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PROCESSING of CONVERTER SLAG of BALKHASH COPPER-SMELTING PLANT. PART I – STRUCTURE and PHASE COMPOSITION

Abstract: The objects of studies were converter slag samples of Balkhash Copper-Smelting Plant (BCP) in initial state and after heat treatments. There were used methods of mineralogical analysis, X-ray phase semi-quantitative analysis, scanning electron microscopy with electron probe microanalysis, and chemical analyses. It was demonstrated that initial converter slag and its heat treated samples have identical matrices with almost complete coincidence of the mineral and phase compositions. A distinguishing feature is a quantitative ratio of mineral components in the slag bulk. Almost all iron is oxidized and has the form of fayalite, hortonolite, magnetite, and magnetite with other elements (silicon, copper, zinc, aluminum) included into its lattice. The structure of all slag samples indicates on the association of sulphur with copper only. Copper in slag occurs both in the native state and in the sulphide form. A slow cooling of converter slag contributes to decreasing of sulphide-metallic suspension in the melt bulk and its coarsening.

Keywords: converter slag, heat treatment, structural studies, mineral and phase composition

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Introduction. Upon pyrometallurgical processing of copper-containing ores blister copper is obtained by way of matte converting over fluxes in whose capacity siliceous ores are usually used [1]. Earlier converter slag were processed in melting furnaces together with furnace burden, but after passing to autogenous melting resulted to magnetite-containing slag, almost all copper-smelting plants have moved to their flotation. Herewith the flotation is applied to slowly cooling slag to enlarge mechanical suspension with the copper weight ratio redistribution towards the formation of secondary copper minerals, or to granulated one after its preliminary depletion together with furnace slag from autogenous melting in separate facility [2-5].

Despite a large number of works dedicated to this issue [3, 6-9] it is inappropriate to extrapolate the available data into slag obtained by the other copper-smelting plants because for such complex systems because metallurgical melts general regularities have not been established yet.

Mattes containing 45-58 % of copper, 3-6 % of lead, and 1.5-4.2 % of zinc obtained upon melting of concentrates in a Vanyukov furnace are converted at the Kazakhmys Smelting LLP Balkhash Copper-Smelting Plant (BCP). Not only copper and precious metals, but also iron, lead, zinc, arsenic, and antimony move into matte upon melting. Subsequently in the process of converting some portion of metals moves into a gaseous phase and dust, and the other one moves into white matte and slag, and then into copper and slag.

Rich slag after second blow and slag poured off white matte are usually put back to the converting process start-up.

The processing of copper mattes results to converter slag containing 1.5-3.5 % of copper characterized by the ratio of mechanic and electrochemical losses of 20-40 and 60-80 %, respectively [2]. At BCP this ratio is reverse: 70-80 and 20-30 %, respectively, with copper content in slag about 4-8 %.

Such abnormal content of mechanical suspension is related mainly to the converter cold run, which in its turn is related by the following circumstances:

- feeding of cold matte melt to converting; it is particularly noticeable when one furnace is working;
- processing of polymetallic mattes (rich in copper); it proceeds with an intense heat balance because in case of lead sulphide oxidation approximately thrice as less heat is released than that in case of iron sulphide oxidation;
- unavailability of additional heating of the converters with fuel oil, gas or powdered coal upon any process shutdowns; it is a regular practice at 80 % of all smelters in the world [10];
- usage of flux ore having a wide range of silica content.

Converter cold run has sharply increased magnetite content in slag (up to 20 % of Fe_3O_4 for 9 months in 2013). Moreover it lead to growth of copper solubility therein and increased its viscosity due to magnetite precipitation into a separate phase resulted to increase of copper mechanical losses.

This overoxidized slag together with its own poor ore are milled to the fraction -0.07 mm and floated with the obtaining of off-grade copper concentrate containing, % wt.: 14.1-22.2 Cu; 13.0-22.1 S; 13.4-18.2 SiO_2 and 25.0-28.7 $Fe_{gen.}$. According to 2013 data in this concentrate from 4.2 to 12.1 % of iron is in the form of magnetite. Tailings after processing contain about 0.9 % of copper. It is noteworthy that the initial ore used for obtaining of copper concentrate contains about 0.65 % of copper (ore taken from the Zhezkazgan deposit) and less. It means that technology-related wastes containing more copper than the ore fed to the copper production are dumped.

Thus the processing flow-sheet for converter slag adopted at BCP does not meet the requirements of rational utilization of raw materials. This work aims at the assessment of the effect of the converter slag cooling mode on its composition and the form of metal occurrence therein.

Experimental Part. Several options of slag heat treatment have been tested. A crucible with a charge of industrial converter slag of the composition, % wt.: 5.40 Cu; 4.6 Pb; 3.9 Zn; 40.6 Fe; 0.056 Sb; 1.81 S; 21.5 SiO_2; 0.86 CaO; 0.86 MgO; 3.8 Al_2O_3 has been placed in a furnace heated preliminary up to the preset temperature (1,250°C). It was held for slag melting for 0.5 hours, and then cooled down to 1,100; 1,000 or 900°C at the rate of 0.2-0.3 degree/s, held at this temperature for 1 hour and the furnace has been shut down. The crucible with the slag charge was kept in the furnace. The melt cooling rate has been constantly decreasing down to 0.08-0.03 degree/s. According to the second option after slag melting at 1,250°C for 30 min the furnace has been shut down, and the crucible has been cooled together with the furnace or has been quenched on a massive metal plate. All experiments have been carried out in the air atmosphere; the temperature in the isothermal area of the furnace has been controlled by a platinum-rhodium thermocouple.

The initial slag and its heat-treated samples passed through mineral optical mineralogical analysis (microscope Leica DM2500P) in polished and transparent sections (the initial and quenched samples) and immersion media, X-ray phase semi-quantitative analysis (D8 ADVANCE «BRUKER»), scanning electron microscopy with electron probe microanalysis - SEM-EPMA (JEOL JXA-8230), as well as X-ray fluorescence (Venus 200 PANalytical B.V.), and chemical analyses.

Results and Discussion. The results of X-ray phase and chemical analyses for the initial samples and after heat treatment are shown in Tables 1 and 2, respectively. To identify the mineral phases a thorough research has been carried out using SEM-EPMA. The following phases have been identified: metallic copper, chalcosine (Cu_2S), magnetite (Fe_3O_4), maghemite (Fe_2O_3), silicon and iron oxides ($Fe_{2.95}Si_{0,05}O_4$), fayalite (Fe_2SiO_4) and hortonolite [$2(Fe_{0.85}$, $Mg_{0.15})O{\cdot}SiO_2$], zinc, aluminum and iron oxide [$Zn(Al_{0.5}Fe_{1.5})O_4$], copper and iron oxide [$Cu(Fe_2O_4)$], lead oxide (Pb_2O_3), aluminum silicate with iron admixture, $Cu_2(Sb,Pb)O_2$, and Cu_2PbS.

Non-availability of sulphide and metallic copper phases upon X-ray phase analysis using Kα radiation of Cu (Table 1) with the sufficiently high content of copper in slag (up to 5-8 %, see Table 2) seems to be explained by overlapping of the main diffraction lines of copper (a=2.08) and magnetite.

Table 1 – Phase composition of slag before and after their heat treatment

Sample #	Fayalite*, hortonolite	Magnetite	Silicon and iron oxide	Heat treatment parameters
1	71.2*	28.8	-	Initial slag
2	38.6	12.2	49.2	Smelting−1,250 °C -30 min, quenching
3	41.1	11.5	47.5	Smelting−1,250 °C -30 min, cooling down to 1100 °C – 1 hour, cooling with the furnace
4	42.9	11.8	45.3	Smelting−1,250 °C -30 min, cooling down to 1000 °C – 1 hour, cooling with the furnace
5	40.7	25.1	34.2	Smelting−1,250 °C -30 min, cooling down to 900 °C – 1 hour, cooling with the furnace
6	45.1	15.3	39.6	Smelting−1,250 °C -30 min, cooling with the furnace
Note: fayalite is identified only in sample # 1				

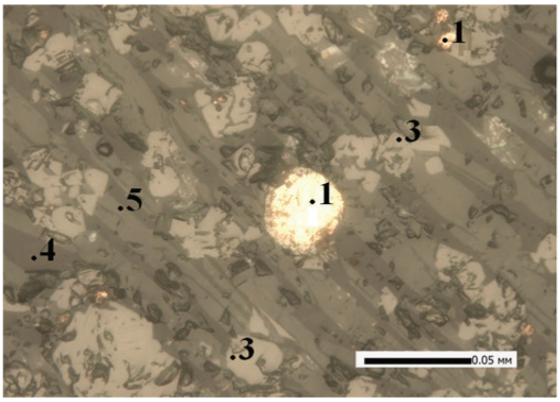
Considerable fluctuations of the values of copper content (and to a lesser extent of lead content) in the experimental slag (Table 2) are generally related to the non-uniform distribution of mechanical suspension.

Table 2 – Chemical composition of slag before and after their heat treatment

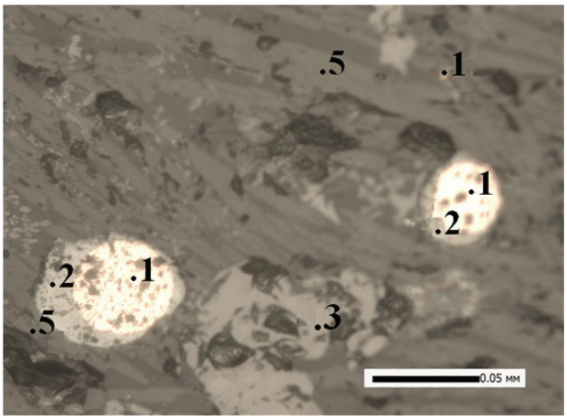
Sample #	Content of the components, %								
	Cu	Pb	Zn	Fe	SiO_2	CaO	MgO	Al_2O_3	S
1	4.97	5.3	3.3	41.3	20.1	0.30	0.58	4.2	1.08
2	7.60	4.8	2.2	40.6	18.8	0.27	0.58	4.7	1.61
3	8.00	4.8	3.2	42.0	18.5	0.30	0.63	4.5	1.85
4	6.10	4.8	3.4	41.3	17.4	0.32	0.63	4.4	1.39
5	7.10	5.5	3.6	42.7	18.3	0.27	0.63	4.2	1.57
6	5.10	5.4	3.5	42.7	19.9	0.20	0.35	4.2	1.09

Mineralogical and X-ray structural analyses of the samples have shown that the composition of the matrix (barren minerals and oxidized iron) of the initial sample and the heat-treated samples is characterized by almost complete similarity of mineral compositions. Only their quantitative ratios differ: content of the magnetite phase decreases, but there appears magnetite with silicon included into its lattice - $Fe_{2.95}\cdot Si_{0.05}\cdot O_4$; fayalite content decreases almost by half due to the oxidation of ferrous iron and its residual portion is transformed into hortonolite; it is also a mineral of the olivine group - $2(Fe_{0.85}Mg_{0.15})O\cdot SiO_2$, an intermediate mineral type in the isomorphous fosterite-fayalite series, but somewhat richer in the forsterite molecule (Table 1). The latter data are in consistency with results of studies on converter slag [11].

Optical microphotographs of samples ## 1 and 2 are shown in Figure 1. It demonstrates that magnetite and iron and silicon oxide do not differ in the polished section. They form grains of tetragonal, rhombic, hexahedral and irregular forms and their aggregations. The grain size has interval from 0.02 to 0.35 mm.



Sample 1



Sample 2

1 – metallic copper; 2 – chalcosine; 3 – magnetite and iron and silicon oxide; 4 – fayalite (hortonolite); 5 – silicate slag phase

Figure1 – Polished sections of samples 1 and 2 in reflected light, X400

The structure of the slag phase is granular and banded, with alternation of thin- and long prismatic crystals of fayalite and the silicate slag phase. On its background there are magnetite, maghemite, and iron oxide with silicon having the form of idiomorphic grains. The similar structure of converter slag was observed before [4, 11]. All samples have a glass filling the gaps between hortonolite grains (in sample #1 – fayalite grains) and the slag phase and constituting the first percentage.

Copper has the form of thin fine-dispersed and fine-grained suspension in free grains as well as in the form of precipitates in chalcosine. The latter is represented by a high temperature type of cubic syngony - digenite. The similar structure of fine copper-containing suspension in iron-silicate slags after their quick cooling has been observed earlier [4, 5, and 12]. In the area of view of the microscope (X400) some enlargement of dispersed copper precipitates and beads of chalcosine has been observed:

№	Cu	Cu_2S	Qty of grains
1	0.005-0.010 mm	up to 0.25	12-15
2	0.010-0.015 mm	up to 0.70	from 4-5 to 10-12
3	<0.01-0.02 mm; rarely 0.04 mm	0.15-0.25	2-4
4	< 0.005-0.025 mm	0.25-0.30	Considerable precipitation of fine-dispersed copper and 3-4 grains
5	< 0.005-0.030 mm	0.20-0.35	

Table 3 – Element composition of oxidized phases, %

Phase	Sample #	O	Na	Mg	Al	Si	K	Ca	Fe	Cu	Zn	Pb
Magnetite	1	33.3	-	-	1.6	0.19	-	-	61.6	-	3.15	-
	2	35-38	-	-	2-2.4	0-0.7	-	-	56-60	-	2.4-3.5	-
	3	35.7	-	-	1.7	0.13	-	-	59.7	-	2.7	-
	4	31-35	-	-	1.7-2.5	0-0.3	-	-	59-64	-	3-3.3	-
	5	33.3	-	-	1.3	-	-	-	62.7	-	2.8	-
	6	34.8	-	-	2.0	-	-	-	60.2	-	3.0	-
Hortonolite	1	39-40	0-3	0.3-0.6	0-1.3	12-14	0-0.9	0-0.3	30-44	-	4.5-4.8	0-6
	2	38-39	-	0.4-0.8	1.4-1.9	13-16	0.7-1.1	0.4-0.5	32-33	0.3-1.4	4.6-4.8	4-6
	3	38.4	-	0.8	-	11.6	-	-	44.6	-	-	-
	4	35-38	-	0.8	-	12-13	-	-	44-46	-	4.5-6.0	-
	5	36.8	-	0.7	-	11.8	-	-	45.0	-	5.7	-
	6	38.7	3.1	0.3	-	11.8	-	0.15	39.8	0.03	6.23	-
Silicate slag with lead oxide	1	30-33	3.1	-	3.1-4.5	16-17	1.2-1.5	1.1-1.5	9.6-9.8	-	3.6-4.5	30-31
	2	34-38	-	0-0.4	1.4-3.1	12-18	0.7-1.3	0.5-1.8	12-33	0-1.4	4.4-5	10-25
	3	34-35	-	-	3-5	11-18	1-1.3	0.6-1.8	12-36	-	4.4-5	10-25
	4	33-38	-	-	3.3-5.3	19-20	1.8-2.1	1.2-1.7	7.2-15	-	3.1-4.0	17-26
	5	36.0	-	-	4.1	20.0	2.0	1.8	7.2	0.51	3.0	25.6
	6	37.7	2.7	-	3.7	20.6	1.8	1.8	7.7	-	3.1	20.5

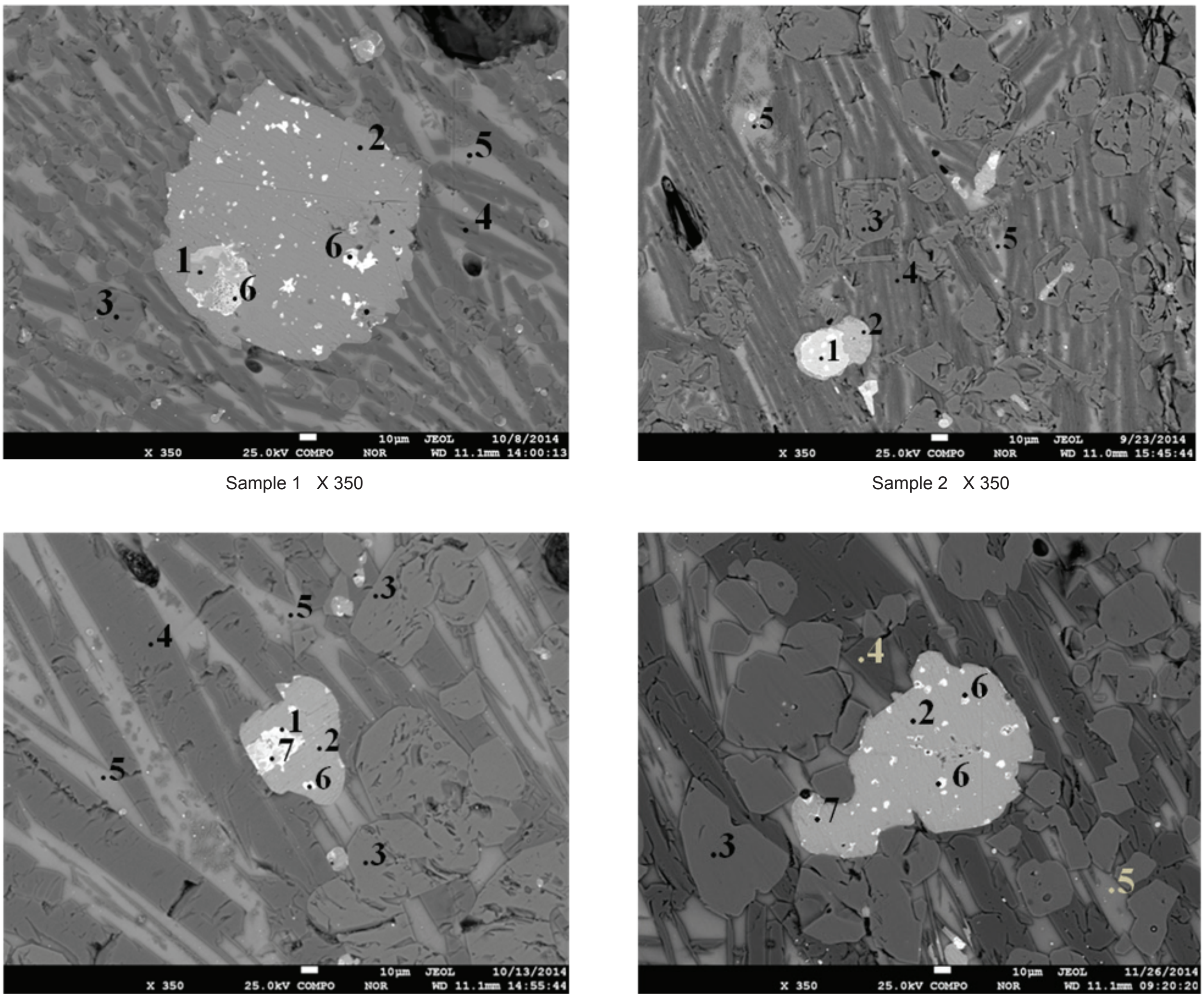
The mineralogical analysis data for the heat treated slag were clarified with SEM-EPMA methods (see Table 3); also the structure and composition of the suspended beads have been identified (see Figure 2).

In the visual field of sample # 1 (Figure 2) an almost pure chalcosine bead of about 0.17 mm is observed (point 2) in the matrix of the silicate slag phase (point 5) and fayalite (point 4), with zinc and lead dissolved therein. Small-sized and bright spots in the bead body (point 6) are lead globes with a small quantity of chalcosine (about 10 %). A medium-sized spot (to the left and top) is an intermetallic compound of copper (77.4 %), arsenic, antimony, and iron; below – lead and copper (97 % Pb).

A small chalcosine bead was also observed in sample # 2 (Figure 2, point 2). There is a copper (point 1) with antimony and iron, % wt: 91.66 Cu; 7.05 Sb and 1.29 Fe. The brighter inclusions in copper-antimony alloy contain up to 71 % of lead.

Figure 2 shows the structure of converter slag of sample # 3. In the bead of non-reacted white matte with 2.22 % of Fe (point 2) the metal copper (point 1) and lead containing about 10 % of copper and 1.5 % of antimony (point 6). Lower and darker area of copper grain is copper-arsenic-antimony alloy, % wt: 41.9 Cu; 48.4 As and 9.7 Sb (point 7).

After slow cooling of initial slag melted at 1,250 ^{o}C (sample # 6, Figure2) there were detected inclusions of lead (and lead with copper as well) in the bead of pure white matte (point 6) whereas in left and narrow portion of bead a complex sulphidized alloy containing, % wt.: 57.55 Cu; 14.33 Pb; 21.56 Sb; 1.57 Fe and 4.99 S, has been revealed (point 7).



Sample 1 X 350

Sample 2 X 350

Sample 3 X 350

Sample 6 X 350

1 – metallic copper; 2 – chalcosine; 3 – magnetite and iron and silicon oxide; 4 – fayalite (hortonolite); 5 – silicate slag phase; 6 – lead; 7 – alloy

Figure 2 – SEM micrographs for the converter slag sections of samples 1, 2, 3, and 6. Numbers show the microanalysis spots

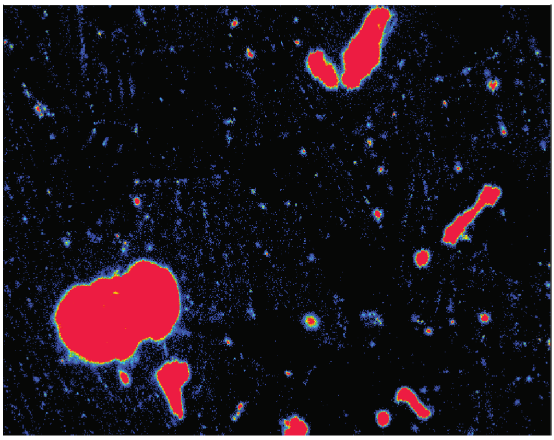
White matte beads of sample # 4 include intermetallic compounds of the type Cu-Sb-Pb containing copper from 23 up to 90 %.

In the heat-treated sample of converter slag # 5 a copper-containing bead has more complex structure: white matte contains a metalized copper phase with arsenic – 18.08 %, antimony – 3.13 % and iron – 1.19 % dissolved therein. Nearby there is a metalized phase with lesser contents of copper (60.41 %) and arsenic (4.66 %), and a larger content of antimony (29.83 %). Outside the metallic phase the sulphide metalized phase is observed again containing, % wt: 62.97 Cu; 18.52 As; 3.54 Sb; 2.96 Fe, and 12.02 S.

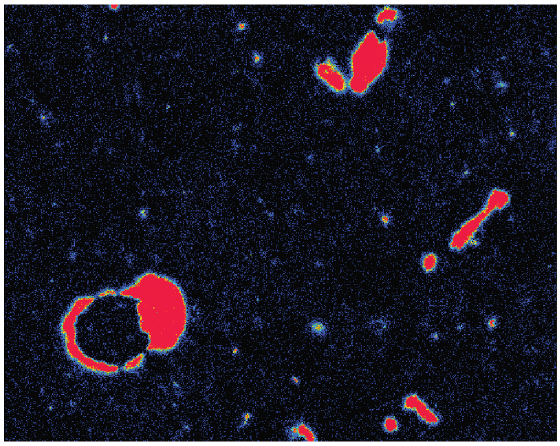
For a number of studied slag samples EPMA wave-dispersion mapping on chemical elements has been performed. It was shown that different schedules of heat treatment of converter slag does not effect on its structure. It remains granular and banded one with alternation of thin- and long prismatic crystals of silicate (with small content of iron) and fayalite slag phase. There are magnetite and iron oxide with silicon in the form of idiomorphic grains. Lead concentrates preferably in iron-depleted slag phase and zinc – in fayalite phase. Copper and sulphur maps on their characteristic X-ray radiation (Kα) are shown in Figure 3 after the standard graphic transformation for brightness and contrast; it is clearly demonstrated that after slag remelting and quenching followed after its holding for 30 min at 1,250°C the substantial quantity of suspension remains in the slag.

The structure of all slag samples revealed by EPMA indicates on the association of sulphur with copper only, which is in complete inconformity with the data of the works [1-6].

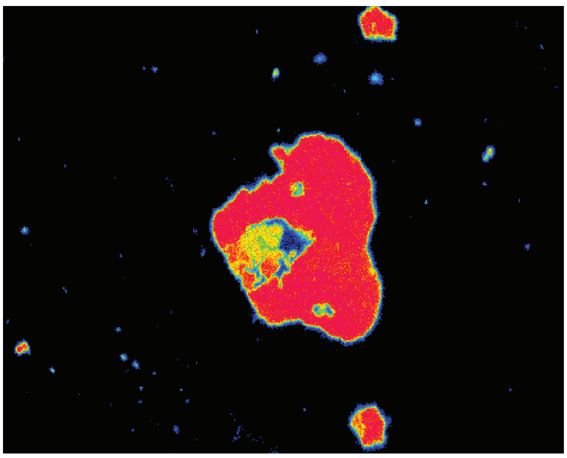
Smelting of the same slag followed by heat treatment considerably decreases the quantity of dispersed suspension only in the sample passed through treatment 1,100 °C for 1 hour.



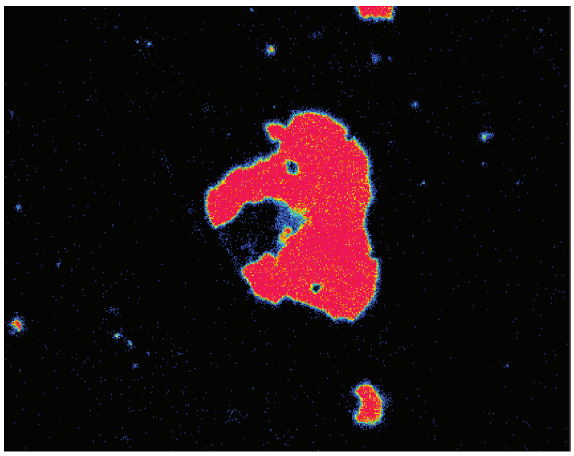
Sample 2 Copper X 350



Sample 2 Sulphur X 350



Sample 3 Copper X 350



Sample 3 Sulphur X 350

Figure 3 – Copper and sulphur distribution pattern in slag samples 2 and 3

Conclusions. The studies carried out with the use of mineralogical, X-ray phase analyses, scanning electron microscopy with electron probe microanalysis have shown that the initial converter slag and its heat treated samples demonstrate identical matrices with almost complete coincidence of the mineral and phase compositions. A distinguishing feature is a quantitative ratio of mineral components in the slag bulk.

In the studied converter slag of the Balkhash Copper-Smelting Plant almost all iron is oxidized and is in the form of fayalite, hortonolite, magnetite and magnetite with other elements (silicon, copper, zinc, aluminum) included into its lattice. The structure of all slag samples revealed by SEM-EPMA indicates on the association of sulphur with copper only.

Copper in slag occurs both in the native state and in the form of sulphide. Lead occurs both in copper-containing grains and in melted slag. Zinc occurs both in silicate and ferrous oxide melts.

In metalized copper and lead compounds the presence of arsenic and antimony is observed sometimes in high quantities.

A slow cooling of converter slag after its heat treatment contributes to some decreasing of sulphide-metallic suspension in the melt bulk and its coarsening. Nevertheless considerable quantity of copper in form of particles with sizes 0.01 mm and smaller is still in slag. It apparently cannot permit to obtain flotation tails to be recognized as dumps on metal and to recover the smelting mass entirely. It is expected that pyrometallurgical treatment of liquid smelting in the specially designed facilities can permit to recover the smelting mass entirely through correction of its composition.

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ТҮЙІНДЕМЕ

Зерттеу нысандары ретінде Балқаш мыс зауытының бастапқы және термиялық өңдеуден кейінгі конверторлық қождарының сынамалары алынды. Минералогиялық, рентгенфазалық талдаулардың, растрлық электрондық микроскопияның және рентгенспектралдық микро талдаудың көмегімен бастапқы конверторлық қождың және оның термиялық өңдеуден кейінгі сынамаларында минералдық және фазалық құрамы іс жүзінде толық сәйкес келетін ұқсас матрицалар болатыны анықталды. Айырмашығы тек қож массасындағы минералдық құраушылардың мөлшерінде. Темір түгел тотығады және фаялит, гортонолит, магнетит және торына басқа элементтер (кремний, мыс, мырыш және алюминий) кіретін магнетит түрінде болады. Қождың барлық үлгілерінің құрылысы күкірттің тек мыспен бірігетінін көрсетті. Қождардағы мыс таза және сульфид түрінде болады. Конверторлық қожды қайта балқытқаннан кейін баяу суыту арқылы балқыма көлеміндегі сульфидті-металдық жүзгіннің мөлшері азайтылып, оның іріленуі қамтамасыз етіледі.

Түйін сөздер: конверторлық қож, термиялық өңдеу, құрылымдық зерттеулер, минералдық және фазалық құрам.

РЕЗЮМЕ

Объектами исследования являлись пробы конвертерного шлака Балхашского медеплавильного завода в исходном состоянии и после термообработки. С помощью минералогического, рентгенофазового анализа, растровой электронной микроскопии и рентгеноспектрального микроанализа установлено, что исходный конвертерный шлак и термически обработанные его пробы имеют идентичные матрицы с практически полным совпадением минерального и фазового составов. Отличительным признаком является количественное соотношение минеральных составляющих в шлаковой массе. Практически все железо окислено и находится в виде: фаялита, гортонолита, магнетита и магнетита с входящими в его решетку другими элементами (кремний, медь, цинк и алюминий). Структура всех образцов шлака указывает на ассоциацию серы только с медью. Медь в шлаках выявлена как в самородном, так и в сульфидном виде. Медленное охлаждение конвертерного шлака после его переплавки, способствует некоторому снижению сульфидно-металлической взвеси в объеме расплава и её укрупнению.

Ключевые слова: конвертерный шлак, термообработка, структурные исследования, минеральный и фазовый состав.

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