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Niobium mineralization of the Bolshetagninskoye deposit (Eastern Sayan)

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Abstract. The Bolshetagninskoye deposit is one of the largest known endogenous deposits, which is promising for niobium ore development. It is confined to the Ziminsky complex of ultramafic alkaline ultramafic alkaline rocks and carbonatites of Late Riphean age. The purpose of the study is to identify the distribution patterns of niobium in rocks and ores of the Bolshetagninskoye alkaline-ultramafic carbonatite massif at different stages of endogenic and postmagmatic processes using the methods of optical microscopy, scanning electron microscopy, X-ray diffraction electron microprobe analysis (microprobe), and X-ray fluorescence analysis. It has been determined that the primary niobium ores belong to holocrystalline apatite-mica and apatite-pyroxhlore rocks. The main minerals concentrating niobium in these ores are fluorocalciumpyroxhlore and ferrocolumbite. It is noted that a close paragenesis of apatite and pyroxhlore has been observed. The remaining (secondary) ore types are associated with pyroxhlore alteration products from primary ores and formed as a result of the redeposition of niobium in new geochemical conditions. Secondary niobium ores are represented by calcite carbonatites, potassium feldspar, and other metasomatites. The main minerals in these rocks with superimposed niobium mineralization – niobium concentrators – are presented by niobium-bearing rutile, ilmenorutile, niobium-bearing hematite, newly formed fluorocalciopyroxhlore and ferrocolumbite. Among the studied rocks pyroxhlore is represented by fluorocalciopyroxhlore and uraniumpyroxhlore, significantly enriched in tantalum. Unaltered fluorocalciopyroxhlores from apatite-pyroxhlore, apatite-mica, and calcite-feldspar rocks are characterized by a consistent composition of CaO, Na₂O, and SrO. Altered, hydrated varieties (based on the A-site vacancy) are more characteristic of metasomatic rocks and calcite carbonatites with superimposed pyroxhlore mineralization. This reflects varying degrees of fluid processing of these pyroxhlores. The conducted study revealed that the niobium ores of the massif underwent a multiphase, multistage transformation, which is represented by a decrease in the ore component content and the presence of different oxide forms of niobium in secondary ores compared to primary ores.

Keywords: pyroxhlore, niobium, Bolshetagninskoye deposit

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Научная статья
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Ниобиевое оруденение Большетагнинского месторождения (Восточный Саян)

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Резюме. Большетагнинское месторождение – одно из крупнейших известных эндогенных месторождений, перспективное для освоения ниобиевых руд. Оно приурочено к Зиминскому комплексу ультраосновных щелочных пород и карбонатитов позднерифейского возраста. Целью работы является выявление закономерностей распределения ниобия в породах и рудах Большетагнинского щелочно-ультраосновного карбонатитового массива на разных этапах эндогенного и постмагматического процессов с помощью методов оптико-микроскопического анализа, сканирующей электронной микроскопии, рентгеноспектрального электронно-зондового микроанализа (микросонд) и рентгенофлуоресцентного анализа. Установлено, что первичные ниобиевые руды относятся к полнокристаллическим апатит-слюдистым и апатит-пироксоловым породам. Основными минералами – концентраторами ниобия – в этих рудах является фторкальциопироксолом и ферроколумбит, отмечается тесный парагенезис апатита и пироксолом. Остальные (вторичные) типы руд связаны с продуктами изменения пироксолом из первичных руд и образовались вследствие переотложения ниобия в новых геохимических условиях. Вторичные ниобиевые руды представлены кальцитовыми карбонатитами, калишпатовыми и другими метасоматитами. Основные минералы в этих породах с наложенным ниобиевым оруденением – концентраторы ниобия – представлены ниобийсодержащим рутилом, ильменорутилом, ниобийсодержащим гематитом и новообразованным фторкальциопироксолом и ферроколумбитом. Пироксолом среди изученных пород представлен фторкальциопироксолом и уранпироксолом, значительно обогащен танталом. Неизменные фторкальциопироксолом из апатитит-пироксолом, апатит-слюдистых пород и кальцит-полевошпатовой породы характеризуются выдержанным составом по CaO, Na₂O и SrO. Измененные, гидратированные разности (по вакансиям в позиции А) более характерны для метасоматических пород и кальцитовых карбонатитов с наложенной пироксолом минерализацией, что отражает разную степень флюидной переработки этих пироксолом. В результате проведенного исследования выявлено, что ниобиевые руды массива претерпели полифазное многостадийное преобразование, которое выражается в уменьшении содержания рудного компонента и различных оксидных формах нахождения ниобия во вторичных рудах по сравнению с первичными.

Ключевые слова: пироксолом, ниобий, Большетагнинское месторождение

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Introduction

The minerals of the pyrochlore supergroup belong to complex oxides with the general formula $A_{2-m}B_2(X)_{6-w}Y_{1-n}$, where $A = Ca^{2+}, Na^+, Sr^{2+}, Ba^{2+}, Mn^{2+}, Sb^{3+}, Bi^{3+}, REE^{3+}, Th^{4+}, U^{4+}, H_2O^0, \square$; $B = Nb^{5+}, Ta^{5+}, Ti^{4+}, Sb^{5+}, Sn^{4+}, Fe^{3+}, W^{6+}$; $X = O, OH, F$; $Y = OH^-, F^-, O^{2-}, H_2O^0, \square, K, Cs, Rb$ [1]. In modern works, two classifications of minerals of the pyrochlore supergroup are used: the classification

introduced by Atencio et al. [1] and the classification presented by Hogarth et al. [2], which are also used in this work.

Pyrochlore supergroup minerals are important strategic raw materials for niobium and tantalum. They may also contain uranium and light rare earth elements. All of them are included in the List of the main types of strategic mineral raw materials (Executive Order of the Government of



the Russian Federation dated August 30, 2022, No. 2473-p¹). Two of the most sought-after rare metals are niobium and tantalum, alloying additives to alloys, whose metals are resistant to many aggressive media, refractoriness, corrosion resistance, high-temperature strength, superconductivity and other properties. Pyrochlore concentrates (pyrochlore and bariopyrochlore) and less than 10% ferrocolumbite form the basis of the raw material base [3]. In Russia, the demand for raw materials of tantalum and niobium is mostly met by imports [3, 4].

Niobium ores with pyrochlore are formed in various rocks: alkaline ultramafic (pegmatites of nepheline syenites) and acidic rocks (anorogenic granites and pegmatites), carbonatites and related alkaline rocks. Significant deposits of niobium, tantalum, rare earth elements, zirconium, barium, strontium, thorium, and iron, as well as apatite and fluorite, are associated with alkaline-ultramafic carbonatite (AUC) massifs [5].

The object of this study is the Bolshetagninskoye deposit, located in the Irkutsk region and belonging to the Ziminsky ore region, which is promising for the development of niobium ores [4]. The Bolshetagninskoye deposit is confined to the carbonatite complex of the UAC massif of the same name, which is part of the Ziminsky complex of the AUC with an age of 632 ± 2 million years [6].

The Bolshetagninskoye field has been studied repeatedly. In the early 50s, the anomalies revealed by the search party of the All-Russian Scientific Research Institute of Mineral Resources named after N.M. Fedorovsky (Moscow, Russia), using aerial gamma-ray surveying, resulted in the discovery of the Belozyminsky massif of the AUC with tantalum-niobium mineralisation. In 1975, the Urik-lysky party discovered the Bolshetagninskoye field during an additional site exploration GDP-50 of the massif of the same name. The prospecting works were carried out by the Production Geological Association "Irkutskgeologiya" with interruptions from 1957 to 1975. In 1988–1992, evaluation works were carried out. These works made it possible to establish the high prospects of the deposit for

niobium. In 2008–2012, the works resumed at the FSBI "All-Russian Scientific Research Institute of Mineral Raw Materials" field, and the amount of drilling and mining operations necessary to complete the evaluation stage was carried out. As a result, the reserves of the Bolshetagninskoye field were registered in the State Reserve Register in 2012. In May 2023, ST Elements JSC acquired the rights to develop the Bolshetagninskoye deposit at an auction. As a result, it was planned to build a mining and processing plant. As of 2016, Nb_2O_5 balance reserves in the C1 category were estimated at 362 thousand tonnes, in the C2 category at 4.5 thousand tonnes, and off-balance reserves at 9.5 thousand tonnes². Ore processing technologies have been developed at FSBI "All-Russian Scientific Research Institute of Mineral Raw Materials" repeatedly [4]. In the work of V.S. Pikalova² schemes for processing and extracting pyrochlore from microcline, carbonatite-mica and mica ores to produce pyrochlore concentrate were proposed.

The Bolshetagninsky massif has a rounded shape and a zonal ring structure. It is composed of pyroxene-nepheline rocks of the melteigite–ijolite–urtite series, nepheline and alkaline syenites, potassic feldspar syenites and carbonatites, and late dykes of picrite porphyrites-aillikites are also noted [5]. The formation of the Bolshetagninsky massif proceeded in several successive stages. At the first stage, the introduction of intrusive rocks of the melteigite–ijolite–urtite series took place, followed by nepheline and subalkaline syenites. The formation of the massif ended with the introduction of carbonatites and late dykes of aillikites [5]. The introduction of AUC caused the phenitization of the enclosing strata, the introduction of nepheline and sub-alkaline syenites, and their potassium feldspathization. Secondary overprinted mineralization has been in the rocks of the massif, expressed in carbonatization, micaization, hematitization, sulfidization and fluoritization of the enclosing strata.

According to the predecessors' data, it was revealed that the main rich niobium mineralization, unlike other deposits associated with the AUC intrusions, is not confined to carbonatites but to sili-

¹ Executive Order of the Government of the Russian Federation dated August 30, 2022, No. 2473-p. On approval of the List of the main types of strategic mineral raw materials. Moscow; 2022, 3 p.

² Pikalova V.S. Geological and economic assessment of a new potentially industrial type of niobium ores on the example of the Bolshetagninskoye deposit. Cand. Sci. (Geol. and Mineral.) thesis. 25.00.11. Moscow; 2018, 136 p.



cate rocks – microclinities (80–85% of ores, potassic feldspar syenites based on alkaline ultramafic rocks) and mica (metasomatites of carbonate-microcline-biotite composition²) [4]. There is also an ore-bearing apatite-pyrochlore lenticular deposit [4]. The following varieties are noted among the ore-bearing carbonatite rocks: carbonate-microcline (the content of carbonates is 15–20%); carbonate-biotite (up to 25% of the criss-cross carbonate of the ankerite-dolomite series); significantly carbonate (more than 30% of carbonates) rocks [7].

Earlier, pyrochlore mineralization was studied by L.K. Pozharitskaya in the work [8], where the primary pyrochlore of the Bolshetagninskoye deposit from microclinities and mica is represented by fluorocalciopyrochlore, which constantly contains an impurity of strontium (0.021–0.034 f. u.) [7]. In the work of L.A. Azaranova [7] K pyrochlore formed as a result of hypergene alteration of ore microcline rocks was described and hydrated pyrochlore was established.

The aim of the work is to identify patterns of niobium distribution in rocks and ores of the Bolshetagninsky alkaline-ultramafic carbonatite massif at different stages of endogenic and post-magmatic processes. Identification of the distribution of ore minerals in rocks and ores of the Bolshetagninsky massif and determination of niobium contents in them are necessary in the future to clarify the prospecting criteria for more accurate delineation of ore deposits and development of technology for extracting useful components from new complex types of ores, as well as to create a model of the ore formation process.

Materials and methods

The work uses the results of analytical studies of rocks and ores performed on the equipment of the Shared Research Facility of “Isotope-Geochemical Research” at the A.P. Vinogradov Institute of Geochemistry, Siberian Branch of the Russian Academy of Sciences. 137 lump samples, more than 120 sections and more than 20 polished sections were examined. The samples were characterised, and the diagnostic properties of minerals were determined using a Micromed MC-2-ZOOM

Digital stereomicroscope. Textural and structural features of rocks and ores, their mineral composition, morphological features of minerals and aggregates, and paragenetic mineral associations were studied in sections and polished sections using the petrographic mineralogical method using an Olympus BX-51 polarising light microscope with a 20–500-fold magnification range in transmitted and reflected light. The sections were stained using a special technique that makes it possible to assess the percentage of feldspar and feldspathoids, as well as calcium and iron carbonates, most effectively. The contents of rare elements (niobium, zirconium, and yttrium) in rocks and ores (120 samples) were analysed by the X-ray fluorescence method using a VRA-30 spectrometer (analysts are Candidates of Chemical Sciences V.M. Chubarov, A.A. Amosova, T.S. Aisueva, and E.V. Chuparina; Doctor of Technical Sciences A.L. Finkelstein). The limits of the element detection according to the X-ray fluorescence analysis method used were niobium – 0.0005%, zirconium – 0.0010%, and yttrium – 0.0010%. The elemental composition of the main rock-forming and ore minerals was determined by scanning electron microscopy using a MIRA 3 LMH TESCAN electron microscope (6 preparations, analyst – A.G. Chueshova). The results of the quantitative analysis of pyrochlore (6 preparations) were obtained by electron probe X-ray spectral microanalysis using wave spectrometers on a Superprobe JXA-8200 microanalyser (JEOL Ltd, Japan; analyst – Candidate of Chemical Sciences O.Y. Belozeroва).

Results and discussion

Based on the 122 samples studied, it was found that 20 samples contain abundant niobium mineralization (with a cut-off grade of Nb₂O₅ more than 0.3 wt. %). According to the guidelines³, The run-of-mine ores of this deposit contain Nb₂O₅ from 0.3 to 1.2 wt. % and with an average Nb₂O₅ content in the ore of 1 wt. %. In the analysed ores, the average Nb₂O₅ content is 1.5 wt. %. 13 samples meet the criteria of run-of-mine ores, and 7 samples are classified as rich and very rich ores: the Nb₂O₅ content in them falls within the range

³ Guidelines on the application of the Classification of deposit reserves and prognostic resources of solid minerals. *Niobium, tantalum ores and rare earth elements*. Moscow, 2007, 42 p.



of values from 1.2 to 6.1 wt. % (Fig. 1). We have classified two types of ores: primary and secondary ores. The primary ores are represented by apatite-mica and apatite-pyrochlore (apatite) rocks belonging to the majority of the ores studied (11 out of 20 samples). Primary apatite-mica ores with pyrochlore are very rich ($Nb_2O_5 \sim 6.1$ wt. %), rich ($Nb_2O_5 > 1,2$ wt. %) and run-of-mine ($Nb_2O_5 \sim 0,3$ wt. %) ores. The content of P_2O_5 in these rocks reaches 12 wt. %. The main niobium ore minerals of primary apatite-mica ores are pyrochlore and ferrocolumbite. The remaining (secondary) types of niobium ores were formed by the redeposition of niobium in the rocks of the massif due to metasomatic and hydrothermal processes. Secondary ores are rich ($Nb_2O_5 \sim 2$ wt. %) and run-of-mine ores ($Nb_2O_5 \sim 0,3$ wt. %). The main ore minerals of these rocks are represented by various oxides of iron, titanium, and niobium: niobium-bearing rutile, ilmenorutyl, niobium-bearing hematite, newly formed pyrochlore, and ferrocolumbite. There is a decrease in the Nb_2O_5 content among secondary ores compared to primary ores. This is probably due to the dispersion of niobium during metasomatism and its dispersion in less concentrated mineral forms: niobium-bearing rutile and/or hematite. Secondary niobium ores are represented by calcite carbonatites, carbonate-apatite-aegirine-sulfide rock, and potassium-feldspar metasomatites.

In the Bolshetagninsky massif, pyrochlore group minerals are the main niobium concentrators and are found in apatite-pyrochlore, apatite-mica ores with sulfides, calcite carbonatites, and metasomatites.

Apatite pyrochlore ore (apatite) (Fig. 2, a) is a massive holocrystalline rock of light beige colour, composed mainly of apatite (~ 85 vol. %), pyrochlore (~ 10), potassium feldspar (~ 5). Against the background of hypidiomorphic and xenomorphic rounded isometric and prismatic aggregates of apatite grains, large (~ 0.2 mm) idiomorphic pyrochlore phenocrysts of cubo-octahedral crystal habit are distinguished (see Fig. 2, e–h). Potassium feldspar is observed in the form of “schlieren” segregations among the groundmass of the rock (see Fig. 2, b, d). In apatite-pyrochlore rocks, the colour of pyrochlores under oblique illumination varies from colourless, pale yellow, amber yellow to brown (see Fig. 2, b, d). Pyrochlore is often zonal, with a sectoral colour distribution, which is also observed in reflected light (Fig. 2, c, d). The central parts of the grains of some pyrochlores have surface defects and brown colour, and in reflected light they are observed with a higher reflection coefficient, probably due to the fact that some central parts of the grains have increased concentrations of iron oxides.

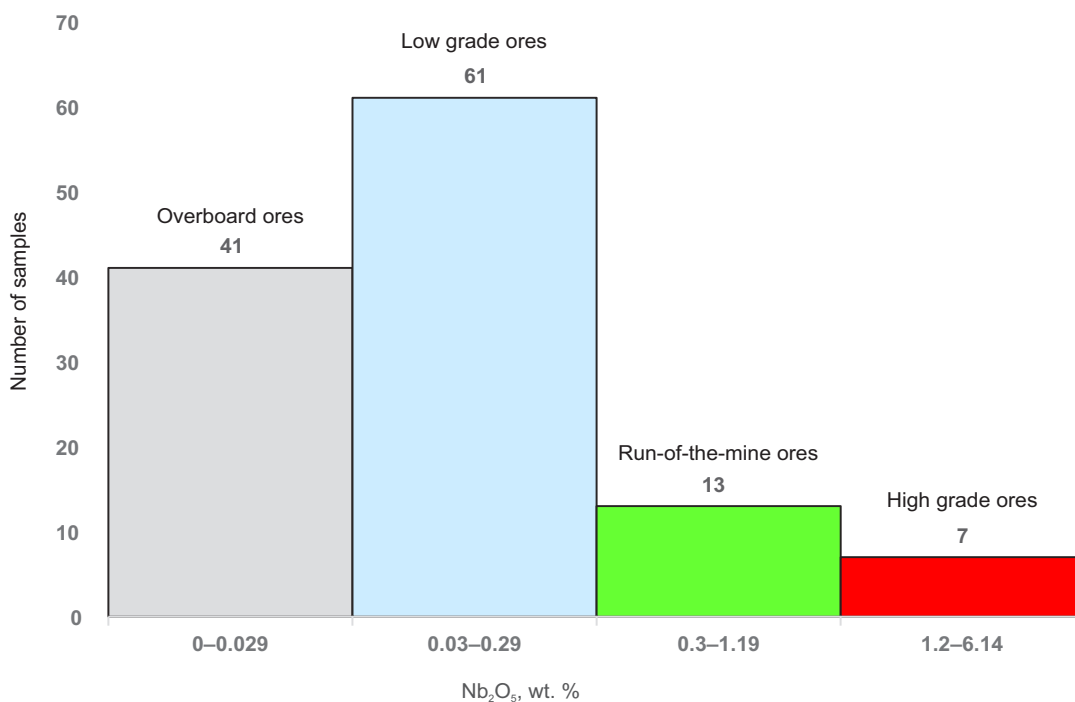


Fig. 1. Histogram of niobium ore occurrence in the studied rocks of the massif

Рис. 1. Гистограмма встречаемости рудных содержаний ниобия в изученных породах массива



Apatite-mica ores are holocrystalline, medium- and fine-grained rocks of black colour. Ore apatite-mica rocks (Fig. 3, a) consist of (vol. %) mica – tetraferriannite (20–60), sulfides (10–15), apatite (1–12), potassium feldspar (0–10), pyrochlore and/or ferrocolumbite (1–6), magnetite (less than 5), and carbonate (less than 5). The texture of the ore is spotted, impregnated, or veined-impregnated. Mica is observed in the form of idiomorphic plates and scales of a pseudo-hexagonal habit, brownish orange in colour. Apatite occupies interstices between mica flakes and forms prismatic grains. Potassium feldspar is observed in the form of “schlieren” segregations among the groundmass of the rock. Pyrochlore in apatite-mica rocks is observed in the form of large and small idiomorphic brown grains, which form impregnation, separate rounded clusters of irregularly shaped grains and veins (see Fig. 3, b–e). There is a close paragenesis of pyrochlore and apatite. In apatite-tetraferriannite rocks, pyrochlore is homogeneous in colour and reflective power. Its colour varies from colourless to pale yellow in oblique illumination (see Fig. 3, f, g). Ferrocolumbite in the form of idiomorphic grains with numerous pores is also

common in apatite-mica rocks. It forms partial and complete pseudomorphs of pyrochlore. The rate of change in primary apatite-mica ores is insignificant. Iron sulfides are developed to varying degrees in apatite-mica rocks and ores, mainly in the form of pyrrhotite and pyrite. Modified (carbonatized and potassium feldspathized) apatite-mica rocks are characterised by a lower value than the cut-off grade of niobium oxide (~ 0.2% or less).

Ore calcite carbonatites are medium-grained and fine-grained rocks of grey, pink, ochre or dark brown colours. Ore textures are banded, lenticular, veined, and veined-impregnated.

The sample of apatite-calcite carbonatite (БТГ-50) (Fig. 4, a) is composed of medium-grained pink calcite (~ 80 vol. %) and apatite (~ 20); siderite and dolomite are also noted (see Fig. 4, k), as is quartz in the form of idiomorphic grains. The ore textures are spotted, banded, and veined-impregnated. Niobium mineralization is associated with the products of pyrochlore alteration: niobium-bearing rutile, ilmeno-rutile, newly formed ferrocolumbite, as well as oxides of niobium, iron and titanium. Rutile, as the main ore mineral, is noted in the form of allotriomor-

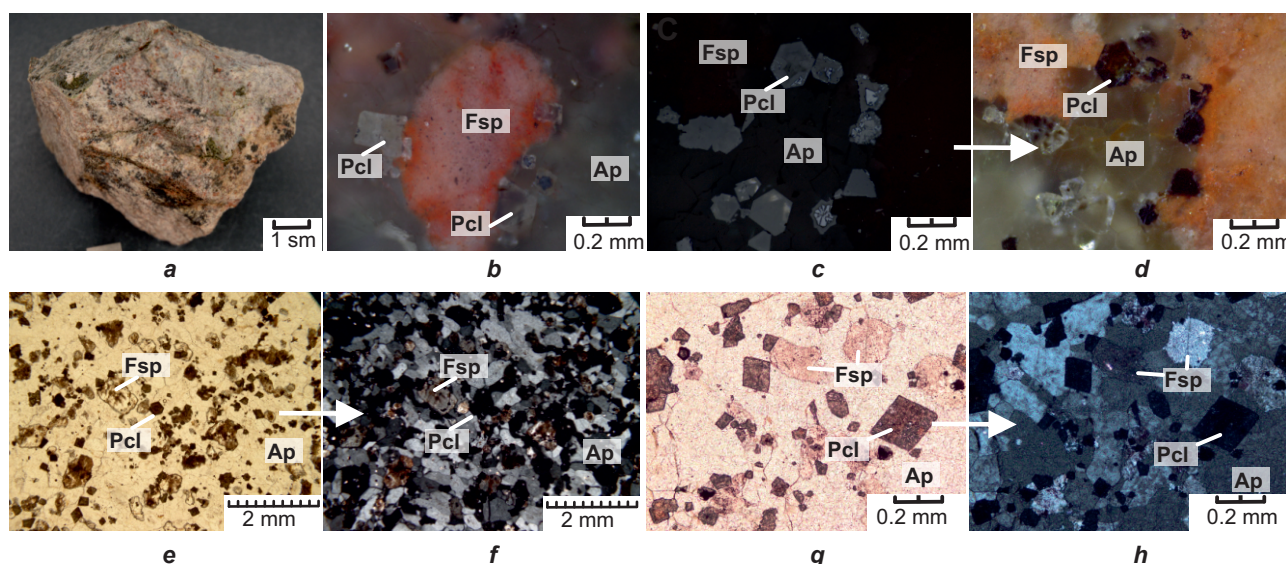


Fig. 2. Apatite-pyrochlore ore rock (apatite) (sample БТГ-8-21):

a – photograph of the sample; b–d – micrographs of a polished section: c – in reflected light, b, d – with side illumination; e–h – micrographs of a thin section in transmitted light:

e, g – with a single polarizer, f, h – with crossed polarizers

Pcl – pyrochlore; Fsp – potassium feldspar; Ap – apatite

Рис. 2. Рудная апатит-пироклоровая порода (апатитит) (образец БТГ-8-21):

a – фотография образца; b–d – микрофотографии шлифа: c – в отраженном свете, b, d – при боковом освещении; e–h – микрофотографии шлифа в проходящем свете:

e, g – при одном поляризаторе, f, h – при скрещенных поляризаторах

Pcl – пироклор; Fsp – калиевый полевоый шпат; Ap – апатит

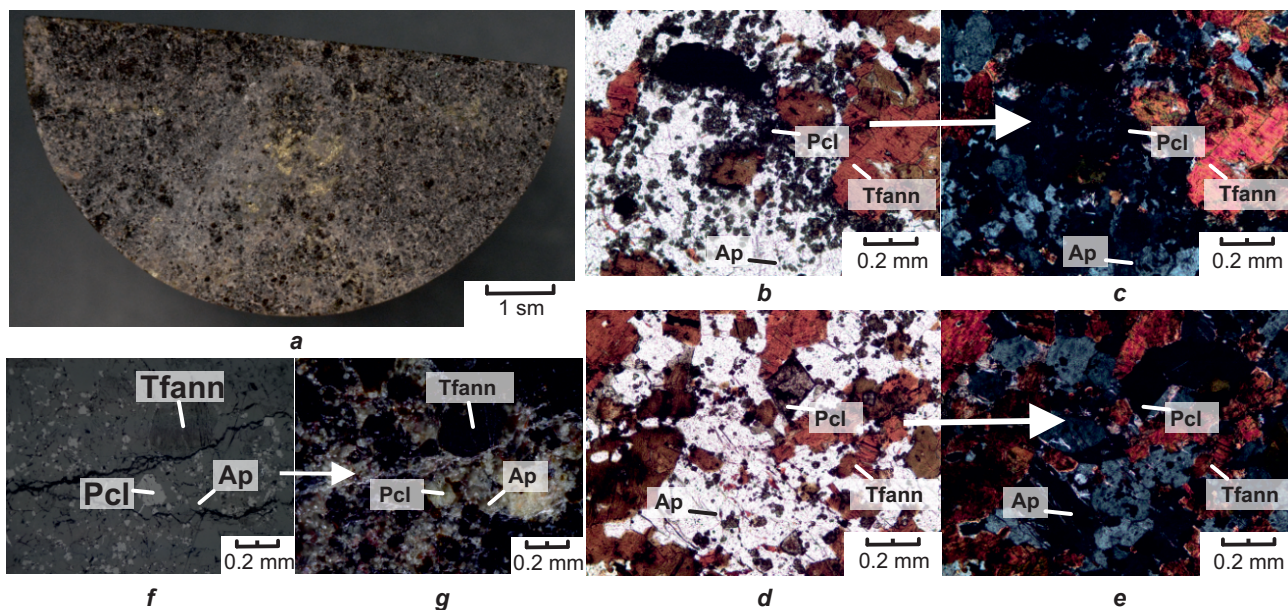


Fig. 3. Apatite-mica ore rock (sample БТГ-15-2):

a – photograph of the sample; *b–e* – micrographs of a thin section in transmitted light:
b, d – with a single polarizer, *c, e* – with crossed polarizers; *f, g* – micrographs of a polished section:
f – in reflected light, *g* – with side illumination

Pcl – pyrochlore; *Tfann* – tetraferriannite; *Ap* – apatite

Рис. 3. Рудная апатит-слюдистая порода (образец БТГ-15-2):

a – фотография образца; *b–e* – микрофотографии шлифа в проходящем свете:
b, d – при одном поляризаторе, *c, e* – при скрещенных поляризаторах; *f, g* – микрофотографии анишлифа:
f – в отраженном свете, *g* – при боковом освещении

Pcl – пирохлор; *Tfann* – тетраферрианнит; *Ap* – апатит

phic grains and aggregates forming clusters and veins (see Fig. 4, *i–l*). Pyrochlore is observed in the form of single idiomorphic grains, grain fragments, and relict grains, according to which ferrocolumbite is developed (see Fig. 4, *g, l*). Apatite-rutile paragenesis in calcitic carbonatites forms nests and veins. The rock was largely exposed to secondary hydrothermal processes, such as hematitisation and leaching of pyrochlore. There is also an uneven distribution of the ore component in the sample due to the migration of niobium under the influence of overprinted secondary processes (hematitization).

In the modified carbonate-apatite-aegirine-sulphide rock (Fig. 5, *a*), pyrochlore occurs in the form of idiomorphic large grains of cubo-octahedral habit and allotriomorphic rounded grains and clusters of grains among prismatic grains of apatite and modified aegirine. The texture of the ore is spotted, veined-impregnated, and veined. In transmitted light, the colour of pyrochlore is brown with a darker brown border (see Fig. 5, *d*). In oblique illumination, pyrochlore has an unstable

colour, varying from amber yellow, lemon yellow to light yellow within a single grain (see Fig. 5, *h, i*). Probably, the colour of pyrochlore is related to the heterogeneous chemical composition and the presence of uranyl ion in the composition of uranium pyrochlore (explained below). Idiomorphic pyrochlore is often zonal, with zones of development of ferrocolumbite and uranium pyrochlore, as well as with inclusions of hematite (see Fig. 5, *b, c*). Ferrocolumbite in these ores is observed as partial pseudomorphs, it forms rims along idiomorphic pyrochlore crystals (see Fig. 5, *b*). Iron sulfides in the form of pyrrhotite and pyrite are significantly developed in the rock, hematite and sphalerite are also noted.

Pyrochlore in all the studied rocks corresponds in composition to fluorocalciopyrochlore, the composition points in the diagram of niobium, titanium, and tantalum in position B fall into the field of pyrochlore group minerals, and there is a trend towards titanium-mineral enrichment (Fig. 6, *a*). The empirical formula of fluorocalciopyrochlore is presented as follows:

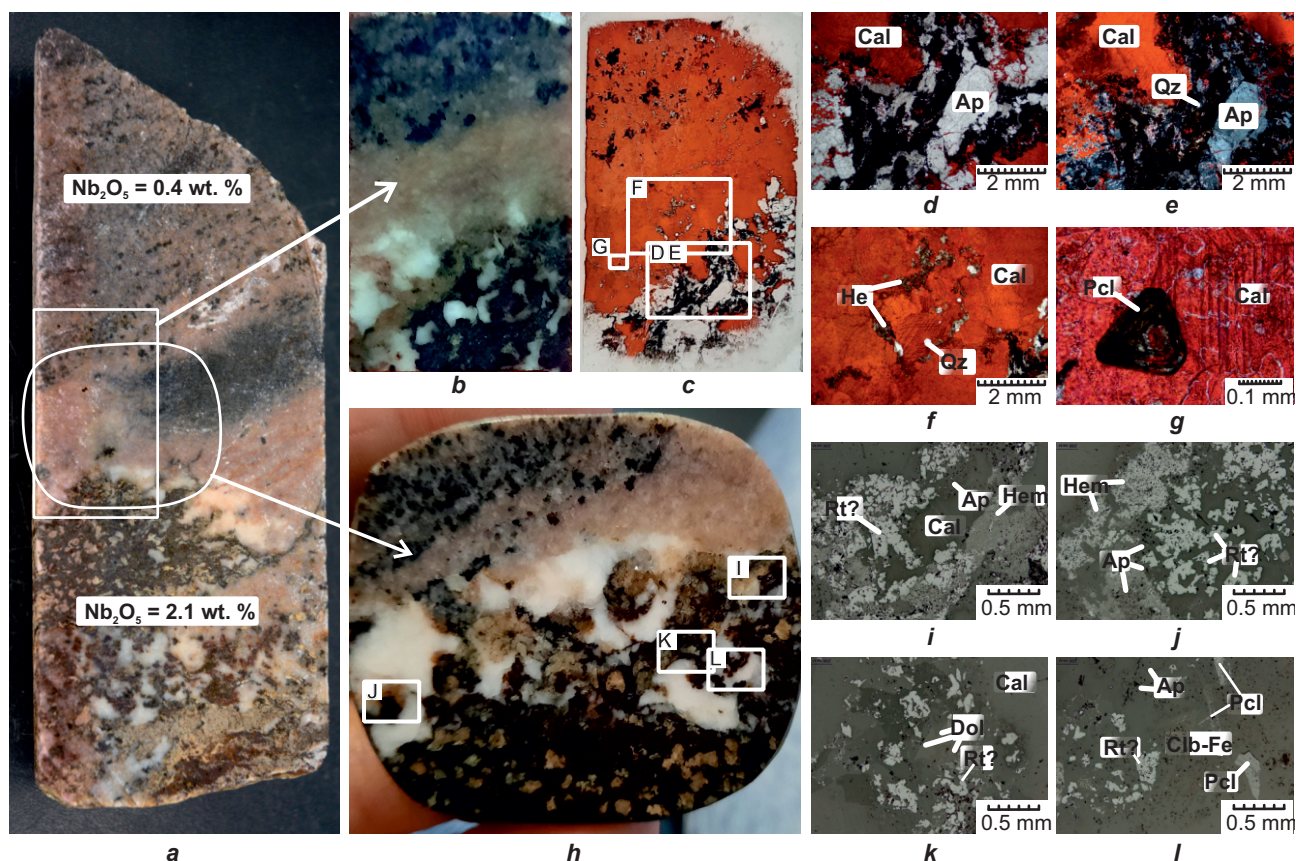


Fig. 4. Ore apatite-calcite carbonatite (sample БТГ-50):

a – photograph of the sample; *b*, *c* – photographs of a thin section; *h* – photographs of a polished section; *d–g*, *i–l* – micrographs of thin sections and polished sections of an apatite-carbonatite rock with pyrochlore mineralization: *d–g* – in transmitted polarized light, *d*, *f* – with a single polarizer, *e*, *g* – with crossed polarizers, *i–l* – in reflected light
Pcl – pyrochlore; *Cal* – calcite; *Ap* – apatite; *Dol* – dolomite; *Hem* – hematite;
Qz – quartz; *Clb-Fe* – ferrocolumbite; *Rt* – rutile

Рис. 4. Рудный апатит-кальцитовый карбонатит (образец БТГ-50):

a – фотография образца; *b*, *c* – фотографии шлифа; *h* – фотографии аншлифа;
d–g, *i–l* – микрофотографии шлифов и аншлифов апатит-карбонатитовой породы с пироксеновым оруденением: *d–g* – в проходящем поляризованном свете, *d*, *f* – при одном поляризаторе, *e*, *g* – при скрещенных поляризаторах, *i–l* – в отраженном свете
Pcl – пироксенол; *Cal* – кальцит; *Ap* – апатит; *Dol* – доломит; *Hem* – гематит;
Qz – кварц; *Clb-Fe* – ферроколумбит; *Rt* – рутил



The composition of fluorocalciopyrochlores is similar to the compositions of fluorocalciopyrochlores from the neighboring Beloziminsky massif in the work of E.A. Khromova [9], but in the studied pyrochlores there is more titanium and iron in the B position due to impurities of FeO up to 3.7% and TiO₂ up to 7 wt. % (see Fig. 6, *b*).

Pyrochlores are probably of primary fluid-magmatic origin, corresponding in composition to fluorocalciopyrochlores, and have a stable composition for cations in position A (with no or insignificant vacancy in position A) (see Fig. 6, *c*).

Pyrochlores of late hydrothermal origin (or too modified) are characterised by a significant vacancy in position A and a nonstoichiometric ratio of sodium and calcium in position A, corresponding to non-ideal fluorocalciopyrochlore compositions. The stoichiometry of the pyrochlore mineral composition, which does not correspond to the ideal one, is due to a low sodium content and too high calcium content in position A (see Fig. 6, *c*), or too high niobium and iron content in position B (see Fig. 6, *b*, *d*), or an analysis error.

An impurity of SrO up to 1.8 wt. % was found in fluorocalciopyrochlores. At the same time, pyrochlore compositions corresponding to those of

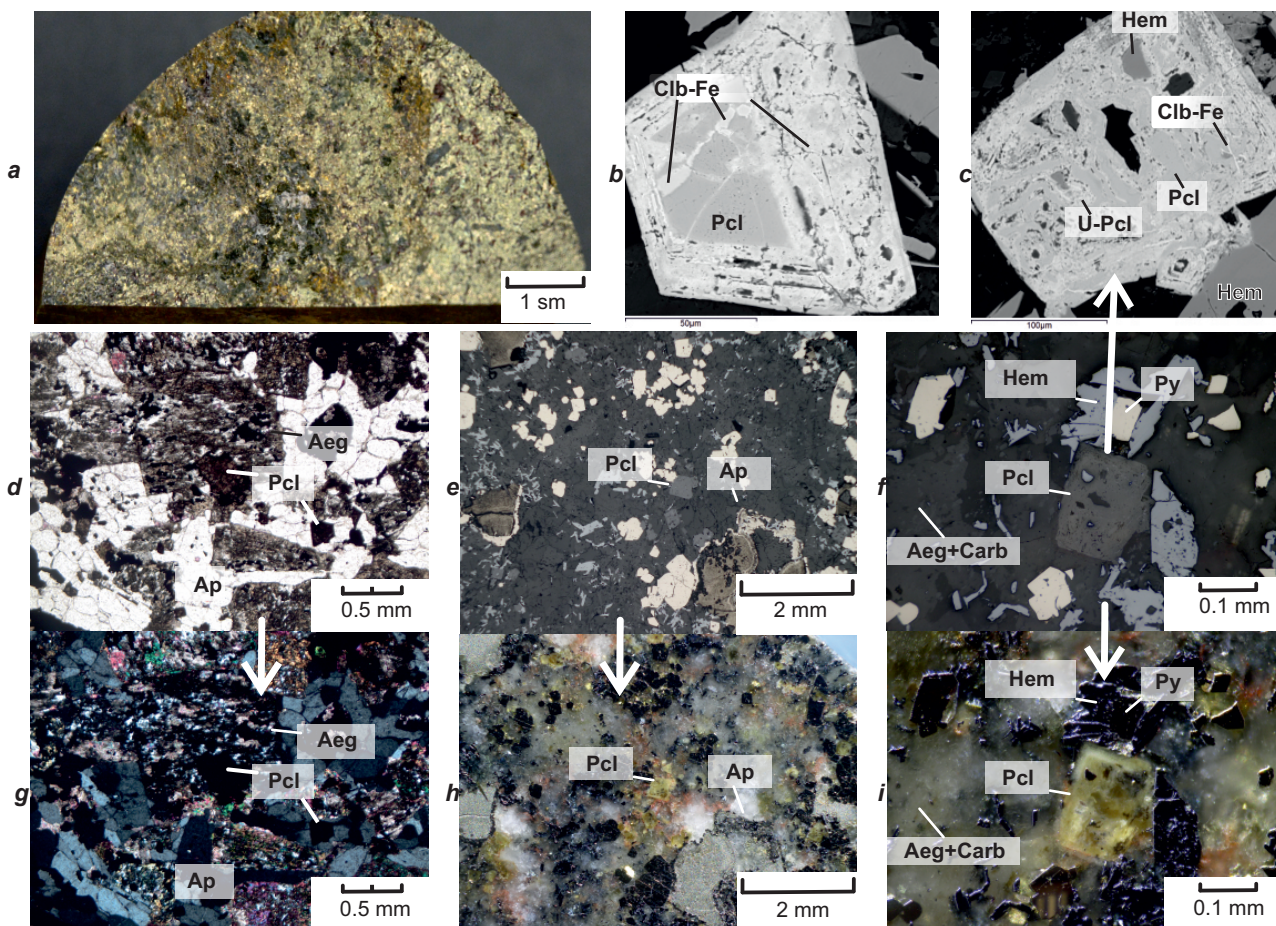


Fig. 5. Carbonate-apatite-aegirine-sulfide ore rock (sample БТГ-18):

a – photograph of the sample; *b, c* – micrographs in back scattered electrons;
d, g – micrographs of a thin section in transmitted light: *d* – with a single polarizer,
g – with crossed polarizers; *e, f, h, i* – microraphs of a polished section:
e, f – in reflected light, *h, i* – with side illumination

Pcl – pyrochlore; *U-Pcl* – uraniumpyrochlore; *Aeg+Carb* – aegirine and carbonate; *Ap* – apatite;
Dol – dolomite; *Hem* – hematite; *Clb-Fe* – ferrocolumbite; *Py* – pyrite

Рис. 5. Рудная карбонат-апатит-эгирин-сульфидная порода (образец БТГ-18):

a – фотография образца; *b, c* – микрофотографии в обратно-рассеянных электронах;
d, g – микрофотографии шлифа в проходящем свете: *d* – при одном поляризаторе,
g – при скрещенных поляризаторах; *e, f, h, i* – микрофотографии шлифа:
e, f – в отраженном свете, *h, i* – при боковом освещении

Pcl – пирохлор; *U-Pcl* – уранпирохлор; *Aeg+Carb* – эгирин и карбонат; *Ap* – апатит;
Dol – доломит; *Hem* – гематит; *Clb-Fe* – ферроколумбит; *Py* – пирит

fluorocalciopyrochlores from probably primary fluid-magmatic apatite-pyrochlore and apatite-mica rocks are characterised by sustained SrO values ranging from 0.5 to 1.7 wt. %. A significant variation in SrO values is observed in ores with newly formed (or altered) pyrochlore (in calcitic carbonatites and metasomatites).

Among the pyrochlores, uranium-bearing pyrochlore (UO_2 up to 1.6 wt. %) was found in a carbonate-apatite-aegirine-sulfide rock sample with zones of development of uranium pyrochlore (gatchetolite) with UO_2 contents of 6.4 wt. % and Ta_2O_5 contents of 0.9 wt. % (see Fig. 5, c).

Ferrocolumbite is a minor ore mineral of the niobium of Bolshetagninskoye deposit. Ferrocolumbite is observed as idiomorphic porous grains in apatite-mica rocks, in the form of complete and partial cellular pseudomorphs in pyrochlore crystals (see Fig. 4, l), forms rims in pyrochlore crystals (see Fig. 5, b), as well as in the form of allotriomorphic and needle-plate aggregates among calcitic carbonatites. The composition of ferrocolumbite is very variable and is characterised by the following variations of compositions (wt. %): Nb_2O_5 (58.88–68.55), FeO (12.13–19.98), MnO (0.84–7.22), TiO_2 (3.27–14.26).

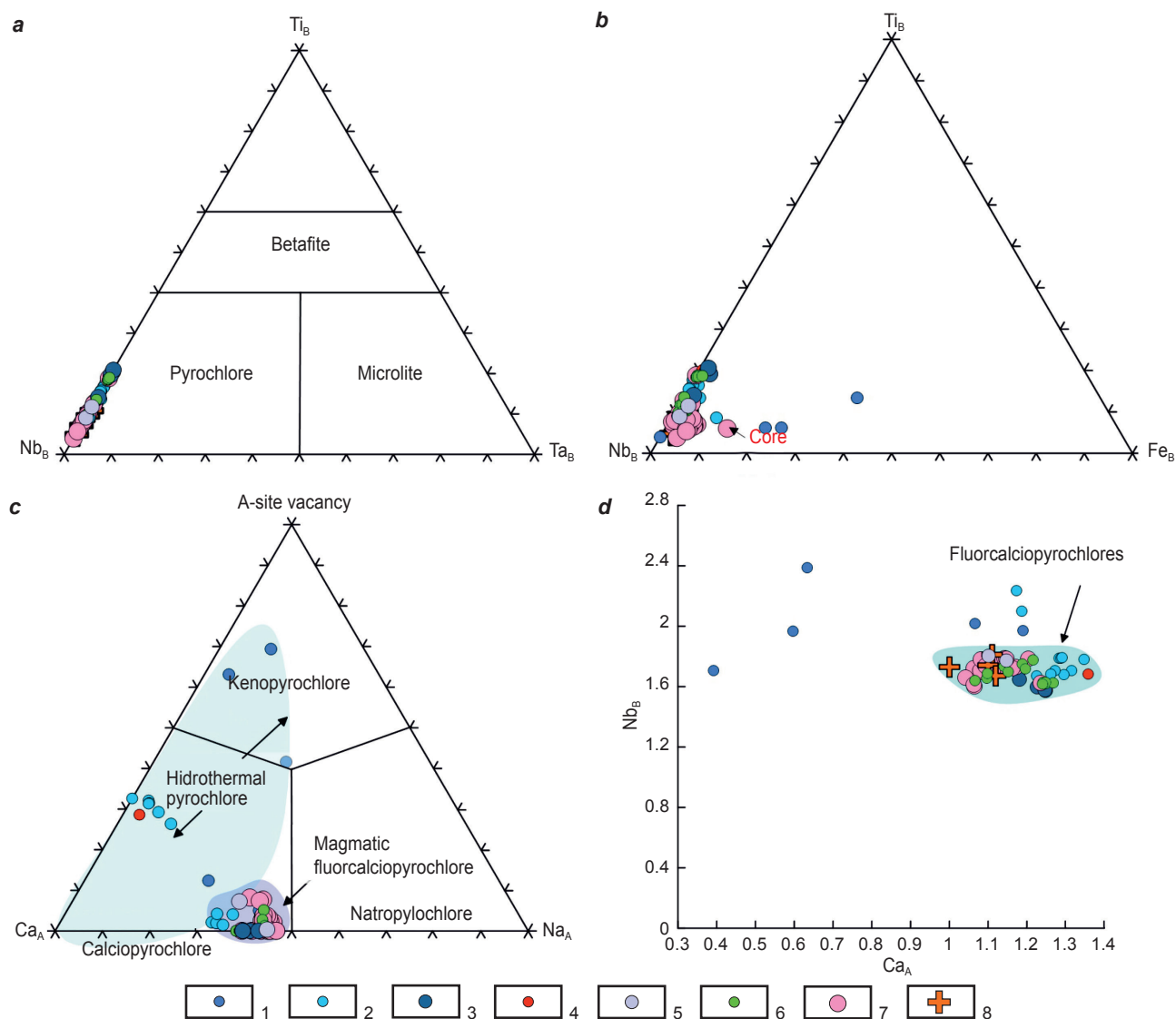


Fig. 6. Pyrochlore composition:

a – classification diagram for pyrochlore group minerals Nb, Ti, Ta in B-site according to source [2];
b – Nb, Fe, Ti in B-site in formula units; *c* – classification diagram for pyrochlores Na, Ca – in A-site, A-site vacancy according to source [10] (the field of hydrothermal pyrochlores is marked in blue, the field of magmatic pyrochlores is marked in purple); *d* – binary diagram Nb in B-site and Ca in A-site;
 1 – БТГ-50 – calcite carbonatite; 2 – БТГ-18 – carbonate-apatite-aegirine-sulfide rock;
 3 – БТГ-74 – calcite-feldspar rock; 4–6 – БТГ-76, БТГ-23, БТГ-15/2 – apatite-mica rock;
 7 – БТГ-8/21 – apatite-pyrochlore ore; 8 – fluorocalciopyrochlore compositions from work [9]

Рис. 6. Состав пироксенов:

a – классификационная диаграмма для минералов группы пироксена Nb, Ti, Ta в позиции В по источнику [2];
b – Nb, Fe, Ti в позиции В в формульных единицах; *c* – классификационная диаграмма для пироксенов Na, Ca – в позиции А, вакансия в позиции А по источнику [10] (голубым цветом обозначено поле гидротермальных пироксенов, сиреневым – магматических пироксенов); *d* – бинарная диаграмма Nb в позиции В и Ca в позиции А
 1 – БТГ-50 – кальцитовый карбонатит; 2 – БТГ-18 – карбонат-апатит-эгирин-сульфидная порода;
 3 – БТГ-74 – кальцит-полевошпатовая порода; 4–6 – БТГ-76, БТГ-23, БТГ-15/2 – апатит-слюдистая порода;
 7 – БТГ-8/21 – апатит-пироксеновая руда; 8 – составы фторкальциопироксенов из работы [9]

Among the secondary ore rocks, niobium minerals are represented (Nb_2O_5 , content, wt. %) by niobium-bearing rutile (up to 7), ilmenorutyl (up to 25), niobium-bearing hematite (up to 7.4), newly formed pyrochlore and ferrocolumbite. Also, pyrochlore transformation products are oxides of niobium, iron and titanium with a very variable

composition. The mineral dmitriyvarlamovite with the composition [11] (wt. %): Nb_2O_5 (40,08), FeO (20,77), TiO_2 (37,72), MnO (0,11), V_2O_3 (0,89). However, the composition of the studied mineral is slightly different, which is most likely due to the phenomena of isomorphism (wt. %): Nb_2O_5 (43,43), FeO (18,44), TiO_2 (31,48), V_2O_3 (2,29).



It was noted in the predecessors' works that the main mineralization of the Bolshetagninsky massif is associated with "microclinitic" pyrochlore ores and "micaites", which are metasomatic formations derived from primary ultramafic alkaline and medium-moderate alkaline rocks² [7, 12]. During petrographic research, V.S. Pikalova's² work revealed that "micaites" (apatite-biotite metasomatites) were formed by the substitution of ijolites, and "microclinites" (dolomite-ankerite-calcite-microcline rocks) were presumably developed by syenites, while the primary source of the ore component, niobium, was not specified, and the metasomatic genesis of pyrochlore is assumed. In this work², it was noted that the composition of pyrochlores in "microclinites" and "micaites" is similar, which indicates a single stage of ore formation. However, during petrographic work, we found that ore apatite-pyrochlore and apatite-mica rocks, which are primary ores, most likely have an early magmatic fluid nature.

Silicate phosphate rocks spatially and genetically associated with carbonatites of ultramafic alkaline complexes are phoscorites and glimmerites. Glimmerites are rocks consisting mainly of (≥ 75 vol. %) mica (tetraferriphlogopite or biotite), apatite and carbonate minerals; amphibole may also be present [13, 14]. The metasomatic (more generally accepted) [14, 15] or magmatic [13, 16] genesis of glimmerites is assumed. An example of the formation of phlogopitic metasomatites (phlogopites, biotites, or glimmerites) [17, 18] from pyroxenites due to the action of carbonatite melt is observed in the Yakupiranga rock complex in Brazil [15]. At the same time, there is a reaction zone between rocks that differ sharply in composition and a change of typical skarn ("anti-skarn") associations (olivine, phlogopite, calcite, titanite, amphibole) in the rock contact zone. Glimmerites are also the main ore in the Sokli and Siilinjärvi massifs in Finland, which are associated with industrial reserves of phosphate raw materials [13]. Phoscorites are rocks consisting of apatite, magnetite, and silicate minerals (olivine and/or pyroxene and/or phlogopite) [19]. According to some models, the formation of such rocks is associated with direct crystallization from a phoscorite melt or with fractional crystallization from a phosphate melt or liquation from a carbonatite melt [19, 20]. Apatite-mica rocks and apatites from the Bolshetagninskoye deposit may be genetically

related to phoscorites and glimmerites, differing from them in a different way of evolution of an alkaline phosphate melt solution significantly saturated with fluids. This is confirmed by the observed morphological and optical properties of minerals and their textural and structural relationships of mineral parageneses and associations in ores. Apatite-mica rocks were not formed from modified ultramafic alkaline rocks. Pyrochlore in apatite-mica rocks is observed in the form of large idiomorphic and small idiomorphic grains forming impregnation and separate clusters; close paragenesis of pyrochlore and apatite is noted. At the same time, pyrochlore was not detected in potassium feldspar. Potassium feldspar in apatite-mica rocks and in apatite-pyrochlore ores forms "schlieres" – fine-crystalline aggregates of rounded shape, while no metasomatic reaction rim and potassium feldspathization of mica are observed. The contact of apatite-mica rocks and potassium-feldspar rocks is smooth, without reaction rims.

The main ores of the deposit ("microclinite" and "biotite-microclinite" and "biotites", classified as glimmerites) were probably formed from the initial potassium feldspar metasomatites due to the action of a carbonate-phosphate melt solution with a significant role of HF, PO₄, CO₂ in the fluid concentrating niobium. This is confirmed by the fact that ore "microclinites" are characterised by a heterogeneous distribution of the ore component, which more often forms veined and veined-spotted ore textures, while potassium feldspar is breccated, recrystallized and corroded (dissolved). From the experimental work [21], where melting was carried out in systems of this kind at 800°C, it was found that when a dolomite carbonate melt is exposed to potassium feldspar, phlogopite and calcite are formed, while pyrochlore and niobium phases crystallize along the metasomatic contact next to phlogopite between potassium feldspar and carbonate (or between glimmerite and carbonatite) [21]. In this work [21], a model was proposed for the formation of ore glimmerites associated with the action of a carbonate melt on the enclosing potassium feldspar metasomatites. This model can explain the formation of ore metasomatites of potassium feldspar, mica-feldspar and mica rocks of the Bolshetagninskoye deposit.

Apatite-pyrochlore ores from the Chuktukon-skoye carbonatite deposit (Chadobetskoye uplift,



outskirts of the Siberian Platform) are similar to similar ores from the Bolshetagninskoye deposit [22]. The data obtained may indicate the important ore producing importance of fractionation of the carbonatite system with the separation of the alkaline phosphate rare metal phase [22], which contains significantly increased concentrations of niobium compared with the enclosing metasomatites and carbonatites.

The phenomenon of immiscibility in phosphate-silicate and phosphate-aluminosilicate systems has long been studied and confirmed experimentally [23, 24]. These experiments have shown the area of wide immiscibility in silicate and phosphate melt and suggest the liquation nature of the genesis of a number of apatite deposits associated with ultramafic alkaline rocks, for example, apatite-nepheline ores of the Khibinsky massif [24]. Experimental work [24] showed that in the “dry” system of the $Ab - Di - NaPO_3 - Nb_2O_5 - Ta_2O_5$ composition, niobium and tantalum are concentrated mainly in the phosphate phase rather than in the silicate phase, so it can be assumed that the formation of pyrochlore is possible in the phosphate phase in such systems. Experimental studies of niobium-bearing haplocarbonatites have shown that pyrochlore can be the initial crystallizing phase in a wide range of pressures and temperatures in pyrochlore apatites [25–27]. For example, Roger Mitchell in his work [27] showed that in the $CaCO_3 - Na_2CO_3 - (Ca, Na)Nb_2O_6 - F$ system, pyrochlore is the initial liquidus phase when the fluorine content in the melt exceeds 1 wt. % at temperatures ≥ 900 °C; a decrease in fluorine concentrations and temperature leads to the formation of a sodium niobate phase.

The work² indicates that igneous calcite carbonatites of the Bolshetagninsky massif are characterised by a very low Nb_2O_5 content (hundredths, rarely tenths of wt. %), and no ore bodies with industrial mineralization have been identified in them. Among the ores studied, in addition to apatite-mica and apatite-pyrochlore rocks, we found calcite ore carbonatites with different niobium contents (from cut-off grades to rich contents) and potassium-feldspar-carbonate rocks (Nb_2O_5 content ~ 0.5 wt. %).

Pyrochlore in ores is observed in the form of idiomorphic inclusions in apatite-pyrochlore rocks and idiomorphic grains and clusters of rounded grains among apatite-mica rocks, as well as in the

form of idiomorphic single grains among calcitic carbonatites. Secondary niobium ores were most likely formed by changes in the primary apatite-mica rocks, which is confirmed by a decrease in the niobium content in the altered apatite-mica rocks and in various numerous oxide mineral forms of niobium, which form overprinted mineralization in other rocks of the massif. Secondary ores are associated with newly formed pyrochlore and with the products of changes in the original pyrochlore deposited in the altered rocks of the massif due to hydrothermal and metasomatic processes with a significant role of the introduction of iron. Secondary overprinted mineralization is represented by ferrocolumbite, which could pseudomorphically completely or partially replace pyrochlore, ilmenorutyl, niobium-bearing rutile, and niobium-bearing hematite.

Conclusion

As a result of the study, it was concluded that niobium mineralization in the Bolshetagninskoye deposit is associated not only with pyrochlore-apatite, potassium feldspar metasomatites and apatite-mica rocks (as revealed by the predecessors) but is also found in calcite carbonatites and other various metasomatites.

It has been established that primary niobium ores are associated with apatite-mica and apatite-pyrochlore rocks. The key primary mineral concentrator of niobium in these rocks is fluorocalciopyrochlore, and ferrocolumbite is also noted. Pyrochlore is significantly associated with apatite. Primary niobium ores, unlike secondary ores, are characterised by a low degree of metasomatic and postmagmatic changes, which led to the discovery of niobium in new mineral forms and, as a result, a decrease in niobium concentrations.

Unmodified fluorocalciopyrochlores from apatite-pyrochlore, apatite-mica rocks and calcite-feldspar rocks are characterised by a stable composition of CaO , Na_2O и SrO . More modified, hydrated differences (according to the vacancy in position A) and pyrochlore compositions that do not correspond to the composition of fluorocalciopyrochlore are more typical for metasomatic rocks (БТГ-18) and calcitic carbonatites (БТГ-50) with overprinted pyrochlore mineralization, which reflects the varying degree of fluid processing of these pyrochlores.



Intensive postmagmatic processes significantly affected the redistribution of niobium and its redeposition in new geochemical conditions, forming secondary ore deposits. In these metasomatic formations, niobium is contained in the form of large, newly formed fluorocalciopirochlore, enriched significantly with uranium and tantalum. In calcite carbonatites, mineralization has an overprinted veined-impregnated, veined character. It was also revealed that the ores of the massif underwent a multiphase, multistage transformation, secondary niobium mineralization is associated with the products of pyrochlore change – newly formed ferrocolumbite, niobium-bearing rutile,

ilmenorutyl and niobium-bearing hematite. The hydrothermal transformation of ores in the final stages completed the multi-stage history of the formation of niobium mineralization. These secondary ores may be of interest as complex raw materials for niobium, titanium and iron. Primary types of ores (apatite-pyrochlorite and apatite-mica rocks) may also be of industrial interest for phosphate raw materials. The new data obtained can be taken into account when constructing a scheme for mining ore deposits to fully extract the ore component and to develop a scheme for processing new complex types of ores from the deposit.

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