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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 was 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 indicated that the decrease of copper content was achieved by reducing Fe_3O_4, $CuFe_2O_4$, *and* Cu_2O *in the slag, decreasing the magnetism of slag and lowering the viscosity of slag, which was feasible at the operating temperature of the molten bath. Experiments showed that the optimal combination of operating conditions were found to be the addition of copper matte between 5,000 – 7,000 kg/h, a lance airflow rate of 13,000-14,000* Nm^3/h, *and a lance submergence depth into the molten bath of 700-900 mm, in which the copper content in the slag was 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 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 efficiently remove heavy metals, including Cu^{2+}, from waste water and soil, adsorption have been used for removal of Cu^{2+}. 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 was discarded in the dump.

As an improved converting unit processes, the Ausmelt Top Submerged Lancing (TSL) matte converting technique was adopted at the Copper

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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 SO_2 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 1,543 to 1,593 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 slag produced from the Ausmelt Converting Process for copper matte was 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 was tapped continuously from the furnace while solid copper matte was fed continuously into the furnace. The Converting stage 2 slag was tapped from furnace when final converting process was finished. The granulated copper matte was produced from the upstream of Ausmelt TSL smelting process was added into the Ausmelt converting furnace with belt conveyor systems. Limestone and quartz were industrial grade. Other chemicals, including hydrochloric acid (HCl, 36 %, Xilong Chemical Group Co. Ltd.), and sulfuric acid (H_2SO_4, 98%, Xilong Chemical Group Co. Ltd.) were 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 2θ range of 5-90 ° with the scan rate of 5°/min. The metallic copper particles contained in the slag samples were difficult to grind during sample preparation; therefore, these particles were screened out through a 400-mesh sieve and the undersize fraction was used for the 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 H_2SO_4 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 had the phase magnetite (Fe_3O_4), followed by cuprous oxide (Cu_2O), and a small amount of white matte (Cu_2S). At the Converting 2 stage, the strongest peak was also Fe_3O_4, then $CuFe_2O_4$, and Cu_2O.

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 had the Fe_3O_4 phase and about 6 wt.% of metallic copper particles was suspended in the slag in both Converting 1 and Converting 2 stage, which indicated that the content of magnetite in the slag was high. When a magnetic analyzer was used to test the slag, the measured results showed more than 40 wt.% of the slag belonged to magnetism, suggesting that the viscosity of the slag was very high [17]. This is the main reason for the slag being contaminated with metallic copper particles, which was due to the highly oxidizing atmosphere in the TSL converting process. This high oxygen potential of the slag caused some oxidation of Cu to Cu_2O during the Converting 1 stage [24]. In addition, some Cu_2O reacted with Fe_3O_4 and formed $CuO·Fe_2O_3$ 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_3O_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 could settle and be separated from the slag. Furthermore, when the oxygen potential was decreased, dissolved Cu_2O and $CuFe_2O_4$ in the slag would be reduced to Cu_2S, which was insoluble in the slag. Then, Cu_2S could react with Cu_2O 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]:

$$Fe_3O_4+C=3FeO+CO \quad (1)$$
$$Fe_3O_4+FeS+O_2=4FeO+SO_2 \quad (2)$$
$$2CuFe_2O_4+3C=Cu_2O+4FeO+3CO \quad (3)$$
$$2CuFe_2O_4+2FeS=Cu_2S+6FeO+SO_2 \quad (4)$$
$$FeS+Cu_2O=FeO+Cu_2S \quad (5)$$
$$Cu_2S+2Cu_2O=6Cu+SO_2 \quad (6)$$

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 (DG^0) and the temperature for the reactions (1)-(6) were calculated and shown in Fig. 2.

$$2FeO+SiO_2 = 2FeO·SiO_2 \quad (7)$$

Fig. 2 shows that the initial reaction temperatures were 850 K (reaction 1), 560 K (reaction 3), 400 K (reaction 4), and 955 K (reaction 6). The reaction followed the descending sequence: (2) > (5) > (4) > (3) > (1) > (6) at the operating temperature. The Gibbs free energy of reactions (2) and (5) was always negative [29, 30], indicating that both reactions occurred readily. Therefore, Fe_3O_4 and Cu_2O in the

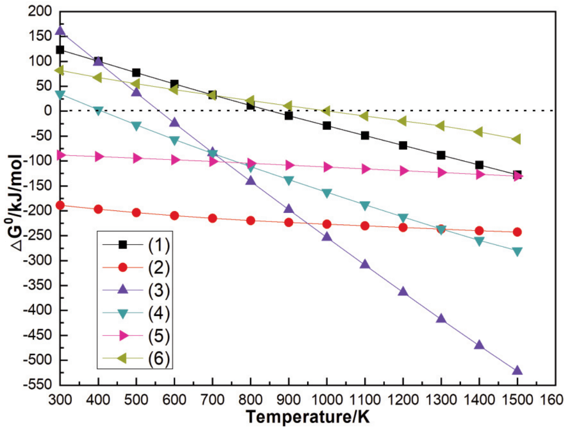

Figure 2. *Relationship between Gibbs free energy and temperature for reactions (1) - (6)*

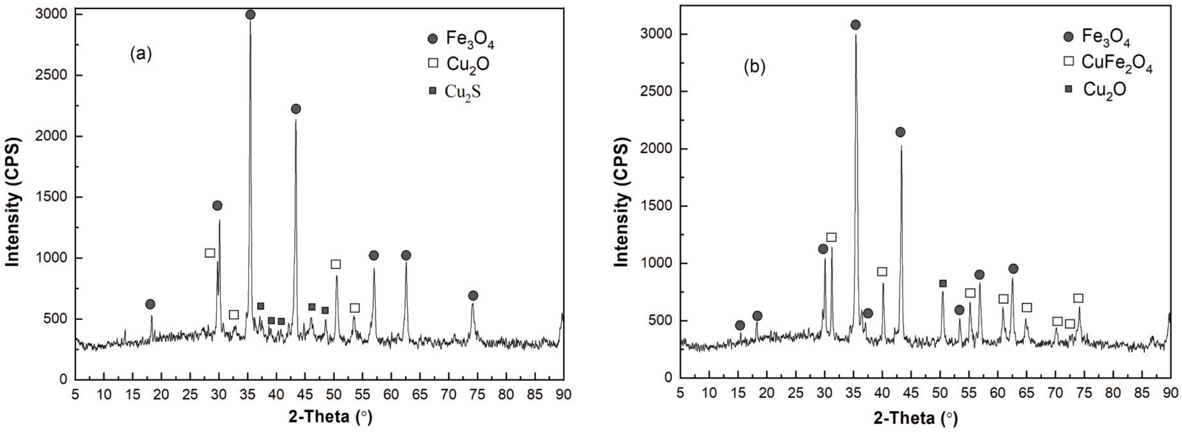

Figure 1. *XRD patterns for (a) Converting 1 slag and (b) Converting 2 slag*

slag would be easily reduced by FeS when FeS existed in the molten bath.

Considering that the temperature of the molten bath would be up to 1,523-1,573 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_3O_4 and Cu_2O 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 was a copper matte continuous feeding and slag making operation, during which Cu_2S was oxidized to blister copper and then can further be oxidized to Cu_2O 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 was tapped from furnace. Also, as part of the blister, copper was strongly oxidized to Cu_2O and its solubility in the slag was much higher than the Cu_2S, leading to higher total copper content in the slag.

During the Converting 1 stage, the copper matte of 58 - 60 wt.% Cu was 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_2S would also be oxidized, which would result in the increase of copper content in the slag. When the air factor was 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_3O_4 and Cu_2O, thus decreasing copper content in the slag effectively. However, if the smelting air factor was further reduced, since the heat of slagging reaction was not enough, it would result in lower bath temperature and this was not conducive for slag making normally at C1 stage in the TSL process.

Therefore, lowering the lance air smelting factor the oxygen potential of the molten bath decreased and then the amount of Cu_2S, which was 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 remained in the molten bath and promoted reactions (2), (4), and (5), and then reduced Fe_3O_4 content of slag to ensure that slag had good fluidity, which helped copper metal particles settling from slag. The Cu_2O in the slag can be reduced by FeS to form Cu_2S [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_2S 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 was lower than 1,563 K, the reduction impact was not significant and the copper content in the slag was above 15.67 wt.%. However, when the temperature of the bath was above 1,573 K, the copper in the slag was lowered to below 13.65 wt.%. The reason was that when using coal or matte to reduce the slag, the reactions were endothermic and the initial bath temperature was essential for the reduction effect [32, 33]. Low initial temperature and endothermic reactions would cause the bath temperature to further decrease, which would result in low temperature, poor mobility, and high viscosity, and copper particles or Cu_2S not settling and so becoming entrained in the slag. Therefore, to obtain a better reduction effect, initial bath temperature must be maintained at around 1,573 K before proceeding with reduction to ensure adequate heat was available for endothermic reaction requirements in the reduction process.

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 was reduced from 18.88 wt.% to 7.79 wt.% when the amount of copper matte added increased from 1000 to 9000 kg/h. This demonstrated the effect of copper matte as a reductant to the copper content was significant due to reactions (2), (4), and (5). The precondition for this was that specific gravity of copper matte was close to Fe_3O_4 or $CuFe_2O_4$ and the stirring action of injected air from the lance accelerated those reactions. This resulted 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 Cu_2S [26-28]. All these factors supported a lower copper content in the slag.

Although the addition of copper matte had a significant effect on the reduction of copper content in the slag during the Converting 2 stage, the amount of matte could not be added excessively. This was because the copper matte particles feeding into molten bath would absorb heat to melt into liquid phase. If the amount was excessive, the molten bath temperature would be decreased which was not ideal for reactions (2), (4), and (5) to occur. Under these conditions, the best addition rate of copper matte was controlled in the range of 5,000 – 7,000 kg/h. It should be recognized that excessive addition of copper matte would also lead to longer operation time and higher sulfur content of the blister copper.

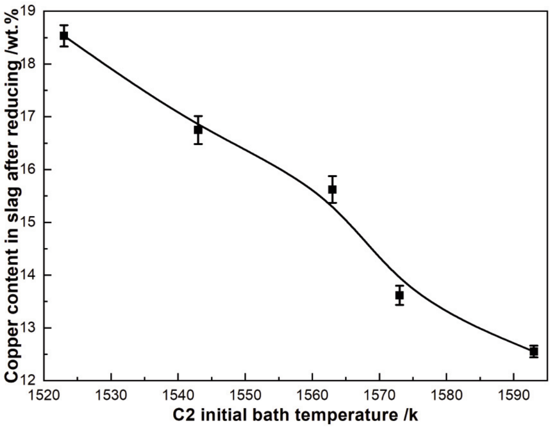

Figure 3. *Relationship between Converting 2 initial molten bath temperature and copper content in the slag after reduction*

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 7,000 kg/h and reduction time maintained at 15 min. The results are shown in Fig. 5.

When the airflow rate was controlled in the range of 18,000 - 20,000 Nm^3/h, the reduction of slag was ineffective and copper content in the slag had only slightly reduced. The main reason was that FeS in copper matte was preferentially oxidized [24, 34], which resulted 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 was weakened.

With the airflow rate decreased to 13,000 - 14,000 Nm^3/h, copper content in the slag began to decrease

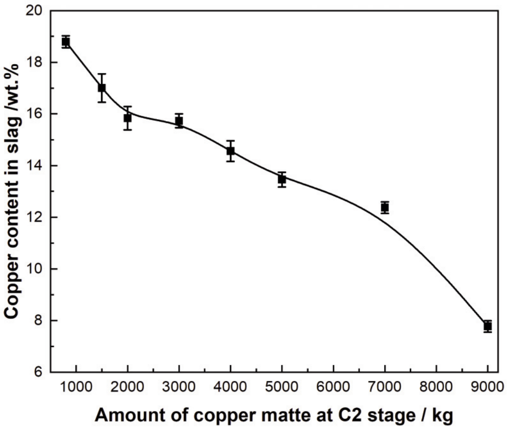

Figure 4. *Relationships between copper content in the slag and amount of copper matte at Converting 2 stage*

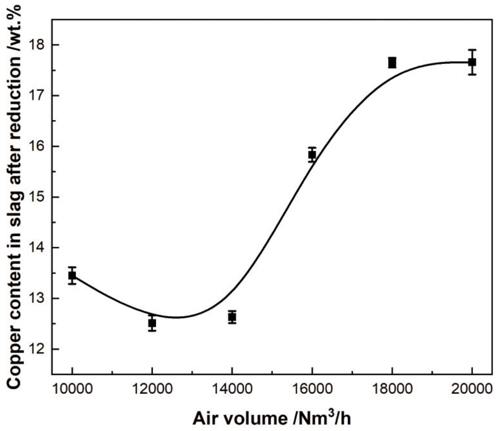

Figure 5. *The relationship of airflow rate with copper content in the slag after reduction*

significantly. When airflow rate further declined, there was a slight decrease of copper content in the slag. However, the difference of melt depth inserted into the melt caused 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 was not favored for the operation as it could cause lance tip blockage, less stirring of molten bath, and result in difficulties in achieving stable normal operation. It was found for this case that the best airflow rate should be controlled in the range of 13,000 - 14,000 Nm^3/h, which could 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 7,000 kg/h, the reduction time was set to 15 min, and lance airflow rate was controlled at 14,000 Nm^3/h. The results are shown in Table 1.

Too deep (>900 mm) or too shallow (< 700 mm) lance submerging depth was not beneficial for slag reduction. The reason was that, if the lance submerging depth was too deep, blister copper at the bottom of molten bath would be stirred and then mixed with slag phase, causing the copper content of slag to increase. However, if it was too shallow, the mixing effect on the molten bath was poor, which was not good for the reaction of copper matte with slag. It can be seen that the best submergence depth was 700 - 900 mm, while the copper content in the slag would be controlled at around 12.54 wt.%, which meant that the copper content in the slag 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 Fe_3O_4 phase, then the magnetic iron phase. A small amount of copper metallic phase was also observed. By comparing Fig. 1 and Fig. 6, $CuFe_2O_4$, Cu_2O, and Cu_2S in the slag were all gone after the slag reduction at the end of Converting 2 stage, indicating that the measures were effective for the reduction of copper content in the slag. The copper-containing phase in the slag was effectively decreased and the chemically dissolved copper in the slag was lowered. Chemical analysis of the slag samples showed that the copper content in the slag was 7.70 wt.%, which was far below the previous value of 22.74 wt.% prior to the experiment.

Table 1.** **The relationship of lance injection depth with copper content in the slag after reduction

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

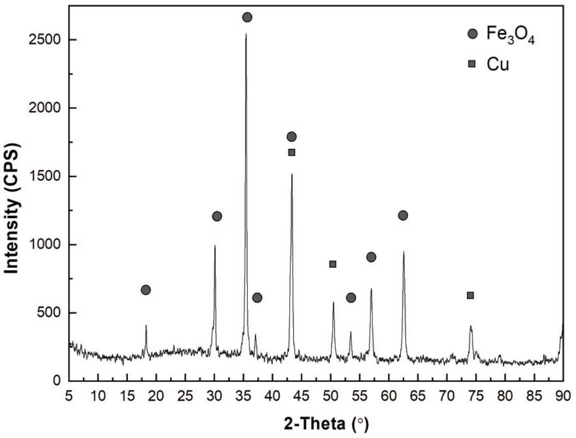

***Figure 6.** XRD analysis of the comprehensive slag*

4. Conclusions

In the study, theoretical analysis showed that the copper content decreased in the slag by reduction of Fe_3O_4, $CuFe_2O_4$, and Cu_2O in the slag, diminishing the magnetism of slag and lowering the viscosity of slag was feasible at the process operating temperatures. When the Smelting Air Factor was set to 700-750 Nm^3/tonne, the oxygen potential of the bath could be reduced; thus, a small amount of FeS would exist to inhibit the formation of Fe_3O_4 phase and reduce the oxidized Cu_2O, which could 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 5,000 - 7,000 kg/h, a lance airflow rate of 13,000-14,000 Nm^3/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.

Acknowledgments

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SMANJENJE SADRŽAJA BAKRA U ŠLJACI PROIZVEDENOJ TOKOM AUSMELT POSTUPKA KONVERTOVANJA

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Apstrakt

Kontrola postupka smanjenja sadržaja bakra u šljaci prilikom konvertovanja je značajna za topionice bakra. U ovom radu je analizirana šljaka dobijena tokom Ausmelt postupka za konvertovanje bakrenca pomoću rendgenske difrakcije i hemijske analize. Termodinamički proračun i efekti različitih uslova, uključujući dubinu potapanja koplja u rastopljenoj kupki, temperaturu rastopljene kupke, dodavanje bakrenca i brzinu protoka vazduha, izvršeni su kako bi se smanjio sadržaj u šljaci. Termodinamička analiza je ukazala da je smanjenje sadržaja bakra postignuto smanjenjem sadržaja Fe_3O4*,* $CuFe_2O_4$ *i* Cu_2O *u šljaci, dovodeći do smanjenja magnetizma šljake i snižavanja njene viskoznosti, što je bilo izvodljivo na operativnoj temperaturi rastopljene kupke. Eksperimenti su pokazali da je optimalna kombinacija operativnih uslova dodavanje bakrenca između 5000 - 7000 kg/h, brzina protoka vazduha u koplju 13000-14000* Nm^3/h *i dubina potapanja koplja u rastopljenu kupku od 700 – 900 mm, kada je sadržaj bakra u šljaci uspešno smanjen sa 22,74 wt. % na 7,70 wt. %. Ovo istraživanje pruža teorijsku podršku i tehničke smernice za promovisanje iskorišćenja šljake dobijene tokom Ausmelt postupka konvertovanja.*

Ključne reči: *Konvertovanje sa produvavanjem odozgo; Sadržaj bakra u šljaci; Bakrenac; Blister bakar; Ausmelt peć*