

Investigation of Cu, Pb, Zn, As, Sb distribution during the lead semiproducts and copper-zinc concentrate comelting

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The issues of smelting of difficult for treatment high-sulfur copper-zinc concentrate together with semiproducts and reverts of lead manufacture are considered. The possibility of direct extraction of copper, lead and zinc from copper-zinc concentrate to target products in a one-stage process is substantiated. Presented are the results of laboratory meltings for coprocessing of lead semiproducts, reverts and copper-zinc concentrate at a temperature of 1250 °C under the melt bubbling conditions.

High indexes of metal extraction into target products were achieved: copper into matte – up to 95% against 88.9% according to the existing technology; lead into lead bullion – up to 94% against 70.8% and zinc into slag – up to 95% compared to 83.7%. Extraction of arsenic and antimony into dust – 91 and 92.3% compared to 69.5% and 59.8%, respectively, which provides circumstances for their removal from the overall process chain of lead production.

The use of the proposed solution in lead production will lessen the load on the environment; improve the process figures of the blast concentrating smelting process by reducing the loss of copper, lead with slag and high recovery of arsenic and antimony into dust. Compared to the existing technology, the consumption of coke and quartz flux is significantly reduced: from 6.7 to 2.5% and from 12.5 to 4.7% of the charge weight, respectively.

The results of comelting of lead semiproducts and copper-zinc concentrate can form the basis for the development of a new manufacturing method.

Key words: lead semiproducts, copper-zinc concentrate, charge, distribution, non-ferrous metals, impurities, comelting, direct smelting.

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Introduction

One of the options for special-purpose treatment of lead reverts and semiproducts is a technology of blast concentrating smelting (BCS), which is used in Kazzinc LLC for a rather long time. The current state of the BCS technology does not ensure the attainment of acceptable indicators for the extraction of copper into matte (~83%), zinc into slag (less than 60%), lead into crude metal (no more than 60%), arsenic and antimony into dust (about 60 and 50%, respectively). Notwithstanding obvious disadvantages, the blast concentrating smelting technology continues to be used as a forced measure, at least partially solving the issue of processing a large volume of lead semiproducts and reverts [1].

Blast concentrating smelting is in need of an advanced improvement, both in the technology management and in the design of the proposed solutions. It is also relevant to adopt another, more promising solution for processing the resulting lead semiproducts together with copper-zinc concentrate with the use of modern continuous

or batch furnace units (electric, combustion (for example, of a short drum-type), bubbling furnaces). An important argument in favor of simultaneous progress on these two, far from equivalent ways of solving the main task, namely, deep processing of lead semiproducts and reverts, is a realization of manufacturing the high-sulfur copper-zinc concentrate at Nikolayevskoye deposit of the Eastern Kazakhstan region.

Direct smelting of high-sulfur copper-zinc concentrate with its simultaneous use as a forming and sulfidizing agent in a BCS would allow a one-stage highest possible extraction of lead into crude metal, copper into matte, and zinc into slag from the processed charge materials, including the concentrate. From the practical point of view, this way of perfection of the lead semiproducts and reverts processing means looks attractive due to the familiarity with BCS (availability of the accumulated operating experience) and minimum capital outlays for manufacturing application.

Application of modern processes of direct lead smelting is restrained by objective reasons: the composition complexity of the polymetallic charge to be processed,

from which it is necessary to extract maximum number of valuable components with great completeness [2–3], the complexity of instrumentation and technology management [4–5]. The positive results of current bubbling technologies (Isasmelt, SKS, furnace with side blowing (VF), etc.) successfully used in lead production as applied to the processing of the main flow of natural lead raw materials are widely represented in the technical literature [6–8]. In spite of this, the behavior of accompanying metals-impurities (As, Sb, etc.) in the lead processes is not fully covered, whereas they are given a lot of attention in copper production [9–10].

The works [11–12] are concerned with individual melting of copper-zinc concentrates in oxidative bubbling processes; almost all the studies have there been performed in relation to copper production and have pursued the only goal — the elaboration of a direct one-stage processing of copper-zinc concentrates. However, the results of [13] show the availability of not only direct processing of copper-zinc concentrate in converters, but also its simultaneous use as a sulfidizing agent which improves the quality of the resulting products and rises the transition of arsenic and antimony to dust.

In our opinion, the development of the **direct smelting technology** for processing the lead semiproducts and reverts in a mixture with copper-zinc concentrate is of great practical interest.

The purpose of this work is to study the comelting of the lead production semiproducts and reverts with high-sulfur copper-zinc concentrate under equilibrium conditions, to determine the concentrate consumption influence on the distribution of parent and impurity metals between the melting products and, accordingly, on their quality.

Routine of experiments

To conduct experiments on reduction and sulfiding smelting, there have been used semiproducts and reverts of lead production at Kazzinc LLC (Table 1) and high-sulfur copper-zinc concentrate of Nikolayevskoye deposit with the following composition, wt.%: 18.57 Cu; 2.3 Pb; 12.5 Zn; 24.6 Fe; 33.4 S; 0.22 As; 0.25 Sb. Coke containing 87.9% carbon was used as a reducing agent.

Experiments were performed with the addition of 10, 20 and 30% of copper-zinc concentrate to the initial mixture of semiproducts and reverts (Table 1). Quantitative ratios of initial materials in all the experiments remained

constant, which has corresponded to the factory practice [1]. The quartz flux and coke consumption was determined by calculation depending on the copper-zinc concentrate being dosed to the initial charge, from considerations of keeping up the ratio of iron and silica contents in the slag (Fe)/(SiO₂) at the constant level of ~0.8. Temperature of the experiments was 1250 °C.

The experiments were carried out on a specially designed laboratory unit with a continuous supply to the reaction zone of a CO–CO₂ gas mixture having given composition with a CO₂/CO = 0.4 ratio, which has been corresponded to the oxygen partial pressure in a real blast concentrating furnace at the level of P_{O₂} = 2.74·10⁻⁴ Pa. The CO–CO₂ gas mixture consumption in all the experiments was constant and amounted to 1 l/h. The procedure of experiments is described in [14].

Preliminary experiments have shown that the composition of waste gases reaches stability when the melt is blown for 10–15 minutes regardless of the initial charge composition. The duration of the melt blowing with a CO–CO₂ gas mixture in all experiments was assumed to be 15 minutes.

On completion of blowing, the melt was settled for 20 minutes, after which the crucible was removed from the furnace and subjected to air quenching. The resulting products in a solid form were separated from each other, weighed and subjected to chemical analysis and mineralogical studies. Each experiment was repeated three times to ensure reliability of the results. All the samples have demonstrated clearly visible separation of slag, matte and lead bullion.

The chemical composition of samples of slags, matte and lead bullion was determined using an inductively coupled plasma mass spectrometer Agilent 7700 Series ICP-MS (USA). Mineralogical studies of the polished surfaces of solid samples of slags and matte were performed on a Carl Zeiss Neofot microscope (Germany). The composition of waste gases during experiments has been periodically monitored using a Crystall-2000 chromatograph (Russia).

The results of the chemical compositions analysis as well as quantitative data on the yield of melting products for each series of experiments provided the base for making a balance sheet of materials with calculation of the element distribution in the source materials and produced products (Table 2).

Table 1.
Chemical composition of semiproducts, reverts and flux of lead production

| Material | Content, wt. % | | | | | | | | | |
|--|----------------|------|------|------|------|------|------|------------------|------|-------|
| | Cu | Pb | Zn | Fe | S | As | Sb | SiO ₂ | CaO | Other |
| Copper slips | 29 | 36 | 4 | – | 8.77 | 3.87 | 1.4 | – | – | 16.96 |
| Poor matte of shaft reduction smelting | 20.85 | 19.5 | 11.4 | 16.7 | 11.1 | 1.1 | 0.56 | – | – | 18.79 |
| Converter slag | 3.83 | 33.5 | 4.54 | 15 | – | 2.3 | 0.94 | 21.66 | 5.4 | 12.83 |
| Alkaline fusion cake | 0.08 | 61.7 | 24.1 | – | – | 0.97 | 1.4 | – | – | 11.75 |
| Quartz flux | – | 0.62 | – | – | – | – | – | 74.8 | 15.7 | 8.9 |

Table 2.

Balance sheet of materials of laboratory smelting of the initial charge with a 30-percent consumption of copper-nickel concentrate

| Materials | Total | | Cu | | | Pb | | | Zn | | | Fe | | | As | | |
|-----------------|--------------|--------------|-------------|----------|--------------|-------------|----------|--------------|------------|----------|--------------|-------------|----------|--------------|------------|----------|--------------|
| | g | % | I | II | III | I | II | III | I | II | III | I | II | III | I | II | III |
| Fed | | | | | | | | | | | | | | | | | |
| Charge | 130.0 | 97.6 | 20.8 | 16.0 | 100.0 | 30.1 | 23.2 | 100.0 | 9.8 | 7.5 | 100.0 | 15.1 | 11.6 | 100.0 | 2.4 | 2.4 | 100.0 |
| Coke | 3.1 | 2.4 | – | – | – | – | – | – | – | – | – | – | – | – | – | – | – |
| Total | 133.1 | 100.0 | 20.8 | – | 100.0 | 30.1 | – | 100.0 | 9.8 | – | 100.0 | 15.1 | – | 100.0 | 2.4 | – | 100.0 |
| Produced | | | | | | | | | | | | | | | | | |
| Lead bullion | 29.3 | 22.0 | 0.9 | 3.1 | 4.3 | 28.2 | 96.2 | 93.6 | – | – | – | – | – | – | – | – | – |
| Matte | 40.9 | 30.7 | 19.7 | 48.3 | 94.8 | 1.6 | 3.8 | 5.2 | 0.6 | 1.5 | 6.4 | 6.8 | 16.6 | 45.0 | 0.2 | 0.5 | 8.5 |
| Slag | 51.8 | 38.9 | 0.1 | 0.3 | 0.7 | 0.3 | 0.5 | 0.9 | 9.1 | 17.5 | 92.7 | 8.2 | 15.8 | 54.5 | 0.01 | 0.02 | 0.5 |
| Dust, gases | 11.2 | 8.4 | 0.0 | 0.4 | 0.2 | 0.1 | 0.8 | 0.3 | 0.1 | 0.8 | 0.9 | 0.1 | 0.7 | 0.5 | 2.2 | 19.4 | 91.0 |
| Total | 133.1 | 100.0 | 20.8 | – | 100.0 | 30.1 | – | 100.0 | 9.8 | – | 93.6 | 15.1 | – | 55.0 | 2.4 | – | 100.0 |

I – amount, g; II – content, %; III – distribution, %.

Results and discussion

The use of high-sulfur copper-zinc concentrate instead of copper-zinc ore in the charge composition of a blast or another concentrating smelting requires ensuring fusibility and, accordingly, acceptable fluidity of slag. A distinctive feature of the slags of lead melts is the ability to dissolve (to slag) significant amounts of ZnO with keeping the fluid state [15]. In case of excessive accumulation of ZnO in the slag, resulting in its heterogenization, it is necessary to adjust the slag composition.

In the conditions of the copper-zinc concentrate and lead semiproducts comelting, key adjustment of the slag composition is carried out by picking up the appropriate consumption of copper-zinc concentrate, which to the point provides the slag base of a requires composition with the specified FeO:CaO:SiO₂ ratio. The choice of the appropriate composition of the FeO – CaO – SiO₂ system slag is very important in the context of minimizing the concentrations of copper in it [16] combined with providing the highest possible solubility of ZnO while preserving fusibility of the multicomponent slag of the final composition [17]. It was found that the CaO content in slags should not exceed 16% for maximum concentration of ZnO in them; ZnO in slags becomes practically insoluble with the calcium oxide content of 24%. Lessening of the SiO₂ content increases the ZnO solubility in slags [15].

Replacement of copper-zinc ore with high-sulfur copper-zinc concentrate is associated with the need to ensure its controlled interaction in the conditions of blast smelting. For this purpose, one can apply widely used methods and devices for local feeding of fine materials (an Inco process, ejection devices, etc.), the variety of which allows to choose the most suitable method for loading the concentrate into a shaft furnace.

Analysis of the yield of smelting products depending on the consumption of copper-zinc concentrate showed a modest increase in the yield of slag from 37.9 to 39.4%

with an increase in the concentrate consumption from 20 to 30%. This does not seem to be a matter of principle, since its maximum level remains below the output of the slag formed by the existing technology – 46.1%.

The revealed slight increase in the yield of matte along with an increase in the concentrate consumption seems quite natural: the introduction of copper-zinc concentrate into the charge adds an additional amount of copper, the transition of which to matte affects its output. On the other hand, the yield of matte will be under the opposite effect of reducing the lead content in it on rising the consumption of copper-zinc concentrate.

Mutual compensation of these two trends provides almost constant yield of matte in the studied range of the copper-zinc concentrate consumption. This is confirmed by the dependence of a sharper decrease in the lead content in matte than the increase of the copper content in it depending on the concentrate consumption, shown in Fig. 1.

X-ray phase analysis and mineralogical studies of matte samples obtained during charge melting in the experiments with different consumption of copper-zinc concen-

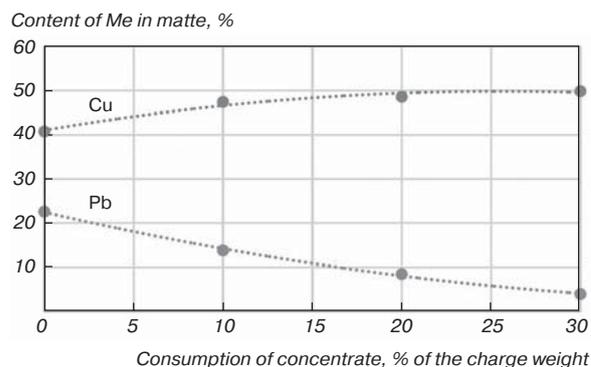


Fig. 1. Dependence of copper and lead content in the matte upon the copper-zinc concentrate consumption

| Sb | | | S | | | O ₂ | | | SiO ₂ | | | CaO | | | C | | | Other | | |
|-------|-------|-------|------|------|-------|----------------|-----|-------|------------------|------|-------|------|------|-------|-----|------|-------|-------|------|-------|
| I | II | III | I | II | III | I | II | III | I | II | III | I | II | III | I | II | III | I | II | III |
| 1.0 | 1.0 | 100.0 | 15.2 | 15.2 | 100.0 | 4.4 | 4.4 | 100.0 | 14.9 | 14.9 | 100.0 | 5.4 | 5.4 | 100.0 | – | – | – | 11.0 | 8.5 | 96.7 |
| – | – | – | – | – | – | – | – | – | – | – | – | – | – | – | 2.8 | 88.0 | 100.0 | 0.4 | 12.0 | 3.3 |
| 1.0 | – | 100.0 | 15.2 | – | 100.0 | 4.4 | – | 100.0 | 14.9 | – | 100.0 | 5.4 | – | 100.0 | 2.8 | – | 100.0 | 11.4 | – | 100.0 |
| 0.1 | 0.2 | 6.6 | – | – | – | – | – | – | – | – | – | – | – | – | – | – | – | 0.2 | 0.6 | 1.5 |
| 0.01 | 0.02 | 0.9 | 10.6 | 26.0 | 70.0 | – | – | – | – | – | – | – | – | – | – | – | – | 1.3 | 3.2 | 11.6 |
| 0.002 | 0.004 | 0.2 | – | – | – | 4.3 | 8.3 | 99.0 | 14.8 | 28.6 | 99.4 | 5.4 | 10.3 | 99.4 | – | – | – | 9.6 | 18.6 | 84.5 |
| 0.9 | 8.5 | 92.3 | 4.6 | 40.8 | 30.0 | 0.04 | 0.4 | 1.0 | 0.1 | 0.8 | 0.6 | 0.03 | 0.3 | 0.6 | 2.8 | 24.8 | 100.0 | 0.3 | 2.5 | 2.4 |
| 1.0 | – | 100.0 | 15.2 | – | 100.0 | 4.4 | – | 100.0 | 14.9 | – | 100.0 | 5.4 | – | 100.0 | 2.8 | – | 100.0 | 11.4 | – | 100.0 |

trate have showed the absence of intermetallics of copper and iron ($\text{Me}_3\text{As-}$, $\text{Me}_3\text{Sb-}$ type compounds) and metallic copper and lead dissolved in matte, which are typical for the mattes of the existing technology [15]. This allows us to assert that even minimum consumption of copper-zinc concentrate (10% of the charge) excludes the possibility of the metallized matte formation. Low lead concentrations are found in the matte obtained by melting with 30% of copper-zinc concentrate. The lead content in them is ~8%, versus 22% corresponding to the existing technology.

The results of X-ray phase analysis of powder samples and mineralogical studies of solid samples of the slags obtained in the melts with different concentrate consumption have also confirmed the absence of copper and iron intermetallics in them. This indicates that smelting of lead semiproducts and reverts with copper-zinc concentrate in any amounts (from 10 to 30%) completely eliminates the losses of copper and lead in the mentioned forms. On the contrary, the presence of copper and lead in the slags of the widely applied BCS technology is so significant that their presence determines the final distribution of metals. The share of the revealed copper and lead sulfide inclusions in all slag samples can be ignored, due to their minimal concentrations. Therefore, the losses of copper and lead in the slags during comelting of the lead charge with copper-zinc concentrate are mainly determined by an oxide form. The pronounced character of the dependence of the copper and lead content in the slag on the copper-zinc concentrate consumption, shown in Fig. 2, confirms the stated assumption.

The dust amount in each melt was determined by the difference between the amount of metals in the initial charge and their amount in the resulting melting products: in slag, matte and lead bullion (Table 2). Dusts are the smallest (less than 1 μm) solid particles (sublimates) formed as a result of condensation of volatile metal com-

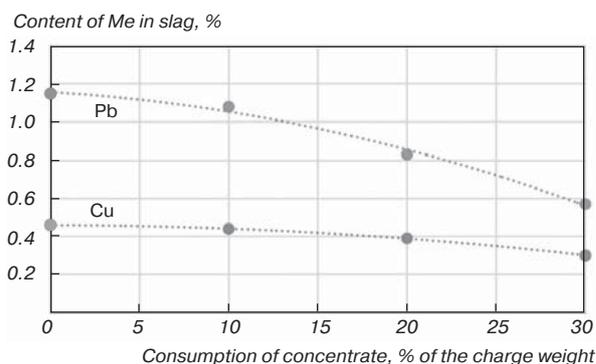


Fig. 2. Dependence of copper and lead content in the slag upon the copper-zinc concentrate consumption

pounds from the vapor-gas phase when its temperature decreases. The dust yield decreases slightly with an increase of the copper-zinc concentrate consumption: from 6.7% according to the existing technology to 5.6% when melting with a 30% concentrate consumption.

The best process indicators are achieved in case fo a melt with the copper-zinc concentrate consumption of 30%. A comparative analysis of the balance sheets of materials of the existing BCS technology and test smelting with the copper-zinc concentrate consumption of 30%, shown in Fig. 3, clearly demonstrates the significant improvement in the main process indexes.

At the same time, attention is drawn to the more than the two-fold decrease of the quartz flux consumption: from 12.5% (according to the existing technology) to 4.7% when melting the initial charge with copper-zinc concentrate.

A detailed analysis of the quartz flux consumption dependence on that needed for obtaining the slag of the required composition is considered in [10]. It was found that the excess amount of silica is ~40% of its total content in factory slags. Mineralogical studies of solid sam-

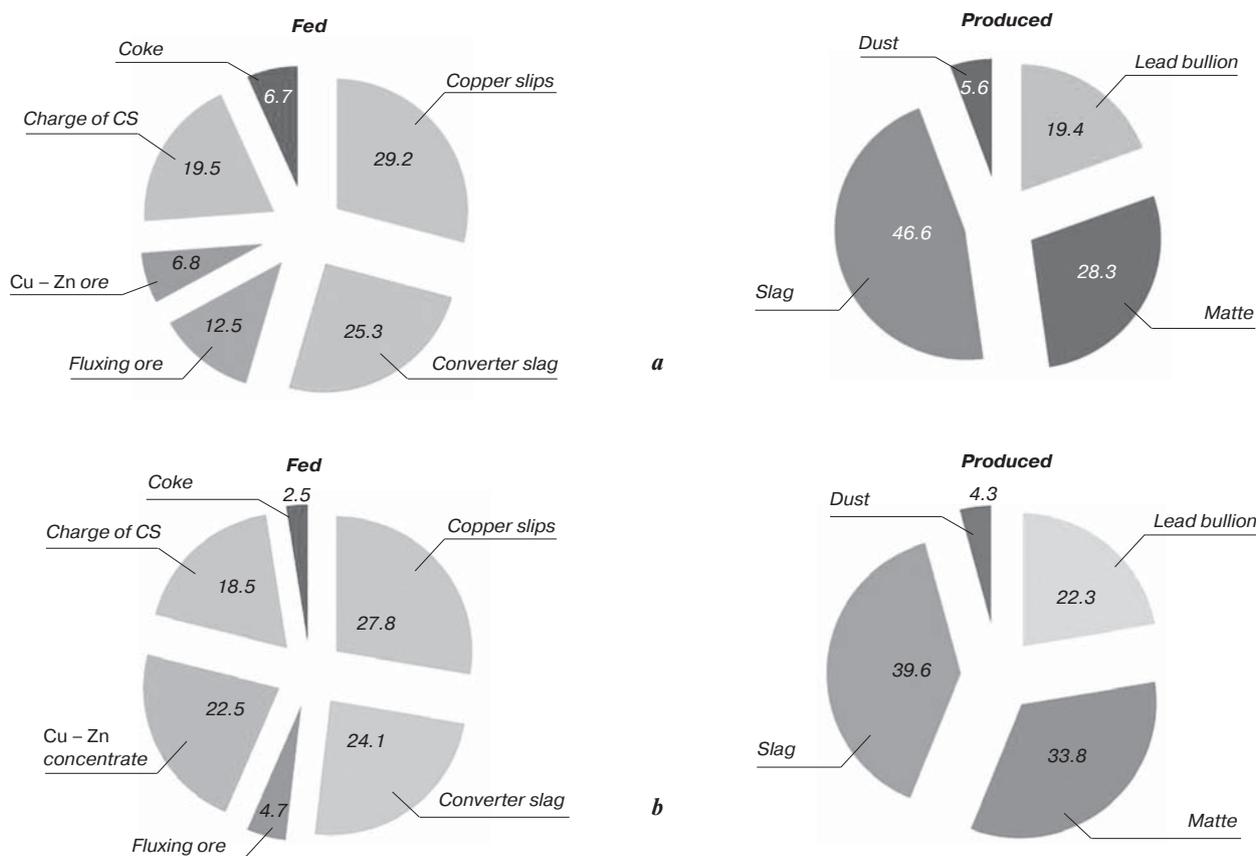


Fig. 3. Balance sheets of materials of the melts:

a — an existing technology of blast concentrating smelting; *b* — smelting of the charge with copper-zinc concentrate

ples of factory slags, along with fayalite, have found the clearly distinguished inclusions of relic quartz belonging to the original flux and not participating in the formation of a homogeneous slag phase of a given composition. This attested to the “non-assimilation” of a part of the flux, which causes a heterogenization of the slag melt, the viscosity rise and significant decrease of its fluidity (mobility), which renewal will require an increased coke consumption amounted 6.7% of the charge weight to increase a temperature in the operating zone of the BCS furnace.

The generalized results of the analysis of the chemical compositions of the melting products (by main elements), shown in Table 3, and the distribution of metals between the melting products (Table 4), show a significant quality improvement when smelting using copper-zinc concentrate compared to the existing BCS technology.

The dependences of copper, lead, zinc, arsenic and antimony extractions into target products on the copper-zinc concentrate consumption for the entire array of test melts are shown in Fig. 4.

Table 3.

Chemical compositions of the lead semiproducts, reverts and copper-zinc concentrate comelting products

| Product | Content*, wt% | | | | | | | | |
|--------------|---------------|-------|-------|-------|-------|-------|-------|------------------|------|
| | Cu | Pb | Zn | Fe | S | As | Sb | SiO ₂ | CaO |
| Lead bullion | 5.73 | 91.09 | – | – | – | – | 2.34 | – | – |
| | 3.02 | 96.28 | – | – | – | – | 0.23 | – | – |
| Matte | 40.78 | 22.58 | 2.34 | 12.6 | 15.15 | 2.92 | 0.87 | – | – |
| | 49.81 | 4.0 | 1.08 | 16.92 | 26.25 | 0.52 | 0.02 | – | – |
| Slag | 0.46 | 1.15 | 9.81 | 10.26 | – | 0.16 | 0.06 | 19.69 | 8.74 |
| | 0.3 | 0.57 | 19.23 | 16.94 | – | 0.03 | – | 23.1 | 5.39 |
| Dust | 2.36 | 5.84 | 3.66 | 1.51 | – | 37.19 | 19.55 | 3.2 | 0.73 |
| | 0.9 | 1.98 | 1.9 | 1.62 | – | 47.45 | 20.67 | 1.45 | 0.34 |

*Numerator — an existing smelting technology (BCS).

Denominator — charge smelting with copper-zinc concentrate.

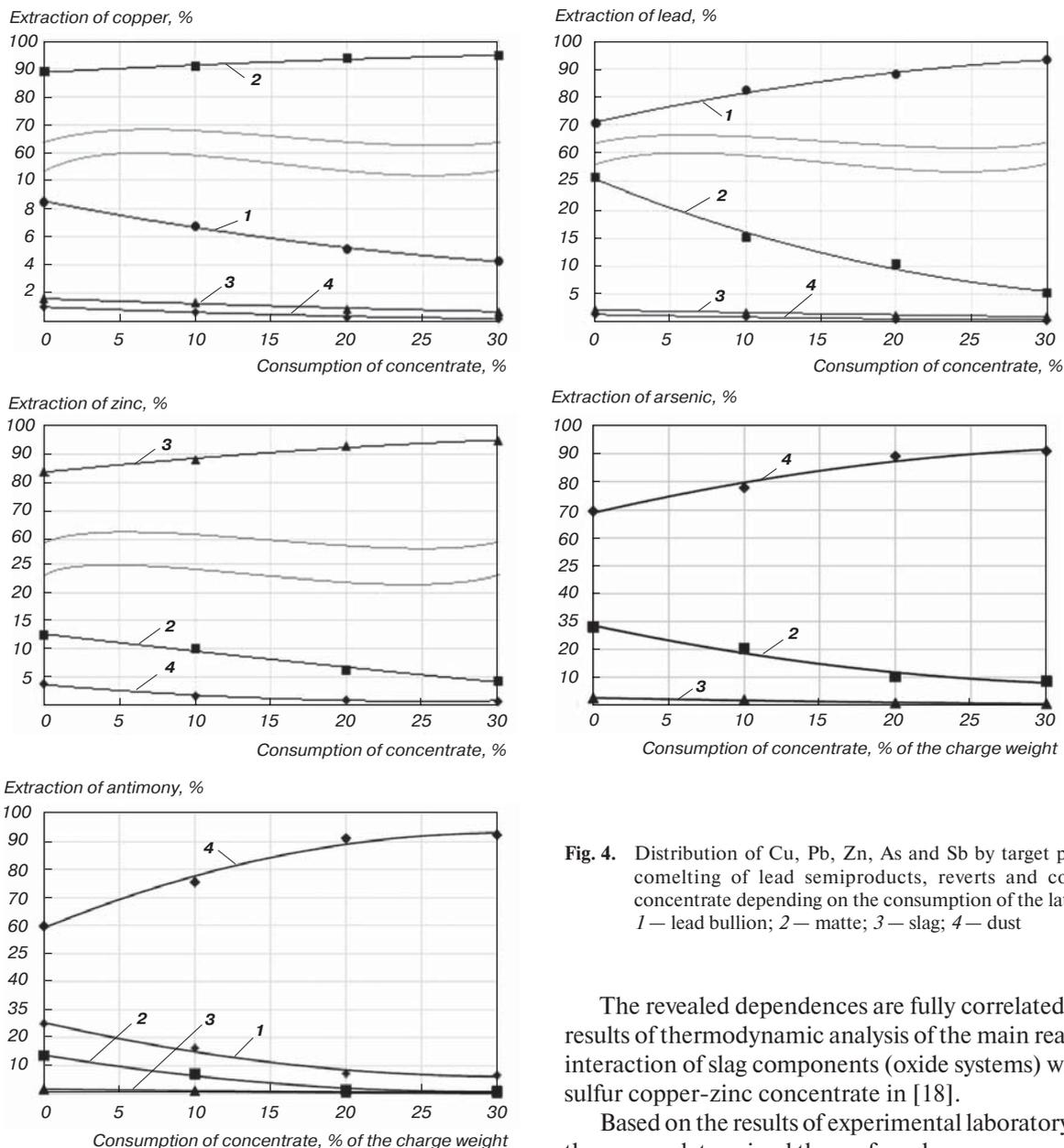


Fig. 4. Distribution of Cu, Pb, Zn, As and Sb by target products of comelting of lead semiproducts, reverts and copper-zinc concentrate depending on the consumption of the latter: 1 – lead bullion; 2 – matte; 3 – slag; 4 – dust

The revealed dependences are fully correlated with the results of thermodynamic analysis of the main reactions of interaction of slag components (oxide systems) with high-sulfur copper-zinc concentrate in [18].

Based on the results of experimental laboratory studies, there were determined the preferred process parameters of the semiproducts, reverts and copper-zinc concentrate comelting at a temperature of 1250 °C under reducing conditions that can serve as a basis for the development of a new direct smelting technology:

- consumption of high-sulfur copper-zinc concentrate is 30% of the charge weight, including lead production semiproducts and reverts, quartz flux;
- the duration of blowing the melt in a periodic duty is 15 minutes, the settling time for separating the melting products after blowing is 20 minutes;
- consumption of coke (or coal) as a reducing agent is 2.5% of the charge weight.

Conclusions

1. There has been shown an opportunity to improve the existing technology of blast concentrating smelting by co-processing of semiproducts and reverts with high-

Table 4. Metal distribution among the lead semiproducts, reverts and copper-zinc concentrate comelting products

| Product | Metal distribution*, % | | | | |
|--------------|------------------------|------|------|------|------|
| | Cu | Pb | Zn | As | Sb |
| Lead bullion | 8.5 | 70.0 | – | – | 25.0 |
| | 4.3 | 90.7 | – | – | 3.1 |
| Matte | 85.0 | 25.0 | 12.5 | 32.0 | 9.0 |
| | 93.3 | 10.9 | 4.4 | 8.5 | 4.2 |
| Slag | 3.5 | 3.5 | 83.7 | 5.0 | 6.0 |
| | 1.4 | 1.2 | 93.5 | 1.4 | 1.6 |
| Dust | 1.0 | 1.5 | 3.8 | 63.0 | 60.0 |
| | 1.0 | 1.2 | 2.1 | 94.1 | 88.1 |

*Numerator — an existing smelting technology (BCS).
Denominator — charge smelting with copper-zinc concentrate.

sulfur copper-zinc concentrate. The best results that ensure high extraction of copper, lead, zinc, arsenic and antimony into the target products of melting are achieved at the concentrate consumption is equal to 30% of the charge weight.

2. It is established that comelting provides a one-stage high selective extraction of copper into matte and zinc into slag from the copper-zinc concentrate. At the same time, the simultaneous use of copper-zinc concentrate as a sulfidizing agent reduces the content of copper and lead in the slag, increases the arsenic and antimony extraction into dust. Compared to the existing technology, the coke and quartz flux consumption is reduced (from 6.7 to 2.5% and from 12.5 to 4.7% of the charge weight, respectively), which significantly reduces the spending for processing.

3. There have been determined the preferred process parameters of the semiproducts, reverts and copper-zinc concentrate comelting, which can form the basis for the development of a new engineering process.

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