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Research article
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Thermodynamic premises of fire refining of blister copper considering the interaction parameters of the melt

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Abstract: The process of fire refining of copper is based on the removal of impurities that have a high affinity for oxygen through their oxidation by gaseous oxygen. Since the main component of blister copper is copper itself, according to the law of mass action and its affinity for oxygen, during air blowing the metal primarily reacts with the oxygen in the blast. The resulting copper (I) oxide is transported from the zone of direct contact with gaseous oxygen into the region of lower oxygen concentration, where the oxidation of impurities (Me_i) occurs. In practice, the actual copper melt deviates from ideal behavior; therefore, it is necessary to consider the activities of the components and the interaction parameters of the system when evaluating the thermodynamic premises of fire refining. It is known that the oxygen activity in copper melts depends on the oxygen affinity of the impurities. Impurities with a high affinity for oxygen (e.g., Al, Si, Mn) significantly reduce the oxygen activity, whereas those with a lower affinity (e.g., Zn, Fe, Sn, Co, Pb) only partially decrease it. Thermodynamic calculations were performed to estimate the final concentration of impurities in the copper melt and to theoretically evaluate the influence of impurities on oxygen activity in blister and anode copper. The calculations showed that fire refining of copper by air blowing under a weighted ideal slag has thermodynamic limitations. The final impurity concentration depends on both the oxygen activity in the melt and the activity of the impurity oxide in the slag. A decrease in the impurity oxide activity in the slag enhances refining efficiency by shifting the oxidation reaction equilibrium toward the reaction products. The theoretical effect of impurities on the oxygen activity in copper is substantiated for two melts differing in chemical compositin.

Keywords: oxidation, blister copper, anode copper, oxygen activity, activity coefficient, weighted ideal slag.

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Термодинамические предпосылки огневого рафинирования черновой меди с учетом параметров взаимодействия расплава

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Аннотация: В основе огневого рафинирования меди лежит процесс удаления примесей, обладающих повышенным сродством к кислороду, за счет их окисления кислородом газовой фазы. Поскольку основным компонентом черновой меди является медь, то, согласно закону действующих масс и сродства к кислороду, при продувке расплава воздухом она преимущественно вступает во взаимодействие с кислородом дутья. Образовавшийся оксид меди (I) в результате перемешивания потоками воздуха перемещается из зоны непосредственного контакта с газообразным кислородом в зону низких концентраций кислорода, в которой осуществляется протекание реакции окисления примесей (Me_i) [1]. На практике реальный расплав меди отличается от идеального, поэтому для оценки термодинамических предпосылок огневого рафинирования меди целесообразно учитывать активности компонентов и параметры взаимодействия системы. Известно, что активность кислорода в медных расплавах зависит от сродства примесей к кислороду. Примеси, обладающие высоким сродством к кислороду (например, Al, Si, Mn), достаточно хорошо снижают активность кислорода. Примеси, обладающие меньшим сродством к кислороду (например, Zn, Fe, Sn, Co, Pb), частично снижают его активность. Для оценки термодинамической возможности окисления примесей (Me_i) в расплаве меди, с учетом параметров взаимодействия расплава, проведены расчеты конечной концентрации примесей в расплаве меди и теоретическая оценка влияния примесей на активность кислорода в расплаве черновой и анодной меди. Расчеты показали, что возможность огневого рафинирования меди путем продувки расплава воздухом под средневзвешенным идеальным шлаком имеет термодинамические ограничения, при этом конечная концентрация примеси зависит от активности кислорода в расплаве и от активности оксида примеси в шлаке. С уменьшением активности оксида примеси в шлаке улучшается рафинирование за счет сдвига равновесия реакции окисления примеси в сторону продуктов взаимодействия. Теоретически обосновано влияние примесей на активность кислорода в меди для двух различных по химическому составу расплавов.

Ключевые слова: окисление, черновая медь, анодная медь, активность кислорода, коэффициент активности, средневзвешенный идеальный шлак.

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Introduction

The fire refining technology of copper is based on the partial oxidation of the melt with free oxygen from the gas phase and the formation of oxides of impurities (Me_xO_y) that are only partially soluble in liquid copper. These oxides, due to their lower density, form a slag layer on the surface of the melt. The oxidation reactions of copper and impurities occur in a system consisting of a liquid phase of impurity solution, condensed oxide phases, and the gas phase of volatile compounds. The oxygen content during the oxidation stage is between 0.4—0.8 %, which ensures the transfer of copper (I) oxide (Cu_2O) into the liquid copper and facilitates the oxidation of impurities (Me_i). The actual copper melt deviates from the ideal, and thus it is appropriate to consider the thermodynamic aspects of the process, including the activities of the components and the system's interaction parameters, when evaluating the potential technological possibilities of fire refining.

Problem statement

It is known that fire refining of copper is carried out by blowing the melt with air at temperatures ranging from 1150 to 1170 °C. According to the law of mass action, due to the high concentration of copper in the melt, the oxidation reaction of copper predominates:

$$4[Cu] + \{O_2\} = 2(Cu_2O). \quad (1)$$

The formed copper (I) oxide moves from the zone of direct contact with gaseous oxygen into the depth of the melt, where the oxygen concentration is low, and the direction of reaction (1) changes to the opposite, with Cu_2O dissolving in the copper melt, thereby increasing the oxygen concentration:

$$(Cu_2O) = 2[Cu] + [O], \quad (2)$$

This facilitates the direct oxidation of impurities with dissolved oxygen:

$$[Me] + [O] = (MeO). \quad (3)$$

Additionally, Cu_2O acts as a condensed oxidant for impurities according to the reaction:

$$[Me] + (Cu_2O) = (MeO) + 2[Cu]. \quad (4)$$

According to the Cu—Cu_2O phase diagram, the solubility of Cu_2O in copper increases with temperature. At temperatures above 1200 °C, copper (I) oxide transitions into the slag, while copper (II) oxide (CuO) does not form due to the dissociation pressure exceeding the partial oxygen pressure in the air.

Thus, the primary condition for fire refining of copper is maintaining the oxygen concentration in copper higher than the equilibrium concentration for impurity oxidation reactions [2—18].

It is known that the potential for oxidation and removal of impurities under primary slag is determined by the Gibbs free energy of the oxidation reactions [1]. The reduction of the Gibbs free energy for the main oxidation reactions of impurities indicates the degree to which their affinity for oxygen changes, their ability to oxidize, and their removal from the melt.

To prevent over-oxidation of copper and its conversion into copper (I) oxide in the slag, it is necessary to limit the oxygen saturation of the copper melt to 12 % Cu_2O.

Assuming that the metallic and oxide phases in reaction (3) are in equilibrium, the following expression holds for each impurity present in these phases:

$$x[Me] + y[O] = (Me_xO_y). \quad (5)$$

Considering the deviation of the properties of the real copper solution from the ideal, the equilibrium constant for reaction (5) is determined by the formula:

$$K_{Me} = \frac{a_{(Me_xO_y)}}{a^x_{[Me]}\, a^y_{[O]}} = \frac{N_{(Me_xO_y)}\gamma_{(Me_xO_y)}}{[Me]^x [O]^y \gamma^x_{Me}\, \gamma^y_O}, \quad (6)$$

where $N_{(Me_xO_y)}$ is the molar fraction of the impurity oxide in the slag; $[Me]^x$ and $[O]^y$ represent the molar fractions of the impurity and oxygen in the melt, respectively; $\gamma_{(Me_xO_y)}$ is the activity coefficient for the impurity in the slag; γ^x_{Me} and γ^y_O are the activity coefficients for the impurity and oxygen in the melt.

Based on the reduction of the Gibbs free energy for the main oxidation reactions of the impurities and its relationship with the equilibrium constant

$$\Delta G^0 = -RT \ln K_{Me} \quad (7)$$

the final concentration of the impurity in the copper melt (mole fraction) can be estimated by converting equation (6) as follows [3]:

$$[Me]^x = \frac{N_{(Me_xO_y)}\gamma_{(Me_xO_y)}}{K_{Me}[O]^y \gamma^x_{Me}\, \gamma^y_O}. \quad (8)$$

It is known that the oxygen activity in copper melts depends on the impurity's affinity for oxygen. Impurities with a high affinity for oxygen (e.g., Al, Si, Mn) significantly reduce the oxygen activity, while impurities with a lower affinity (e.g., Zn, Fe, Sn, Co, Pb) only partially reduce it.

Copper melt with low impurity concentrations is considered diluted. In this case, a 1 % ideal diluted solution of the *i*-th impurity in copper is assumed to be the standard state. The oxygen activity in the copper melt with *n* impurities is described by the following expression:

$$\lg(a_{[O]}) = \lg[\%\,O] + \sum_{j=1}^{n} e^j_{[O]}[\%\,j], \quad (9)$$

where $e^j_{[O]}$ is the interaction parameter between oxygen and the *j*-th impurity of the first order.

The objective of this work is to evaluate the thermodynamic feasibility of oxidizing impurities (Me_i) in copper melt, considering the interaction parameters of the melt, and to theoretically assess the impact of impurities on the oxygen activity in the copper melt.

Methodology

To evaluate the thermodynamic feasibility of oxidizing impurities (Me_i) in copper melt, considering the interaction parameters of the melt, calculations were

performed to estimate the final impurity concentration in copper at a temperature of 1150 °C, characteristic for the oxidation stage of refining. The following steps were taken:

The real weighted composition of blister copper was used as input data (wt. %):

Cu	98.86	S	0.16
Ag	0.076	Pb	0.0658
Sb	0.147	Zn	0.041
As	0.128	Sn	0.058
Ni	0.2	Fe	0.047
Bi	0.0012		

It is known from the practice of fire refining that the oxygen concentration in the melt is maintained at an elevated level due to continuous blowing of air into the melt, which fluctuates between 0.4 and 0.8 wt. %.

At the initial stage of oxidation, a substantial amount of oxygen is introduced into the melt, so an average oxygen concentration of 0.6 wt. % was assumed.

The oxidation process of impurities is determined by their affinity for oxygen; therefore, the composition of the slag phase changes during smelting proportionally to the oxidation rate of the *i*-th impurity. Accordingly, to evaluate the thermodynamic feasibility of impurity oxidation, a weighted ideal slag composition was selected, wt. %: ~70 Cu_2O, 29 SiO_2, ~1 Me_xO_y, under the assumption that the activity of the impurity oxide in the slag equals its molar fraction:

$$a_{Me_xO_y} = N_{Me_xO_y}. \quad (10)$$

The equilibrium constant and Gibbs free energy for the oxidation reaction of the *i*-th impurity (5) were calculated using the thermodynamic software package HSC Chemistry 9.

To determine the final impurity concentration from equation (8), the molar fraction of the *i*-th impurity oxide was calculated based on the condition of the weighted ideal slag composition.

Since the activity coefficient of the *i*-th impurity is affected by all components of the melt, the activity coefficients of the impurity and oxygen in the melt were calculated using the following formulas [19—25]:

$$\ln\gamma_{[Me]} = \ln\gamma^{\infty}_{[Me]} + N_{[Me]}\varepsilon^{Me}_{[Me]} + \sum_{j=1}^{n} N_{[j]}\varepsilon^{j}_{[Me]}, \quad (11)$$

$$\ln\gamma_{[O]} = \ln\gamma^{\infty}_{[O]} + N_{[O]}\varepsilon^{O}_{[O]} + \sum_{j=1}^{n} N_{[O]}\varepsilon^{j}_{[O]}, \quad (12)$$

where $\gamma^{\infty}_{[Me]}$, $\gamma^{\infty}_{[O]}$ are the activity coefficients of the impurity and oxygen in an infinitely dilute copper solution; $\varepsilon^{Me}_{[Me]}$, $\varepsilon^{j}_{[Me]}$, $\varepsilon^{O}_{[O]}$, $\varepsilon^{j}_{[O]}$ are the interaction parameters between identical and dissimilar components of the melt.

The interaction parameters of the *i*-th impurity and oxygen ($\varepsilon^{O}_{[Me]}$ and $\varepsilon^{Me}_{[O]}$) in the temperature range 1100—1200 °C for the Cu—Me_i—O system are given below [1]:

Ag	−0.2	Pb	−6.15
Sb	−1.3962	Zn	−2.52
As	3.3	Sn	−0.35
Ni	−5.9625	Fe	−239.92
Bi	−2.408	O	−7.7
S	−0.081		

At t = 1150 °C, the maximum oxygen solubility in liquid copper is 3.01 at. % (0.79 wt. %):

$$\lg[\%O]_{max} = -\frac{9260}{t} + 7.15. \quad (13)$$

To calculate the final impurity concentration, the chemical compositions of blister copper (see data above) and the ideal slag were expressed in molar fractions. The activity coefficients of the components were calculated using equations (11) and (12). Based on these assumptions, and using equation (8), the following final impurity concentrations were obtained (converted to wt. %):

Sb	0.08	Pb	0.09
As	0.27	Zn	0.0009
Ni	0.11	Sn	0.0087
Bi	0.0043	Fe	0.0068

The calculated results were compared with practical data for anode copper, which has the following chemical composition (wt. %):

Sb	0.14	Pb	0.13
As	0.11	Zn	0.003
Ni	0.17	Sn	0.0052
Bi	0.0011	Fe	0.0012

To theoretically evaluate the influence of impurities on the oxygen activity in the melt, data on the chemical composition of blister and anode copper, as well as the assumptions used in the thermodynamic assessment of impurity oxidation (Me_i), were applied. The evaluation of impurity influence was carried out in the following sequence:

The molar interaction parameters (see above) were recalculated to mass-based parameters using the equation:

$$e_i^j = \frac{1}{100}\frac{1}{\ln 10}\left(\frac{A_{r\mathrm{Me}}}{A_{ri}}\varepsilon_i^j + \frac{A_{ri} - A_{r\mathrm{Me}}}{A_{ri}}\right), \qquad (14)$$

where $A_{r\mathrm{Me}}$ is the atomic mass of copper (g/mol); A_{ri} is the atomic mass of the i-th impurity (g/mol); ε_j^i is the molar interaction parameter.

Mass-based interaction parameters in the Cu—Me_i—O system:

Ag 0,00125	S..... −0,226
Sb..... 0,000083	Pb..... −0,00544
As..... 0,0129	Zn ..... −0,01
Ni..... −0,0257	Sn..... −0,001162
Bi 0,000075	Fe..... −0,58

An average oxygen concentration in the melt of 0.6 wt. % was assumed.

Using expression (9), the oxygen activity in the copper melt was calculated. For blister copper, the oxygen activity was $a_{[O]}$ = 0.515 %, and for anode copper — $a_{[O]}$ = 0.59 %.

Results

The calculation results, ordered by the impurity values, show good convergence with the practice of fire refining of copper. The residual concentration of impurities in copper decreases with an increase in their affinity for oxygen, a reduction in the impurity oxide activity in the slag, and an increase in the oxygen activity in the copper melt. The calculations of the residual concentration of impurities such as As, Sb, Pb, and Ni showed values that differed from the practical data of anode copper. This discrepancy is explained by the following: first, the oxidation of copper during refining does not reach the saturation limits, so the copper oxide activity in the slag is not equal to 1; second, the activity of impurities in the slag is much lower than that of the pure impurity in the melt; finally, impurity solutions in metals are not ideal. Overall, the calculations indicate that the maximum impurity content in copper melt depends on the refining temperature, the oxygen saturation of the melt, and the slag phase composition.

The calculation of oxygen activity in copper for two melts with different chemical compositions revealed a significant difference in the extent to which impurity concentrations affect it. Overall, impurities reduced the oxygen activity in the blister copper melt by 14.2 %. This indicates that, at such impurity concentrations, they do not have a major impact on the physicochemical properties of the melt; however, they do considerably decrease the activity of dissolved oxygen. In anode copper, the reduction in oxygen activity was 1.6 %.

Analysis of the chemical compositions of blister and anode copper presented above shows that impurities with a higher affinity for oxygen exert a greater influence on oxygen activity. Theoretical calculations of the effect of impurities on oxygen concentration in copper melt should be taken into account at the stage of impurity oxidation, particularly at its initial stage, in order to optimize the air-blowing rate.

Conclusion

Considering the activity coefficients of impurities and the selected weighted ideal slag composition, the thermodynamic possibilities of oxidizing impurities (Me_i) in copper melt were assessed by calculating the final impurity concentrations at a temperature of 1150°C, characteristic for the oxidation stage of refining. It was shown that fire refining of copper by blowing the melt with air under a weighted ideal slag has thermodynamic limitations, with the final impurity concentration depending on the oxygen activity in the melt and the impurity oxide activity in the slag. As the impurity oxide activity in the slag decreases, the refining process improves due to the shift in the equilibrium of the oxidation reaction towards the interaction products.

The theoretical assessment of the impact of impurities on the oxygen activity in copper for two melts with different chemical compositions was also justified.

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