The importance of sample preparation in the hydrometallurgical processing of refractory gold concentrates

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Abstract. The article indicates the importance of thorough preparation of concentrate samples before hydrometallurgical operations; the experiments on studying of the factor of the size of the material are described. The comparative analysis of particle size after conventional and fine grinding is carried out. As obligatory stage of preparation of the crushed concentrate before autoclave leaching, the decarbonization process is studied. On the basis of the conducted experiments, the optimal regime of the autoclave leaching process is revealed, which provides the oxidation degree of sulfide sulfur of the order of 99%.

1. Introduction

One of the main trends in the development of the raw material base of the gold mining industry is the decline in the quality of the initial mineral raw materials. This is due to the depletion of rich gold-bearing ores. Therefore, the sphere of production requires a more complex material composition resistant raw materials [1]. The most common reason for the persistence of gold ores is the fine impregnation of gold into rock-forming minerals. Refractory ores account for more than 30% of the world's total reserves [2, 8].

The method of hydrometallurgical autoclave oxidation of the latter in an acidic environment is an effective method of gold extraction from sulfide concentrates. Most often, fine gold is associated with sulfide minerals. Therefore, autoclave technologies are used for oxidation of sulfide minerals in the processing of pyrite and arsenopyrite refractory gold ores. The content of gold in pyrite-arsenopyrite ores is about 3.5-4.0 g/t, sulfides (mainly pyrite) – 2-3%. Sulfide content in raw materials should provide the necessary dissipation to maintain autogenous process of pressure oxidation.

Technological scheme of processing of refractory gold ore is based on the use of flotation enrichment to obtain concentrates. After that, the concentrates are subjected to autoclave opening and subsequent sorption cyanidation of oxidation products.

The actual direction of development of autoclave technology is application of processes of leaching of sulfide ores and concentrates at rather low parameters. The attractiveness of the technology based on low-temperature leaching is due to the low cost of autoclave equipment. This allows reducing capital costs in the realization of the industrial process [9].

The successful realization of the autoclave leaching process largely depends on the preparatory stages. These operations include grinding and decarbonization. The most complete processing of gold-containing minerals requires fine and ultra-fine grinding, where 80% of the particles have a fineness of less than 10-15 microns [3, 4, 7]. The process of ultra-fine grinding of the material leads to a high degree of deformation set into the crystal lattice of minerals. This increases the activity of minerals...
and promotes their leaching [10]. Therefore, the question of thorough preparation of the concentrate sample for processing is very important.

2. Characteristics of raw materials
Granulometric characteristics of fine and fine-disperse gold of the initial ore sample (size range from 3 to 25 microns) showed that the proportion of gold of this size in the total content of the precious metal in the sample is 90-92%, the average diameter of the gold is 7 microns. The scheme of preparation of such ore includes one-stage crushing and two-stage grinding. Grinding is carried out in mills operating in semi-autogenous mode at the first stage and ball grinding – at the second stage. According to the test results, the mass fraction of the class - 0.074 mm in the ore sample in the flotation concentrate grinding unit was 95%.

According to mineralogical data, pyrite concentrate is represented by rock components such as quartz (25.1%) and feldspar (16%). The main ore mineral is pyrite, the content of which is 44.5%, there are arsenopyrite (1.5%) and pyrrhotite (1%). Arsenopyrite concentrate is the components of rocks (quartz – 9.5%; feldspar – 2%; carbonate to 2.4%; mica-hydrologist components – 26.3%). The main ore minerals are pyrite (38%) and arsenopyrite (17.8%).

The general scheme of pyrite-arsenopyrite concentrates processing includes the following main processes:
- grinding of flotation gold-containing concentrate in a ball mill;
- acid processing (prior to the removal of the carbonate compounds - the process of decarbonization);
- autoclave oxidative leaching;
- thickening and filtration of oxidized pulp;
- neutralization of the solution with limestone and lime;
- sorption cyanidation of cake.

3. Research methods
Grinding. Fine grinding is necessary to prepare the concentrate for the low-temperature process [5]. In laboratory studies, the required size of the grinding is achieved by using a planetary mill "Pulverisette 6" (Fritsch, Germany). The size of the source and ground material is measured on the laboratory analyzer "Analysette 22" (Fritsch, Germany). In the study of the size factor, experiments were carried out to grind the sample in two modes:
- conventional ($P_{80} = 34-35$ microns);
- fine grinding ($P_{80} = 8-9$ microns).

Grinding was carried out in a planetary ball mill. Loading material, balls and rotation speed were identical for both modes. Time of stay in the mill varied [6]. The size of concentrates at all stages of the experiment is presented in table 1.

| Study stage                           | Fineness          | $P_{80}$, microns |
|---------------------------------------|-------------------|------------------|
| The initial concentrate               | not ground        | 47-48            |
| Grinding                              | conventional      | 34-35            |
|                                        | finely divided    | 8-9              |
| Auto clave oxidative leaching         | conventional      | 30-32            |
|                                        | finely divided    | 8-9              |

The particle size distribution characteristics were determined by the laser analyzer "Analysette 22" manufactured by the German company Fritsch. This type of analyzer allows reliable measuring the
specified characteristics at the particle sizes in the sample from 0.3 mm to 0.3 microns. Figures 1 and 2 show the measurement results. The graphs show 2 functions: differential and integral. The differential function reflects the distribution of particles for each class of size, the integral function sums up these indicators. The crushed material after the particle size measurement by laser granulometry was sent to the decarbonization stage and then to leaching.

Figure 1. Granulometric composition of fine concentrate ($P_{80} = 34-35$ mcm).

Figure 2. Granulometric composition of the crushed concentrate ($P_{80} = 8-9$ mcm).

Decarbonization. The raw material supplied to the autoclave leaching, as a rule, contains carbonates. A mandatory stage for persistent gold-containing concentrates is the preliminary removal of carbonate compounds from their content (the decarbonization process). Their presence in the autoclave process leads to the release of gaseous CO$_2$, reduces the partial pressure of oxygen and to a large extent its use. The process of decomposition of sulfides (pyrite and arsenopyrite) at relatively low parameters differs from the traditional high-temperature leaching in that during the oxidation of sulfide sulfur, part of it is oxidized to sulfate, and part is inevitably transformed into elemental sulfur. The optimal modes of acid processing are:
- reagent – sulfuric acid (dilution 1:1);
- liquid to solid ratio on operation: 2:1;
- temperature: 60°C;
- processing time 0.5 h,
- pH of the final pulp 2.0 - 2.2;
- acid consumption is ~30 kg/t.

Autoclave leaching. The prepared material (sample of crushed and decarbonized concentrate) is subjected to hydrometallurgical low-temperature processing under the following conditions:
- liquid to solid ratio – 3;
- temperature – 130°C;
- partial pressure of oxygen – 1.0 MPa;
- the speed of rotation of the agitator was selected in such a way as to provide an oxygen absorption rate of 0.5 mol O$_2$/(l·h·ATM);
- the duration of the autoclave oxidative leaching was estimated by stopping the absorption of oxygen.

The parameters of the pulp after unloading from the autoclave are presented in table 2.
Table 2. Parameters of the pulp after autoclave leaching of low-temperature

| Concentrate          | Fineness          | The degree of oxidation of sulfide sulfur, % | Analysis of the cake, % |
|----------------------|-------------------|---------------------------------------------|-------------------------|
| Pyrite               | conventional      | 90.4                                        | 0.2 8.9 7.24            |
| Pyrite               | finely divided    | 99.5                                        | 0.2 5.25 7.21           |
| Pyrite-arsenopyrite  | finely divided    | 99.2                                        | 5.5 10.0 6.58           |

4. Conclusion
The conducted studies show that the sample preparation of the material for technological operations plays a decisive role. Thus, the degree of sulfur oxidation in the experiment with a concentration in usual size is not high enough and is about 90%. With such a degree of decomposition of sulfides, a significant part of gold remains in the structure of the initial pyrite, which leads to its loss. Therefore, it is advisable to apply ultra-fine grinding to a class content of 10-12 microns of at least 80% in order to avoid the loss of gold with rock-forming minerals. In addition, the important aspect of the autoclave technology is the careful blending of ores in order to average ores over gold and maintain an appropriate level of mineral content - acid generators (sulphides) and its consumers (carbonates).

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