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Research article

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Prospects for refractory gold-sulfide ore processing

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Abstract: Cyanide-refractory ores constitute 30 % of the world’s gold mineral resource base. With the global decrease in the availability of high-grade and free-milling ores, low-quality ores, including those rich in sulfur and arsenic, are increasingly being processed. The authors have conducted an assessment of the primary factors complicating the leaching process of refractory gold. These factors include the influence of gold distribution within the ore, the presence of preg-robbing effects, and the impact of cyanicidal minerals, notably pyrrhotite, on the leaching process. Sulfide minerals significantly affect the kinetics of gold leaching and associated reagent costs. The behavior of Fe_5S_6 is elucidated through the concept of “chemical depression”. Under cyanide leaching conditions, pyrrhotite actively and directly reacts with NaCN/KCN , undergoing surface oxidation by dissolved oxygen in the pulp. This leads to the formation of ferrocyanide complexes and rhodanides, which are unable to leach gold. Presently, there are two approaches to enhance the process parameters of refractory ore processing technology. The first approach involves the inclusion of preparation operations for cyanidation, aimed at liberating gold from the sulfide matrix (including hydrometallurgical and pyrometallurgical oxidation technologies and mechanical activation). An alternative approach is to use alternative reagents as leaching agents (notably thiourea, sodium and ammonium thiosulfates, and halides). The article explores means of modifying the technological process for gold extraction when ores contain substantial amounts of pyrrhotite or concentrates.

Keywords: gold, refractory ores, cyanidation, preg-robbing, pyrrhotite, pyrite, arsenopyrite, leaching.

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Перспективы переработки упорного золотосульфидного сырья

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Аннотация: Мировая минерально-сырьевая база золота на 30 % представлена упорным по отношению к цианиду сырьем. На фоне глобальной тенденции к снижению добычи богатых и легкообогатимых руд в переработку вовлекается низкосортное сырье, в том числе и с высоким содержанием серы и мышьяка. Авторами оценены основные факторы, затрудняющие процесс выщелачивания упорного золота: влияние форм нахождения золота в сырье, наличие эффекта прег-роббинга, влияние на процесс выщелачивания минералов-цианисидов, в частности пирротина. Сульфидные минералы оказывают

значительное влияние на кинетику процесса выщелачивания золота, а также на расходы реагентов. Поведение Fe_3S_4 описывается понятием «химическая депрессия». В условиях цианидного выщелачивания пирротин активно вступает в прямое взаимодействие с NaCN/KCN , подвергается реакциям поверхностного окисления растворенным в пульпе кислородом с образованием ферроцианидных комплексов, роданидов, не проявляющих выщелачивающую способность в отношении золота. На сегодняшний день существуют два подхода к способам повышения технологических показателей технологии переработки упорного сырья. Первый метод предполагает включение в технологическую схему операций подготовки к цианированию, направленных на раскрытие заключенного в сульфидную матрицу золота (гидрометаллургические и пирометаллургические технологии окисления, механоактивация). Альтернативным подходом является использование в качестве выщелачивателя иных реагентов (наиболее известные из них — тиомочевина, тиосульфаты натрия и аммония, галоиды). В статье рассматриваются способы модификации технологического процесса извлечения золота при значительных содержаниях пирротина в составе руд или продуктов обогащения.

Ключевые слова: золото, упорные руды, цианирование, прег-роббинг, пирротин, пирит, арсенопирит, выщелачивание.

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Introduction

Gold mining is a global industry with operations on every continent except Antarctica. It is recognized as one of the priority sectors in the global economy. However, as the annual production and consumption of gold continue to increase, the accessible reserves that can be economically processed using conventional methods are diminishing [1]. According to the state report titled “On the state and use of mineral resources of the Russian Federation in 2019” [2], China, which contributes 12 % of the world’s total gold production, holds the position of the largest gold producer globally. Russia ranks among the top three gold producers in the world, with a 10 % share of global produc-

tion [2]. In 2020, the output of refined gold decreased to 340.2 tons, a decline of less than 1 % compared to the previous year (Fig. 1).

Over the past 25 years, significant advancements in gold cyanidation have been driven by a variety of factors. These include the diminishing quality of gold deposits, the shift from open-pit to underground mining, the increasing complexity of ore processing, and heightened concerns regarding environmental regulations. As shown in Fig. 2, the primary trends in gold mining innovation are closely tied to the utilization of three key chemical agents: thiosulfate, cyanide, and halides/ HCl [3]. A comprehensive evaluation of gold recovery technologies employed at contemporary industrial facilities reveals that the use of cyanide remains the prevailing method.

Approximately 30 % of the world’s gold reserves are considered highly refractory, characterized by high NaCN consumption and suboptimal gold recovery rates. To tackle this issue, two primary approaches have been employed [4]:

- continuing with cyanide technology;
- employing alternative leaching reagents, to which the ore is less refractory.

Traditionally, the classical method involved oxidizing the sulfide component of the ores or concentrates through roasting. However, the high costs associated with purifying the resulting gases, which contain dust, arsenic, and sulfur, have raised questions about the feasibility of this technology [20]. Modern ore pre-treatment methods for cyanidation have shifted towards hydrometallurgical processes, such as autoclave oxidative leaching [5–7], bioleaching [8–10], and oxidation of materials after ultrafine grinding

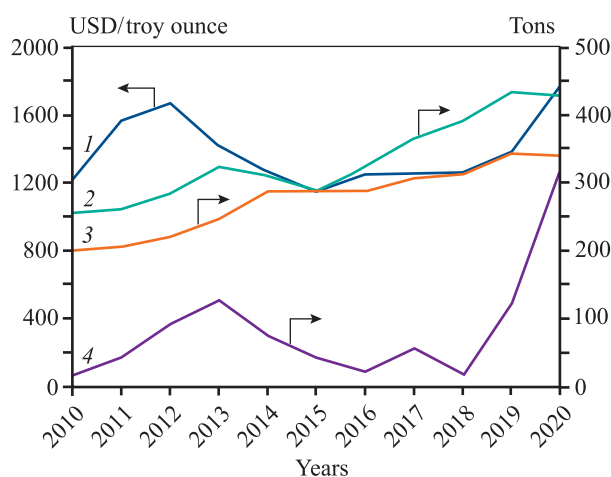


Fig. 1. Dynamics of the price (1), mining volumes (2), production from minerals and scrap (3), and gold exports (4) of the Russian Federation

Рис. 1. Динамика показателей цены (1), объемов добычи (2), производства из минерального сырья и скрапа (3) и экспорта золота (4) РФ

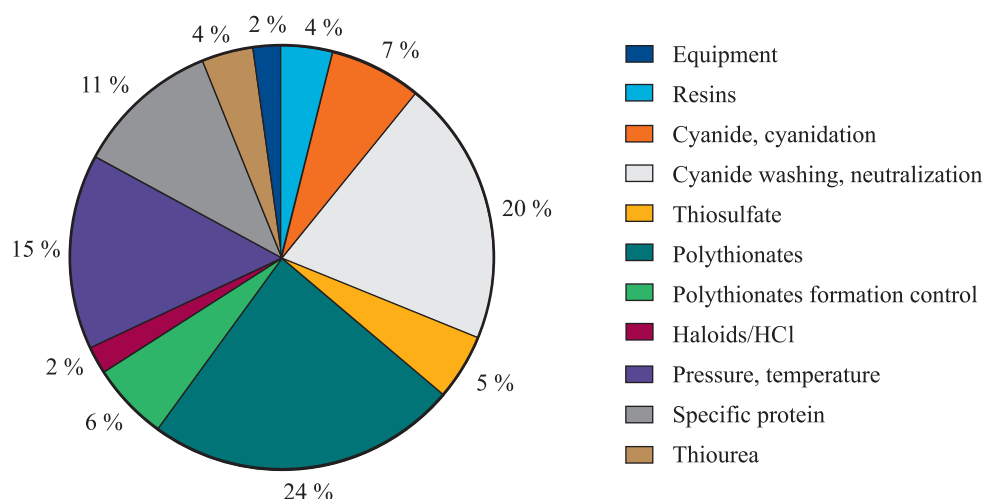


Fig. 2. Percentage of U.S. patents on gold leaching categorized by leaching approaches or reagents [3]

Рис. 2. Доля патентов США по теме выщелачивания золота, разделенных по заявленным подходам к выщелачиванию или реагентам [3]

under atmospheric conditions (Albion process, oxygen-lime treatment) [11; 12].

With the discovery of new deposits, the composition of ores has evolved, leading to challenges caused by a significant pyrrhotite content. To sustain growth in the gold mining industry and maintain metal production at current levels, it's essential to incorporate low-grade gold resources into processing.

However, pyrrhotite presents a challenge as it acts as a chemical depressant and a cyanicide, rendering the standard cyanidation process ineffective. Even concentrating on pyrrhotite-bearing ores through flotation is not straightforward. A study referenced in [13] highlights that the high rate of pyrrhotite oxidation under standard conditions for this method results in waste products that are rich in valuable components, causing significant losses during ore processing.

The low recovery of gold can be attributed to several factors. Firstly, the presence of minerals that absorb cyanide radicals and dissolved oxygen in the pulp inhibits the gold dissolution process. Additionally, the presence of sorption-active carbonaceous material leads to a preg-robbing effect [14]. Furthermore, some researchers have suggested that aurocyan ions are adsorbed by the surfaces of sulfide minerals (such as pyrite, pyrrhotite, and copper sulfides), quartz, and layered silicates [15]. Cyanicides readily dissolve in NaCN solutions and create secondary films on the surface of gold particles. Pyrrhotite, in addition to consuming cyanide, releases sulfide anions into the solution, similar to copper. For the reaction involving just 1 % (10 kg/t) of pyrrhotite in the ore or concen-

trate, the stoichiometric consumption of cyanide is 38.9 kg [16].

The diminishing quality of ores available for processing leads to increased waste from gold extraction operations, which can contaminate water and soil, resulting in environmental degradation [17]. The development of resource-saving technologies that reduce emissions, enhance working conditions for personnel, and improve the technical and economic efficiency of the processing is a key concern for professionals in the mining industry [18].

The objective of this research is to review existing studies on the processing of refractory gold-bearing ores and concentrates and to assess the effectiveness of well-established technologies for processing pyrrhotite ores.

Impact of ore compositional characteristics

A significant portion of gold-bearing ore deposits consists of a diverse range of minerals, including refractory quartz, carbonate, or sulfide ores. These minerals are notable for containing finely disseminated gold within the host minerals. To determine the direction of technological research for developing an efficient processing method, ore mineral composition is analyzed according to the framework depicted in Fig. 3. The anticipated influence of these factors on the process should be considered in the early stages of research to prevent adverse effects on the economics of a given process.

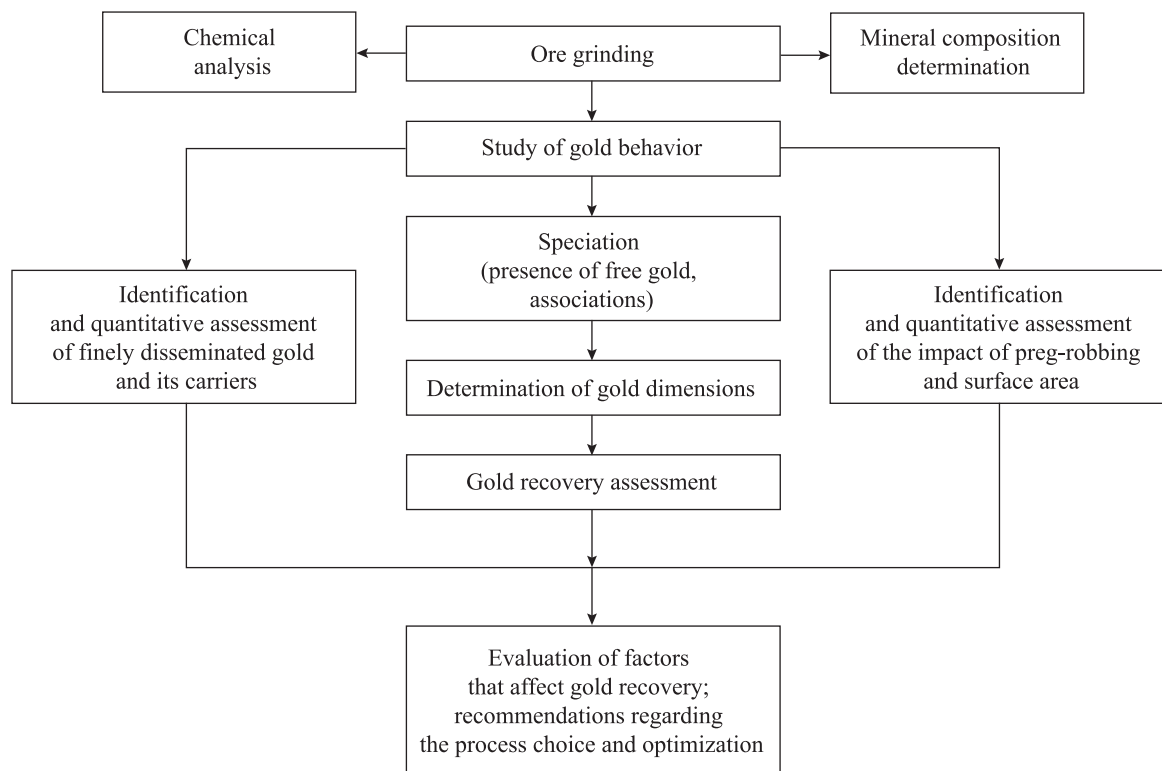


Fig. 3. Framework for gold-bearing ore preparation for investigation

Рис. 3. Блок-схема подготовки золотосодержащего сырья к исследованиям

During a scientific conference, V.V. Lodeishchikov [21] introduced a classification system for refractory gold-bearing ores, categorizing them into four distinct groups (Fig. 4). Group *A* includes ores that do not present complications and can be processed using conventional cyanide technology. They are considered readily cyanidable. Group *B* consists of ores with finely disseminated gold occurring in other mineral forms, which hinders the penetration of the leaching solution and the contact with gold particles. Ores in Group *B* contain cy-

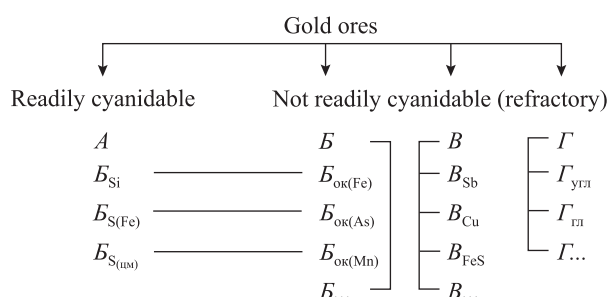
anicides, which consume cyanide during the dissolution process, forming inactive complexes that impede gold leaching. Additionally, this group may include minerals that promote the formation of films on the gold surface. And, finally, ores of Group *I* contain substances of both organic and inorganic origin that exhibit heightened sorption activity.

When studying the compositional characteristics of ores, special attention is devoted to the phase analysis of gold deportment. The behavior of gold during cyanidation is influenced by various factors, with a significant focus on its association with ore and rock-forming minerals, and whether the composition includes organic carbon [21–23]. Metallic gold is primarily found in ores and concentrates in four distinct forms:

- free gold;
- associated with host minerals (gold is intergrown with host minerals);
- finely disseminated and submicroscopic (often within quartz and sulfides);
- covered with surface films.

Fig. 4. Classification of gold ores by the degree and nature of their technological refractoriness [21]

Рис. 4. Классификация золотых руд по степени и характеру их технологической упорности [21]



tion [25; 26]. The presence of large, free gold grains may necessitate an extended leaching time for complete dissolution. When gold is disseminated within sulfides or quartz, it becomes less accessible to the leaching solution, causing cyanide and/or oxygen to be consumed in reactions with other mineral forms [27–29], including arsenopyrite, pyrrhotite, copper sulfides, stibnite, realgar, and orpiment.

The association of gold with arsenic-bearing minerals in the ores of the Kanowna Belle Gold Mine in Western Australia was determined, justifying the need to oxidize arsenic-bearing sulphide minerals prior to cyanidation.

Influence of pyrrhotite on cyanide leaching

Rapidly oxidizing iron sulfides, namely pyrite, marcasite, and pyrrhotite, fall into the category of active chemical depressors of the first class. Russian scientists, including V.Ya. Mostovich, G.V. Ilyuvnieva, and I.N. Maslenitsky, have extensively studied the reactions of pyrrhotite in alkaline cyanide compound solutions.

Pyrrhotite is characterized by an excess of sulfur and a structurally defective composition. During ore mining, it is highly likely to undergo decomposition, resulting in the formation of sulfuric acid, sulfates, carbonates, basic iron sulfates, and hydrates. Pyrrhotite possesses one weakly bonded sulfur atom that readily reacts with cyanide to produce thiocyanide and iron sulfide. The iron sulfide is rapidly oxidized by oxygen to sulfate, which, in turn, reacts with cyanide to generate ferrocyanide:



As a result, pyrrhotite not only acts as a cyanide mineral but also consumes oxygen necessary for the gold dissolution reaction in pulps and solutions (Fig. 5 and 6).

To mitigate these effects, the ore or concentrate can be aerated in an alkaline medium before processing. During this procedure, a protective film forms on the pyrrhotite surface, inhibiting its reaction with the cyanide solution.

Preg-robbing effect

Another common factor contributing to refractoriness in the standard cyanide process is the presence of

sorption-active substances and clay minerals [31; 32]. Initially, the term “preg-robbing” [33; 34] referred to carbonaceous materials that competed with activated carbon for the adsorption of gold cyanide complexes. In recent decades, this concept has been expanded to en-

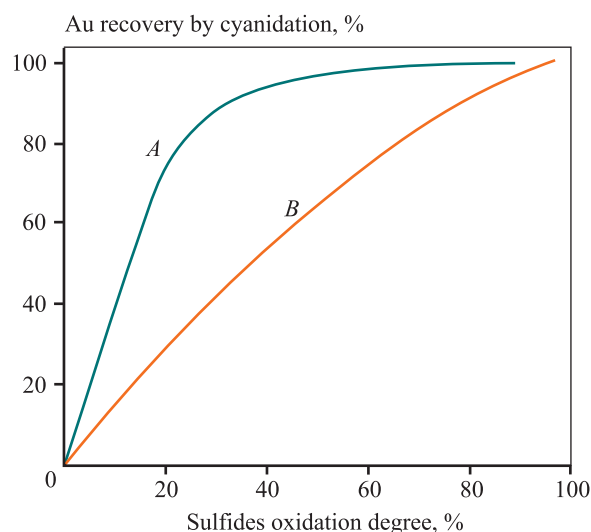


Fig. 5. Effect of oxidation rate on gold recovery, depending on the ore compositional varieties [30]

A: 82 % pyrite, 15 % arsenic pyrite, 3 % pyrrhotine, Au mostly with arsenic pyrite

B: 91 % pyrite, 6.5 % arsenic pyrite, 0.5 % pyrrhotine, 2 % sphalerite, Au is associated with pyrite and arsenic pyrite

Рис. 5. Влияние степени окисления на извлечение золота, в зависимости от минерального состава сырья [30]

A: 82 % пирит, 15 % арсенопирит, 3 % пирротин, Au преимущественно с арсенопиритом

B: 91 % пирит, 6,5 % арсенопирит, 0,5 % пирротин, 2 % сфалерит, Au ассоциировано с пиритом и арсенопиритом

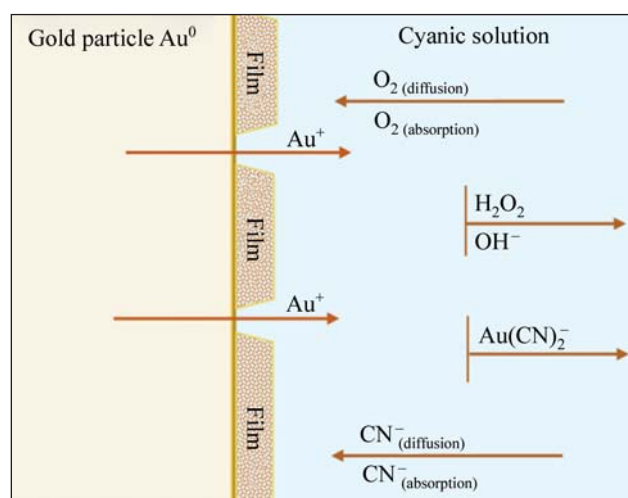


Fig. 6. Process of the gold particle dissolution

Рис. 6. Процесс растворения частицы золота

Table 1. **Mechanisms of preg-robbing depending on the material responsible for the effect**

Таблица 1. Описание механизма прег-роббинга в зависимости от материала, вызывающего данный эффект

Materials	Mechanism
Carbon	Surface adsorption from the gold complex solution
Heavy hydrocarbons	Slight reduction of the preg-robbing effect due to carbon coating
Organic acids (humic substances)	Formation of complexes with gold; low surface adsorption
Kerogen	Physical encapsulation of gold (cannot be considered actual preg-robbing)
Iron-bearing minerals (pyrite, chalcopyrite), silicates	Reduction to the elemental gold state and adsorption on the surface
Clays	Adsorption from rich solutions

compass minerals, clays, and other carbon compounds that, in addition to absorbing soluble gold, can also reduce it to its elemental form. The presence of organic carbon may necessitate roasting, a process that eliminates the sorption-active material that absorbs gold during cyanidation.

The mechanism by which gold dissolves during the cyanidation process varies depending on the nature of the substances causing this effect, as summarized in Table 1.

Another method is chloride preg-robbing, in which gold sorption during pressure oxidation forms the gold chloride complex $[\text{AuCl}_4]^-$ [35; 36].

Existing methods for refractory gold-sulfide ore processing

There is currently no universally applicable technology that ensures cost-effective processing of such ores [37]. This is primarily due to variations in the overall chemical, material, and phase composition of the ore, which includes the ratio of gold forms that can be leached by cyanide and those that cannot, as well as the presence of minerals that interfere with the process, such as cyanicides and chemical depressors. As a result, a tailored technological approach is often necessary for each specific deposit [38]. Given these distinctive characteristics, practical methods for ore dressing and metallurgical processing can vary significantly from one deposit to another.

At present, two main strategies are employed to overcome the challenge of processing refractory gold-bear-

ing ores. The first method maintains the use of the cyanide process but includes additional steps to condition the ore or concentrate. Fig. 7 provides an overview of the most common and commercially utilized methods for ore preparation.

Another approach involves the special treatment or leaching of gold using non-cyanide media, where the material is less refractory [40–42].

Considerable attention is devoted to the conditioning and processing of refractory sulfide and carbonaceous ores [43], as well as gravity or flotation concentrates. Typically, fine and ultrafine grinding of refractory ores significantly reduces gold losses in tailings [44]. This process involves a mechanical impact that induces changes in the mineral structure at the molecular level, making it a mechanochemical method. However, fine grinding without subsequent oxidation treatment is suitable for non-sulfide materials only. The mechanochemical method for preparing arsenopyrite-pyrite ores for cyanidation proves inefficient due to incomplete liberation of finely dispersed gold particles and increased chemical reactivity of crushed sulfides.

The objective of oxidative disintegration of ores is to transform sulfides into oxides or sulfates [45]. This process disintegrates the sulfide matrix, allowing the leaching solution to reach previously inaccessible gold particles and dissolve them easily [46].

Table 2 outlines the primary methods for preparing sulfide concentrates for cyanidation, along with some operational characteristics. The economic viability of these technologies is often limited due to high rea-

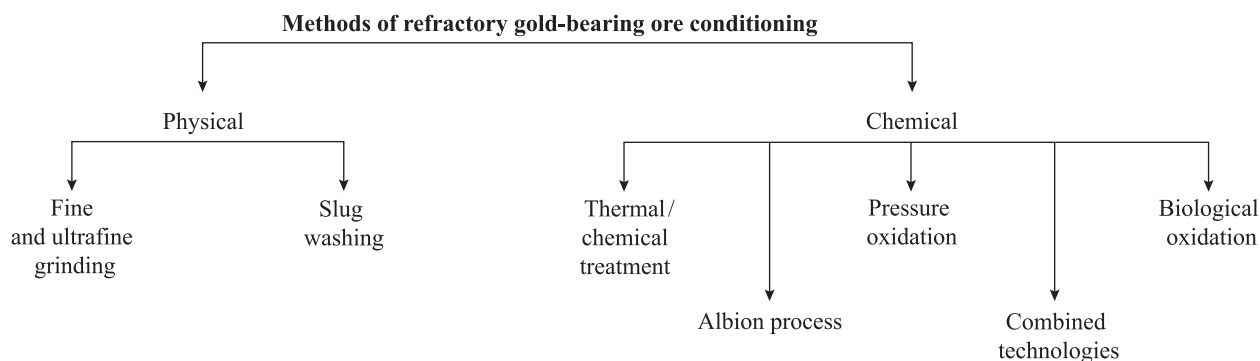


Fig. 7. Classification of conditioning methods [39]

Рис. 7. Классификация способов кондиционирования [39]

gent consumption, energy costs, extended processing times, and other factors. Recent research has been focused on developing alternative methods for extracting gold from refractory sulfide products, primarily based on ultrafine grinding of the material [47; 48]. For example, a study described in [48] presents the optimization of cyanide leaching of pyrrhotite concentrate, highlighting three critical factors influencing the gold leaching rate:

- the extent of pre-crushing to a particle size of $P_{80} = 10 \mu\text{m}$;
- the volume of oxygen introduced into the system;
- the addition of lead nitrate.

When gold miners select hydrometallurgical technologies for the initial preparation of ores for gold leaching operations, they consider several crucial technological factors [37]:

- a decline in the quality of the ores being processed, marked by an increased presence of detrimental components, particularly arsenic and sulfur;
- the utilization of pyrometallurgical processes for ore preparation, which necessitate costly gas cleaning systems and the management of flue gases and the heat generated during roasting/pyrolysis;
- the suboptimal technical and economic performance of refractory ore treatment when employing direct cyanide technology.

Pyrrhotite, frequently encountered in many gold ores alongside pyrite and/or arsenopyrite, poses numerous challenges during the processing of refractory ores. Unlike pyrite, pyrrhotite reacts most vigorously with cyanide and oxygen, often acting as a reactive anode [50].

Oxidative disintegration of gold-bearing minerals under alkaline conditions is a logical step. This approach eliminates the need for alkalization after oxidation processes in an acidic medium, which is

typically required when the pH is above 10.5 for cyanidation. The alkaline process is suitable for disintegrating minerals such as pyrite, arsenopyrite, selenides, or tellurides.

While investigating the oxidation process based on Albion technology (involving ultrafine grinding followed by oxidation), researchers have examined the reactions of sulfide minerals, particularly pyrite and arsenopyrite [51; 52]. However, the oxidation mechanism of pyrrhotite under these conditions has not been thoroughly explored. Earlier experimental studies have shown that the oxidation of arsenopyrite is more pronounced at a $\text{pH} \approx 7$. These investigations differ from patented technologies related to oxidizing gold-bearing materials in an alkaline medium after preliminary ultrafine grinding [50; 53]. The mechanism of pyrrhotite oxidation remains underexplored, and further experimental studies are needed to describe the physicochemical model of the process.

The study [54] highlights that additional grinding of refractory gold-bearing material containing pyrrhotite inclusions, using ceramic media, significantly enhances gold recovery, with a threefold increase compared to when steel grinding media are employed. This improvement can be attributed to galvanic interactions between the forged steel medium and pyrrhotite, which can lead to the formation of iron hydroxide. Iron hydroxide subsequently reacts with free cyanide to produce ferrocyanide, which, in contrast to free cyanide, is incapable of dissolving gold (resulting in the conversion of 75 % of the free cyanide to ferrocyanide). Moreover, these galvanic interactions markedly reduce the levels of dissolved oxygen and cyanide, making the gold leaching process more complex. Enhanced mechanical activation, therefore, plays a crucial role in improving the recovery of this precious metal.

Table 2. **Methods for treating refractory gold-bearing ores [49]**

Таблица 2. Способы обработки упорного золотосодержащего сырья [49]

Processes		Basic principles	Notes
Pyrometallurgical	Standard roasting [55]	S, As and C in the ores are oxidized and volatilized, and the mineral structure, including gold, is destroyed	This process is dependable but demands significant investments and a rather intricate purification system to eliminate the off-gases
	Roasting by microwave radiation [56; 57]	The material composed of polar molecules is more effective at absorbing microwaves and converting them into heat	High impurities removal rate; thermal efficiency; complex and expensive equipment; off-gas generation
	Roasting with additives [58]	The introduction of additives alters the phase transformation or reaction process, resulting in the formation of new phases during roasting	Calcite voids and gold exposure increase, sulfur and arsenic are present in the calcine
Hydrometallurgical	Acid leaching [59]	Inclusions in minerals are destroyed by acid (e.g., sulfuric acid, hydrochloric acid)	This is typically followed by a roasting treatment; the operation is simple but poorly adapted to the ore
	Alkaline leaching [60; 61]	Sulfides or oxides, which have a significant impact on the gold leaching process and encapsulate the gold, are eliminated by the use of alkaline compounds, such as sodium hydroxide or sodium sulfide	The process is highly efficient, doesn't produce harmful gases, and has low energy consumption. However, it is not well-suited for oxide ores
	Pressure leaching [62; 63]	This is typically accomplished by raising the leaching pressure, often referred to as pressure oxidation (POX) leaching, and conducting fine grinding (mechanical activation)	It is well-suited to the ore and enhances gold recovery efficiency, but the equipment is complex and expensive
	Biological leaching [64]	Microorganisms dissolve sulfides or harmful elements in minerals to aid in the extraction of valuable elements	The method is cost-effective and generates minimal waste. Nevertheless, it is time-consuming, leading to excessive operating costs and may not be well-suited for certain types of ores

Conclusion

The trend of decreasing mined ore quality is persistently growing, necessitating the processing of ores with lower target component content and complex chemical and mineralogical compositions. Presently, the gold mining industry has seen considerable success in dealing with refractory ores in which gold is dispersed with-

in pyrite and arsenopyrite. Bio- and pressure oxidation followed by cyanide leaching are commonly employed for this purpose. However, pyrrhotite is increasingly common in gold-bearing ores, in addition to the traditional sulfide minerals. The behavior of pyrrhotite in the technological process differs significantly from the extensively researched pyrite and arsenopyrite. During cyanide leaching, it reacts with NaCN/KCN and dis-

solved oxygen in the pulp, leading to increased reagent consumption, reduced gold dissolution rates, and decreased overall process efficiency.

Currently, lime-air pretreatment of the ore or concentrate is used to mitigate the adverse impact of pyrrhotite on the cyanidation process. This operation, although not highly intensive, enhances process performance but offers potential for improvement.

Upon reviewing available sources, it is apparent that the physicochemical transformations of pyrrhotite during oxidation in an alkaline medium remain insufficiently studied. A deeper understanding of this area will facilitate more effective process management and increased efficiency. Consequently, a more comprehensive investigation of the pyrrhotite oxidation mechanism, considering various technological parameters, emerges as an urgent scientific and practical task for the future.

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A.Ya. Boduen — creation and validation of the concept of the work (formulation of the idea), annotation of data, formulation of the conclusion, revision of the manuscript text.

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