Insight on the Reduction of Copper Content in Slags Produced from the Ausmelt Converting Process

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Abstract

The reduction of copper content in converting slag using process control is significant to copper smelter. In this study, the slags produced from the Ausmelt Converting Process for copper matte have been analyzed using X-ray diffraction and chemical analysis. Thermodynamic calculation and effects of various conditions including the lance submerging depth in molten bath, the molten bath temperature, the addition of copper matte, and airflow rate were carried out to lower the content in the slag. Thermodynamic analysis indicates that the decrease of copper content was achieved by reducing Fe$_2$O$_4$, CuFe$_2$O$_4$ and Cu$_2$O in the slag, decreasing the magnetism of slag and lowering the viscosity of slag, which is feasible at the operating temperature of the molten bath. Experiments show that the optimal combination of operating conditions were found to be the addition of copper matte between 5000 - 7000 kg/h, a lance airflow rate of 13000-14000 Nm$^3$/h and a lance submergence depth into the molten bath of 700-900 mm, in which the copper content in the slag can be effectively reduced from 22.74 wt. % to 7.70 wt. %. This study provides a theoretical support and technical guidance for promoting the utilization of slags from the Ausmelt Converting Process.

Keywords: Top-blown converting; Copper content in slag; Copper matte; Blister copper; Ausmelt Furnace

1. Introduction

More than 80% of copper (Cu) is produced by pyro-metallurgical processing world-wide [1-5], with the main converting unit processes including flash converting, Peirce Smith (PS) converting, bottom blowing, top blowing and other improved converting processes. The copper content in the slag is typically less than 5 wt.% in the operation of PS converters and other improved furnaces [5-7], while it is higher than 16 wt.% Cu in some converting processes [8-10]. Numerous studies on reducing copper content in the converting slags have been carried out by experts and scholars [11-13] with their researches mainly focused on increasing feed of matte grade, process control

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system improvements of the converting process, converting slag reduction, converting slag beneficiation and copper recovery.

Increasing matte grade [14-15] will only reduce the output rate of slag, the process control system for the blowing process has no substantive improvement and copper content in the slag will not be reduced effectively.

PS converter slag was re-reduced in the modified PS converter by Yunnan Copper Corporation, but the effect of reduction is not significant because the copper content in PS converter slag is only about 3 wt.% prior to the re-reduction operation [16-17]. Copper was recovered in the form of a copper concentrate from the copper slag flotation process used at most PS converter operating sites in China. This method has many advantages when the copper content in the slag is low, but it is not economically sound to reduce the copper content using this method when the copper content in the slag is more than 16 wt.%, which is due to the low recovery efficiency of copper into the copper concentrate from the slag. Currently, the study of the reduction of copper content in the converting slag by using process control technology is rarely reported. Additionally, the toxicity and environmental issues of the solid waste including copper slags have been received wide attentions in recent years [18]. To efficient remove heavy metals including Cu²⁺ from waste water and soil, adsorption have been used for removal of Cu²⁺. However, the purification such as adsorption [19, 20] and bioremediation [21-23] usually faced the high cost and the waste of value metal resource if the high copper-bearing copper slag is discarded in the dump.

As an improved converting unit processes, the Ausmelt Top Submerged Lancing (TSL) matte converting technique was adopted at the Copper branch of Yunnan Tin Company Limited (YTCL). During the converting operation, limestone and quartz fluxes are fed into the furnace, with oxygen enriched air and pulverized coal injected into the molten bath via the Ausmelt lance. The resulting SO₂ in the flue gas is transported to the sulfuric acid production system to produce concentrated sulfuric acid whilst the dust containing valuable metals in the off-gas is collected by the ESP system [24]. The matte converting process is operated in a batch mode with the Converting 1 stage used for slag making/converting stage and the Converting 2 stage for the final production of blister copper. During Converting 1, solid copper matte is continuously fed to the Ausmelt furnace at about 60 tonnes per hour with a feeding time of 4 hours. Slag is tapped from the furnace twice during this stage while matte continues feeding; the first slag tap after 100 tonnes of matte is fed with the second tap after 180 tonnes of matte fed. Bath temperature is maintained at 1543 to 1593 K. In the Converting 2 stage, matte feeding is stopped and operating parameters are adjusted for converting the bath to produce blister copper. At the completion of this stage, the Converting 2 stage slag is tapped out from Ausmelt furnace, then blister copper is tapped at the end of slag tapping from the Ausmelt Converting furnace and is sent into the anode furnace for refining of copper via blister launder.

Herein, to gain a deeper understanding into this technical aspect, a study of the reduction of copper content in the slag produced from the Ausmelt TSL top-blown converting process of matte was carried out. The slags produced from the Ausmelt Converting Process for copper matte have been analyzed using X-ray diffraction (XRD)
and chemical analysis. Thermodynamic calculation and effects of various conditions including the lance submerging depth in molten bath, the molten bath temperature, the addition of copper matte, and airflow rate were carried out to lower the content in the slag. This study would provide a theoretical support and technical guidance for promoting the utilization of mineral resources.

2. Materials and Experimental Methods

2.1. Materials

In this study, the slag was produced in the converting process of YTCL, i.e. Converting stage 1 slag and Converting stage 2 slag. The Converting stage 1 slag is tapped continuously from the furnace while solid copper matte is fed continuously into the furnace. The Converting stage 2 slag is tapped from furnace when final converting process is finished. The granulated copper matte is produced from the upstream of Ausmelt TSL smelting process is added into the Ausmelt converting furnace with belt conveyor systems. Limestone and quartz are industrial grade. Other chemicals, including hydrochloric acid (HCl, 36 %, Xilong Chemical Group Co. Ltd.) and sulfuric acid (H₂SO₄, 98%, Xilong Chemical Group Co. Ltd.) are of analytical grade.

2.2. Experimental Methods

The experiments were conducted in an industrial experimental facility (inner diameter, 5 m; height, 12 m) located in the Copper Branch of Yunnan Tin Company. During the experimental period, the lance smelting factor and the lance submergence depth were controlled by changing the depth of spray lance to the molten pool to investigate the effect of these two parameters on the copper content in the resulting Converting 1 slags. A reduction of converting slag was carried out before the C2 slag was tapped at the end of the convert. The bath temperature at the end of the converting, reductant matte rate, total fed matte amount, lance airflow rate and lance submerging depth were controlled to different levels to investigate their effects on the copper content in the final tapped slag.

First, the representative slags from converting process including Convert stage 1 and Convert stage 2 were collected. Then, thermodynamic calculation was used to evaluate the reduction of copper in the Ausmelt Converting process. Subsequently, effects of various conditions including lance submerging depth in molten bath, molten bath temperature, the addition of copper matte, and airflow rate were carried out to lower the content in the copper.

The phase composition of Converting 1 and Converting 2 slags was investigated using an XRD (Rigaku IV, Japan) in the 20 range of 5-90 ° with the scan rate of 5°/min. The metallic copper particles contained in the slag samples are difficult to grind during sample preparation; therefore, these particles are screened out through a 400-mesh sieve and the undersize fraction was used for XRD analysis. To determine the content of magnetic components in slag, a magnetic separator was used to select the magnetic components in the slag and the obtained components were weighed for calculating the content of magnetic components in the slag. In addition, 0.10 g of Converting 1 and Converting 2 slag was totally dissolved using 20 mL of concentrated HCl and 15 mL of 50% v/v H₂SO₄ in a 300 mL beaker for the chemical analysis of copper content using an atomic absorption spectrophotometer (AAS, Z-8200, Japan), respectively.
3. Results and Discussions

3.1. XRD analysis of the samples

As can be seen from Fig. 1, at the Converting 1 stage, the strongest peak is the phase magnetite ($\text{Fe}_3\text{O}_4$), followed by cuprous oxide ($\text{Cu}_2\text{O}$) and a small amount of white matte ($\text{Cu}_2\text{S}$). At the Converting 2 stage, the strongest peak is also $\text{Fe}_3\text{O}_4$, then $\text{CuFe}_2\text{O}_4$ and $\text{Cu}_2\text{O}$.

![XRD patterns for (a) Converting 1 slag and (b) Converting 2 slag](image)

**Figure 1.** XRD patterns for (a) Converting 1 slag and (b) Converting 2 slag

3.2. Theoretical assessment of reducing copper content in converting slag

Based on the XRD analysis of the slag along with chemical analysis, it can be found that during the copper matte converting process, the strongest peak is the $\text{Fe}_3\text{O}_4$ phase and about 6 wt.% of metallic copper particles was suspended in the slag in both Converting 1 and Converting 2 stage, which indicates that the content of magnetite in the slag was high. When a magnetic analyzer was used to test the slag, the measured results shows more than 40 wt.% of the slag belongs to magnetism, suggesting that the viscosity of the slag is very high [17]. This is the main reason for the slag being contaminated with metallic copper particles and which is due to the highly oxidizing atmosphere in the TSL converting process. This high oxygen potential of the slag leads to some oxidation of Cu to $\text{Cu}_2\text{O}$ during the Converting 1 stage [24]. In addition, some
Cu$_2$O reacts with Fe$_3$O$_4$ and forms CuO·Fe$_2$O$_3$ during the Converting 2 stage [25, 26].

Therefore, in order to reduce the copper content in the slag during the TSL copper converting process, magnetic strength of the slag must be lowered, so that Fe$_3$O$_4$ would be reduced to FeO to provide a lower viscosity and form a low melting point slag, fayalite (2FeO·SiO$_2$) [26]. When the viscosity of the slag was decreased, metallic copper particles can settle and be separated from the slag. Furthermore, when the oxygen potential was decreased, Cu$_2$O and CuFe$_2$O$_4$ dissolved in the slag would be reduced to Cu$_2$S, which is insoluble in the slag. Then, Cu$_2$S can react with Cu$_2$O to form metallic copper, which would be settled from the slag and transformed to the blister copper. Some related chemical reactions are shown as follows [24; 26-28]:

1. Fe$_3$O$_4$+C=3FeO+CO  
   (1)
2. Fe$_3$O$_4$+FeS+O$_2$=4FeO+SO$_2$  
   (2)
3. 2CuFe$_2$O$_4$+3C=Cu$_2$O+4FeO+3CO  
   (3)
4. 2CuFe$_2$O$_4$+2FeS=Cu$_2$S+6FeO+SO$_2$  
   (4)
5. FeS+Cu$_2$O=FeO+Cu$_2$S  
   (5)
6. Cu$_2$S+2Cu$_2$O=6Cu+SO$_2$  
   (6)

Except for reaction (6), FeO generated in other reactions would react with silica in the molten bath to make slag, as shown in reaction (7). The relationship between Gibbs free energy (Δ$G^0$) and the temperature for the reactions (1)-(6) are calculated and shown in Fig. 2.

$$2\text{FeO} \cdot \text{SiO}_2 = 2\text{FeO} \cdot \text{SiO}_2$$  
   (7)

Fig. 2 shows that the initial reaction temperatures are 850 K (reaction 1), 560 K (reaction 3), 400 K (reaction 4) and 955 K (reaction 6). The reaction follows the descending sequence: (2) > (5) > (4) > (3) > (1) > (6) at the operating temperature. The Gibbs free energy of reactions (2) and (5) is always negative [29, 30], indicating that both reactions occur readily. Therefore, Fe$_3$O$_4$ and Cu$_2$O in the slag would be easily reduced by FeS when FeS exists in the molten bath.

![Figure 2. Relationship between Gibbs free energy and temperature for reactions (1) - (6)](image)

Considering that the temperature of the molten bath would be up to 1523-1573 K during the TSL copper converting process, the above-mentioned reactions (1)-(6) would occur from left to the right. Thus, at such temperatures, Fe$_3$O$_4$ and Cu$_2$O in the
slag would be easily reduced by carbon (C) or FeS.

3.3. Effects of conditions on the copper content in the slag

3.3.1. Effect of Smelting Air Factor on the copper content in the slag at Converting 1 stage

The Converting 1 stage of TSL copper converting process is a copper matte continuous feeding and slag making operation, during which Cu$_2$S is oxidized to blister copper and then can further be oxidized to Cu$_2$O because of the high Smelting Air Factor of lance air and strong oxygen potential of molten bath. Furthermore, vigorous stirring by the TSL lance results in some blister copper particles being entrained in the Converting 1 slag when it is tapped from furnace. Also, as part of the blister copper is strongly oxidized to Cu$_2$O and its solubility in the slag is much higher than the Cu$_2$S, leading to higher total copper content in the slag.

During the Converting 1 stage, the copper matte of 58 - 60 wt.% Cu is fed at 55 – 60 tonnes per hour using a lance smelting air factor of 800 Nm$^3$/tonne. FeS in the molten bath would be strongly oxidized to magnetic iron [24]. Furthermore, some Cu$_2$S would also be oxidized, which will result in the increase of copper content in the slag. When the air factor is decreased to 700 - 750 Nm$^3$/tonne, the oxygen potential of the bath may be reduced, thus a small amount of FeS would exist to help reduce Fe$_3$O$_4$ and Cu$_2$O, thus decreasing copper content in the slag effectively. However, if the smelting air factor is further reduced, since the heat of slagging reaction is not enough, it will result in lower bath temperature and this is not conducive for slag making normally at C1 stage in the TSL process.

Therefore, lowering the lance air smelting factor decreases the oxygen potential of the molten bath and then the amount of Cu$_2$S which is oxidized to blister copper would be reduced during the slag making process in the Converting 1 stage. Meanwhile, FeS should be prevented from being excessively oxidized from the copper matte. Only by doing so, some FeS remains in the molten bath and will promote reactions (2), (4) and (5), and then to reduce Fe$_3$O$_4$ content of slag to ensure that slag has good fluidity, which helps copper metal particles settling from slag. The Cu$_2$O in the slag can be reduced by FeS to form Cu$_3$S [26-28], separating from slag to decrease copper content in the slag.

3.3.2. Effect of lance submerging depth in molten bath on the copper content in the slag at Converting 1 stage

In addition to controlling the smelting air factor during the Converting 1 stage, the lance submergence within the molten bath must also be adjusted. This is due to that if the lance is submerged too deep, blister copper or Cu$_2$S underneath slag layer will be stirred up, resulting in copper loss with the slag discharged. If the lance submerging depth in the molten bath is too shallow, it will reduce the effective utilization of lance flow for stirring and slagging reactions are incomplete, resulting in lower bath temperature, poor mobility of slag and higher copper content in the slag [31].

Experimental studies during the production process have shown that, as the bath level gradually increased with the feeding process, optimal control of the lance submergence depth is 200-300 mm into the molten bath during the Converting 1 stage. If it is too deep, the lance flow will stir up blister copper at the bottom of molten bath into the slag and leads to copper content in slag increased. Settling and separation of
metallic copper from slag is interrupted.

3.3.3. Effect of molten bath temperature on copper content in the slag during the Converting 2 stage

Keeping other conditions relatively constant and adding reductant coal or copper matte for reduction of the converting slag before blister tapping, the effect of initial bath temperature on the copper content in the slag at the end of Converting 2 stage of TSL was examined. The relationship of initial molten bath temperature and the copper content in slag after reduction are shown in Fig. 3.

Fig. 3 shows that when initial bath temperature is lower than 1563 K, the reduction impact is not significant and the copper content in the slag is above 15.67 wt.%. However, when the temperature of the bath is above 1573 K, the copper in the slag is lowered to below 13.65 wt.%. The reason is that when using coal or matte to reduce the slag, the reactions are endothermic and the initial bath temperature is essential for the reduction effect [32, 33]. Low initial temperature and endothermic reactions would cause the bath temperature to further decrease, which will result in low temperature, poor mobility, and high viscosity, and copper particles or Cu$_2$S not settling and so becoming entrained in the slag. Therefore, to obtain a better reduction effect, initial bath temperature must be maintained at around 1573 K before proceeding with reduction to ensure adequate heat is available for endothermic reaction requirements in the reduction process.

![Figure 3. Relationship between Converting 2 initial molten bath temperature and copper content in the slag after reduction](image)

3.3.4. Impact of the addition of copper matte on copper content in slag at Converting 2 stage

By keeping other conditions relatively stable, the effect of the addition of copper matte used as reductant on the copper content in the slag at Converting 2 stage was examined. The results are shown in Fig. 4.

The copper content in Converting 2 slag is reduced from 18.88 wt.% to 7.79 wt.% when the amount of copper matte added is increased from 1000 to 9000 kg/h. This demonstrates the effect of copper matte as a reductant to the copper content is significant due to reactions (2), (4) and (5). The precondition for this is that specific gravity of copper matte is close to Fe$_3$O$_4$ or CuFe$_2$O$_4$ and the stirring action of injected
air from the lance accelerates those reactions. This results in a lowered magnetic strength of the slag and improved the slag fluidity, allowing blister copper to be separated and be settled from the slag. Oxidized copper dissolved in slag was reduced by FeS to produce Cu₂S [26-28]. All these factors support a lower copper content in the slag.

Although the addition of copper matte has a significant effect on the reduction of copper content in the slag during the Converting 2 stage, the amount of matte cannot be added excessively. This is because the copper matte particles feeding into molten bath will absorb heat to melt into liquid phase. If the amount is excessive, the molten bath temperature will be decreased which is not ideal for reactions (2), (4) and (5) to occur. Under these conditions, the best addition rate of copper matte is controlled in the range of 5000 - 7000 kg/h. It should be recognized that excessive addition of copper matte will also lead to longer operation time and higher sulfur content of the blister copper.

\[ \text{Figure 4. Relationships between copper content in the slag and amount of copper matte at Converting 2 stage} \]

3.3.5. Effect of airflow rate on copper content in the slag at slag reduction process of Converting 2 stage

The lance airflow rate is an important factor in the reduction of Converting 2 stage slag process. The impact of lance airflow rate on the copper content in the slag after reduction was examined with addition of copper matte controlled at 7000 kg/h and reduction time maintained at 15 min. The results are shown in Fig. 5.
When the airflow rate is controlled in the range of 18000 - 20000 Nm$^3$/h, the reduction of slag is ineffective and copper content in the slag has only slightly reduced. The main reason is that FeS in copper matte was preferentially oxidized [24, 34], which results in the content of FeS required by reactions of slag reduction (2), (4) and (5) being decreased and the extent of the reduction of slag is weakened.

With the airflow rate decreased to 13000 - 14000 Nm$^3$/h, copper content in the slag began to decrease significantly. When airflow rate further declined, there is a slight decrease of copper content in the slag. However, the difference of melt depth inserted into the melt will lead to the difference of spraying lance airflow rate and melt stirring intensity, thus affecting the relatively static sedimentation separation of copper in the slags [31]. Low airflow rate is not favored for the operation as it can cause lance tip blockage, less stirring of molten bath and result in difficulties in achieving stable normal operation. It is found for this case that the best airflow rate should be controlled in the range of 13000 - 14000 Nm$^3$/h, which can ensure reduction reactions at the end of Converting 2 stage completed smoothly.

### 3.3.6. Effect of lance submergence depth on copper content in the slag during Converting 2 stage

The effect of lance submergence depth in molten bath on copper content in the slag during the Converting 2 stage was examined when the addition rate of copper matte was fixed at 7000 kg/h, the reduction time was set to 15 min and lance airflow rate was controlled at 14000 Nm$^3$/h. The results are shown in Table 1.

| Lance Injection depth (mm) | 1700 | 1300 | 1100 | 900 | 700 | 500 | 300 |
|----------------------------|------|------|------|-----|-----|-----|-----|
| Copper content in slag after reduction (wt.%) | 16.76 | 14.36 | 13.79 | 12.86 | 12.54 | 15.53 | 16.45 |

Too deep (>900 mm) or too shallow (< 700 mm) lance submerging depth is not beneficial for slag reduction. The reason is that, if the lance submerging depth is too deep, blister copper at the bottom of molten bath will be stirred and then mixed with slag phase, causing the copper content of slag to increase. However, if it is too shallow, the mixing effect on the molten bath is poor, which is not good for the reaction of copper matte with slag. We can see that the best submergence depth is 700 - 900 mm, while the copper content in the slag would be controlled at around 12.54 wt.%, which means...
that the copper content in the slag is effectively decreased.

3.4. Analysis of copper content in mixed slag from both Converting 1 and Converting 2 stages

Following adjustment of the Smelting Air Factor, the lance submergence depth and after reduction of the Converting 2 final slag, a mixed slag sample was obtained from Converting 1 and Converting 2 stages, namely comprehensive slag. The sample was tested using XRD and its result is shown in Fig. 6. The strongest peak was the Fe₃O₄ phase, then the magnetic iron phase. A small amount of copper metallic phase was also observed. By comparing Fig. 1 and Fig. 6, CuFe₂O₄, Cu₂O and CuS in the slag were all gone after the slag reduction at the end of Converting 2 stage, indicating that the measures are effective for the reduction of copper content in the slag. The copper-containing phase in the slag is effectively decreased and the chemically dissolved copper in the slag is lowered. Chemical analysis of the slag samples showed that the copper content in the slag is 7.70 wt.%, which is far below the previous value of 22.74 wt.% prior to the experiment.

![Figure 6. XRD analysis of the comprehensive slag](image)

4. Conclusions

In the study, theoretical analysis shows that the copper content was decreased in the slag by reduction of Fe₃O₄, CuFe₂O₄ and Cu₂O in the slag, diminishing the magnetism of slag and lowering the viscosity of slag is feasible at the process operating temperatures. When the Smelting Air Factor is set to 700-750 Nm³/tonne, the oxygen potential of the bath may be reduced; thus, a small amount of FeS would exist to inhibit the formation of Fe₃O₄ phase and will reduce the oxidized Cu₂O, which will effectively reduce the copper content of slag. To get a better reduction efficiency, the initial bath temperature must be maintained at 1573 K and adequate heat must be provided. The best combination of operating conditions for this case were found to be: the addition of copper matte between 5000 - 7000 kg/h, a lance airflow rate of 13000-14000 Nm³/h and a lance submergence depth into the molten bath of 700-900 mm. Under these conditions, the copper content of the converting slag can be effectively maintained at 7.70 wt.%. This study would provide a theoretical support and technical guidance for promoting the utilization of mineral resources. Moreover, the other metal distribution and energy flow in the system will be considered in the future study.
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Figure captions

**Figure 1.** XRD patterns for (a) Converting 1 slag and (b) Converting 2 slag

**Figure 2.** Relationship between Gibbs free energy and temperature for reactions (1) - (6)

**Figure 3.** Relationship between Converting 2 initial molten bath temperature and copper content in the slag after reduction

**Figure 4.** Relationships between copper content in the slag and amount of copper matte at Converting 2 stage

**Figure 5.** The relationship of air rate with copper content in the slag after reduction

**Figure 6.** XRD analysis of comprehensive slag

Table captions

**Table 1** The relationship of lance injection depth with copper content in the slag after reduction
Uvid u smanjenje sadržaja bakra u šljaki proizведенog iz procesa konverzije Ausmelt

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Apstrakt
Smanjenje sadržaja bakra u konvertovanju šljake pomoću kontrole procesa značajno je za topionicu bakra. U ovoj studiji, šljake proizvedene iz Ausmelt procesa konvertovanja bakra su analizirane pomoću rendgenske difrakcije i hemijske analize. Termodinamički proračun i efekti različitih uslova uključujući dubinu potapanja koplja u rastopljenoj kupki, rastopljenu temperaturu kupatila, dodavanje bakarnog mat-a i brzinu protoka vazduha izvršeni su kako bi se smanjio sadržaj u šljaci. Termodinamička analiza ukazuje na to da je smanjenje sadržaja bakra postignuto smanjenjem Fe3O4, CuFe2O4 i Cu2O u šljaci, smanjenjem magnetizma šljake i snižavanjem viskoznosti šljake, što je izvodljivo na operativnoj temperaturi rastopljene kupke. Eksperimenti pokazuju da je utvrđeno da je optimalna kombinacija operativnih uslova dodavanje bakarnog mat-a između 5000 - 7000 kg/h, stopa protoka koplja od 13000-14000 Nm3/h i dubina potapanja koplja u rastopljenu kupku od 700-900 mm, u kojoj se sadržaj bakra u šljaci može efikasno smanjiti sa 22.74 wt. % na 7.70 wt. %. Ova studija pruža teorijsku podršku i tehničke smernice za promovisanje korišćenja šljake iz procesa konverzovanja Ausmelt.

Ključne reči: vrhunsko konvertovanje; Sadržaj bakra u šljaci; Bakarni mat; Blister bakar; Ausmelt Peć