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Research article
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Influence of high-temperature oxidation kinetics of copper on the electrical properties of the melt

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Abstract: The kinetics of high-temperature oxidation of copper of various chemical compositions by gaseous oxygen follows a parabolic rate law within the temperature range of 350–1050 °C. For specialists engaged in the theory and practice of fire refining, of particular interest is the kinetics of copper oxidation over a broader temperature interval of 350–1160 °C. Within this range, the processes include oxidation of solid copper and its melting, oxidation of liquid copper by oxygen introduced into the melt, oxygen solubility in copper, and slag formation. The duration of high-temperature interaction between copper and oxygen has a considerable effect on both the technical and economic indicators of anode smelting and the electrical properties of copper. Therefore, investigating the kinetics of high-temperature oxidation of copper and its influence on the electrical properties of the metal is essential for the optimal organization of fire refining. In the temperature range of 1100–1200 °C, copper oxidation occurs predominantly due to oxygen introduced into the melt with air. The copper(I) oxide formed migrates from the zone of direct contact with gaseous oxygen into the depth of the melt with lower oxygen concentration, where it dissociates into copper and oxygen, thus increasing oxygen concentration in the melt. Overoxidation of copper and excessive saturation of the anode metal with gases lead to its transfer into slag in the form of oxides, to excessive consumption of resources and refractory materials, and to a deterioration of the electrical properties of the metal. To identify optimal oxidation modes and to assess the influence of copper oxidation kinetics on the electrical properties of the melt, a comparative analysis was conducted of the kinetic patterns of oxidation of solid and liquid copper of various chemical compositions under identical experimental conditions, using differential thermogravimetric analysis (DTA) and by surface blowing of the copper melt with an air mixture. The results show that copper samples oxidize at nearly the same rate and that the presence of impurities does not affect the process. All oxygen intended for copper (I) oxide formation dissolves in copper up to the thermodynamic limit (up to 12 % Cu_2O). It was established that oxygen concentrations in the melt above 0.06% adversely affect the electrical properties of copper.

Keywords: kinetics of high-temperature oxidation, blister copper, anode copper, fire refining of copper, oxygen concentration, electrical resistivity of the melt.

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Влияние кинетики высокотемпературного окисления меди на электротехнические свойства расплава

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Аннотация: Кинетика высокотемпературного окисления меди различного химического состава кислородом газовой фазы протекает по параболическому закону окисления в температурном интервале 350–1050 °С. Для специалистов, занимающихся теорий и практикой огневого рафинирования, интересна кинетика высокотемпературного окисления меди в более широком температурном диапазоне 350–1160 °С, в пределах которого осуществляются окисление твердой меди и ее плавление, окисление жидкой меди кислородом воздуха, вводимого в расплав, растворимость в ней кислорода, процесс шлакообразования. Продолжительность высокотемпературного взаимодействия меди с кислородом оказывает большое влияние на технико-экономические показатели анодной плавки и электротехнические свойства меди. Поэтому исследование кинетики высокотемпературного окисления меди и влияния ее на электротехнические свойства металла имеет принципиальное значение для оптимальной организации огневого рафинирования. В температурном интервале 1100–1200 °С окисление меди осуществляется преимущественно за счет кислорода воздуха, вводимого в расплав. Образовавшийся оксид меди (I) перемещается из зоны непосредственного контакта с газообразным кислородом в глубину расплава с низкой концентрацией кислорода и диссоциирует на медь и кислород, что способствует повышению концентрации кислорода в расплаве. Переокисление меди и чрезмерное насыщение анодного металла газами приводят к переводу ее в шлак в виде оксидов, перерасходу материальных ресурсов и огнеупорных материалов и оказывают отрицательное влияние на электротехнические свойства металла. Для поиска оптимальных режимов окисления и оценки влияния кинетики окисления меди на электротехнические свойства расплава проведен сопоставимый анализ кинетических закономерностей окисления твердой и жидкой меди различного химического состава, полученной в одинаковых условиях эксперимента, методом дифференциально-термогравиметрического анализа (ДТА) и путем поверхностного обдува воздушной смесью медного расплава. Проведенные исследования показали, что образцы меди окисляются практически с одинаковой скоростью и наличие примесей не влияет на этот процесс. Весь кислород, предназначенный для образования оксида меди (I), растворяется в нем до предела термодинамического ограничения (до 12 % Cu_2O). Установлено, что наличие кислорода в расплаве больше 0,06 % отрицательно влияет на электротехнические свойства меди.

Ключевые слова: кинетика высокотемпературного окисления, черновая медь, анодная медь, огневое рафинирование меди, концентрация кислорода, электросопротивление расплава.

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Introduction

The kinetics of high-temperature oxidation of copper by gaseous oxygen was the subject of extensive theoretical and practical research in the second half of the 20th century. Domestic and international studies examined in detail the mechanisms of oxidation of copper of various chemical compositions. A parabolic rate law for oxidation was established in the temperature interval of 350—1050 °C, conventionally divided into three ranges: low-temperature (350—550 °C), intermediate (550—850 °C), and high-temperature (850—1050 °C) [1]. For specialists engaged in the theory and practice of fire refining, the oxidation kinetics of copper in the wider temperature interval of 350—1170 °C are of particular interest. Within this range, the fol-

lowing processes occur: oxidation of solid copper (in a furnace oxidizing atmosphere with an oxidizer excess coefficient $\alpha = 1.1 \div 1.15$), melting of copper, oxidation of liquid copper by oxygen from the air introduced into the melt, dissolution of oxygen in the melt, and slag formation.

The duration of high-temperature interaction between copper and oxygen has a considerable effect on the technical and economic indicators of anode smelting and on the electrical properties of the metal. Therefore, investigation of the kinetics of this process and its effect on the electrical properties of copper is of fundamental importance for the optimal organization of fire refining.

Studies [1; 2] have shown that impurities in copper exert different effects on the oxidation rate. These effects are present in the low- and intermediate-temperature ranges, while in the high-temperature range the oxidation rates of copper of different purities are practically identical [1]. In general, the kinetic behavior of the process depends on the formation of a copper (I) oxide film on the surface of solid copper. In the high-temperature interval (600—1050 °C), it is mainly governed by the growth of the copper(I) oxide phase, limited by the oxygen partial pressure (P_{O_2}).

At 1100—1200 °C, copper oxidation proceeds predominantly by oxygen from the air introduced into the melt:

$$4Cu + O_2 = 2Cu_2O.$$

The Cu_2O formed migrates from the zone of direct contact with gaseous oxygen into the deeper layers of the melt, where the oxygen concentration is lower. The direction of reaction (1) then reverses: Cu_2O dissolves in the copper melt, thereby increasing the concentration of O_2 in the melt. In this case, the kinetic patterns of high-temperature oxidation of copper are limited by the transfer of copper into slag when the melt is saturated with oxygen (1.4 %) up to 12.4 % Cu_2O.

Overoxidation of copper and excessive oxygen saturation of the melt result in transfer of the metal into slag in the form of oxides, leading to excessive consumption of materials and refractories and to deterioration of the electrical properties of the metal.

The aim of the present study was to carry out a comparative analysis of the kinetic patterns of oxidation of solid and liquid copper of different chemical compositions, obtained under identical experimental conditions and by surface blowing of the copper melt with an air mixture, as well as to evaluate the degree of oxygen solubility in liquid copper and its influence on the electrical properties of the melt, particularly at oxygen concentrations above 0.08 wt. %.

Methods

The comparison of the kinetic patterns of copper oxidation was carried out using simultaneous thermogravimetric and differential scanning calorimetry (TG—DSC) on a NETZSCH STA 449 F3 thermal analyzer (Germany), coupled with a Tensor 27 Fourier-transform infrared (FTIR) spectrometer (Bruker, USA). The instrument simultaneously recorded changes in sample mass (Δm_i) and heat release/absorption (DSC) during continuous heating at a rate of 10 °C/min up to 1150 °C.

The objects of study were solid copper samples: 49.5 mg of pure copper (99.98 % Cu) and 48.47 mg of blister copper (98.7 % Cu). The samples were placed in a crucible with an inner diameter of 6 mm and a height of 4 mm. During the tests, the working space above the crucible surface was blown with air at a flow rate of 250 mL/min. In the experiment with pure copper, the change in sample mass (Δm_i) was interpreted as the oxygen mass $m_{[O]}$ incorporated into copper oxides of the Cu—O system. For blister copper, Δm_i was taken as the integral amount of oxygen incorporated into oxides of the Cu—Mei—O system.

To assess the degree of oxygen solubility and evaluate its effect on the electrical properties of copper, surface oxidation of blister and pure (anode) copper melt samples was performed using an air mixture. The experiments were carried out under laboratory conditions with the following equipment:

— an electric furnace with silicon carbide heaters and a temperature control unit, operating at approximately 1170 °C;

— a crucible with batch loading of blister and anode copper chips, with a melt surface area S of approximately 7539 mm^2;

— a laboratory compressor with a capacity of 7 m^3/h and an air-blowing rate of 1.75—2.45 m^3/h.

Three samples of blister copper and three samples of anode copper of different chemical compositions, each weighing approximately 5000 g, were selected for study. All samples were melted under a charcoal layer. After the copper melted, slag was removed from the crucible surface, and oxidation was carried out by blowing with an air mixture or approximately 12—18 min.

The degree of oxygen solubility was determined by periodic sampling (one sample per measurement), fol-

lowed by analysis on an ELTRA CS-2000 (Germany) carbon/sulfur analyzer, a SPECTROLAB S (Germany) optical emission spectrometer for copper composition, and an ELTRA ON-900 (Germany) oxygen/nitrogen analyzer. The effect of copper oxidation kinetics on its electrical properties was studied by measuring the electrical resistivity of the anode copper melt [3].

Results and discussion

The TG—DSC curves obtained during 120 min of heating pure and blister copper samples to 1150 °C were plotted on the same coordinate system for clarity (Fig. 1). The numerical values of Δm_i, τ, and t_i at the most characteristic points are shown separately in the diagram field [4].

The kinetic dependences of sample mass change on oxidation time in the temperature interval of 300—1050 °C exhibit a complex rate law close to parabolic, consistent with most previous studies (Fig. 1, curve 2*a* — pure copper, curve 2*b* — blister copper) [5].

The complex oxidation behavior can be explained by thermodynamically probable reactions in the Cu—O system: oxidation of copper to Cu_2O by atomic and molecular oxygen, and recombination reduction of CuO to Cu_2O.

At temperatures of approximately 650 °C and above, bulk diffusion of copper cations within the Cu_2O crystal lattice begins to dominate over grain-boundary diffusion. As a result of grain growth, both the size and the mass fraction of Cu_2O in the oxide layer increase, ensuring complete oxidation of copper to Cu_2O [6—15]. The oxygen diffusion rate also rises, which is reflected in the increase in sample mass and confirms the parabolic rate law (Fig. 1).

A comparison of the Cu_2O masses formed during oxidation of pure and blister copper shows close agreement (3.55 and 3.01 mg, respectively). This indicates that the samples oxidize at nearly the same rate and that the presence of impurities has little effect on the process.

The oxygen required for Cu_2O formation during oxidation of pure and blister copper was calculated based on the mass change. It was established that, to obtain 3.55 mg of Cu_2O in the pure-copper experiment, 0.267 mL of oxygen is required, whereas in the blister-copper experiment 0.234 mL is required; this corresponds to 0.00355 at. % ($8.8 \cdot 10^{-4}$ wt. %) and 0.00301 at.% ($7.5 \cdot 10^{-4}$ wt. %), respectively.

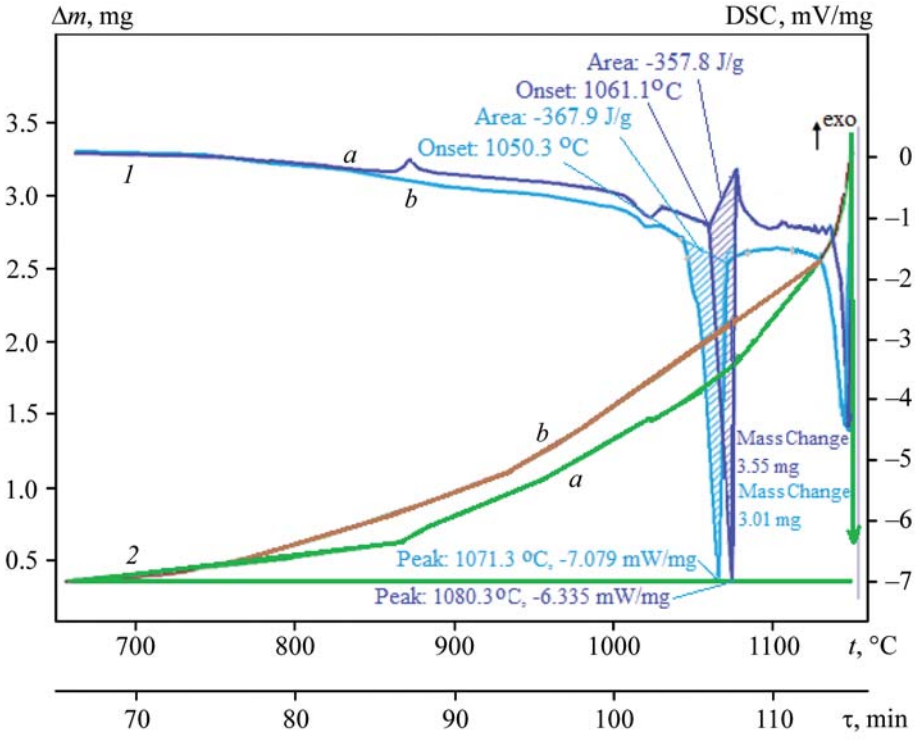

Fig. 1. TG—DSC curves of copper oxidation: pure copper (***a***), blister copper (***b***)
1 – DSC signal, ***2*** – sample mass change, mg

Рис. 1. Кривые ДТА окисления чистой (***a***) и черновой (***b***) меди
1 – ДСК, ***2*** – изменение массы навески, мг

The maximum solubility of oxygen in copper (at. %) at 750—1030 °C is determined according to the following equation [11; 12]

$$\lg[\%O]_{max} = -\frac{6600}{t} + 3{,}41.$$

At 1030 °C, the solubility limit of oxygen in solid copper is 0.02215 at. % (0.0055 wt. %). This indicates that at the given temperature all oxygen intended for copper (I) oxide formation dissolves in the metal, and a continuous oxide layer does not form at the Cu—Cu_2O interface.

During fire refining in the temperature range of 1100—1200 °C, copper is oxidized primarily by atomic oxygen from the air introduced into the melt [16]. The maximum solubility of oxygen in copper in this interval is determined as follows:

$$\lg[\%O]_{max} = -\frac{9260}{t} + 7{,}15.$$

At 1170 °C, the solubility limit of oxygen in liquid copper is 5.4 at. % (1.41 wt. %).

To assess the degree of oxygen solubility and to evaluate its effect on the electrical properties of blister copper, three samples with an average chemical composition were used (Table 1).

The kinetic oxidation curves of blister copper samples with different chemical compositions at 1170 °C are shown in Fig. 2.

Analysis of the empirical curves (Fig. 2) shows that blister copper samples of different chemical compositions exhibit complex oxidation behavior. At the initial stage (up to 6 min), deviations from linear oxidation kinetics are observed. This is explained by the fact that most of the oxygen is consumed in oxidizing impurities (Zn, Fe, Ni, S) and transferring them into the slag and gas phase. The deviation from linearity is most pronounced for curve *2* (Fig. 2). After 3 min of blowing, the oxygen concentration decreases from 0.06 to 0.047 % before starting to rise again. This behavior is attributed to the relatively high sulfur content in this blister copper sample (0.23 %) compared with the other

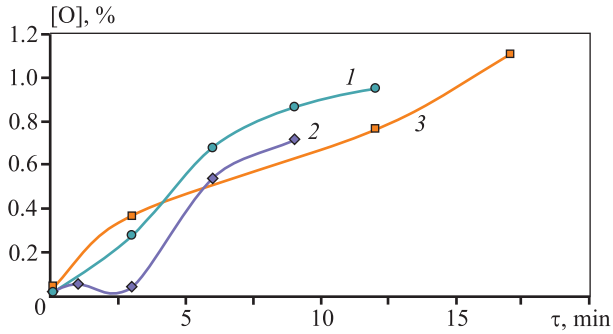

Fig. 2. Empirical dependence of oxygen concentration in copper melt on oxidation time

1–3 – blister copper samples of different chemical compositions (see Table 1)

Рис. 2. Эмпирическая зависимость концентрации кислорода в расплаве меди от продолжительности окисления

1–3 – образцы черновой меди различного химического состава (см. табл. 1)

samples (0.019 and 0.083 %). Since the experiments were conducted under isothermal conditions, 1 min of air blowing was sufficient to saturate the melt with oxygen (from 0.02 to 0.06 %) and to oxidize Zn and Fe. However, this time was insufficient for complete degassing of the copper melt as SO_2, so sulfur largely remained in the melt as dissolved SO_2 bubbles. Prolonged air blowing subsequently removed SO_2 from the melt, while simultaneously reducing the oxygen concentration from 0.06 to 0.047 % [17—22].

After oxidation of the impurities, slag removal, and further oxidation of the melt (from the 6th minute onward), the oxidation of copper follows an almost linear course, with the oxygen concentration in the melt increasing at an average rate of Δ[O] = 0.07 %/min. This is due to the fact that, at the given temperature, all oxygen dissolves in copper, forming Cu_2O and acting as a reactive impurity that promotes overoxidation of the melt.

To assess the degree of oxygen solubility and to evaluate its effect on the electrical properties of anode copper, three samples with an average chemical composition were used (Table 2). The kinetic oxidation curves of ano-

Table 1. **Average chemical composition of blister copper samples**

Таблица 1. Средневзвешенный состав образцов черновой меди

Sample No.	Content, %								
	Pb	Sn	As	Sb	Zn	Fe	Ni	O	S
1	0.03	0.001	0.048	0.076	0.0027	0.0017	0.115	0.28	0.083
2	0.041	0.001	0.045	0.077	0.0055	0.037	0.015	0.02	0.23
3	0.13	0.004	0.156	0.24	0.0039	0.0059	0.4	0.047	0.019

de copper of different chemical compositions at 1170 °C are shown in Fig. 3. Analysis of these data shows a linear increase in the oxygen concentration in the melt, with an average rate of Δ[O] = 0.05 %/min. This trend can be attributed to the fact that molten anode copper contains almost no impurities, allowing its atomic structure to undergo continuous short-range ordering and disordering. This dynamic process creates fluctuating free volume (up to 6 %) and alters interatomic distances by 1—2 %. Because the atomic radius of oxygen (r_O = = $48 \cdot 10^{-12}$ m) is smaller than that of copper (r_{Cu} = = $145 \cdot 10^{-12}$ m), oxygen atoms are generally accommodated uniformly in interstitial positions, saturating the copper with oxygen up to its solubility limit.

The influence of oxygen on the electrical properties of copper in the solid state (binary Cu—O system) has been extensively studied in the literature. Experimental data show that the dependence of copper resistivity on oxygen concentration is nonlinear [23]. To obtain a quantitative description of this dependence, the published data were digitized with WebPlotDigitizer. The results are shown in Fig. 4. The graph indicates that low oxygen concentrations, up to 0.06 %, enhance the electrical conductivity of copper [23]. This effect is explained by the fact that oxygen in copper may exist not only as copper (I) oxide, but also in solid solution, where it interacts with detrimental metallic impurities (e.g., nickel), oxidizing them into less harmful forms [24—26].

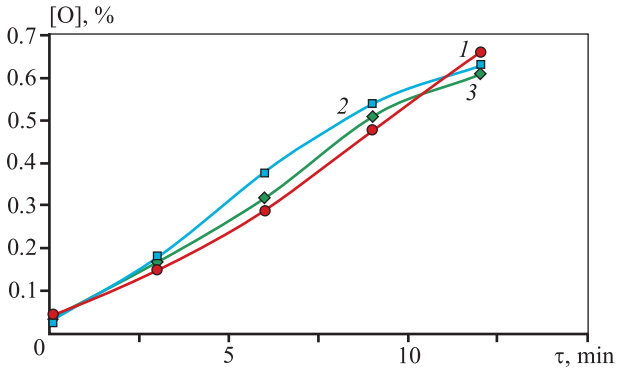

Fig. 3. Empirical dependence of oxygen concentration in copper melt on oxidation time

1–3 – anode copper samples of different chemical compositions (see Table 2)

Рис. 3. Эмпирическая зависимость концентрации кислорода в расплаве меди от продолжительности окисления

1–3 – образцы анодной меди различного химического состава (см. табл. 2)

In the oxygen concentration range above 0.06 %, copper resistivity increases linearly (Fig. 4), with oxygen acting as a detrimental impurity that reduces electrical conductivity.

By contrast, the effect of oxygen on the electrical conductivity of liquid copper of different chemical compositions has been studied only to a limited extent, and the available information is fragmentary. To evaluate the influence of oxidation kinetics of liquid copper on its electrical conductivity, resistivity of anode copper melts was measured simultaneously with oxygen sampling.

The empirical relationships between melt resistivity and oxygen dissolution in anode copper samples of different chemical compositions (Fig. 5) are comparable with the dependence of resistivity on oxygen content in solid copper in the region above 0.06 % (Fig. 4). Analysis of curves *1* and *3* (Fig. 5) shows a similar increase in melt resistivity with oxygen concentration, which can be approximated by an exponential function. This suggests that these anode copper samples have similar chemical compositions and contain only minor impurity levels. Curve *2* in Fig. 5 shows no increase in melt resistivity during oxygen saturation in the initial range of 0.066—0.3 %. This indicates that this anode copper sample has elevated impurity levels. In this case, the dissolved oxygen was consumed by further oxidation of impurities (Pb, Zn, Fe, S) that were not removed during anode smelting, which was manifested in slag formation during melt oxidation. After slag removal and continued oxygen dissolution, an increase in melt resistivity was recorded.

In conclusion, these results demonstrate the kinetic regularities of oxidation of solid and liquid cop-

Table 2. **Average chemical composition of anode copper samples**

Таблица 2. Средневзвешенный состав образцов анодной меди

Sample No.	Content, %								
	Pb	Sn	As	Sb	Zn	Fe	Ni	O	S
1	0.081	0.0056	0.0055	0.099	0.0028	0.0016	0.09	0.018	0.0071
2	0.08	0.0056	0.0054	0.099	0.0049	0.0032	0.092	0.023	0.0086
3	0.081	0.0054	0.0054	0.099	0.0023	0.0015	0.088	0.024	0.0055

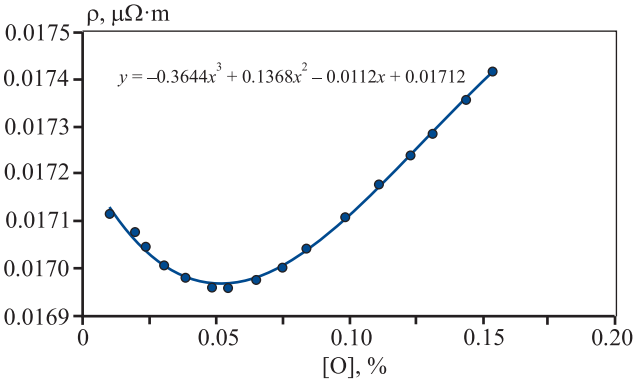

Fig. 4. Dependence of copper resistivity on oxygen concentration [23]

Рис. 4. Зависимость удельного сопротивления меди от содержания кислорода [23]

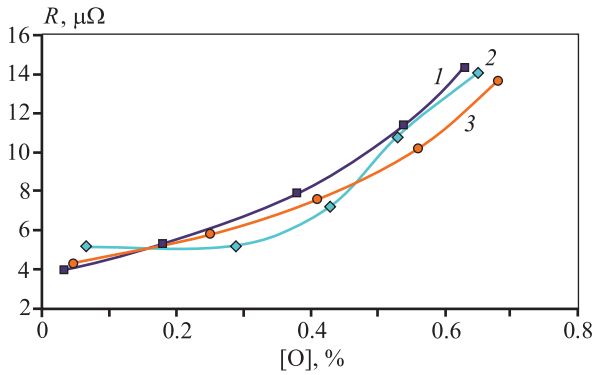

Fig. 5. Dependence of the resistance of copper (***1–3***) melt samples of different chemical (see table 2) composition on the oxygen content

Рис. 5. Зависимость электросопротивления образцов (***1–3***) расплава меди различного химического состава (см. табл. 2) от содержания кислорода

per of different chemical compositions and the effect of oxygen solubility on the electrical properties of the melt.

According to the Cu—Cu_2O phase diagram, the main condition for fire refining is to maintain the oxygen concentration in copper above the equilibrium level required for impurity oxidation (0.4—0.8 % at temperatures up to 1170 °C). Therefore, it is important to analyze the kinetics of oxygen dissolution in liquid copper during the oxidation stage to evaluate impurity transfer into slag and to avoid overoxidation. The influence of oxygen dissolution kinetics on the electrical conductivity of liquid copper should be assessed only after maximum impurity removal. This approach makes it possible to calculate and control the residual oxygen concentration in the copper melt by measuring melt resistivity [3].

Conclusion

The macroscopic oxidation kinetics of pure and blister copper samples of different chemical compositions were investigated using TG—DSC and surface oxidation by air blowing. The results show that all samples oxidize at almost the same rate and that the presence of impurities does not significantly influence copper oxidation.

All oxygen available for copper (I) oxide formation dissolves in copper up to the thermodynamic limit of 12 % Cu_2O. The effect of oxygen solubility in copper melts on their electrical properties was demonstrated. It was established that oxygen concentrations in the melt exceeding 0.08 % deteriorate the electrical properties of copper.

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