2028; 345(2):16-28 ISSN-L 2616-6445, ISSN 2224-5243

DOI: 10.31643/2028/6445.13
Metallurgy and Metallurgical Engineering

Deep Copper Recovery from Autogenous Smelting Slags under Strongly Reducing Conditions

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Received: *March 17, 2026*
Peer-reviewed: *April 21, 2026*
Accepted: *June 1, 2026*

ABSTRACT

Deep reduction of copper smelting slags is a promising route to recover entrained non-ferrous metals and to generate slags suitable for further processing. This work investigates the depletion of fayalite–magnetite slags formed during autogenous smelting of copper concentrates in conditions simulating the oxidation and reduction zones of a two-zone Vanyukov furnace. Laboratory charges of 100 g, containing 14.63–16.82 wt.% Cu, 25.6–27.6 wt.% Fe, 30 wt.% S and 15 wt.% SiO_2, were smelted at 1350 °C with controlled oxygen injection to produce slags containing 0.93–1.54 wt.% Cu, 30.05–32.30 wt.% SiO_2 and 7.8–9.8 wt.% Fe_3O_4. Subsequent reduction at 1300 °C was carried out with activated carbon in a fivefold stoichiometric excess relative to magnetite, at an oxygen-containing blast flow rate of 5 L/h and a 1 h holding time. Chemical analysis shows that Fe_3O_4 in slag decreases from 7.8–7.95 wt.% to 2.5–2.6 wt.%, while copper content drops from 0.93–1.033 wt.% to 0.43–0.50 wt.% under oxygen partial pressures of 10^{-12}–10^{-11} atm. X-ray diffraction and electron microscopic studies reveal a transition from fayalite–magnetite slags with dispersed metallic copper and sulfides to fayalite-dominated matrices containing ferruginous sphalerite, copper minerals of the bornite–chalcopyrite type, iron oxides and glassy phases. Simultaneous thermal analysis demonstrates that all major endothermic and exothermic events are completed by 1300 °C, supporting this as an optimal temperature for deep slag depletion. The results define an operating window—slag composition, temperature, reductant dosage and pO_2—under which copper losses to slag can be reduced to about 0.5 wt.% in industrially relevant fayalite slags.

Keywords: copper slag processing, pyrometallurgical treatment, copper-bearing sulfide materials, reductive melting, sem analysis, metallurgical waste.

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Introduction

Intensive, or deep, reduction of metallurgical slags is increasingly important for maximising metal recovery and minimising waste. The effectiveness of such reduction processes is largely controlled by slag composition, process temperature and oxygen partial pressure, all of which influence phase equilibria, viscosity and metal solubility [[1],[2],[3],[4],[5],[6]].

The processing of metallurgical waste, including copper smelting slags, has been the subject of extensive research in many scientific communities. Researchers from Kazakhstan have investigated the thermodynamic modelling of mineral formation during the processing of toxic metallurgical waste, including the behaviour of zinc-containing phases [[7],[8]], and the utilisation of technogenic raw materials from South Kazakhstan metallurgical enterprises as secondary resources [[9],[10]]. These studies highlight the broader challenge of valorising metallurgical by-products — a context directly relevant to the present work.

Only a limited number of studies have explored deep slag reduction at extremely low oxygen potentials near 10^{-10} atm, leaving the kinetics and mass-transfer mechanisms under such conditions poorly constrained [11]. This knowledge gap is critical for autogenous copper smelting technologies, where copper losses to slag and magnetite build-up strongly depend on pO_2.

Available thermodynamic data suggest that decreasing pO_2 below 10^{-11} atm, and at the same time, at low iron contents in the alloy, leads to a noticeable reduction in copper activity and its solubility in slag [4]. Moreover, earlier studies [[12],[13],[14]] show that deep slag reduction is a complex and sensitive process that is strongly dependent on oxygen potential. This underlines the need for additional experimental and theoretical investigations to improve both the efficiency and economic performance of copper recovery [[15],[16],[17],[18]].

Several approaches have been proposed for copper recovery from smelting slags. Flotation is a widely used method; however, its efficiency is often limited by the fine dissemination of copper-bearing phases, and copper recovery under standard conditions typically does not exceed 45–50% [19], although higher values may be achieved with intensive grinding and optimised reagent conditions.

Electrothermal reduction represents an alternative route, yet it is constrained by the absence of bath agitation and the strongly endothermic nature of reduction reactions, which cause rapid temperature decrease, while electrode power is often insufficient to maintain stable thermal conditions, negatively affecting reaction kinetics and phase separation [[20],[21],[22]].

In contrast, the approach investigated in the present study employs intensive gas injection (bubbling) within a Vanyukov (PV) furnace configuration, which promotes bath mixing, enhances mass transfer, and stabilises the thermal regime. These conditions are more favourable compared to conventional methods, enabling more efficient reduction and improved separation of copper-containing phases.

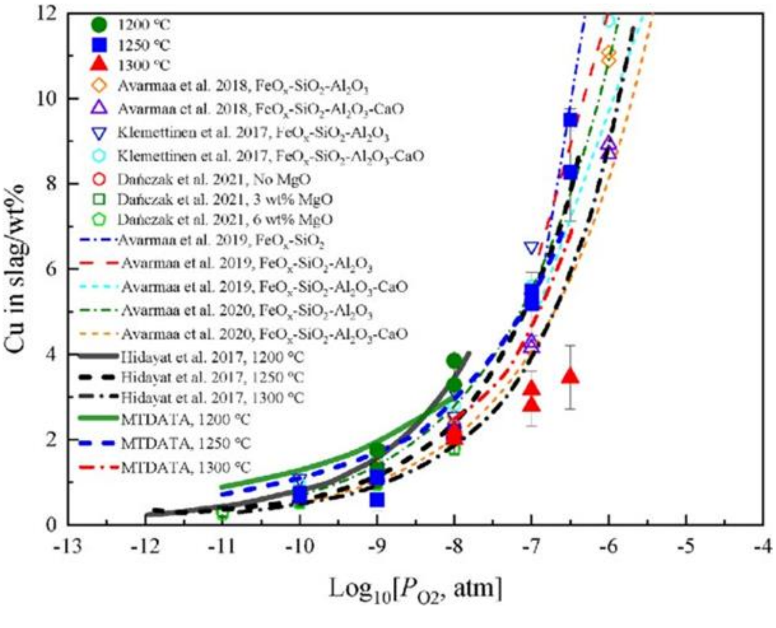


Figure 1 - Dependence of the copper content in the slag on the pO_2. Adapted from [23]

Figure 1 illustrates how the partial pressure of oxygen influences the copper concentration in the slag. In autogenous smelting, processes are conducted under oxidising conditions, with the oxygen partial pressure usually in the range of 10^{-6} to 10^{-8} atm. By contrast, establishing a deep reducing atmosphere (by adding coal or natural gas) significantly increases the thermodynamic possibility of reducing metal oxides.

For a Vanyukov (PV) furnace, achieving such deeply reducing conditions requires sufficient heat to enable effective interaction between the reducing agent and the slag. The present study focuses on experimentally defining the conditions for deep copper recovery from autogenous smelting slags, and on characterising the resulting slag structure and properties.

Experimental part

Laboratory experiments were conducted on 100 g charges smelted in a pre-calcined alund crucible (4) at 1350 °C in a silite furnace (1) designed to model autogenous smelting with gas purging (Figure 2). The charge consisted of copper concentrate containing 30 wt.% S and 27.0–27.6 wt.% Fe and a quartz flux with 70 wt.% SiO_2, added in an amount of 21.2 g per 100 g charge to form fayalite slags. For selected samples, experiments were carried out in triplicate to ensure reproducibility, and the reported values correspond to the average results.

After complete melting, oxygen was blown into the bath through an alund tube (3–5 mm inner diameter) at 1.0 L/min. The total oxygen input (43 L per 100 g charge) was calculated to oxidise iron sulfide and sulfur completely according to the reaction:

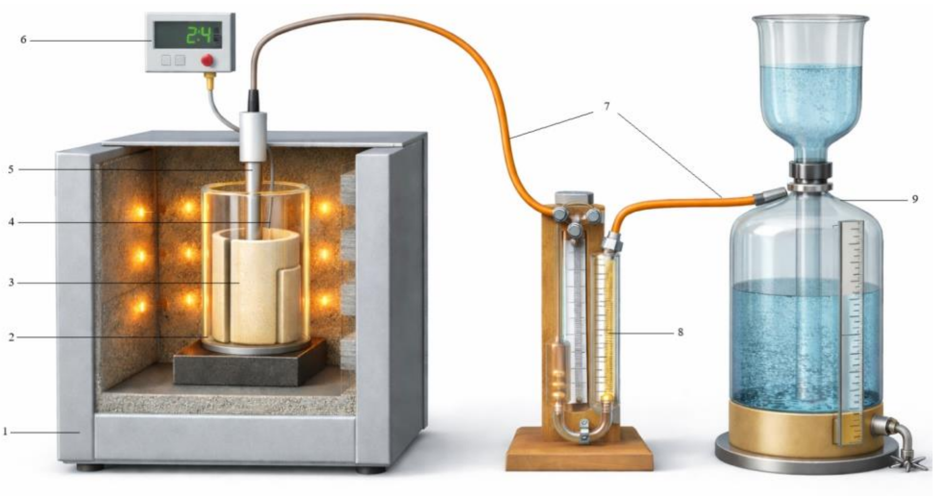
$$2FeS + 3O_2 \rightarrow 2FeO + 2SO_2 \qquad (1)$$

and to convert FeO to fayalite in the presence of silica. After purging, the crucibles were held for 30 min and air-cooled.

Slags and mattes were studied using X-ray diffraction with a Bruker D8 Advance diffractometer (Cu Kα radiation); phase identification and quantification were carried out using DIFFRAC.SUITE EVA Version 5.2.0.5 (Bruker AXS, 2020) with the PDF-2 (2023) reference database applying the RIR-based S-Q method. Scanning electron microscopy with microanalysis (JEOL JXA-8230), simultaneous thermal analysis (NETZSCH STA 449 F3 Jupiter, processed with Proteus software), and reflected-light petrography (OLYMPUS BX-51, 50×–1000× magnification).

For depletion experiments simulating the reduction zone, slags obtained in the first stage were reheated to 1300 °C using activated carbon (74.3 wt.% C) as a reducing agent. The reduction of magnetite proceeds according to the reaction:

$$Fe_3O_4 + C \rightarrow 3FeO + CO\uparrow \qquad (2)$$



1 – silite furnace; 2 – alund glass; 3 – Pt-Pt-Rh thermocouple; 4 – alund crucible; 5 – alund tube;
6 – millivoltmeter; 7 – rubber hose; 8 – rheometer; 9 – gasometer

Figure 2 - Diagram of a laboratory installation for modelling autogenous melting with purging of a melt with an oxygen-containing mixture

The amount of carbon added (2.5 wt.%) corresponds to approximately a fivefold excess relative to the stoichiometric requirement for magnetite reduction, considering the Fe_3O_4 content of the slag and the carbon purity of the reducing agent. An oxygen-containing blast of 5 L/h was maintained, and the melt was held for 1 h before cooling. The experimental procedure was based on our previous study [3], in which the reduction-stage methodology was described in detail.

Oxygen partial pressures were calculated from measured copper contents in slag using a published empirical relationship between Cu in slag and pO_2 for such systems [[13],[24]].

Results and Discussion

Table 1 summarises the compositions of charge, slag and matte. Each value represents the average of three independent experiments conducted for each sample. Within the narrow variation of charge composition, slag chemistry remained stable: 0.93–1.033 wt.% Cu, 31.3–32.05 wt.% SiO_2, 37.3–37.4 wt.% total Fe and 7.8–7.95 wt.% Fe_3O_4, with matte copper contents around 46 wt.% A modest increase in magnetite from 7.80 to 7.95 wt.% was accompanied by a rise in slag Cu from 0.93 to 1.03 wt.%. This relationship demonstrates a strong positive correlation (R^2 = 0.82), indicating that an increase in Fe_3O_4 content is associated with higher copper losses in slag (Figure 3), which is further supported by structural analysis. XRD of slag from test No. 2 (0.97 wt.% Cu) show a fayalite–magnetite matrix with small metallic copper inclusions (Figure 4). Elemental mapping reveals copper, lead and zinc sulfides dispersed in the silicate matrix, with magnetite associated with Fe–O-rich regions and fayalite prominent along grain boundaries (Figure 5).

Table 1 - Principal key components in the charge, slag, and matte (average values)

Test No.	Chemical composition, %							Slag mass, g
	Cu* *in charge	***Fe* *in charge***	***Cu* *in slag***	***SiO_2* *in slag***	***Fe*total *in slag***	***Fe_3O_4* *in slag***	***Cu* *in matte***	
1	16.82	27.00	0.930	32.05	37.4	7.80	46.30	44.0
2	16.73	27.60	0.970	31.90	37.3	7.90	46.20	43.6
3	16.35	27.10	1.033	31.30	37.3	7.95	45.90	44.6

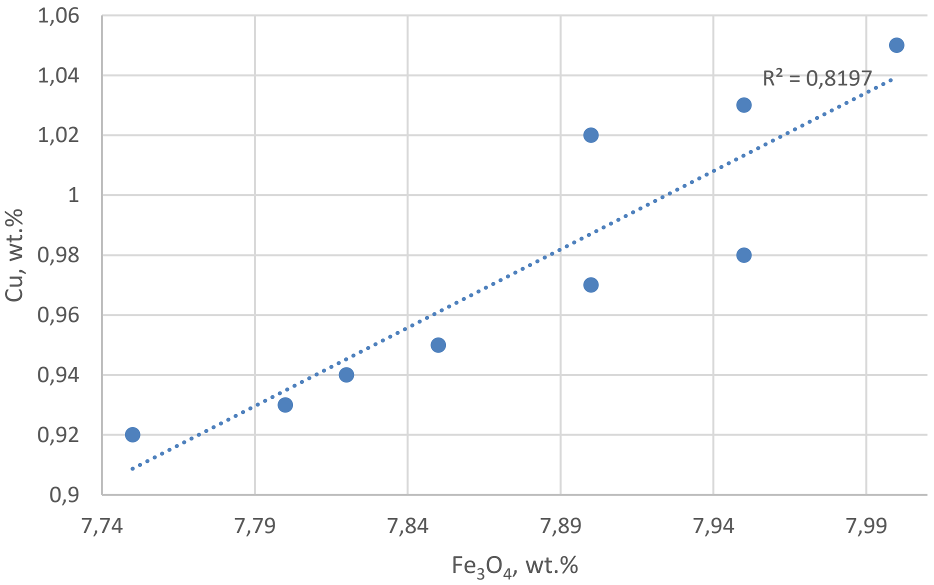


Figure 3 - Dependence of copper content in the slag on the magnetite content in the slag

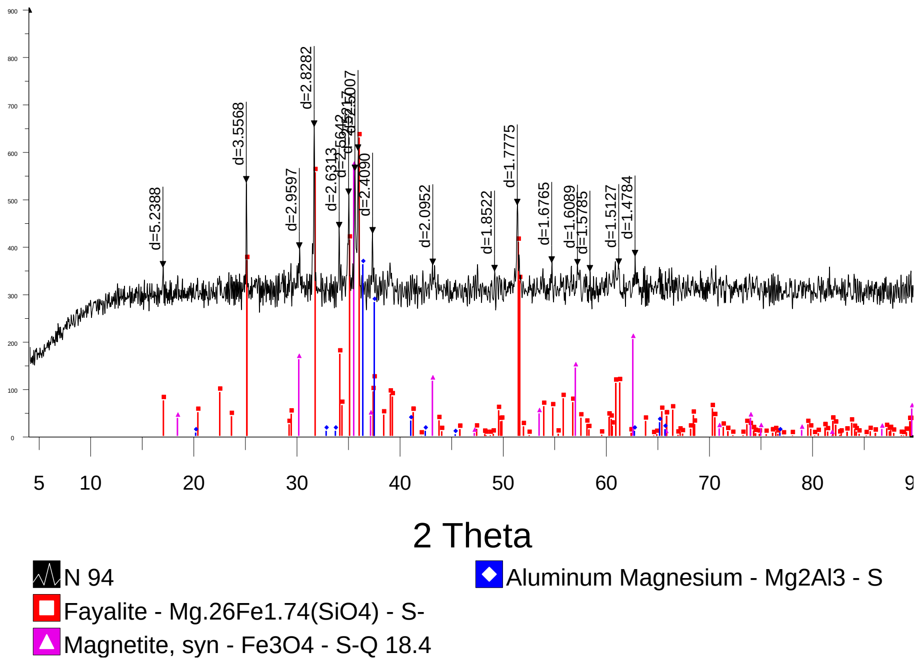


Figure 4 - Diffractogram of a slag sample containing 0.97% copper

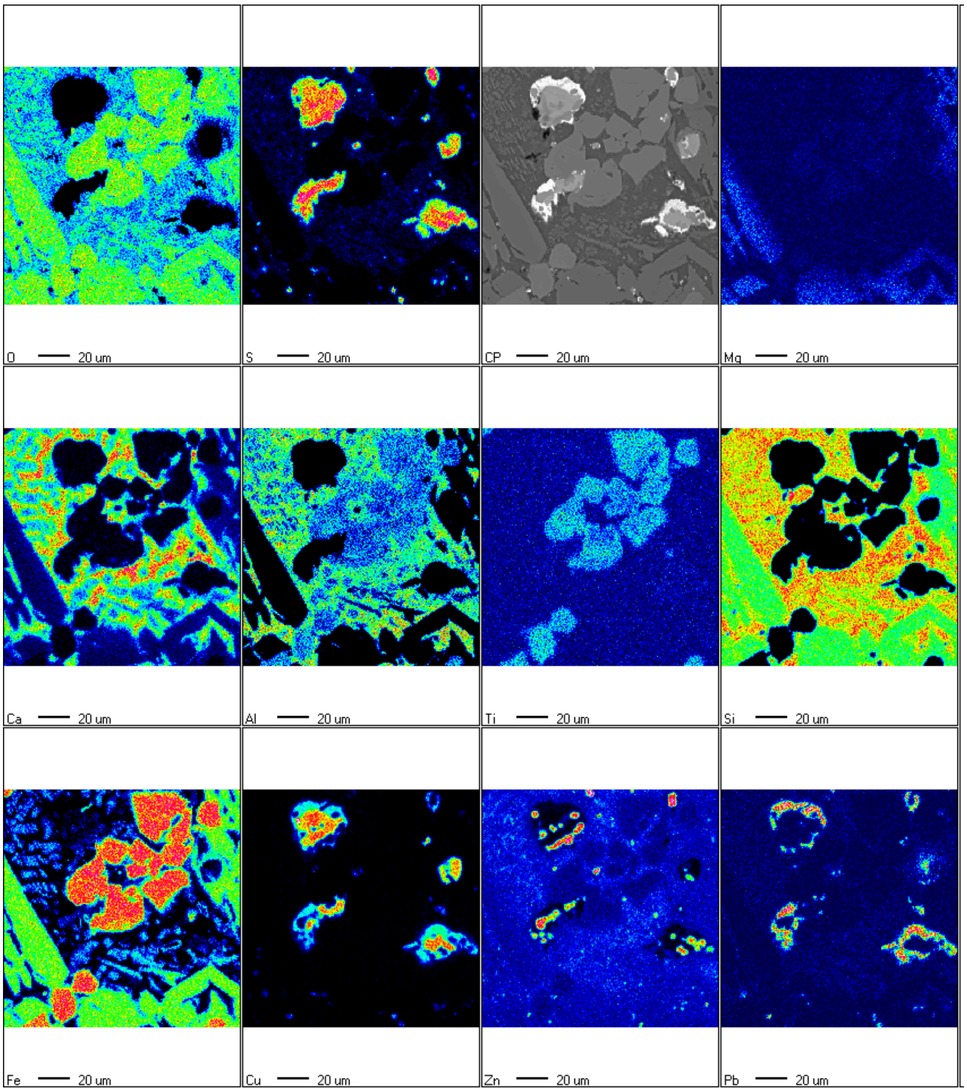


Figure 5 - Elemental mapping of the slag sample from test No. 2 containing 0.97% copper (WDS, x500)

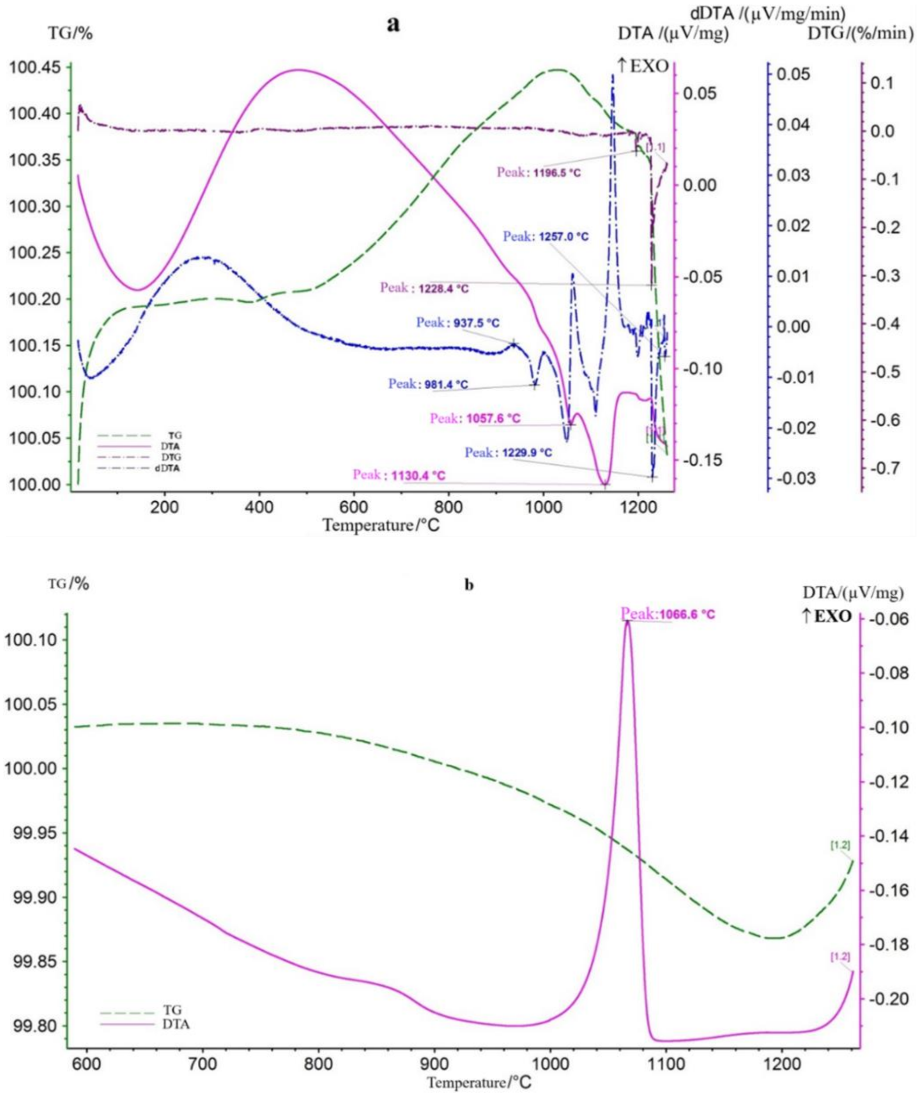


Figure 6 - Thermograms of a slag sample containing 0.97% Cu during heating (a) and cooling (b)

The distribution of elements within the slag sample obtained from test No. 2 is presented in Figure 5. The bright regions observed in the micrographs correspond to areas with elevated concentrations of specific elements. Based on these features, it can be inferred that the slag contains copper, lead, and certain zinc sulfides. The zones enriched in iron coincide with oxygen-rich areas, indicating the presence of magnetite. Fayalite is predominantly observed along the peripheral regions of the sample.

Thermal analysis of this slag indicates complete melting between 1280 and 1300 °C. Endothermic peaks at 1057.6 and 1130.4 °C correspond to the melting of major phases, with remaining phases liquefying around 1229.9 °C; during cooling, an exothermic effect near 1066.6 °C is accompanied by mass gain, consistent with crystallisation and oxidation (Figure 6).

Slags selected for depletion contained 0.93–1.54 wt.% Cu, 30.05–32.30 wt.% SiO_2 and 7.8–9.8 wt.% Fe_3O_4. Such slags require reduction treatment in order to decrease the content of non-ferrous metals.

During reduction at 1300 °C with activated carbon, two phases were formed: a slag phase and a bottom phase, whose compositions were similar to those of the matte phase formed during simulation of the oxidation zone of a two-zone PV furnace. Chemical composition of the obtained slag samples is presented in Table 2. These data indicate that magnetite was partially destroyed during smelting, with its content decreasing by 5.3–6.5 wt.%, which resulted in a lower copper concentration in the slag after reduction.

Table 2 – Chemical composition of major components after reduction of the slag

Test No.	Chemical composition, %			Coal consumption, g	pO_2, atm
	Cu	*SiO_2*	*Fe_3O_4*		
11	0.43	33.1	2.5	0.543	$\approx 10^{-12}$
12	0.47	32.9	2.6	0.545	$\approx 10^{-11}$
13	0.50	32.9	2.6	0.568	$\approx 10^{-11}$

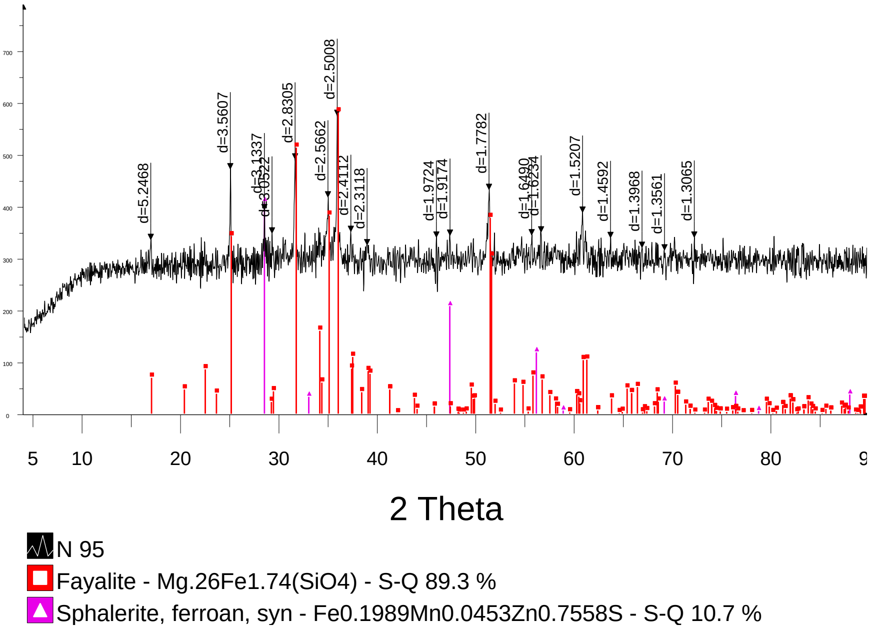


Figure 7 - Diffractogram of a sample of slag from test No. 12 containing 0.47% Cu

The results of the slag analysis after treatment with activated carbon revealed a substantial reduction in copper content, decreasing to 0.43–0.50 wt.% compared with the initial 0.93–1.033 wt.% in slags prior to reduction treatment. This decrease in copper concentration is attributed to the transformation of magnetite and other iron oxides into fayalite under reducing conditions. As a result, the magnetite content in the slag decreases to 2.5–2.6 wt.%, whereas before reduction it ranged from 7.8 to 7.95 wt.%.

Having calculated the partial pressure of oxygen according to the formula [13]:

$$\lg (Cu) = 0.221 \lg PO2 + 2.09 \quad (3)$$

where Cu is the copper content in the slag, % by weight, and pO_2 is the partial pressure of oxygen, atm., we obtain the pO_2 values (Table 2) in the slag under conditions of reducing the depletion of the slag by coal.

Based on the calculations performed, the obtained low oxygen partial pressure values indicate that the processes of depletion of copper slags occur under deeply reducing conditions, at $pO_2 < 10^{-11}$ atm. Under such conditions, the dissolved copper losses from the slag are restored, as well as the magnetite is restored. The reduction of magnetite has a beneficial effect on the viscosity of the slag. Reducing the viscosity of the slag, in turn, helps to reduce the mechanical losses of copper with the slag. Because, from studies conducted in the field of slag reduction treatment, it is known that the presence of magnetite in slag can increase its viscosity, which negatively affects the mechanical losses of copper [[25],[26],[27],[28],[29],[30],[31]]. However, the reduction processes occurring under deeply reducing conditions contribute to the transformation of magnetite and a decrease in thecontent of iron oxides, which favourably affects the physico-chemical properties of the slag, including its viscosity, which contributes to a more efficient removal of copper from the slag phase [[32],[33]].

X-ray diffraction analysis indicated that the slag sample of test No. 12, containing 0.47% Cu, consists of more than 85% fayalite with an admixture of iron-containing sphalerite (Figure 7).

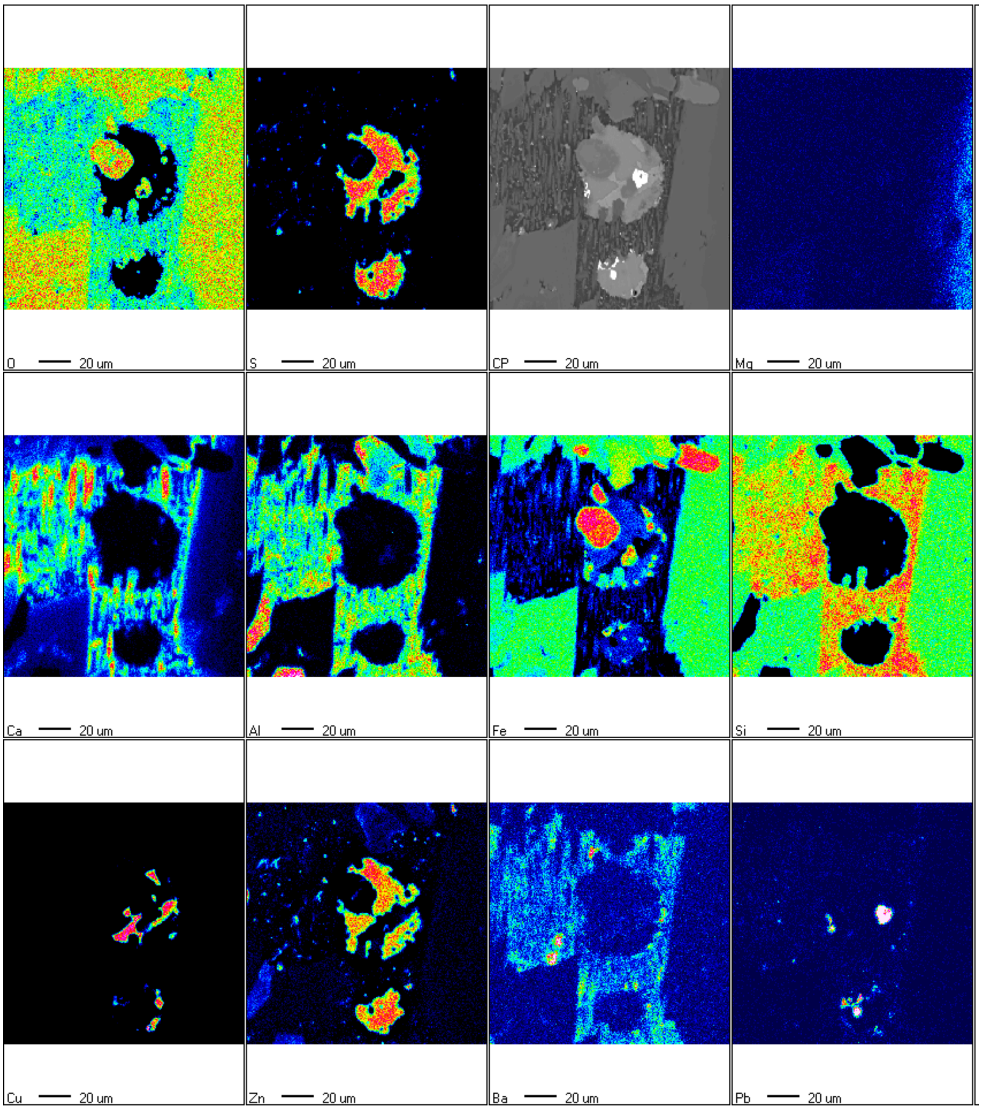


Figure 8 - Elemental mapping of the slag sample from test No. 12 containing 0.47% copper (WDS, x500)

According to the analysis data, the host matrix in the sample is fayalite, which contains iron oxides $FeCl_2$, FeO, Fe_2O_3, ferruginous sphalerite, and an isotropic glass phase in the form of inclusions. The distribution of the elements in the slag sample of test No. 12 is shown in Figure 8.

The presented thermograms (Figure 9) show that during heating, low-temperature effects (≈350–500 °C) are associated with minor structural changes and removal of physically bound components. At higher temperatures (≈870–980 °C), broader thermal effects indicate progressive phase transformations within the silicate system. More pronounced peaks in the range of ≈1090–1130 °C correspond to the onset of melting, while the strong effect near ≈1240–1250 °C is associated with the completion of melting and formation of a liquid phase.

During cooling, a distinct exothermic peak at ≈1067 °C corresponds to crystallisation of the melt.

It is observed that all significant endothermic and exothermic effects occur below 1300 °C. This is further supported by the absence of additional thermal effects above ≈1250 °C and the stabilisation of the thermal curves, indicating that the main phase transformations are completed within this temperature range. Therefore, this temperature range is considered optimal for the slag reduction process.

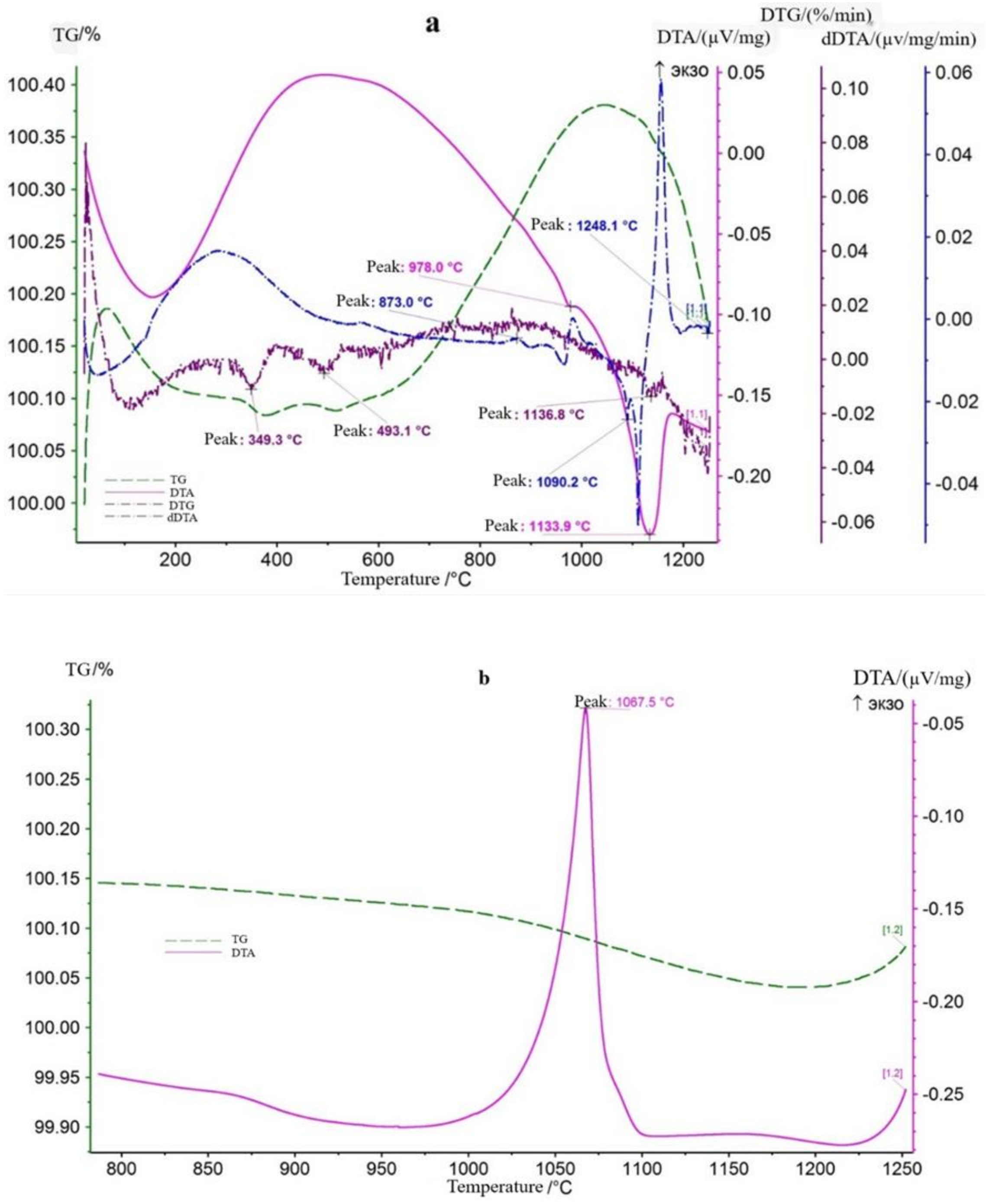


Figure 9 - Thermograms of the slag sample of test No. 12, during heating (a) and during cooling (b)

Conclusions

The study demonstrates that fayalite slags produced from the autogenous smelting of copper concentrates can be effectively depleted in a two-zone Vanyukov-type process when operated at 1300 °C and under strongly reducing conditions ($pO_2 < 10^{-11}$ atm). Slags with 0.93–1.54 wt.% Cu, 30.05–32.30 wt.% SiO_2 and 7.8–9.8 wt.% Fe_3O_4 are suitable feed for depletion; after reduction with activated carbon, magnetite decreases to 2.5–3.3 wt.% and copper to 0.43–0.80 wt.%.

The results revealed that ensuring sufficient heat input and controlling pO_2 are key to achieving deep reduction of metal oxides, lowering slag viscosity via magnetite destruction, and reducing both chemical and mechanical copper losses in industrial practice.

Conflicts of interest. On behalf of all authors, the corresponding author states that there is no conflict of interest.

CRediT author statement: **D. Altybayeva:** Writing draft preparation; **B. Kenzhaliyev**: Conceptualization, Project administration; **S. Kvyatkovskiy**: Supervision, Methodology; **M. Dyussebekova**: Data curation, Reviewing and Editing; **B. Abdikerim**: Visualization, Investigation; **A. Semenova**: Resources, Software, Validation; **A. Gemeal**: Validation, Formal analysis. All authors have read and agreed to the published version of the manuscript.

Funding. The research was funded by the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan (Grant AP26101273).

Cite this article as: Altybayeva D Kh, Kenzhaliyev BK, Kvyatkovskiy SA, Dyussebekova MA, Abdikerim BE, Semenova AS, Gemeal A. Deep Copper Recovery from Autogenous Smelting Slags under Strongly Reducing Conditions. Kompleksnoe Ispolzovanie Mineralnogo Syra = Complex Use of Mineral Resources. 2028; 345(2):16-28. https://doi.org/10.31643/2028/6445.13

Жоғары тотықсыздандыру жағдайында автогенді балқыту қождарынан мысты терең бөліп алу

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Мақала келді: *17 наурыз 2026*
Сараптамадан өтті: *21 сәуір 2026*
Қабылданды: *1 маусым 2026*

ТҮЙІНДЕМЕ

Мыс балқыту өндірісінің қождарын терең тотықсыздандыру олардың құрамындағы түсті металдарды бөліп алудың және әрі қарай қайта өңдеуге жарамды қождар алудың перспективалы тәсілі болып табылады. Бұл жұмыста екі аймақты Ванюков пешінің тотығу және тотықсыздану аймақтарын имитациялайтын жағдайларда мыс концентраттарын автогенді балқыту кезінде түзілетін фаялит-магнетитті қождарды жұтаңдануы зерттелді. Массасы 100 г шихта үлгілері (құрамында 14,63–16,82 % Cu, 25,6–27,6 % Fe, 30 % S және 15 % SiO_2) 1350 °C температурада оттегімен реттелген үрлеу жағдайында балқытылып, құрамында 0,93–1,54 % Cu, 30,05–32,30 % SiO_2 және 7,8–9,8 % Fe_3O_4 бар қождар алынды. Кейінгі тотықсыздандыру 1300 °C температурада магнетитке қатысты стехиометриялық мөлшерден бес есе артық енгізілген активтендірілген көмір қатысында, оттегі құрамды үрлеу шығыны 5 л/сағ және ұстау уақыты 1 сағат жағдайында жүргізілді. Химиялық талдау нәтижелері бойынша қождағы Fe_3O_4 мөлшері 7,8–7,95 %-дан 2,5–2,6 %-ға дейін, ал мыс мөлшері 0,93–1,033 %-дан 0,43–0,50 %-ға дейін төмендегені анықталды (оттегінің парциалдық қысымы ~10^{-12}–10^{-11} атм). Рентгенодифракциялық және SEM зерттеулер фаялит-магнетитті қождардың құрамындағы дисперсті металл мыс пен сульфидтерден темірлі сфалерит, борнит-халькопирит типті мыс минералдары, темір оксидтері және шынытәрізді фазалар бар фаялитті матрицаға өтуін көрсетті. Қосымша термиялық талдау барлық негізгі эндотермиялық және экзотермиялық процестердің 1300 °C дейін аяқталатынын көрсетті, бұл температураның қождарды терең жұтаңдануы үшін оңтайлы екенін дәлелдейді. Алынған нәтижелер қож құрамының, температураның, тотықсыздандырғыш мөлшерінің және оттегінің парциалдық қысымының жұмыс диапазонын анықтайды, бұл жағдайда қождағы мыс шығындарын шамамен 0,5 %-ға дейін төмендетуге болады, бұл өнеркәсіптік тұрғыдан маңызды.

Түйін сөздер: мыс қожын өңдеу, Пирометаллургиялық өңдеу, Құрамында мыс сульфидті материалдар, Тотықсыздандыру балқыту, SEM талдау, Металлургиялық қалдықтар.

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Глубокое извлечение меди из шлаков автогенной плавки в условиях интенсивного восстановления

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Поступила: *17 марта 2026*
Рецензирование: *21 апреля 2026*
Принята в печать: *1 июня 2026*

АННОТАЦИЯ

Глубокое восстановление шлаков медеплавильного производства является перспективным способом извлечения содержащихся в них цветных металлов и получения шлаков, пригодных для дальнейшей переработки. В данной работе исследуется обеднение фаялит-магнетитовых шлаков, образующихся при автогенной плавке медных концентратов в условиях, имитирующих зоны окисления и восстановления двухзонной печи Ванюкова. Пробы шихты массой 100 г, содержащие 14,63–16,82% Cu, 25,6–27,6 % Fe, 30 % S и 15 % SiO_2, плавили при 1350 °C с контролируемой продувкой кислородом для получения шлаков, содержащих 0,93–1,54 % Cu, 30,05–32,30 % SiO_2 и 7,8–9,8 % Fe_3O_4. Последующее восстановление при 1300 °C проводилось с использованием активированного угля в пятикратном стехиометрическом избытке по отношению к магнетиту при потоке кислород содержащего дутья 5 л/ч и времени выдержки 1 ч. Химический анализ показал, что содержание Fe_3O_4 в шлаке снизилось с 7,8–7,95 % до 2,5–2,6 %, а содержание меди в шлаке с 0,93–1,033 % снизилось до 0,43–0,50 % при парциальном давлении кислорода $\sim10^{-12}$–10^{-11} атм. Рентгенодифракционные и SEM исследования выявляют переход от фаялит-магнетитовых шлаков с диспергированной металлической медью и сульфидами к фаялитсодержащим матрицам, содержащим железистый сфалерит, медные минералы борнит-халькопиритового типа, оксиды железа и стеклообразные фазы. Одновременный термический анализ показывает, что все основные эндотермические и экзотермические процессы завершаются при температуре до 1300 °C, что подтверждает оптимальность этой температуры для глубокого обеднения шлаков. Результаты определяют рабочий диапазон — состав шлака, температуру, дозировку восстановителя и pO_2 — при котором потери меди в шлаке могут быть снижены примерно до 0,5 % шлаках, имеющих промышленное значение.

Ключевые слова: переработка медного шлака, пирометаллургическая обработка, медьсодержащие сульфидные материалы, восстановительная плавка, SEM-анализ, металлургические отходы.

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