USA) energy dispersive spectrometer integrated into TM-3000 (Hitachi) scanning electron microscope.

X-ray diffractograms registering was carried using the Dron-3 X-ray diffractometer (“Petrel”, Russia) in the $2\theta = 5\text{--}70^\circ$ angle range.

Solid products composition was determined using X-ray fluorescence analyzer (PANalytical Axios Advanced).

RESULTS AND DISCUSSION

Ore composition

Ores are complex, mainly of iron-manganese type, Fe_2O_3 content varies within the deposit from 38.0 to 71.4 %, MnO – from 1.2 to 19.7 %; concentrations of barium of 0.6–14.5 % (based on BaO), P_2O_5 1.2–27.0 %, Nb_2O_5 0.2–4.85 %, lanthanides 1.8–30.4 %, Y 0.7–2.3 %, Sc 0.005–0.05 %, Mo to 0.1 %, V 0.1–0.4 % (V_2O_5), Zn 0.2–1.5 %. Ores are weakly radioactive because of the presence of uranium and thorium in minor amounts.

Small trial material is presented everywhere by dark-colored ochres and small-leaved fries with a gradation of colours from almost black to greenish-gray with a predominance of dark colors, which corresponds to a high content of iron oxides in the form of Fe_2O_3 (30–50 %) and manganese oxides (to 10 %). Samples of yellowish and light brown shades also turned out to be highly ferriferous (30–40 % of Fe_2O_3).

According to X-ray phase analysis data, the main components of a representative sample of ore are the following: iron oxides hematite ($\alpha\text{-Fe}_2\text{O}_3$) and goethite ($\text{FeO}(\text{OH})$, $\text{FeO}_{1.833}(\text{OH})_{0.5}\text{O}_{2.5}$), the manganese minerals, psilomelane, pyrolusite ($\text{Ba}_x\text{Mn}_2\text{O}_{10} \cdot 2\text{H}_2\text{O}$), MnO_2 , $(\text{Fe}_{0.67}\text{Mn}_{0.33})\text{O}(\text{OH})$, rare earth metal (REM) phosphates (monazite (LnPO_4) and florencite ($\text{LnAl}_3(\text{PO}_4)(\text{OH})_6$)), and apatite. Along with the main minerals, there

TABLE 1

Sample composition of initial ore of Chuktukon deposit

Oxide	Content, mass %
Al_2O_3	4.60
BaO	1.70
CaO	1.00
Fe_2O_3	61.00
$\Sigma\text{Ln}_2\text{O}_3$	7.10
MgO	0.14
MnO_2	11.00
Nb_2O_5	1.30
P_2O_5	3.80
PbO	0.06
SO_3	0.38
SiO_2	4.50

were found niobium minerals, pyrochlore $(\text{CaNa})_2(\text{Nb,Ti})_2\text{O}_6\text{F}$ and in small quantities hydroxyphosphate iron giniite $(\text{Fe}_5(\text{PO}_4)_4(\text{OH}) \cdot 2\text{H}_2\text{O})$ and siderite (FeCO_3) , the fraction of which does not exceed 3 % (determined by the amount of carbon dioxide released during acid decomposition of ore).

Sample calcination to temperatures of 1200 °C is accompanied by a mass loss by about 11 %. The first diapason of mass decrease (to 6 %) is associated with the 200–250 °C temperature range. The process is accompanied by the endothermal effect and is probably due to $\alpha\text{-FeOOH} \rightarrow \alpha\text{-Fe}_2\text{O}_3$ transition that appears at 243 °C. continuous mass loss as further temperature increase to 1100–1200 °C, is probably related to the decomposition of carbonates, and also to dehydration of hydroxide forms of minerals (goethite, florencite, and other aluminophosphates) and degradation of higher manganese oxides (MnO_2 and Mn_2O_3).

Granulometric composition characteristics of ore of the Chuktukon deposit

Ore is by over 50 % presented by 3–5 mm wet frail loose pieces consisting of fine particles: filling pieces fall apart with minor mechanical impact. To carry out sieve analysis, the ore was preliminarily exposed to weak decomposition in a laboratory grinder. Afterwards, to 100 g of the ore, 500 mL of water was added, the mixture was aged for several hours, and divided according to size classes using sieves with a size of 500, 160, 80, and 40 μm , and the resulting fractions were dried at 150 °C.

Figure 1 gives the data on particle size distribution. It can be seen that more than 50 % of the particles belong to the size class less than 40 μm and about 90 % – less than 160 μm (mass fraction below 40 μm – 54 %, +40 μm – 16.9, +80 μm – 14.2, +160 μm – 11.3 %). Table 2 gives the elemental composition of the fractions.

As it follows from the findings, the prevailing part of elements are distributed fairly evenly by size classes, from –40 to +160 μm with a minor increase of silicon and aluminium contents and manganese amount decrease in the fine fraction. The contents of REM and niobium at a fraction to 40 μm increase to 1.3 and 2 times, respectively. At the same time, when the fraction of 54 % is released, the total

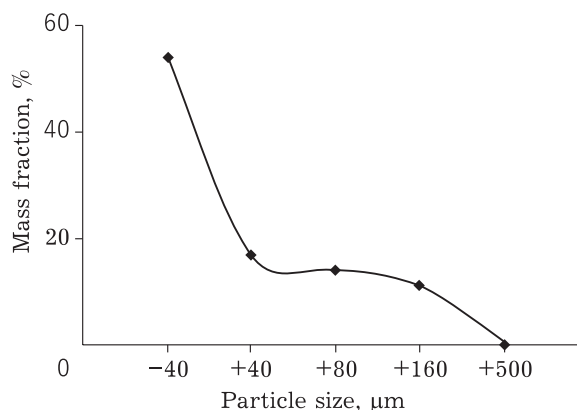


Fig. 1. Particle size distribution of ore of Chuktukon deposit according to sieve analysis data.

amount of REM in it is about 60 %, and niobium – 70 %, which is not enough for the use of gravitational methods of Chuktukon deposit ores.

Figure 2 gives particle size distribution acquired using LA-300 laser diffraction particle analyser (Horiba) with different durations of ore grinding in a ball mill. There are two well-defined maxima with a particle size of ~2 and

TABLE 2

Sieve analysis results of ore sample of Chuktukon deposit

Oxide	Content, %, in fractions, μm			
	-40	+40	+80	+160
	Al_2O_3	6.30	5.40	5
BaO	1.70	1.70	2.00	2.20
CaO	0.87	0.95	1.10	1.20
Ce_2O_3	2.60	2.00	2.00	1.80
Fe_2O_3	60	62	61	62
La_2O_3	1.90	1.40	1.50	1.30
MgO	0.15	0.17	0.13	0.13
MnO_2	9.90	11	12	12
Nb_2O_5	1.90	1.20	1.00	0.85
Nd_2O_3	1.00	0.83	0.82	0.81
P_2O_5	4.30	4.30	4.30	4.30
Pr_2O_3	0.28	0.21	0.32	0.24
SO_3	0.61	0.58	0.69	0.93
SiO_2	5.10	5.40	4.90	4.60
SrO	0.54	0.51	0.50	0.29
ThO_2	0.15	0.11	0.10	0.09
TiO_2	1.30	1.30	1.20	1.10
V_2O_5	0.26	–	0.29	0.28
Y_2O_3	0.21	0.18	0.18	0.17
ZnO	0.77	0.75	0.87	0.96

~12 μm in the distribution curve; moreover, the first maximum substantially increases, while the second one decreases with grinding time increase.

The sizes of the particles in the bleached suspension in settling the crushed ore are determined by the method of dynamic light scattering spectroscopy, simultaneously with the measurement of the Zeta potential. The results of the measurements (Fig. 3) demonstrate the unimodal distribution of the hydrodynamic particle diameter with a maximum in the region of just under 1 μm after 10 min of settling, the position of which slowly shifts towards smaller values during settling, reaching 700 nm after 50 min. It worth noting that the measurements are complicated by the presence of larger particles formed during aggregation. The measured zeta potential for a pH value of ~6 naturally created in a water suspension is -18 mV, and this value is steady in time.

Using the hydrodynamic method, the ore fraction with a size of ~40 μm was divided into two products containing mainly particles with an average size of 1–10 μm (product yield of 8 %) and 10–40 μm (92 %). Table 3 gives the data for the content of the main components in these fractions. It can be seen that the compositions differ slightly, therefore, it one may assume the aggregation of particles of a

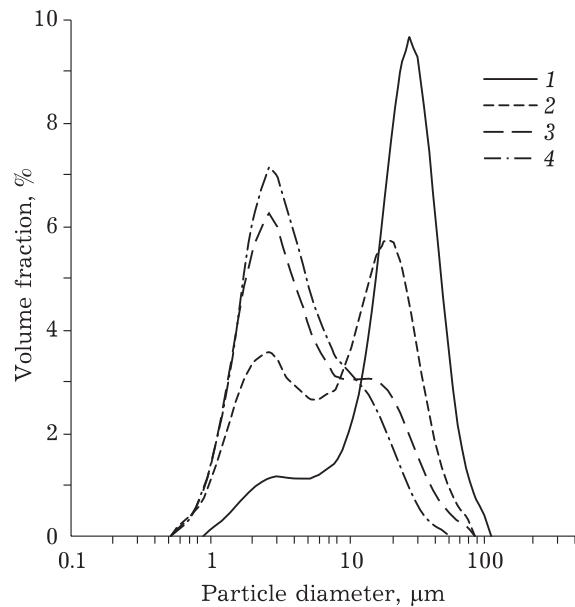


Fig. 2. Particle size distribution of ore of Chuktukon deposit according to laser diffraction data. 1 – after breaking ore lumps in laboratory grinder, 2–4 – after wet grinding in a drum mill for 0.5 (2), 1 (3), and 1.5 h (4).

size of ~2 μm with generating larger globules with an average size of 12 μm .

To increase particle separation efficiency, the ore was treated with a NaOH solution,

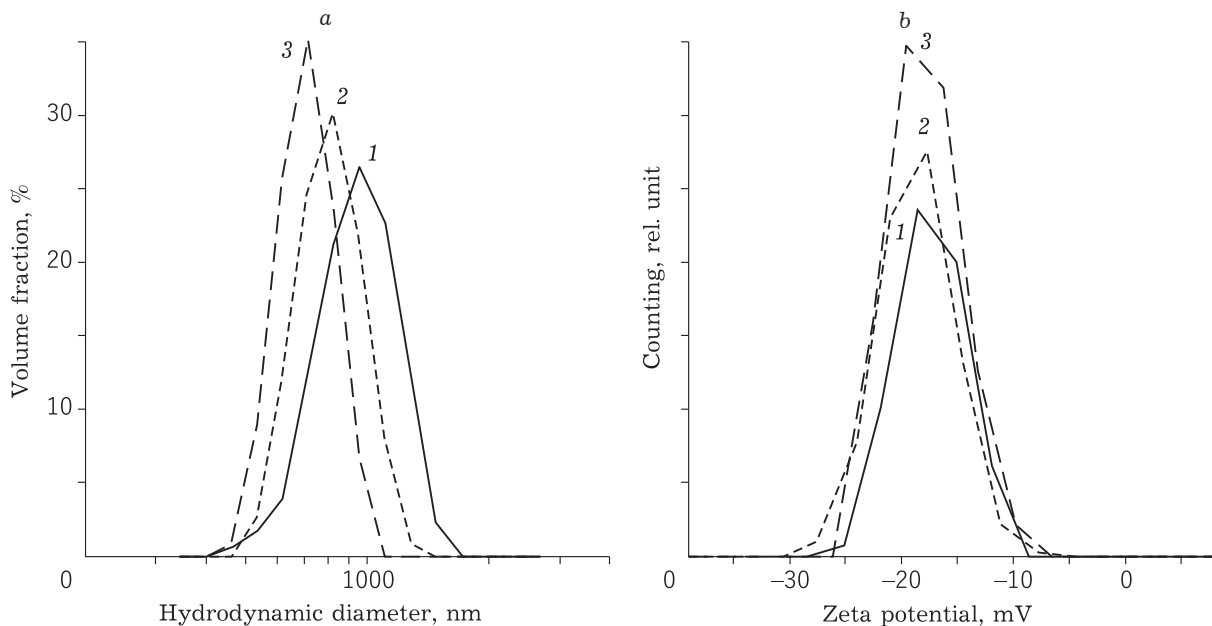


Fig. 3. Hydrodynamic diameter (a) and Zeta potential (b) of colloidal particles after settling pulp of ore of Chuktukon deposit for 10 (1), 20 (2), and 50 min (3).

TABLE 3

Composition of ore fractions isolated by washing out $-40 \mu\text{m}$ fraction by water

Oxide	Content, %, in fraction, μm	
	10-40	0-10
Al_2O_3	5.90	6.60
BaO	1.70	1.50
CaO	0.90	1.10
Ce_2O_3	2.70	2.40
Fe_2O_3	63.00	60.00
La_2O_3	1.90	1.80
MgO	0.12	0.13
MnO_2	10.00	9.70
Nb_2O_5	1.90	2.10
Nd_2O_3	0.94	1.10
P_2O_5	2.90	3.30
Pr_2O_3	0.33	0.31
SiO_2	4.80	5.40
SrO	0.33	0.64
ThO_2	0.15	0.16
TiO_2	1.30	1.30
ZnO	0.83	0.84

bringing the reaction medium to pH 8, more favourable for particle aggregation prevention. Herewith, the NaOH consumption was about 0.1 mol per 1 kg of ore. Apart from that, experimental conditions were the same: 100 g of ore was processed in 500 mL, the reaction medium was brought to pH 8 with a sodium hydroxide solution, the ore was separated on 500, 160, 80 and 40 μm sieves; the fractions were dried at 150 °C, weighed and calcined at 650 °C for X-ray fluorescence analysis.

A more remarkable increase (from 54 to 62 %) of a particle fraction of 40 μm was determined. As demonstrated by analysis of the composition of the separated fractions, the compositions of small and large particles differ to a greater extent compared to the previous experiment (sieve separation of ore in water). This applies mainly to niobium and REM. As a whole, most elements are distributed fairly evenly between fractions.

The results of exploring particle size distribution for the $-40 \mu\text{m}$ fraction by laser diffraction are also comparable with those acquired earlier. Using the hydrodynamic

method, a fraction size of $-40 \mu\text{m}$ is divided into two parts, *i.e.* large (35 % yield) and thin (45 %). As in the previous case, the compositions differ slightly, only in the thin fraction, the contents of aluminum and phosphorus significantly increase (Table 4).

The findings attest to exceptionally high dispersion of Chuktukon deposit ores and a close association of REM minerals with rock-forming ones, primarily, with iron oxides (gothite and hematite), which greatly complicates high yield isolation of concentrates of rare metals (REM or niobium) by gravitation methods.

Ore exploration using electron microscopy

The studies of ore structure using electron microscopy have been continued [6].

Microscopic study results confirm the conclusions about the prevalence of fine mineral particles. The initial ore sample is mainly presented by particles of the micro- (the first micrometres) and nanometer (less

TABLE 4

Composition of fractions isolated by $-40 \mu\text{m}$ ore washing with weakly alkaline solution

Oxide	Content, %, in fraction, μm	
	10-40	0-10
Al_2O_3	2.90	5.10
BaO	1.60	1.60
CaO	0.72	0.82
Ce_2O_3	2.20	2.60
Fe_2O_3	68.00	63.00
K_2O	0.11	0.11
La_2O_3	1.70	1.90
MgO	0.14	0.14
MnO_2	10.00	9.50
Nb_2O_5	2.30	2.00
Nd_2O_3	0.83	1.20
P_2O_5	2.80	4.50
Pr_2O_3	0.26	0.32
SiO_2	2.50	3.40
SrO	0.26	0.67
ThO_2	0.12	0.15
TiO_2	1.50	1.30
V_2O_5	0.29	0.26
Y_2O_3	0.28	0.21
ZnO	0.78	0.82

than 10 nm) range. Globules with a size to several tens of micrometres are loose porous structures composed of thin (by an order) particles. Micrometre species are also not monolithic, and are dense, closely linked aggregates, probably with a semiporous structure (Fig. 4). Their constituent particles are nanoscale.

Nanodust with a diameter of below 3 nm covers the surface of larger particles (1–3 μm) and shades their structure. After washing-off (elutriation), the morphology and surface structure of micrometre particles become clear. Morphologically, the particles can be divided into needle disordered and ordered fragmental assemblies (also 1–2 μm in size), loose rounded aggregates of oval fine particles or flakes, dense chip species (5–6 μm) and rare dendritic aggregates (1–3 μm) collected from small spherical globules (10–20 nm).

Treatment of ore samples with a solution of hydrochloric acid and EDTA (dissolution to 10 % of oxides mass) also leads to the disappearance of nanodust, and microscopic

images of particles became clearer. Herewith, the overall picture did not change, and the typical sample was mainly presented by iron oxide particles of a size of 1–2 μm , massive ones or aggregations of smaller species (50–10 nm).

As a whole, ore particles are composed of iron oxides and are of clearly hydrothermal origin. Manganese is mainly present in the scattered state. Individual particles with a high content of manganese oxides are agglomerates of needle-shaped particles with a crystal thickness of 50–60 nm. They are associated with silicon and barium and have patches of iron oxide particles (100–200 nm) (Fig. 5).

Loose aggregates of round particles or flakes are presented by iron oxides associated with manganese and silicon, and dense fragments – mainly, with calcium phosphate (apatite) and associated with silicon, carbon, and possibly fluorine. Individual particles containing niobium or REM are extremely rare.

Mineral REM particles (see Fig. 4 and 6) have a shape of complex aggregates. Some of

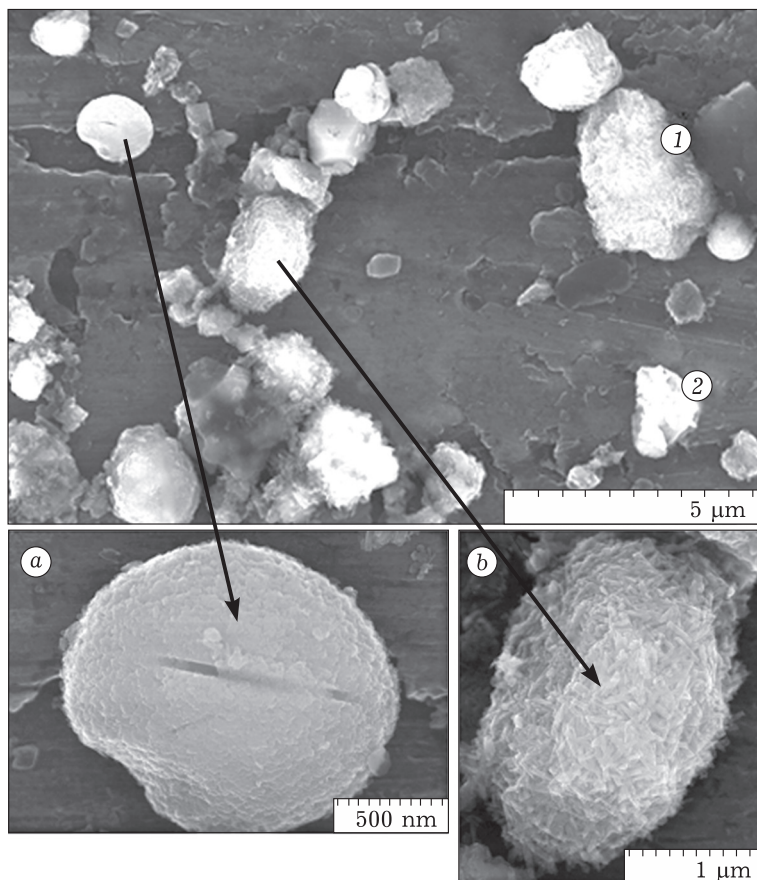


Fig. 4. Cerium-neodymium-lanthanum-oxide particles (1, 2) surrounded by iron oxide species of different morphology.

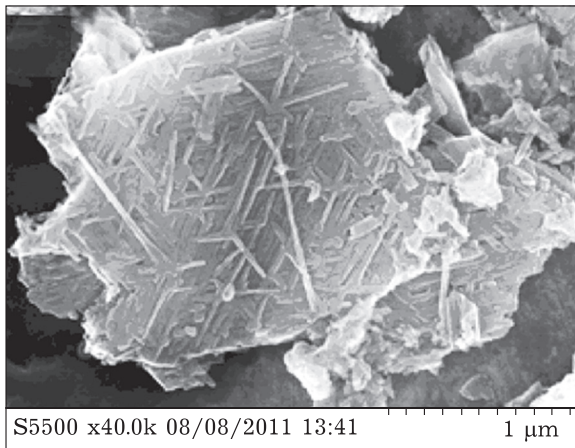


Fig. 5. Ordered needle aggregates of manganese oxides and hydroxides.

them are fully disclosed and others are found inside of iron oxide aggregates. During decomposition of REM phosphates in a solution of nitric acid (4 mol/L) at 200 °C, when the main mineral forms of iron were not dissolved [10], it was found that iron oxide particles were embedded with florencite, while monazite was disclosed and, according to XPA data, completely decomposed. The fraction of undisclosed REM minerals was about 15 % of the total amount.

Niobium oxides are rare, as barium and barium-strontium pyrochlore. Unlike other minerals, pyrochlore is much weaker bound with iron oxides and hydroxides.

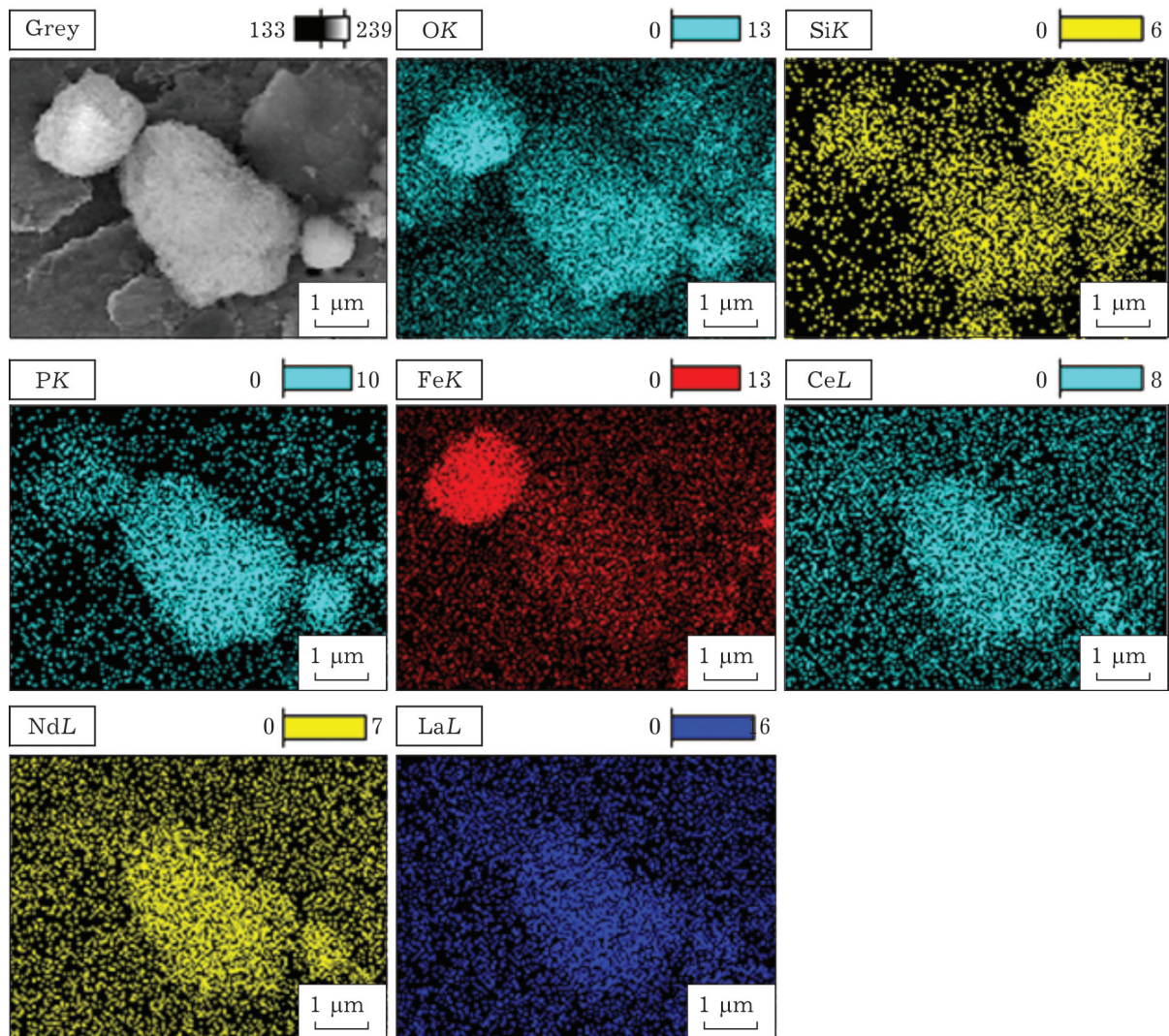


Fig 6. Results of elemental mapping monazite particle.

*Investigation of the surface of ore particles
by means of XPS*

The measurements were carried out using SPECS photoelectronic spectrometer (SPECS GmbH, Germany), excitation by MgK_{α} -radiation of a double anode X-ray tube. The bombardment of samples by Ar^{+} ions (1 min ion etching corresponds to the removal of about 0.2–0.5 nm of the surface layer) was used to remove contaminants and improve the quality of spectra, and also to assess the distribution of elements and compounds along particles depth. In case of a polydisperse sample, the findings allow qualitatively estimating the composition of the surface and the deeper layers of ore particles.

Figure 7 gives survey spectra of the ore sample of the Chuktukon deposit, before and after ion etching with different durations, including for the low bond energy region. Table 5 indicates the content of the main elements in the surface of the initial ore before and after treatment with a hydrochloric acid solution. Carbon that is mainly a part of a layer of hydrocarbon pollutants of the surface was not considered. It can be seen that the highest surface concentrations, in addition to oxygen, are typical for iron, aluminum, silicon, phosphorus, and manganese. During ion etching, Fe and Ce concentrations increase most significantly (to about 10 and 1 at. %, respectively). Hence, the minerals that contain these metals are most shielded by other

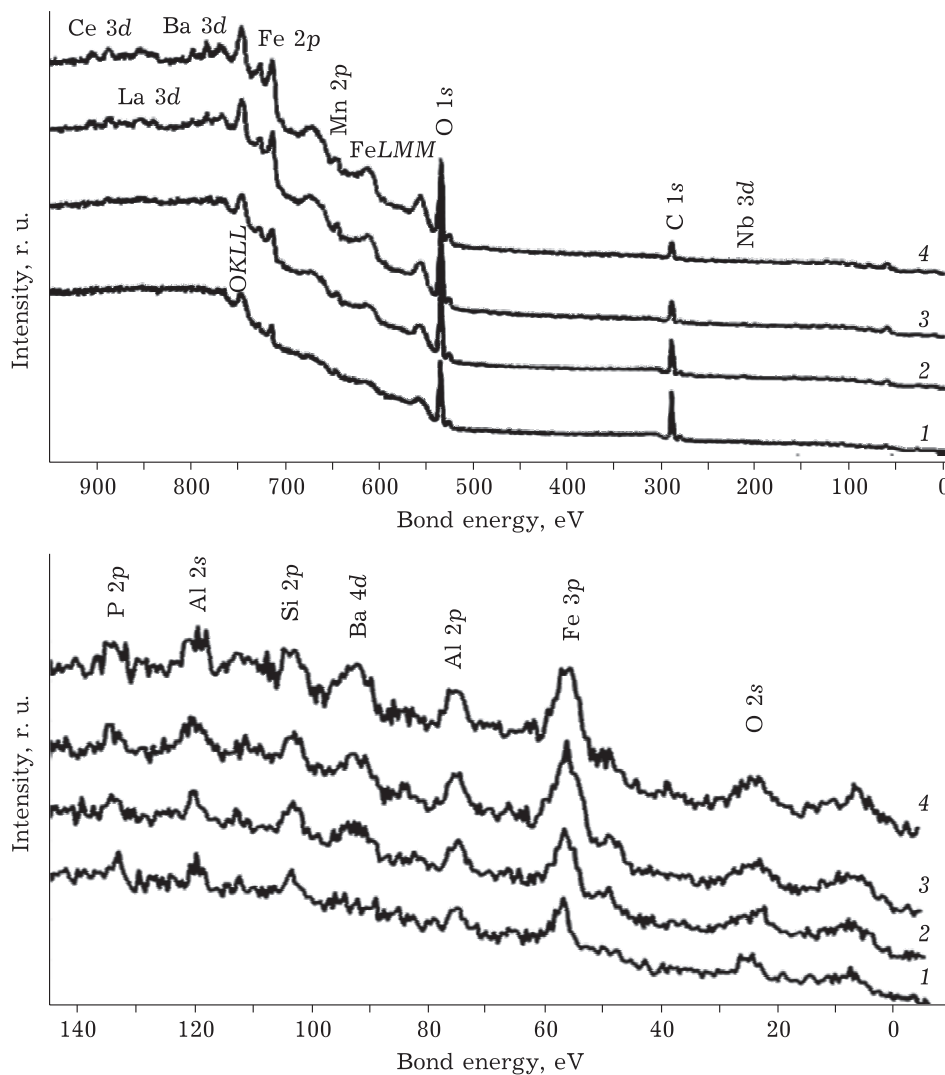


Fig. 7. Survey XPS spectra of ore sample of Chuktukon deposit before (1) and after (2–4) ion etching of different lengths, min: 6 (2), 10 (3), and 20 (4).

TABLE 5

Atomic concentrations of elements in samples surface of ore of Chuktukon deposit before and after treatment with HCl solution (mass loss of 10 %), according to XPS

Material	Ar ⁺ etching time, min	Content, at. %								
		O	Ce	La	Nb	Fe	Mn	P	Si	Al
Initial ore	0	78.7	0.32	Traces	0.34	6.6	1.8	2.5	3.6	4.3
»	6	69.9	0.43	0.37	0.34	9.7	3.4	2.0	4.5	8.2
»	20	59.4	1.50	0.71	0.42	15.5	3.8	2.5	7.0	7.8
Ore, HCl treatment	0	76.5	0.72	Traces	0.48	5.50	2.6	2.1	5.2	5.3
»	6	69.8	0.61	Traces	0.44	12.4	1.7	2.0	5.6	6.9
»	20	62.4	1.34	Traces	0.43	13.7	2.8	3.4	5.6	7.3

compounds: iron – by adsorbed water and possibly organic compounds, cerium (lanthanides) – by oxides of iron and other minerals, which are associated with monazite and florencite. Surface concentrations of Si, Al, and Mn do not increase so significantly. The concentration of Nb in ion etching almost does not change or even somewhat decreases. This indicates that pyrochlore is weakly associated with other mineral particles.

After acid treatment, resulting from which about 10 % of the sample mass is leached, the relative concentration of Ce (lanthanides) considerably increases. This is likely driven by decomposition of associates and dissolution of the surface iron oxide film over monacite and florencite.

Errors of determination of metals with small contents in ore (REM and Nb) and light elements with low sensibility factors (Si, Al, and P) by XPS method may be large, therefore a test using other methods is required.

CONCLUSION

Ores of the Chuktukon deposit are complex, mainly of the iron-manganese composition and are characterised by substantial contents of barium, phosphorus, niobium, lanthanides, yttrium, scandium, vanadium, and zinc. The main mineral forms of iron are hematite and goethite, of manganese – psilomelane and pyrolusite, of REM – monazite and florencite, and of niobium is barium-strontium pyrochlore.

As demonstrated by the investigation of the granulometric composition, above 50 % of ore mass is presented by a particle size of $-40 \mu\text{m}$ (sieve analysis data). Moreover, particles with sizes

of 1–2 μm and about 12 μm prevail. It is worth noting that the yield of fine micrometre particles increases rapidly with the grinding time rising.

Elements are mainly distributed uniformly in all size classes (from -40 to $+160 \mu\text{m}$). Although the contents of REM and niobium increase in fine fractions, producing rich REM concentrates with good yields by means of gravitational methods does not seem to be possible. According to the results of microscopic studies, the ore is mainly formed by ultrafine particles of the micro- (the first micrometres) and nanometre range. Ore globules of a size to several tens of micrometres are loose porous structures composed of thinner micrometre particles that in turn are not monolithic, and are dense aggregates, probably with a low-porous structure. Their constituent particles are nanoscale. As demonstrated by experimental results on acid leaching REM minerals and XPA data, monazite is mostly uncovered, and florencite is subtly embedded into the oxide forms of iron and does not go into solution. The fraction of “closed” forms of REM phosphates is almost 15 %. This is in agreement with X-ray photoelectron spectroscopy results, according to which REM concentrations are lower in the particle surface than in deeper layers. Niobium mineral, *i.e.* pyrochlore, is associated with iron oxides to a considerably lesser extent than other minerals are.

Thus, it is obvious that the enrichment of Chuktukon deposit ores by traditional methods is almost impossible. Apparently, the use of developing new methods for separating fine ores will also be inefficient, both because of a wide range of particle sizes of mineral components and their significant association

with iron oxides and hydroxides. In our opinion, to solve the problems of enrichment of Chuktukon deposit ores, preliminary decomposition of dense mineral aggregates of iron oxide particles on the micronic level is required. Modern grinding methods are hardly suitable for these purposes. The preliminary thermochemical treatment of ore possibly combined with mechanical appears much more promising. When solving the task of destruction of iron oxide aggregates, at least, separation of niobium and rare-earth metals from the bulk of the iron oxides by polygradient magnetic separation would be possible.

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