

BEHAVIOR OF PLATINUM IN THE SYSTEM OF THE MATTE-SLAG IN THE PROCESSING OF COPPER-NICKEL MATERIALS

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Abstract

Copper-nickel sulfide ores are one of the main sources of platinum. One of the ways to extract precious metals from such ores involves melting of a concentrate in electric ore smelting furnaces, where the melt is divided into matte and slag. Platinum is generally concentrated in matte; however, some of it remains in the slag, thus leading to metal losses. In order to reduce platinum losses, the forms of platinum in these phases should be studied. It was found that during the melting of this ore, iron, nickel, and copper are reduced. The mineral composition of matte was studied. Platinum in matte is present in the form of intermetallics with Fe and Ni. The PtFe intermetallic is a dispersed needle formation with a length of 20 to 500 μm and a thickness of up to 10 μm . The size effect is revealed: the content of platinum in the PtFe intermetallic decreases with decreasing the thickness of needle formations. The decreases in the content of platinum in dispersed needle formations can be explained by an increase in the thermodynamic activity and changing properties of the dispersed substance and a corresponding increase in solubility. It was found that matte drops, together with their associated platinum-containing particles of no more than 5-7 μm in size, were carried into the slag by gas bubbles using flotation. The conditions for the rise of a matte drop, together with a bubble in the slag, consist in the fact that the adhesive force of the drop with the bubble and the buoyancy force acting on the bubble must be greater than the gravity of the drop.

Keywords: Platinum; Intermetallic; Matte; Slag; Surface tension; Flotation

1. Introduction

The world's main reserves of platinum are found in layered intrusions of ultrabasic rocks and sulfide copper-nickel ores [1, 2]. Extraction of platinum from sulfide copper-nickel ores is a multi-stage process. One of the stages of this process is melting of a previously prepared concentrate in electric ore smelting furnaces [3], where the melt is divided into sulfide and oxide components (matte and slag). Platinum is generally concentrated in matte; however, some of it remains in the slag, thus leading to metal losses [4, 5]. In order to develop technologies providing reduced platinum losses, the mechanism of transition of platinum into slag and the forms of platinum during all the respective stages should be studied. One of the possible options for this transition is the flotation of matte drops containing platinum by gas bubbles that appear due to sulfide decomposition. From the perspective of physical chemistry, flotation implies floating of bodies whose densities are higher than that of a liquid on the surface of that liquid. During the flotation, the bubble must overcome the matte-slag boundary and hold a

matte drop when floating in the slag. Thus far, researchers have mainly focused either on the mineralogy of platinum in the slag disposal areas formed during copper and nickel production [6] or the distribution of platinum between slag and matte [7-10]. Dependence of the flotation of matte droplets in slags of various compositions on the partial pressure of oxygen, and interfacial tensions in this system were considered in [11-14]; the breakthrough of the surface of two liquids by a gas bubble was analyzed in [15].

Therefore, the aim of the present study was to experimentally study platinum forms in the products of copper-nickel ore melting (matte and slag), to analyze possible chemical reactions in such ores using the HSC Chemistry 9.0 software package and the flotation conditions of matte drops together with the associated platinum-containing particles by gas bubbles. The results can be used in studies aimed at increasing the degree of platinum recovery.

2. Experimental

Copper-nickel sulfide ore widely used for

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extracting copper, nickel, and platinoids was selected as the material for the study. The mineral composition of the ore samples was found to contain (wt%): pyrrhotite 42.8, chalcopyrite 20.0, pentlandite 11.3, magnetite 16.0, and silicates 9.9. The Pt content was 1.5 g/t. In some cases, to increase the basicity of the slag, lime was added to the ore in an amount of 3-10 wt.%. Also, to facilitate the search for platinum-containing phases using microscopes, Pt was added to a part of the samples with the studied ore in the form of a specially prepared platinum sponge with a particle size not exceeding 100 μm , in an amount from 1.0 to 2.5 kg/t. The added platinum interacted with the ore components during heating, soaking at high temperatures and cooling, similarly to natural platinum.

Ore samples were melted in open corundum crucibles in a resistor furnace with a graphite heater at a temperature of 1200 and 1300 °C in the atmosphere of air. The exposure period was 1 hour, and the furnace cooling period was 1 hour. The as-obtained ingots were divided into matte and slag. Some of the samples were crushed to a particle size of 0.050 mm for chemical and mineral analyzes. At the same time, the separation of matte droplets from slag before chemical analysis was not carried out. The mineral composition was determined by X-ray phase analysis on an XRD 7000C X-ray diffractometer. The fraction of each phase was according to the Rietveld method. The platinum metal content was determined using an inductively coupled plasma atomic emission spectrometer Spectroflame Modula S. The chemical composition of slag and matte was determined using an S4 Explorer X-ray fluorescence spectrometer. Polished sections (10 pieces) were made from other parts of samples to study the mineral composition using an Axio Image optical microscope and a Tescan Vega 3 scanning electron microscope equipped with an Oxford Instruments X-act energy dispersive analyser. The electron beam diameter was 2-3 μm . The composition of platinum-containing phases less than 10 μm in size was determined as follows. Compositions of needle-like formations (bright in BSE mode) of different thickness and adjoining area were explored using an analyser. It was established that the needle formations consist of Pt with metal impurities; no Pt was found in the adjoining area. Consequently, platinum is concentrated only in needle formations. The content of elements was determined with an accuracy of 0.1 wt% [16].

3. Results and discussion

After melting copper-nickel sulfide ore with a natural platinum content (1.5 g/t) at a temperature of 1300 °C and soaking for 15 minutes, skeletal FeNi crystals were found in the sulfide part (matte), which

contained single drop-shaped platinum-containing intermetallic particles of no more than 0.5 μm . The formation of the FeNi phase during slow cooling of the matte is described in [17]. The matte consisted of (wt%) troilite 72.4, bornite 10.1, Fe-Ni alloy 7.0, and magnetite 4.9. The phase composition of the matte was formed under the action of reactions on the slag-matte interface during heating, cooling and a given composition of the gas phase. Iron in the metal form as a separate phase was not detected. The chemical compositions of matte and slag are shown in table 1.

The addition of platinum metal to the furnace mixture revealed several phases containing Pt. Pt in the matte after cooling from 1300°C is in the form of intermetallics and probably chemical compounds with Fe and Ni, which are needle formations from 20 to 500 μm long and up to 10 μm thick (at 1300 °C), Fig. 1. These formations are aggregates of crystals, which, as shown by chemical analysis using an energy dispersive analyser, are PtFe intermetallic. The crystals are in a double shell. After cooling from 1200 °C, PtFe intermetallic crystals have a skeletal shape, smaller size (average thickness of 2-3 μm), and contain lower amounts of Pt (from 47 to 63 wt%) compared to samples after cooling from 1300 °C.

Since the cooling was quite long (1 hour), this time was sufficient for the formation of metal alloys, in particular the alloys with platinum. Their formation indicates the reduction of iron, nickel, and copper. Besides, sulphide mattes cannot be quenched without precipitation of metal alloys due to sulfur deficiency.

PtFe intermetallic contains Cu and Ni impurities. Such impurities are characteristic of this alloy [18]. The platinum content decreases, as in the case of dispersed Au particles, which was established earlier [19], with a decrease in the thickness of the PtFe formations, Fig. 2. As a result of statistical processing

Table 1. Chemical composition of matte and slag (in wt%)

Matte		Slag	
S	30.5	SiO ₂	40.9
Fe	57.9	Al ₂ O ₃	18.1
Cu	4.66	FeO	29
Ni	4.28	Fe ₂ O ₃	0.95
O	2	TiO ₂	0.6
Pt	3.68·10 ⁻⁴	CaO	2.38
		MgO	1.68
		Na ₂ O	0.9
		K ₂ O	1.14
		S	3.6
		Cu	0.38
		Ni	0.45
		Pt	0.2·10 ⁻⁴



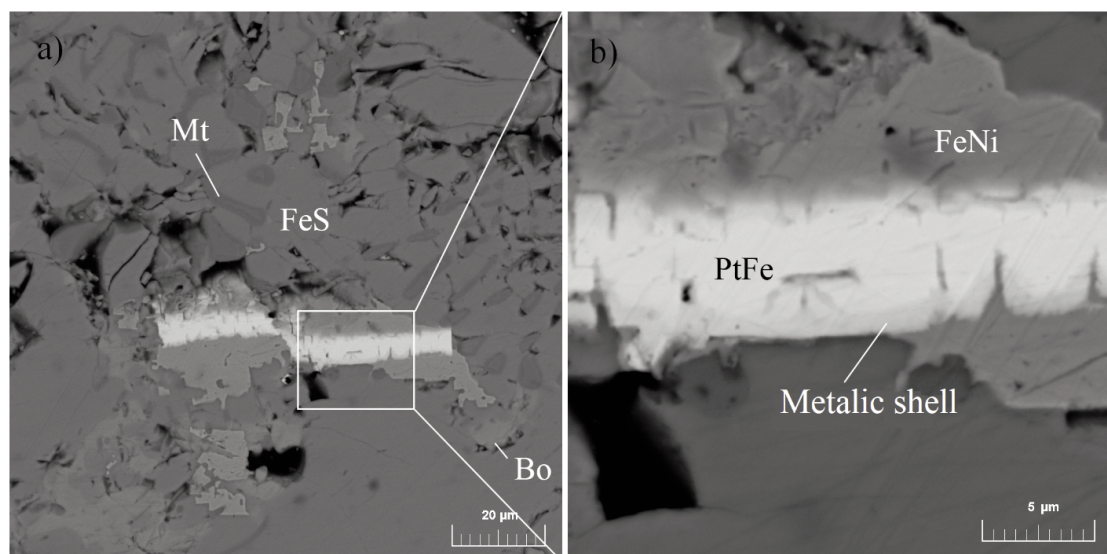


Figure 1. Needle formations of PtFe intermetallic (b) in a double shell in matte. Matte (a) composed of troilite (FeS), bornite (Bo) and magnetite (Mt). Cu-Ni sulfide ore after melting at 1300°C. Image taken in BSE mode

of the experimental data, the value of the pair correlation coefficient $r = 0.52$ was obtained. The calculated value of the Student's criterion is 2.29, the tabular value is 2.1 (the number of degrees of freedom $n = 16$ and the significance level $\beta = 0.95$). Since the calculated value is larger than the tabular, it can be argued that there is a linear relationship between the investigated parameters.

The increase in the impurity content in dispersed needle platinum formations (thickness no more than 10 μm) with a decrease in their thickness L can be explained by an increase in the thermodynamic activity of the dispersed substance. The Gibbs potential G of thin PtFe “needles” is increased by the amount:

$$\Delta G^0 = \sigma S = \sigma S_1 V = \frac{\sigma S_1 M}{d} \quad (1)$$

where σ is an interfacial tension, S is a total surface of the needles per mole of substance, $S_1 = S/V$ is a specific surface area per unit volume, $V = M/d$ is a molar volume, M is a molecular weight, d is a density. For needles with thickness L per unit length S_1 is $\pi L / (\pi L^2 / 4) = 4/L$, therefore equation (1) will have the form:

$$\Delta G^0 = \sigma S = \frac{4\sigma M}{LdRT} \quad (2)$$

The vapor pressure P , the thermodynamic activity a , and, accordingly, the equilibrium solubility, when it is far from unlimited (100%), the equilibrium constants of the K reactions, in which the dispersed

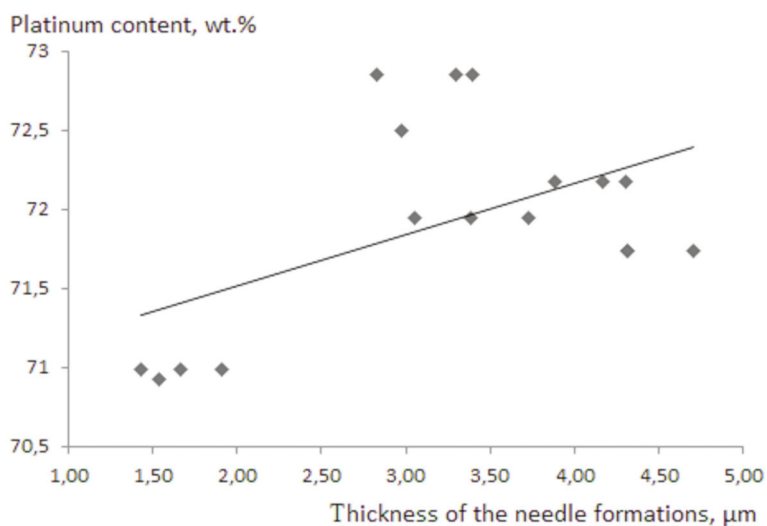


Figure 2. The dependence of the platinum content in the needle formations of PtFe intermetallic on their thickness after cooling on the temperature of 1300°C

substance is involved, depend on the potential G^0 exponentially. Therefore, an increase in the G^0 potential will lead to a significant increase in the listed values, including the solubility of impurities, in exp $(3\sigma M/dLRT)$ times compared to the values for massive substances P_0, K_0 :

$$\frac{P}{P_0} = a = e^{\frac{4\sigma M}{dLRT}} \quad (3)$$

In our case, the change in the activity of the substances of the needle due to only one increase in surface energy by $\Delta G^0 = \sigma S$ would be small (by 0.23%). This is at the interfacial tension characteristic of massive bodies σ is 1 J/m^2 , the average value of the atomic weight of platinum and iron in the PtFe compound M is 0.126 kg/mol , density d is $14,000 \text{ kg/m}^3$, needle thickness L is $1 \text{ }\mu\text{m}$ or 10^{-6} m . In addition, when passing to small particles, apparently, the structure in their volume and properties change. Indeed, metal filaments with a diameter of $\sim 10 \text{ }\mu\text{m}$ and alloys with a grain size of $\sim 10 \text{ }\mu\text{m}$ already have increased strength. At smaller sizes, the strength, and hence σ , increases many times [20]. Such effects cannot but depend on the structure. At present, changes in the volumetric structure of small particles and the corresponding changes in properties have been little worked out theoretically, but the experimental data clearly indicate their existence [20].

In order to predict changes in the chemical and phase composition, the authors [21] analyzed possible chemical reactions in the ore, the mineral composition of which is given above, using the Equilibrium Compositions module of the HSC Chemistry 9.0 software package for changing the thermodynamic parameters and carbon content. It was found that platinum during heating the ore mass in the range of

$0\text{-}1300 \text{ }^\circ\text{C}$ could be in the following form: platinum, as well as PtS, PtFe, PtFe₃, and PtNi. Dynamic changes in sulfide ore start in the temperature range of $300\text{-}600 \text{ }^\circ\text{C}$: the contents of C, Fe₇S₈, CuFeS₂, Fe₃O₄, and PtS fall sharply, while CO, CO₂, FeS, and intermetallics including those containing Pt appear in large quantities (Fig. 3). The phase composition and number of phases at different temperatures are affected by carbon. For example, when the carbon content is high, Fe, Ni, and Cu are actively reduced, and FeNi is formed. With an increase in the amount of introduced platinum metal from 0.1 to 2.5 kg/t in the initial furnace-charge, the number of platinum-containing products shown in Fig. 3 increases proportionally. However, the products remain unchanged. This indicates that the behavior of platinum during ore heating and melting is not affected by its content.

The study of the oxide melt (slag) during the melting of copper-nickel sulfide ore showed the following. The slag is porous due to the release of gas bubbles containing sulfur. These bubbles, as seen from micrographs (Fig. 4), float matte drops up to 1.5 mm in diameter. Irregular-shaped intermetallic particles containing Pt, Fe, and Ni, no more than $5\text{-}7 \text{ }\mu\text{m}$ in size, and found in the surface layers of slag, are associated with these drops (Fig. 5). According to chemical analysis, about 5% platinum, 7.5% copper, and 9.5% nickel deport into the slag. When lime was added to the initial charge as a flux, the value for copper decreased to 3%, and for nickel - to 3%. This is consistent with the production data: 1-3 % for Cu and 1-2 % for Ni [3, 22, 23]. From 1 to 2 % platinum deport into industrial slags after smelting in electric ore smelting furnaces [4, 24]. Thus, the flotation of matte drops is one of the main processes that lead to

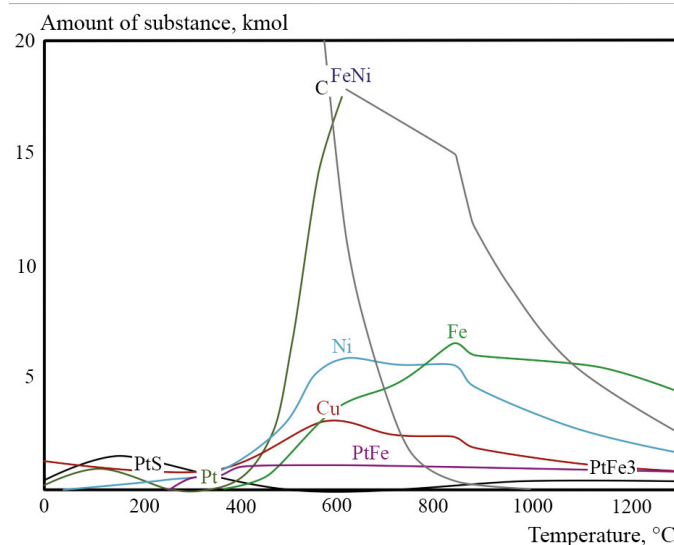


Figure 3. The equilibrium composition in the process of heating the ore to 1300°C and the initial amount of carbon equal to 1.5 wt.%

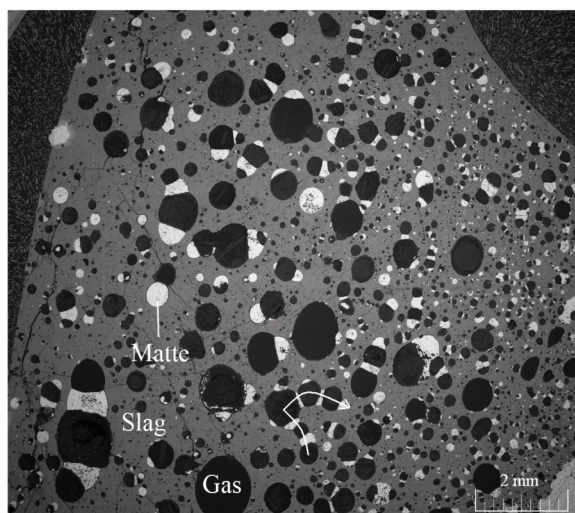


Figure 4. Sulphide drops (Matte) trapped by gas bubbles (Gas) in slag (Slag). The arrow shows a chain of alternating drops and bubbles. Image taken in BSE mode

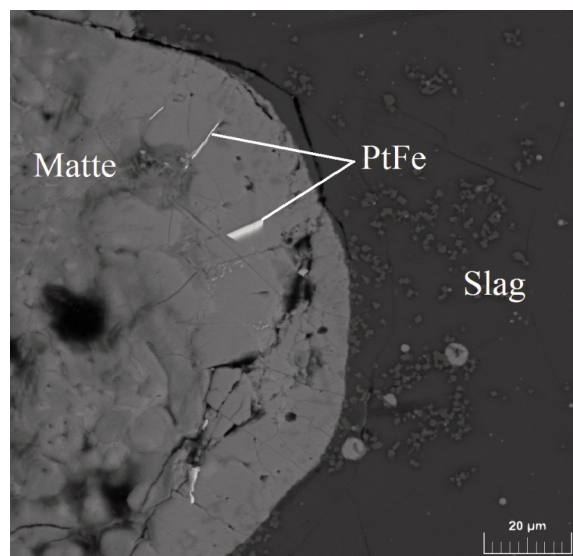


Figure 5. A matte drop in a slag containing platinum phases. Image taken in BSE mode

the losses of valuable metals, including platinum, during the melting of Cu-Ni sulfide ore on matte. This is consistent with the fact that the chemical solubility of PGMs, in this case platinum, in slags is very low (<1 ppmw) [9].

The passage of a gas bubble through the boundary of two immiscible liquids is poorly studied. During the passage of this boundary, the bubble can capture and float a drop of heavier liquid. Let us consider the process using the example of the matte-slag boundary and the conditions for the rise of a matte drop with a bubble. Phases close to equilibrium are shown in Fig. 4. The analysis was carried out based on the photos obtained using a Tescan Vega 3 scanning electron microscope after cooling the matte-slag melt from a temperature of 1300 °C of sulfide Pt-containing materials.

In order for the bubble to “tear” a matte drop from the sulfide melt and float with it in the slag, two conditions must be met:

1) The drops are held on the bubbles by surface tension, which can be called the coupling force. In order for the drop not to separate from the bubble, the adhesion force of the matte drop to the bubble must be greater than its gravity.

2) The buoyancy force applied to the bubble must be greater than the gravity of the drop. Otherwise, the drop will sink.

The adhesion force of the drop to the bubble is $2\pi r\sigma_{m-s}\sin\theta$, where θ is the angle between the tensions at the matte-slag boundary σ_{m-s} and the matte-gas σ_m , Fig. 6, and $\sigma_{m-s}\sin\theta$ is the vertical component of the surface tension at the matte-slag boundary. The first condition is expressed as an inequality:

$$2\pi r\sigma_{m-s}\sin\theta > c \frac{4}{3}\pi r^3\rho_m g \quad (4)$$

where r is the radius of the matte drop, ρ_m is the density of the matte, and c is the fraction of the volume of the drop from the full sphere.

When the drop size decreases, the adhesive force in Equation (4) decreases proportionally to r , and the gravity decreases proportionally to r^3 . The predominance of surface forces over gravity is typical for all dispersed systems [25]. Therefore, for micron drops and bubbles, it is almost always performed. In Fig. 4, there is also a tendency to form chains of alternating matte drops and gas bubbles (up to 15 pieces), which indicates a strong adhesion along the bubble-drop line.

The interfacial tension at the boundary of two immiscible liquids is determined from photographs of the interphase boundaries. The matte-slag σ_{m-s} tension can be calculated from the equilibrium condition at the three-phase boundary:

$$\Delta\sigma = \sigma_m + \sigma_{m-s} \cdot \cos\theta - \sigma_s \cdot \cos\alpha = 0 \quad (5)$$

where α is the angle between σ_s and σ_m .

Data on the densities and surface tensions of matte and slag, which are close in composition to the experimental ones, were taken from the source [22]: $\sigma_{m-s} = 0.18 \text{ J/m}^2$. The obtained value is close to the literature data for systems similar in composition [23] and, on the whole, agrees with the values obtained using an X-ray television introscope [11, 12]. In these works it was experimentally established that the composition of the phases and temperature have a strong effect on σ_{m-s} and contact angles.

For the experimentally studied slag and matte, the maximum possible radius of the matte drop held by the adhesive force on the surface of the gas bubble is 4.2 mm.

The ratio of the radius of the matte drop r to the gas bubble radius R is determined by the following



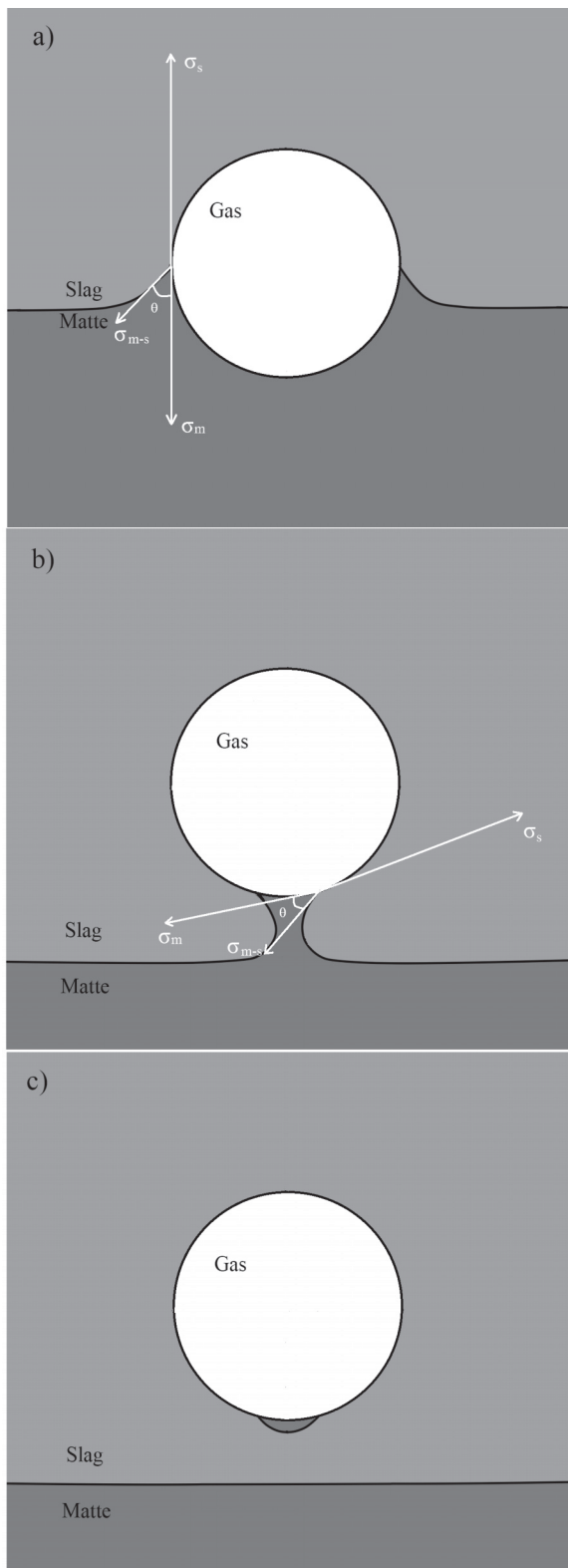


Figure 6. Stages of the passage of a gas bubble through the matte-slag interface: a, b - transition of a gas bubble through the matte-slag interface; c - floating gas bubble with a matte drop

condition given above: the Archimedean force F_a applied to the bubble must overcome the gravity of the matte drop P :

$$F_a \geq P \quad (6)$$

or

$$\frac{4}{3}\pi R^3(\rho_s - \rho_g)g \geq c \frac{4}{3}\pi r^3(\rho_m - \rho_s)g \quad (7)$$

From condition (7), the following ratio of the drop and bubble radii can be obtained:

$$\frac{r^3}{R^3} \leq \frac{(\rho_s - \rho_g)}{(\rho_m - \rho_s)} \leq \frac{2700}{c \cdot 1700}, R \geq 0.7 - 0.9 r \quad (8)$$

where ρ_s, ρ_g is the density of slag and gas.

The size of the bubble that raises a matte drop may be even smaller than its size, as can be seen from Fig. 4. This approach is consistent with the experimental work [14].

Thus, the conditions for the rise of a matte drop together with a bubble of the floating gas were considered. The maximum possible size of floated matte drops is very significant, which is consistent with noticeable losses of valuable components.

4. Conclusions

The mineral composition of Cu-Ni matte sulfide ore, which is widely used for extracting copper, nickel, and platinum, was studied. It was found that during the melting of this ore, iron, nickel, and copper are reduced. Platinum in matte is present in the form of intermetallics with Fe. The PtFe intermetallic is a dispersed needle formation with a length of 20 to 500 μm and a thickness of up to 10 μm . The size effect is revealed: the content of platinum in the PtFe intermetallic (Cu and Ni) decreases with decreasing the thickness of needle formations. The decrease in the content of platinum in dispersed platinum needle formations can be explained by an increase in the thermodynamic activity of the dispersed substance and a corresponding increase in the solubility of impurities, taking into account the fact that when passing to small particles, apparently, the structure in their volume and properties change. The mechanism of the transition of platinum to slag was established: matte droplets, together with the associated Pt and Fe intermetallic particles of no more than 5-7 μm in size, were carried into the slag by gas bubbles using flotation. This leads to the loss of valuable components, in particular, platinum. The conditions for the rise of a matte drop, together with a bubble in the slag, consist in the fact that the adhesive force of the drop with the bubble and the buoyancy force acting on the bubble must be greater than the gravity of the drop.

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PONAŠANJE PLATINE U SISTEMU KAMENAC-ŠLJAKA TOKOM PRERADE RUDE BAKRA-NIKLA

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Apstrakt

Sulfidne rude bakra-nikla su jedan od glavnih izvora platine. Jedan od načina dobijanja plemenitih metala iz ovih ruda uključuje topljenje koncentrata u električnim pećima, gde se otopljen ruda odvaja na kamenac i šljaku. Platina se uglavnom nalazi u kamencu; pa ipak, nešto platine ostaje u šljaci, što dovodi do gubitka metala. Da bi se gubici platine smanjili potrebno je ispitati oblike platine u ovim fazama. Otkriveno je da tokom topljenja ove rude dolazi do redukcije gvožđa, nikla, i bakra. Ispitivan je mineralni sastav kamenca. Platina u kamencu je prisutna u obliku intermetalnih spojeva sa Fe i Ni. Intermetalni spoj PtFe je disperzovana igličasta formacija dužine od 20 do 500 μm i debljine do 10 μm. Otkriven je uticaj veličine: sadržaj platine u PtFe intermetalnim spojevima se smanjuje sa smanjenjem debljine igličaste formacije. Smanjenje sadržaja platine u disperzovanoj igličastoj formaciji može se objasniti povećanjem termodinamičke aktivnosti kao i menjanjem osobina disperzovane supstance i odgovarajućim porastom topljivosti. Nađeno je da kapljice kamenca, zajedno sa pratećim česticama ne većim od 5-7 μm koje sadrže platinu bivaju tokom flotacije odnesene u šljaku mehurićima gasa. Uslovi za to da se kapljice kamenca zajedno sa mehurićem popnu do šljake leže u činjenici da su verovatno adhezivna snaga kapljice sa mehurićem i sila uzgona koja deluje na mehurić veće nego gravitacija kapljice.

Ključne reči: Platina; Intermetalni spoj; Kamenac; Šljaka; Površinski napon; Flotacija

