Issue VII, November 2024
e-ISSN 2707-9481 Institute of Metallurgy and Ore Beneficiation JSC, Satbayev University, Almaty, Kazakhstan
ISBN 978-601-80473-3-6 https://doi.org/10.31643/2024.13

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Physical and chemical studies of substandard ilmenite concentrate from the deposit of Obukhovskoye

Abstract: The results of physical and chemical studies of a representative sample of substandard titanium concentrate from the Obukhovskoye deposit are presented. According to the results of X-ray phase analysis, the main components of the concentrate are ilmenite and synthetic rutile, titanium chromium oxide iron chromate, etc. Mineralogical analysis data indicate that the concentrate consists of rutile and ilmenite. The conducted electron probe studies have established that rare and rare earth elements in ilmenite concentrate are found in various minerals in the form of monazite inclusion. The results obtained will be taken into account when developing methods for the complex extraction of valuable components from this raw material.

Keywords: ilmenite concentrate, titanium, chromium, rare earth elements, physical and chemical study.

Introduction

Titanium-bearing placer deposits of the Republic of Kazakhstan contain mainly ilmenite. There is a shortage of titanium raw materials in Kazakhstan, so it has become urgent to process chromium-containing ilmenite concentrates from deposits such as Obukhovskoye and Shokash. The high chromium content in these concentrates makes it difficult to further process them into titanium-containing slag, titanium tetrachloride, and spongy titanium. This poses challenges with the transition of chromium into production waste, requiring extensive purification of the raw material (Akhmetova et al., 2020; Krysenko et al., 2020; Khursanov, Hasanov, Tolibov, 2023)).

The primary types of chromium impurities found in ilmenite concentrates are chromium-containing spinel minerals, specifically spinels that are rich in chromium, such as chromite ($FeCr_2O_4$) and magnesiochromite ($MgCr_2O_4$). In current industrial processes, chromium impurities are removed from ilmenite concentrates through a magnetization firing procedure (Ahmad et al., 2016; Lv et al., 2016).

The current Obukhovskoye deposit which located in the North Kazakhstan region produces industrial products such as zircon-rutile and ilmenite concentrate. The ilmenite concentrate meets the requirements of consumers in terms of titanium dioxide content, but its high chromium content (7.5-8%) prevents its sale (Kenzhaliyev et al., 2024). The developed technology will allow the production of marketable products: REE concentrate and titanium dioxide, thus diversifying the market of titanium products in the Republic of Kazakhstan.

The Ust-Kamenogorsk Titanium and Magnesium Plant, operating in Kazakhstan (UKTMP JSC), receives ilmenite concentrate from the Satpaevskoye field, which accounts for about 30% of its raw materials (the rest is imported from Ukraine). The plant produces titanium metal in the form of ingots and slabs. Ilmenite concentrates from the Obukhovskoye deposit are currently not processed at titanium-magnesium plants due to their high chromium content, despite having sufficient titanium content (Tuleutay et al., 2018).

In world practice, ilmenite concentrates with a high chromium content are practically not processed. In the Republic of Kazakhstan, technologies for processing titanium-containing raw materials with the formation of titanium slags from conditioned ilmenites have been developed, and the titanium-magnesium combine of JSC UKTMKP operates on their basis (Ultarakova et al., 2021).

There are still no implemented technologies for processing chromium-containing ilmenite concentrates. Thus, the purpose of the research is to study to process of substandard ilmenite concentrate and it is necessary to obtain objective data on the composition of the initial ore for the further complex processing of this raw material with the extraction of all valuable components.

Research Methods

The following methods of analysis were used to investigate the composition of the ilmenite concentrate: chemical, X-ray phase, X-ray fluorescence, and electron scanning microscopy. Also, the following types of equipment were used for the study: atomic emission spectrometer Optima 2000 DV (PerkinElmer, Inc., Waltham, MA, USA); X-ray diffractometer D8 Advance BRUKER, Cu α-radiation (BRUKER AXS GmbH, Karlsruhe, Germany); X-ray fluorescence spectrometer Venus 200 PANalytical B.V. (Malvern Panalytical, Almelo, The Netherlands); LEICA DM 2500 P (Leica Microsystems GmbH, Wetzlar, Germany) and JEOL JXA 8230 Electron Probe Microanalyses (JEOL Ltd., Tokyo, Japan).

Research Results

The physical and chemical properties of a batch of substandard ilmenite concentrate from the Obukhov deposit have been studied.

Chemical composition, weight. %: 39,8 Ti; 31,4 Fe; 2,8 Cr; 0,6 Al; 0,4 Si; 0,004 Sr; 0,018 Y; 0,013 Th. The results of the X-ray phase analysis are shown in Table 1. XRD of ilmenite concentrate is shown in Fig. 1.

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

№	Compound	Content
1	Ilmenite $Fe_{1.04}Ti_{0.96}O_3$	28.4 %
2	Iron chromate $FeCrO_3$	14.4 %
3	Rutile syn. $Ti_{0.912}O_2$	11.7 %
4	Titanium chromium oxide $(Ti_{0.97}Cr_{0.03})O_2$	10.5 %
5	Pseudorutile $Fe_2Ti_3O_9$	10.3 %
6	Titanium iron oxide $Fe_2Ti_3O_9$	10.2 %
7	Panguite $Ti_{1.67}O_3$	8.1 %
8	Iron oxide (III) Fe_2O_3	6.4 %

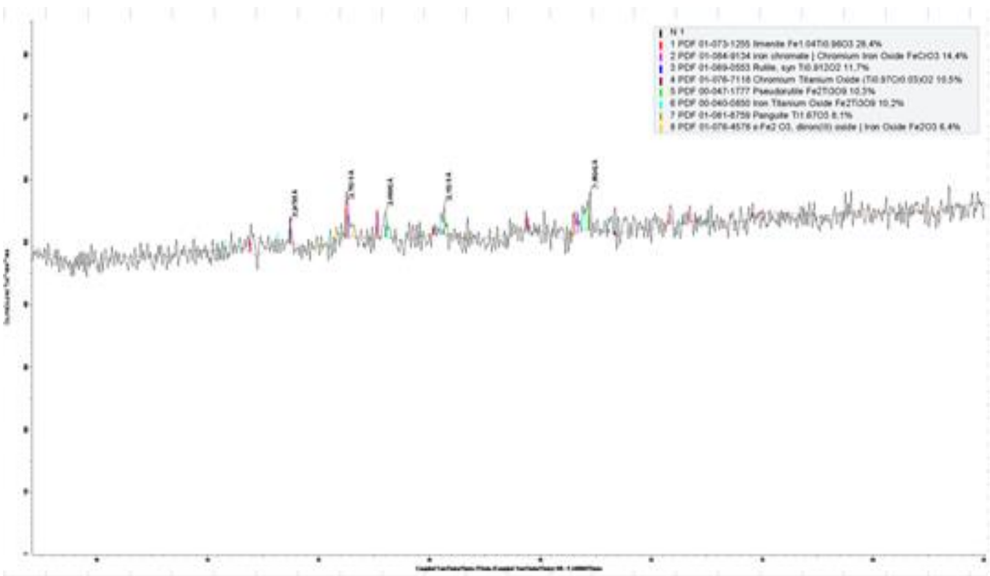


Figure 1 – XRD of ilmenite concentrate

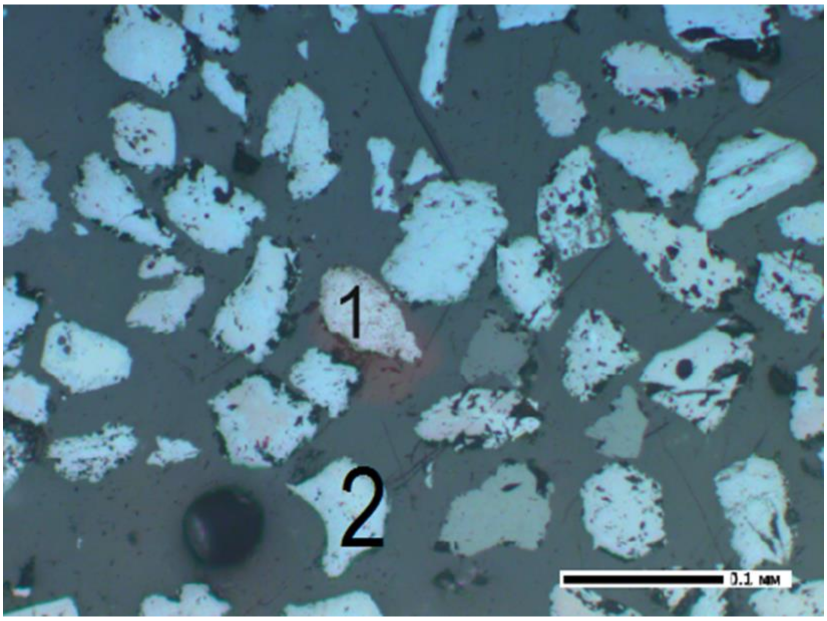
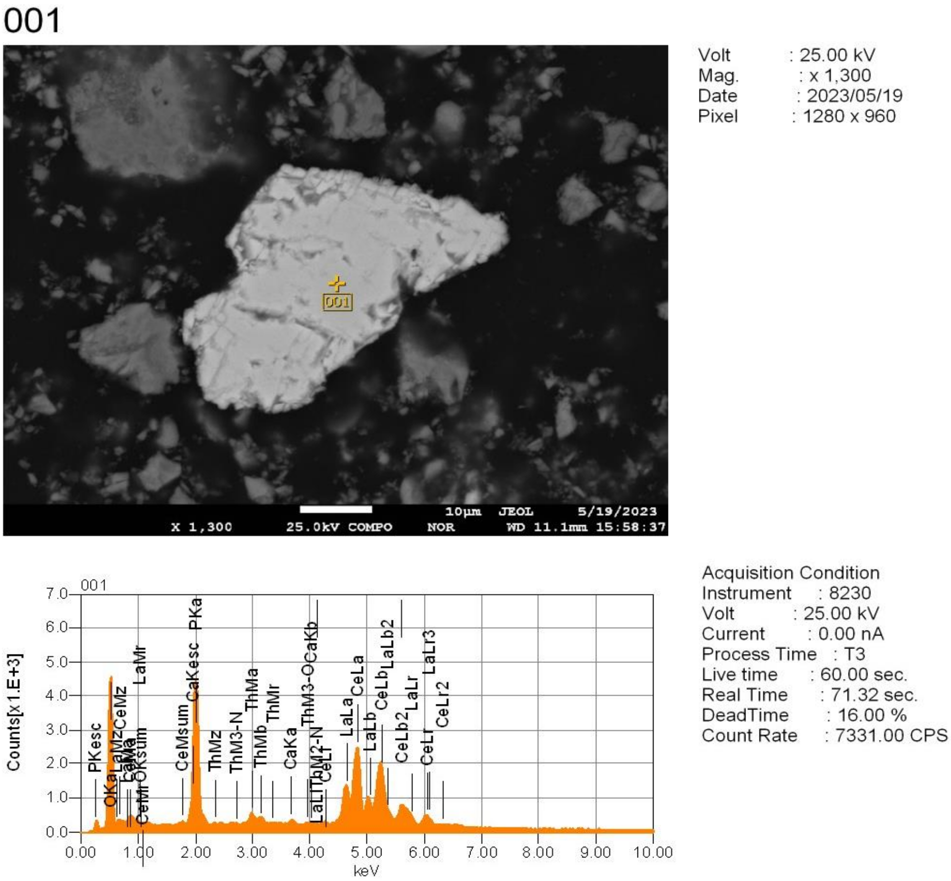


Figure 2 - Rutile (1) and ilmenite (2). Sample №1, ×200



Chemical formula	ms%	mol%	Sigma	Net	K ratio	Line
O	40.23	76.18	0.21	923960	0.1946111	K
P	14.28	13.97	0.12	1479674	0.0982318	K
Ca	0.55	0.41	0.13	72735	0.0045679	K
La	11.47	2.50	0.41	724505	0.0830271	L
Ce	29.92	6.47	0.40	2061241	0.2291432	L
Th*	3.56	0.46	0.43	204496	0.0205343	M
Total	100.00	100.00				

Figure 3 - The results of the EDS analysis of REE containing particles of ilmenite concentrate

According to the results of X-ray fluorescence and chemical analysis, chromium is present in the sample. According to the results of X-ray phase analysis, chromium is represented in the concentrate by minerals of the spinel group, namely spinel (aluminochromite), and chromite (chrompicotite).

The study involved analyzing the material composition of ilmenite concentrate samples from the titanium-zirconium placers of the Obukhovskoye deposit. The samples were crushed for X-ray phase and X-ray fluorescence analyses. Polished artificial plates (briquettes) were made from the samples for further study.

Rutile (TiO_2) is a light gray mineral with low reflectivity. It has thick brown internal reflexes with a reddish tinge and is anisotropic (Sun et al., 2011). It is marked in the form of an anhedral shape (Fig. 2).

Ilmenite ($FeO{\cdot}TiO_2$) is a gray mineral with high hardness, clear reflection, and strong anisotropy. It has an irregular shape with sinuous borders (Fig. 2).

With the magnification of x1300 (Fig. 3), the phases of the rare earth elements monazite (Ce, La, Nd, Th) were detected in the studied sample [PO_4]. Monazite is a mineral belonging to the lanthanide phosphate class, mainly cerium (Ce), lanthanum (La), neodymium (Nd), praseodymium (Pr), thulium (Tm), gadolinium (Gd), samarium (Sm), as well as scandium (Sc), yttrium (Y), along with lanthanides, they are classified as rare earth elements and impurities of actinides — thorium (Th), uranium (U) with the general chemical formula $M(III)PO_4$ (Naumov, 2008). Due to the content of thorium and uranium, it is weakly radioactive. It is an ore of rare earth elements and thorium.

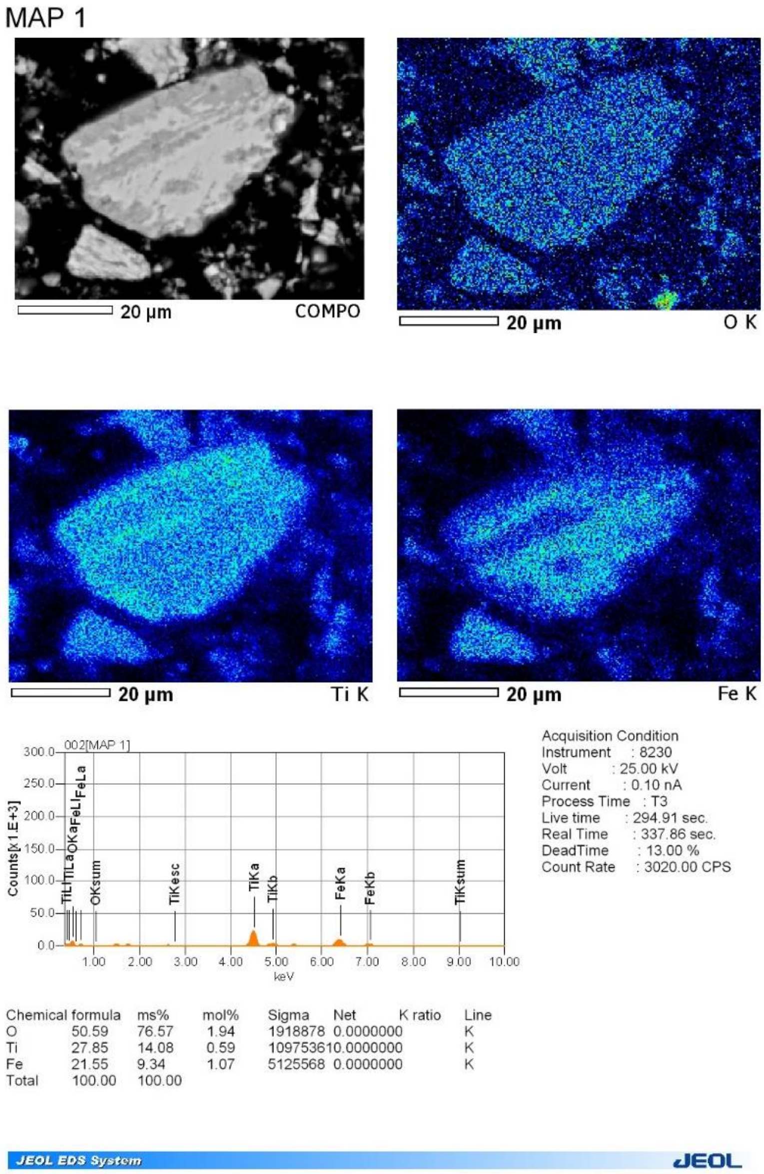


Figure 4 - SEM-EDS elemental mapping of ilmenite concentrate

The elemental mapping using SEM-EDS was performed to obtain information on the distribution of Fe, Ti and O elements between phases in the samples. Furthermore, this supported the findings of the chemical analysis and EDS analysis.

Conclusion

Ilmenite concentrates from the Obukhovskoye deposit are currently not processed at titanium-magnesium plants due to their high chromium content, their processing is relevant in terms of expanding titanium raw materials. Thus, in the process of physical and chemical studies, objective data were obtained on the material composition of substandard ilmenite concentrate from Obukhovskoye deposit. It also identifies the features of the minerals present in the composition. The results of the electron probe analysis showed the activation of the REE phosphate lattice using the example of monazite. These results are crucial for developing methods to extract valuable components from this raw material effectively. The obtained results should be taken into account when developing methods for the complex extraction of valuable components from this raw material.

CRediT author statement: **A.Toishybek**: Software, Data curation, Writing draft preparation, Reviewing and Editing **A.Ultarakova**: Conceptualization, Methodology, Supervision, Reviewing and Editing. **N.Sadykov**: Visualization, Investigation. Reviewing and Editing.

Acknowledgement. This work was supported by the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan, Program-Targeted Funding BR18574006.

Cite this article as: Toishybek, A., Ultarakova, A., Sadykov N. (2024) Physical and chemical studies of substandard ilmenite concentrate from the deposit of Obukhovskoye. *Challenges of Science*. Issue VII, pp. 96-100. https://doi.org/10.31643/2024.13

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