5. Now some manganese open pits of the Pokrovsky MPE are under cleaning-up of ore reserves. In this respect researches of a reengineering of a technological scheme of mining operations under cleaning-up of pit area are needed. In this aspect, not enough attention is still being paid to the solution of issues related to the technology for cleaning-up of deposit and the closure of open pits, as well as the production costs for the improvement of their surface.

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X-RAY COMPUTED MICROTOMOGRAPHY AS THE BASIS FOR MINERAL PROCESSING IMPROVEMENT : REVIEW

This article provides a brief review of the application of the X-ray computer microtomography in the studies of mineral raw materials, as well as the relevant generalized results of the authors' own research. It is noted that X-ray computed microtomography is one of the promising introscopic methods for studying mineral raw materials, which has recently been successfully used in researches of ores, rocks, coal, mining and processing waste, composites and other materials. The advantages of tomography, as compared to other x-ray and optical methods, consist in its efficiency and high information content at minimized human factor.

Along with the growing popularity and relevance of the method, the number of problems to be solved using x-ray microtomography is also increasing. In particular, a very urgent problem is to study dynamics of change in internal structure of rocks exposed to various force fields (compression, tension, temperature and other effects) in practical geotechnology and mineral processing. Being a non-destructive research method, X-ray computed microtomography has a number of undeniable advantages in studying processes of deformation and fracture of ores and rocks as it allows examining pore space structure of rock samples without destroying them. At the same time, the review of relevant publications demonstrates that in each case, the tomographic characteristics of separate (or single) samples are studied and described depending on a specific objective. At the moment, no generalized dependencies have been identified to describe the relationship between the structures of pore space and the physical and mechanical properties of rocks. Computed tomography enables quantitative characterization of the pore space structure with regard to the physical and mechanical properties of rocks, which provides the grounds for substantiating energy-efficient crushing methods with allowance for textural and structural features of ores and rocks.

Key words: *computed X-ray microtomography, ores, rocks, pore space.*
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Introduction

The development of advanced resource-saving technologies for mineral processing requires a comprehensive study into material composition of minerals, their structural features and behavior of mineral phases in disintegration and concentration. In this regard, there is a need to improve the methodological approaches to the study of ores and rocks and to apply advanced instrumental diagnostic methods in the mineral engineering.

X-ray computed microtomography is one of the promising introscopic methods for studying mineral raw materials, which has recently been successfully used to study ores, rocks, coal, mining and processing waste, composites and other materials.

This article provides a brief review of the application of the X-ray computer microtomography method in the studies of mineral raw materials, as well as the relevant generalized results of the authors' own research.

Application of X-ray tomography in mineral research

X-ray tomography is a non-destructive method for studying the internal structure of solid materials using the dependence of the linear radiation attenuation coefficient on the chemical composition and density of a substance under analysis. Computer processing of shadow projections obtained in x-ray scanning of samples allows visualizing the internal three-dimensional structure of each sample and performing a detailed analysis of its morphometric and density characteristics both in different sections and throughout the sample volume with quantification of parameters [1].

This method allows identifying structural defects (pores, cracks, inhomogeneities, gas–liquid inclusions), determining mineral composition and reconstructing the distribution of phases and defects in different sections and throughout the volume of the sample studied with quantitative assessments (number of pores, microcracks, particles, etc., their linear dimensions, volumes, surface area, shapes, connectivity and cohesion). The sensitivity of X-ray tomography to local inhomogeneities is much higher than in radiography (X-ray imaging); the spatial resolution of microtomographs is from units to several tens of microns, which corresponds to the size of crystals and pores in rock samples [2].

Tomography, as a method of medical diagnosis, was developed in 1972 by G. Hounsfield and A. Cormack [3]. Subsequently, the method found application in various industries for studying defects in solids (industrial tomography). The use of X-ray tomography for examining the microstructure of ores and rocks in our country was suggested by M. S. Khozyainov and E. I. Vainberg in 1992 [4]. A major contribution to the development of the methodology and technology for computed microtomography of geo-objects was made by O. A. Yakushina and E. G. Ozhogina [5–7].

Regarding advantages over other x-ray and optical methods, tomography is highly informative in assessing phase composition, nature of dissociation of ore minerals and heterogeneity of grains and aggregates in technological and mineralogical studies [8].

Microtomography provides a higher degree of accuracy, as compared to the traditional mineralogical analysis, in estimating the volume fraction of minerals without preliminary sample preparation [9–10]; however, the use of this method in ore dressability studies is somewhat limited due to the impossibility of identifying non-contrasting X-ray phases [11].

Along with the growing popularity and relevance of the method in studying the properties of ores and rocks, the number of problems to be solved using x-ray microtomography is also increasing. In particular, a very urgent problem is to study the dynamics of changes in the internal structure of rocks exposed to various force fields in practical mining and mineral processing.

For instance, microtomography of building stone sample enabled further studies into dynamics of a joint system in freezing and thawing [12–14]. It has been found that in cyclic freezing and thawing of rocks, the pore space structure changes not only in the surface layer but also in the rock volume (**Fig. 1**), which made it possible to formulate a new approach to assessing the durability of natural stone [15].

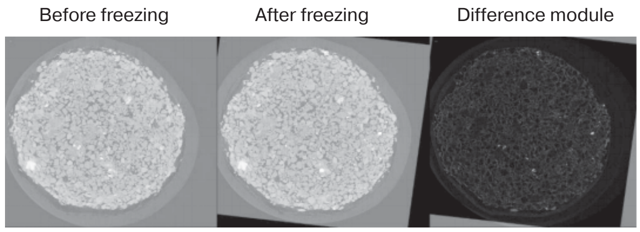

Fig. 1. Combination and comparison of two 3D-models of a sample before and after freezing [26]

The method has been successfully applied to assessing the changes in the structure of the void space in ore disintegration in crushers [16], as well as in studying processes of deformation and fracture of rocks [17–21]. The mechanisms of deformation and fracture of rock and coal samples under dynamic impact have been studied; the results of tomographic researches are of great importance for the geomechanical support of geotechnical solutions in mineral mining, including the research of such phenomena as rock bursts and coal and gas outbursts [18, 19].

Microtomography is the main method for studying the reservoir/filtration properties of rocks oil and gas geology [22–25].

A method has been developed for combining images obtained by tomography (Fig. 1) to study influence of various physical and chemical processes and effects on the mineral skeleton and porosity structure, including acid treatment, mechanical loading, thermal effects, freezing, and diffusion of heavy elements in the pore space [26].

The x-ray computed tomography has been increasingly applied both in theoretical and applied research. In particular, it is used by oil companies to scan rock cores for determining their bulk density. Some research laboratories in the Russian oil and gas sector are equipped with X-ray tomography scanners, which are mainly used to scan core samples to identify their most informative sections for conventional logging. Tomography is applied in certification and digital archiving of samples and core fragments, as well as in determination of their geological (petrophysical) parameters [27, 28].

X-ray computed microtomography has a number of undeniable advantages in studies of disintegration of ores and rocks. This approach to the analysis of rock deformation and fracture is based on the assessment of structural heterogeneity and structural defects at the micro level. This allows solving the problem of developing and substantiating energy-efficient fracturing along the interfaces of mineral phases.

A significant drawback of the existing approaches to the disintegration process investigation is the fact that most of these methods are destructive and exclude long-term observations of the behavior of rocks in various physical fields. Regarding rocks, due to the heterogeneity of their composition and structure, it is practically impossible to find samples with absolutely identical properties; therefore, structural characteristics and mineral composition of rocks and their mechanical properties are studied using separate samples, which introduces significant limitations and assumptions in the interpretation of the experimental data.

Computed tomography allows examining pore space of rock samples without destroying them. In the proposed procedure (**Fig. 2**), it is possible to obtain a tomographic image of the initial (unloaded) sample, then to subject the sample to loading without destruction, and to take a tomographic image of the loaded sample after that. The resultant three-dimensional models demonstrate the spatial location and the quantitative volume of the void space corresponding to the volume of cracks formed under mechanical impacts [29].

A promising approach to studying rock structures in disintegration is represented by the procedure and software developed at the Gubkin Russian State University of Oil and Gas. The computer program for detecting cohesions in a void space allows identifying the structural anisotropy of a structure. In order to analyze a heterogeneous void space, a series of data sets have been created and adapted to solving of particular problems such as determination of connectivity of elements in the void space, or structural modeling of the pore space with elements of orientation, shape, connectivity and pore size [30].

The use of a special add-on device and integrated tomography scanner software for tension/compression testing of specimens enables registration of displacements of inhomogeneities under various deformations.

In ore and rock disintegration research, an undoubted advantage of computed microtomography consists in the ability to identify quantitative characteristics of the pore space structure, which provides the grounds for predicting the relationship between the morphometric parameters of rocks, properties of the crushing product and the production process characteristics, in particular, energy consumption [31].

Basic areas and result of rock disintegration research by microtomography

For some years, the authors have studied the structure of pore space and mineral matter in the main types of igneous rocks, namely, gabbro-diabases and granites of various compositions (plagioclase, microcline-plagioclase), in order to improve the disintegration process.

The current approach to rock deformation and fracture research is based on the assessment of structural heterogeneity and defects in rocks at the micro level. This approach allows solving the problem of developing the methods for energy-efficient fracturing along the interfaces of mineral phases.

Thus, the main objectives of microtomography in solving practical problems associated with disintegration of rocks include:

- acquisition of data for quantification of pore space structure (pore content, size, sphericity, connectivity);
- differential characterization of porosity (pore distribution by quantity, size and sphericity);
- visualization of the structure of samples both in different sections and in the total volume to identify any possible heterogeneity in pore distribution.

Since the experimental studies of rock microstructure are to set a threshold minimum pore size that affects the strength characteristics of rocks, the tomography scanner model should be selected so that to ensure the required resolution. For another thin, after the tomography scanning, the samples will be tested to determine their physical and mechanical properties, and the samples should have fixed dimensions therefore (diameter/height of 40–50 mm). SkyScan-1172 and SkyScan-1173 tomography scanners (Belgium) meet these requirements. The minimum spatial resolution of these tomography scanners is 0.5 µm, which corresponds to the minimum pore sizes in the structure of rocks.

The research was carried out in the areas described below.

1. Comparative assessment of the pore space structure in rocks (granites, gabbro-diabase) with regard to their strength.

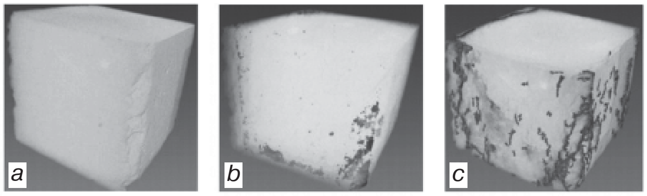

Fig. 2. Three-dimensional model of a rock salt sample: (*a*) before testing; (*b*) after loading stage I; (*c*) after loading stage II [29]

Tomographic characteristics of rock samples (3D system)

Parameter	Values for samples, from-to / average	
	Gabbro-diabases (8 samples, σ_{comp} = = 160÷259 MPa)	Granites (10 samples, σ_{comp} = = 100÷125 MPa)
Pore concentration, mm^{-3}	37–763 222	13–340 150
Pore fraction in sample volume (porosity), %	0.24–0.66 0.39	0.69–1.01 0.82
Largest pore size, µm	24–147 74.6	79–620 350.5
Median pore size, µm	4.4–24.1 12.6	5.6–91.8 31.0
Comment: σ_{comp} is the ultimate compression strength.		

The comparison of the microtomography data with the strength test results shows that gabbro-diabases (ultimate compression strength σ_{com} = 160–259 MPa) have higher porosity as against granites (σ_{comp} = 100–125 MPa), although, there is no dependence between the strength and pore concentration (See **Table**). Namely, the gabbro-diabase sample with the highest pore concentration (763 mm^{-3}) is the sample with the lowest total porosity of 0.24%.

The lack of correlation between the total porosity and pore concentration is explained by the different pore sizes: the largest pore sizes are 24–147 µm in the gabbro-diabase samples and 79–620 µm in the granite samples.

In this regard, the quantitative distribution of pore sizes is very important. The concentration of pores decreases with an increase in their size (**Fig. 3**); this regularity is observed in all samples studied. The number of hypercapillary pores in the samples is insignificant. The overwhelming number of pores in the rocks studied have small capillary sizes. Instead of these pores, the strength of the rocks is determined based on elongated pores, even despite their insignificant contribution to the total porosity.

When determining the pore concentration, it is of fundamental importance to evaluate the minimum pore size. While the maximum pore size can be found quite accurately and, given the geometric shapes, can be estimated by the largest pore diameter, the minimum pore size is governed by the spatial resolution of the tomography scanner. Thus, the pore concentration, as a value depending on the experimental setup, may not be an objective characteristic of the pore space.

The dependence of the pore sphericity on the pore size has been determined: pores smaller than 20 µm are close to isometric in shape; the sphericity of pores of up to 80–100 µm

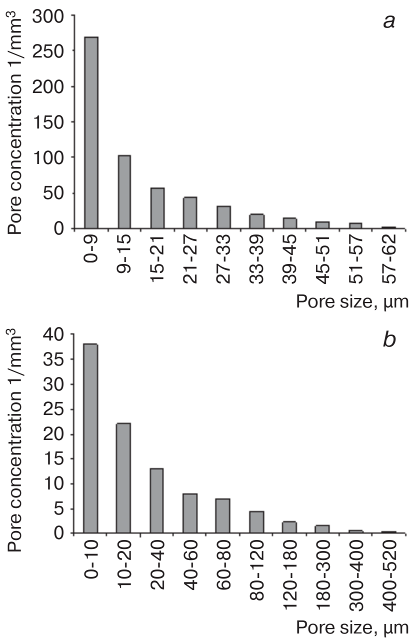

Fig. 3. Pore size distribution:
a) gabbro-diabase sample; *b*) granite sample

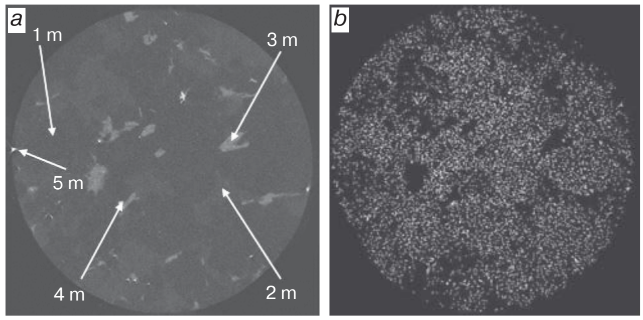

Fig. 4. Spatial distribution of mineral components (*a*) and pores (*b*) in granite sample (2D system):
1m: microcline; 2m: plagioclase; 3m: biotite; 4m: apatite; 5m: titanite

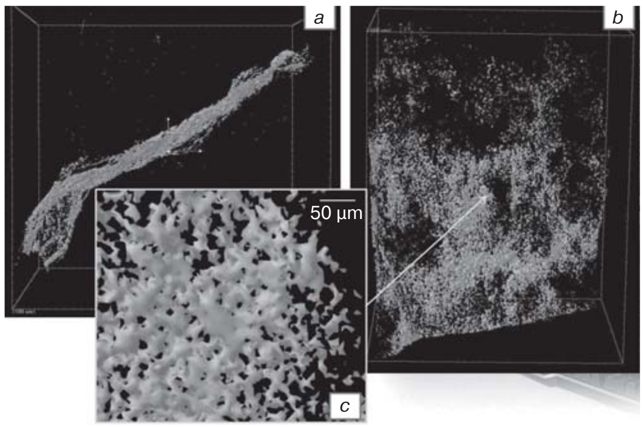

Fig. 5. Crack structure in loaded granite sample (3D system):
(*a*) top view; (*b*) side view; (*c*) cavity detail (white) in cracks

does not exceed 0.25–0.3, and pores of 100–140 µm have an elongated shape (0.1 and less). This dependence explains the high strength of rocks characterized by small capillary pore sizes and is consistent with Griffith's theory, according to which the overall strength of a body depends on its most injurious defect (the longest pore). In some samples, the largest pore size does not exceed 24–30 µm, which a priori suggests the high strength of these rocks.

2. Analysis of the spatial distribution of pores in the structure of granite.

Pores are unevenly distributed in the structures of samples. A high pore concentration is typical of feldspar grains (microcline and plagioclase). In all granite samples studied, pores are mainly concentrated in microcline and plagioclase grains with predominance in microcline (**Fig. 4**). The study of a microcline grain shows that its porosity is more than three times higher than the total porosity of the whole sample. If the total porosity of the test sample is 1.01% and the pore concentration is 166 mm^{-1}, then the microcline grain porosity is 3.28% and the pore concentration is 725 mm^{-1}.

Considering that the pores are distributed unevenly in the rocks, fracture originates in different weakened areas in the sample volume, with the highest concentration of the largest pores. In the granite samples studied, these areas are represented by microcline grains. The results satisfactorily explain the decrease in the strength of granites with increasing content of microcline [32].

3. *Study of granite structure transformation under uniaxial compression.*

It has been found that under uniaxial compression to ~90% of the expected ultimate compression strength value, a region of increased pore concentration is formed in the sample. Structurally, this region is a system of cracks emerging on the surface, with micron-sized mineral particles (**Fig. 5**).

It should be noted that the phenomenon of microparticle emission under dynamic loads was previously experimentally revealed in the tomographic studies of S. D. Viktorov and A. N. Kochanov [18]. Their work proves that the size and quantity of microparticles depend both on the petrographic features of rocks, as well as on the fracturing method and conditions.

In this regard, the phenomenon of microparticle emission requires a further detailed study in solving problems of rock disintegration, when the formation of ultrafine varieties must be minimized.

In the undisturbed areas, rock compaction occurs due to the closure of intergranular and intracrystalline pores. The closure of intracrystalline pores leads to the change in the geometric characteristics of crystals.

The comparison of the shadow projections in x-ray radiation shows the irreversible change in the linear dimensions of the sample after unloading. Longitudinal strains corresponding to normal stresses result in reduction of the sample height and make 20 µm or 0.19% (rel.). Transverse strains lead to the increase in the sample diameter in different sections by 21–65 µm or 0.19–0.58% (rel.) The sample volume increases by 0.3% (from initial 1.068 cm^3 to 1.071 cm^3 after loading). Thus, dilation that develops in the rock sample is manifested not only by initiation of microcracks but also by transformation of the whole volume of rock and its structural elements (pores and crystals).

4. Studies into dynamics of change in the pore space structure of rocks depending on the magnitude of the destructive force.

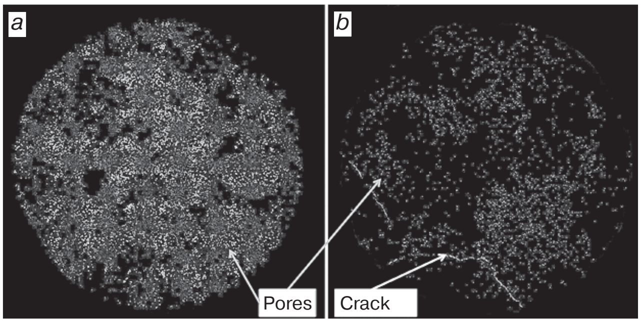

Fig. 6. Tomographic section in granite sample
(*a*) initial state; (*b*) after loading at 27.5% of expected ultimate compression strength

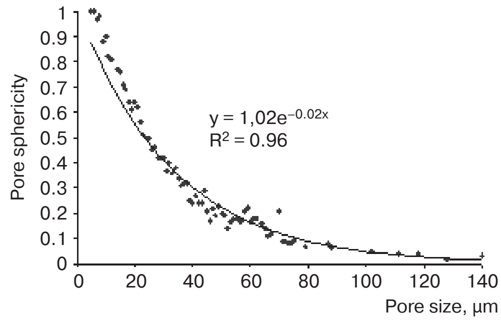

Fig. 7. Dependence of pore sphericity on pore size

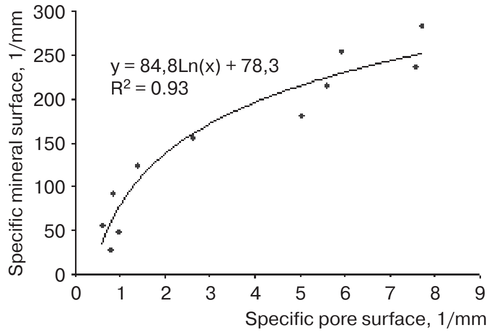

Fig. 8. Relationship between specific surface of minerals and specific surface of pores

In the current theory rock disintegration, fracture is a process that develops in time. The formation and development of microscopic disturbances begin in rock upon application of a loading force, either dynamic or static, and continue over the whole period of loading until fracture.

In connection with this, the effects occurring in rocks under compressive loads corresponding to approximately ~10–90% of the expected ultimate compression strength were studied.

During the tests, x-ray tomography of the initial samples of granite and gabbro-diabase was carried out, with quantitative assessment of contents of mineral components and determination of the pore space parameters such as total porosity, number, concentration and sizes of pores.

Then, the samples were loaded on a hydraulic press to the values corresponding to 10–90% of the expected ultimate compression strength at an increment of ~20 MPa. After each loading cycle, microtomographic survey was repeated.

As a result, the dynamics of change in the pore space structure under compressive loads was studied and it was found that within the test range, the volume of the pore space changed in the samples. At the initial loading stage (10–40% of the expected ultimate compression strength), the rock is compacted, which is accompanied by a decrease in the volume of the pore space. The further increase in the load leads to granite decompaction (**Fig. 6**) owing to an increase in the total porosity and pore size; while gabbro-diabase is characterized by compaction in the entire loading range studied.

5. Studies of interrelations of pore space structure parameters in rocks.

The review of relevant publications demonstrates that in each case of a specific objective, the scope of the studies and description embraces tomographic characteristics of separate (or single) samples. Computed tomography allows finding exact numerical values of the parameters characterizing mineral substance and pore space and, thereby, which offers an opportunity to interrelate them in quantitative terms.

Generalization of the tomographic survey results on a statistically significant number of granite and gabbro-diabase samples enabled identification of certain general dependencies and patterns characterizing relationship between parameters of rock microstructure.

Namely, anisotropy of pore shape, estimated in terms of the sphericity index, lowers in going from small to large pores (**Fig. 7**).

The interrelation of pore sizes and sizes of mineral grains was set (**Fig. 8**). These patterns are observed in all samples studied.

The relationships obtained confirm the theory of Academician P. A. Rebinder, according to which in rocks composed of smaller grains, pores and other defects are smaller, too [33].

Conclusions

X-ray computed microtomography is one of the promising introscopic methods for studying mineral raw materials, This method has recently been successfully used in the analyzes of ores, rocks, coal, mining and processing waste, composites and other materials. The advantages of tomography, as compared to other x-ray and optical methods, consist in its efficiency and high information content at minimized human factor. Microtomography of minerals allows investigating their phase composition, dissociation, heterogeneity of grains, and structure of pore space with quantitative assessments and statistical relationships.

Being a non-destructive research method, x-ray computed microtomography has a number of undeniable advantages in the studies of deformation and fracture of ores and rocks as it enables examining the pore space structure of rock samples without destroying them.

Along with the growing popularity and relevance of the method, the number of problems to be solved using x-ray microtomography is also increasing. In particular, a very urgent problem is to study the dynamics of change in the internal structure of rocks exposed to various force fields (compression, tension, temperature and other effects) in solving certain practical problems of geotechnology and mineral processing.

At the same time, the review of relevant publications demonstrates that in each case, the tomographic characteristics of separate (or single) samples are studied and described depending on a specific objective. At the moment, no generalized dependencies have been identified that would describe the relationship between the structures of the pore space and the physical and mechanical properties of rocks. Computed tomography enables quantitative characterization of the pore space structure with regard to the physical and mechanical properties of rocks, which provides the grounds for substantiation of energy-efficient crushing methods for the specific texture and structure features of ores and rocks.

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STAGE-ACTIVATION LEACHING OF OXIDIZED COPPER–GOLD ORE: THEORY AND TECHNOLOGY

The article emphasizes the urgent need of heap leaching in the current geotechnical conditions. The application prospects for heap leaching are connected with preliminary oxidation of refractory ore. Another important requirement is geo-engineering typification of ore through geotechnical mapping. An alternative of cyanide in gold leaching is chlorine compounds. The article describes the theory of formation of active chlorine–oxygen complexes using electrochemical and photochemical effects. Iron acts as a catalyst of gold complexing by means of change in the oxidation rate +3/+2. The tests of agitation leaching are carried out on oxidized copper–porphyry–gold ore from the Malmyzh deposit. Efficiency of the best reagent scheme is proved by the results of percolation leaching. The laboratory experiment included primary processing of ore in peroxide–sulfuric solution prepared by electrochemical and photochemical treatment. After diffusion oxidation during ageing of treated samples for three days, the samples were irrigated with active hypochlorite–chloride solution for 15 days. The experiment proved recoverability of gold at a rate of 92–93% and copper at a rate of 55–60% without capital input and at minimized operating cost. The new-developed circuit of heap leaching includes layer-by-layer stockpiling. Selectivity of heap leaching is ensured by treatment of layers of qualitatively different ore types and grades by solutions of different reagents, at certain concentrations and in certain hydrodynamic modes of irrigation.

Key words: *copper–porphyry–gold ore, oxidized ore, leaching, geotechnical mapping, stockpiling, electro-photochemical activation, preparatory oxidation, reagents, peroxide–sulfuric solutions, hypochlorite–chloride leaching.*
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Introduction

As against many metals and minerals, the price of gold is governed not only by the demand and supply balance but also depends on numerous industrial, political and economic factors. In gold mines, despite the anticipated rise in the gold price, a sturdy characteristic of a final product is the capital and operating cost dependent on the mineral supplies, as well as on labor, power, material and technical resources. The complex-structure folded ore bodies, with highly heterogeneous distribution of gold per different scales and with varied ratio of gold occurrence forms, need differentiated quality requirements. This necessity was highlighted by such mining scientists as Academician M. I. Agoshkov, Professor Z. A. Terpogosov, Professor S. S. Reznichenko and other as early as the late 1980s. The complex composition of copper–gold ore gives rise to a high scatter of the recovery results and quality data of flotation concentrate. The difference in composition of chalcopyrite, chalcocite and oxidized minerals of copper allows dividing ore into processing types [1]. In this case, the economic performance of processing of various copper–porphyritic ore should be estimated in terms of gold and copper recovery in flotation and heap leaching. At the present time, percentage of gold and copper produced by heap leaching in the world exceeds 40%.

The subject of this research is the Malmyzh copper–gold deposit in the Khabarovsk Krai. The only economically expedient method to recover the low-grade copper and gold ore from the oxidation zone of this deposit is heap leaching [2, 3].

The prospects for gold heap leaching are connected with pre-oxidation of rebellious ore [4], with an increase in the rate and ratio of dispersed gold recovery in order to make this method comparable with the conventional production and processing efficiency reached with both low-grade and high-grade ore. It is advisable to assess applicability of flexible