

Article

Phase Transformation of Arsenic, Antimony and Lead in High-Grade Copper Matte Converting

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Abstract: The duration of the high-grade matte converting process is short, the amount of slag is small, and it is difficult for the original impurity removal operation in the low-grade matte converting process to meet the current production demand. Because the removal method for impurity elements during high-grade matte converting is unclear, the phase transformation of impurity elements during this process is investigated in this study. The results show that arsenic exists mainly in the form of FeAsO_4 and As_2O_5 , antimony in the form of Sb and Sb_2O_5 , and lead in the form of PbS and PbO in high-grade matte. During the converting process, arsenic and antimony mainly exist in the melt in the form of oxides and gradually aggregate into large particles with increasing copper content in the melt. Lead exists in matte in the form of PbS until the end of the converting process, and PbS is not completely oxidized until the matte converted to blister copper phase. The phase transformation characteristics of copper, iron, sulfur and impurity elements in the process of high-grade matte converting were revealed. This study provides a theoretical reference for the formulation of an efficient impurity removal scheme for the converting process.

Keywords: P-S converting; high-grade matte; impurity element; phase transformation



Citation: Qu, W.; Yang, Y.; Zhou, S.; Wei, Y.; Li, B. Phase Transformation of Arsenic, Antimony and Lead in High-Grade Copper Matte Converting. *Minerals* **2024**, *14*, 499. <https://doi.org/10.3390/min14050499>

Academic Editor: Hugo Marcelo Veit

Received: 7 April 2024

Revised: 2 May 2024

Accepted: 7 May 2024

Published: 9 May 2024



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1. Introduction

P-S converters occupy a leading position in the matte converting industry, and are favored by many smelters because of their simple processing, mature technology and large production scale. Since its birth in 1905, this process has gradually been used around the world. The data show that 70% to 80% of the world's converting process still uses a P-S converter, which is more convenient to operate, inexpensive, and shows its special advantages in dealing with self-produced matte and purchased scrap copper [1]. Therefore, P-S converting technology will continue to be used in the foreseeable future. At present, the P-S converting technology has been significantly improved in terms of the operation process, mechanical automation and environmental protection, but there are still some problems with this technology [2]. Due to the periodic intermittent operation of P-S converting, problems such as high heat loss, low sulfur recovery caused by large soot emissions and composition fluctuations, and SO_2 low-altitude pollution caused by unsealed furnace bodies need to be improved and optimized [3,4].

With the progress of modern converting technology, the problems of mineral utilization, production efficiency, direct copper yield and low-altitude pollution have been solved to varying degrees in the matte smelting process [5–9]. To improve the efficiency of the copper pyrometallurgy process, the grade of the matte has also increased. It is difficult for the original low-grade matte converting technology to meet the requirement of removing impurities in the process of modern converting. This makes the impurities in the blister

copper excessively high, seriously affecting subsequent pyrometallurgical refining and electrolytic refining [10].

In modern copper smelting, matte with a grade greater than 70% is generally called high-grade matte. The higher the grade of matte is, the lower the content of other elements such as iron and sulfur, and the reduction in the content of iron is accompanied by a reduction in the amount of slag in the converting process. The heat required for the converting process is provided by the oxidation exotherm of FeS, Cu₂S and other sulfides. The improvement in the matte grade makes it difficult to meet the heat needs of the converting temperature in the oxidation reaction during the converting process, and additional coal injection is often needed to ensure the converting temperature [11,12]. With increasing maturity grade, less oxygen is needed, the time required for the entire converting process is reduced, the converting cycle is shortened, and the indicators that rely on the converting time in the original low-grade matte converting process, such as the copper content in slag and impurity removal, become difficult to determine [13,14].

The oxides of the impurity elements arsenic and antimony are difficult to react with quartz flux, and mainly rely on the volatilization of their monomers or low-valent oxides to remove them [15–18]. According to scholars, in the simulation of the distribution proportion of arsenic in the converting process, 77% of arsenic volatilizes into gas, but the factory production data are far from the difference, which is strongly related to the matte grade in the converting process [19,20]. The P-S converter selects quartz as the converting flux, which can enter a large amount of amorphous PbSiO₃ produced by PbO slagging during the slagging period. However, it is difficult for quartz flux to remove the complex salt of lead, and the lead in the melt can form lead arsenic antimonate during the copper-making period and can form complex compounds with other oxides to remain in the blister copper. In the process of converter production, the removal rate of lead can reach approximately 87%, and the amount of lead converted into gas through volatilization is twice that removed from the slag [21,22]. When scholars studied the distribution of impurity elements in the converting process, it was found that the residual impurities in the blister copper products increased significantly with increasing matte grade because of the high copper content in the matte, the small blast volume of the matte, and the small amount of slag formed [23–25].

At present, research on impurity elements in the converting process has focused mainly on the direction and distribution of impurity elements, and the law of phase transformation of impurity elements remains to be studied. To master the method and principle of impurity removal in the P-S converting process, based on the detection results of matte impurity element content and phase characteristics, the distribution behavior of impurity elements in each phase was studied by analyzing the changes in impurity element content and phase characteristics in different stages of the converting process, and the phase transformation laws of arsenic, antimony and lead in the converting process were revealed. This study provides a theoretical reference for improving the removal efficiency of impurities in the P-S converting process.

2. Materials and Methods

2.1. Materials

The raw material used in this research was a high-grade matte produced by the bottom blow smelting furnace of a copper smelting plant. After the high-grade matte was broken, the sample was finely ground to less than 60 mesh using a vibrating mill. The chemical composition of the high-grade matte sample was analyzed by means of chemical composition analysis and inductively coupled plasma emission spectrometry (ICP-OES), and the results are listed in Table 1. The converting process used a ferrosilicon slag system. The content of SiO₂ in quartz flux was more than 85%, the moisture content was less than 6%, the powder content was less than 5%, and the impurities were less than 3%. We added coal powder at the beginning of the converting to ensure the converting temperature. The proximate analysis of the coal powder is listed in Table 2.

Table 1. Chemical analysis of high-grade matte.

Element	Cu	Fe	S	Pb	Zn	Ca	As	Si	Ni	Sb
Content (wt.%)	72.22	3.72	18.38	1.38	0.39	0.28	0.25	0.2	0.18	0.07

Table 2. Industrial analysis of carbon powder.

Species	Mad	Aad	Vad	Fcad
Content (wt.%)	0.48	15.53	8.68	75.31

The results of X-ray diffraction (XRD) analysis of the high-grade matte are shown in Figure 1. The main phases of high-grade matte are Cu₂S and FeS, but compounds with low contents of As, Sb, Pb and other elements cannot be detected by XRD.

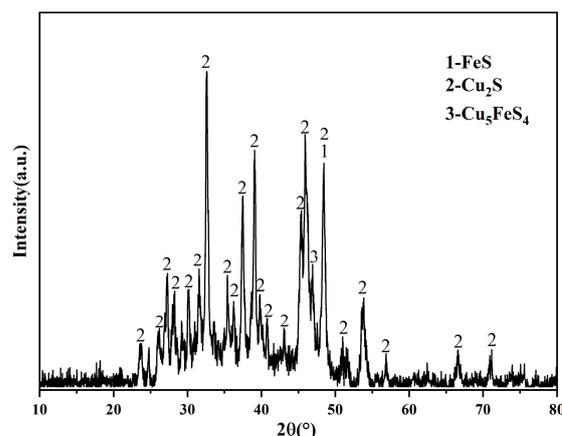


Figure 1. XRD pattern of high-grade matte.

2.2. Research Methods

2.2.1. Experimental Methods

The operating system for high-grade matte converting is shown in Figure 2. In the process of high-grade matte converting, about 110 t matte was added to the converter in 3 batches during the feeding period. Before oxygen blowing, the first batch of 51 t included the 1, 2, and 3 packages of molten matte, and 8 t of the cold charge were added. During the whole converting process, 2 t coal powder were added three times through the tuyere to stabilize the converting temperature. After oxygen blowing for 35 min, the air flow was stopped, and the second batch 20 t of molten matte (package 4) and 6 t of the cold charge was added. The converter proceeds rotated to the converting position for oxygen blowing. The third batch is the fifth package 19 t of molten matte and 6 t of the cold charge. The feeding period lasted 100 min. Subsequently, the furnace continuous oxygen blowing and converting lasted 130 min until end. During the whole converting process, 1.7 t quartz flux was added into the furnace in 4 times. After the converting, the slag was released before the copper was released. 17 t converting slag and 75 t blister copper were produced.

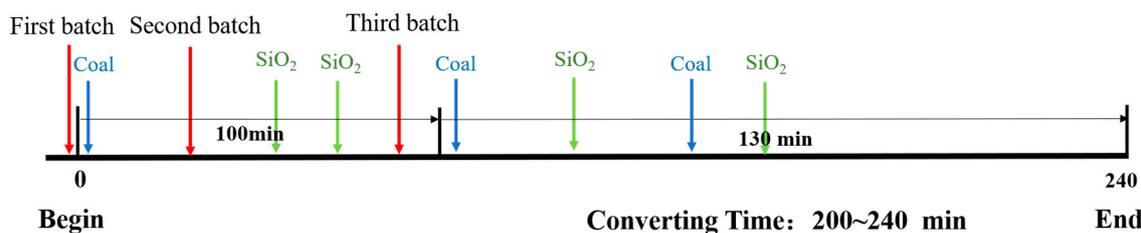


Figure 2. Flow chart of the high-grade matte converting.

A series of experiments were carried out in the process of converting high-grade matte. First, chemical phase analysis of the high-grade matte was performed. Then, the high-grade matte converting process was sampled according to the converting time. The sampling operation determined the sampling time point according to the oxygen converting time. Starting from the addition of molten matte into the converter, sampling was carried out every 30 min, and a total of 7 samples were obtained. The molten sample was obtained by inserting steel into the melt at the air inlet. The extracted sample was water-quenched to reduce the influence of the air composition on the sample.

2.2.2. Analytical Methods

The elemental content in the sample was detected by chemical analysis, and the phase in the raw material and each sample was analyzed by an X-ray diffractometer (Rigaku TTR III, Tokyo, Japan). The phase composition of the impurity elements in the matte was determined by chemical phase analysis. The migration and transformation of each element in the sample during the converting process were analyzed by scanning electron microscopy (CIQTEK SEM5000, Hefei, China) and an Energy Dispersive Spectrometer (EDS) (Oxford Xplore30, Shanghai, China).

3. Results and Discussion

3.1. Transformation of the Main Elements during Converting

The contents of Cu, Fe and S in samples 1–7 are listed in Table 3. Figure 3 shows changes in the Cu, Fe and S contents with the time during the converting process. After oxygenating for 95 min, all the molten matte was added to the converter melt, and the Cu content in the melt increased to 80.44%. The Cu content in the melt only increased 3.68% after continued to oxygenating for 60 min. When continuing oxygen blowing for 30 min, the Cu content in the melt rapidly increased to 97.38%. The Cu content increased from 97.38% to 98.86% in the last 30 min. Compared with the trend for copper, the S content decreased slowly in the slagging stage and rapidly decreased with an increasing Cu content after 160 min. When the Cu content reached more than 97.38%, the S content decreased below 0.02% until it reached 0%. After the first feeding, the Fe content was rapidly reduced by more than 50%, then the Fe content in the melt changed little within 120 min after converting, and the Fe content was again rapidly reduced to 0.3% in the last 30 min of converting.

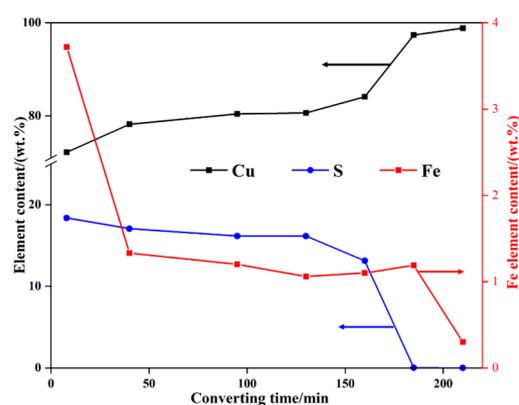


Figure 3. Variation in the Cu, Fe and S contents during the converting process.

Figure 4 shows the XRD pattern analysis of the sample taken according to the converting time in the production process of the P-S converter. The phase transformation with the converting time can be seen.

Table 3. Cu, Fe and S contents during the converting process.

Element	Cu	S	Fe
Sample 1	72.22	18.38	3.72
Sample 2	78.21	17.06	1.33
Sample 3	80.44	16.16	1.20
Sample 4	80.68	16.15	1.06
Sample 5	84.12	13.13	1.10
Sample 6	97.38	0.02	1.19
Sample 7	98.86	0	0.30

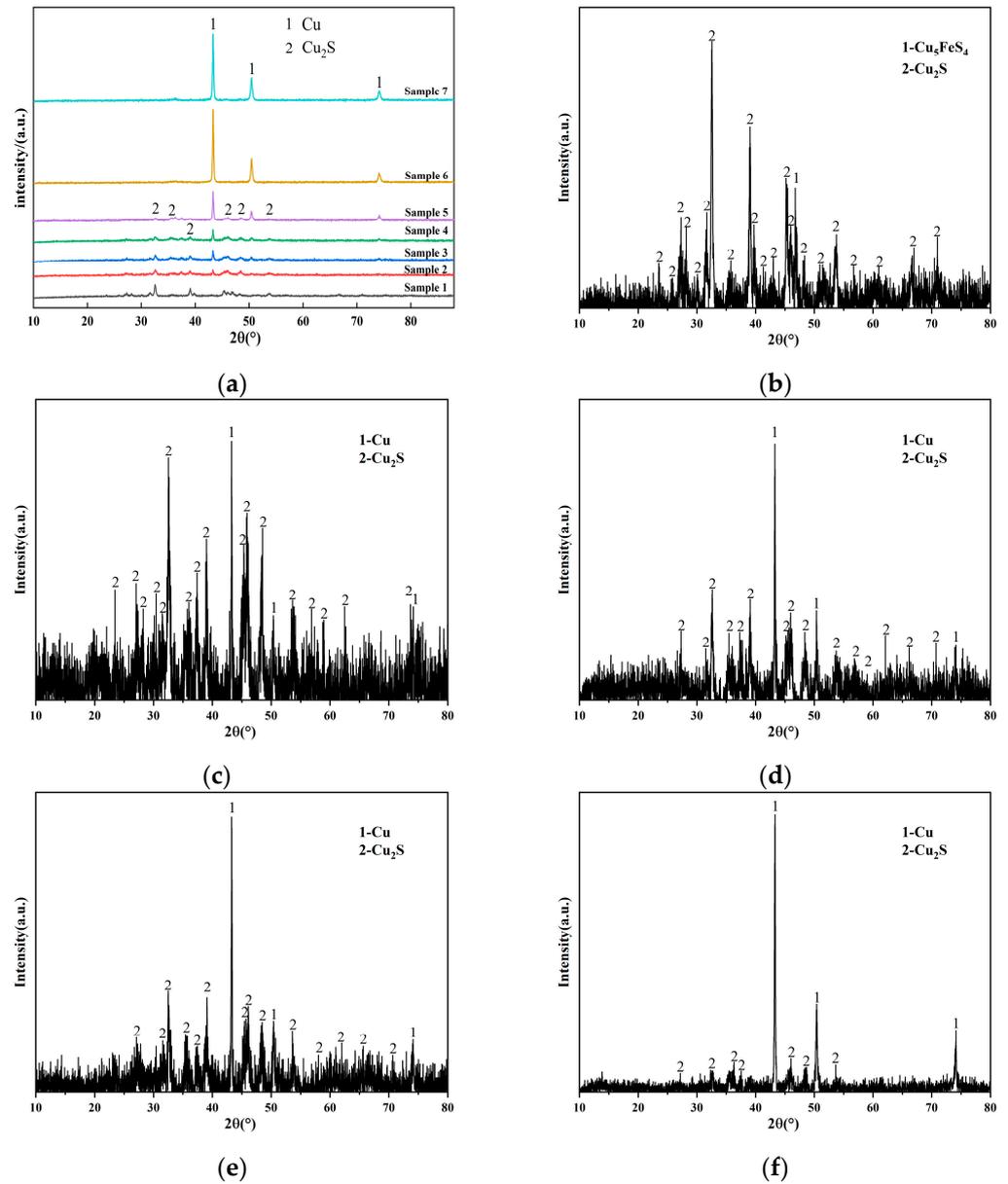


Figure 4. XRD of time sampling during the converting process. (a) XRD of all samples. (b) XRD of sample 1. (c) XRD of sample 2. (d) XRD of sample 3. (e) XRD of sample 4. (f) XRD of sample 5.

The temperature was maintained at around 1200 °C (± 10 °C) during the converting process. Sample 1 was collected when air was blown. As shown in the figure, the diffraction peak of Cu_2S in sample 1 was the strongest, and there was no phase of Cu_2O or metallic copper, so the main component of the raw material used for converting was Cu_2S .

The phases of samples 2–5 in the melt were mainly the gradually decreasing Cu₂S and increasing amounts of copper, which was due to reactions in the melt pool (Reaction (1)) and (Reaction (2)). The sampling time of sample 2 occurred before the fourth package was added, and the XRD pattern showed the presence of the Cu phase, indicating that copper formation reaction had occurred in the melt. It could be considered that there was no slagging stage period or copper formation period, with clear time boundaries in the process of high-grade matte conversion.



In the process of high-grade matte conversion, the iron was first oxidized to magnetic iron and floated to the surface of the melt, resulting in a rapid decrease in the content of Fe in the melt and Cu₂S in the melt pool. After oxygenating for 100 min, the matte was added, and the Cu concentration in the melt reached 80.68%. After further converting, the melt was a mixture of Cu₂S and Cu₂O, and the proportion of Cu₂S decreased with increasing converting time. After oxygenation for 60 min, a large amount of copper reacted, sulfur escaped as SO₂, and the Cu grade in the melt began to increase rapidly. In the last 30 min, Cu₂S was completely transformed into Cu, the grade reached more than 98.86%, the iron in the melt was greatly reduced, and the blister copper was stratified with the slag.

3.2. Transformation of Impurity Elements during Converting

3.2.1. The Form of Impurity Elements in the High-Grade Matte

Table 4 lists the chemical phase analysis results of the high-grade matte. Evidently, As is mainly in the form of As₂S₃ and FeAsO₄. FeAsO₄ is easily decomposed into Fe₃O₄ As₂O₃ and CO₂ under converting conditions. There is also a small amount of As in the matte in the form of As₂O₃ and others. Sb mainly exists in the form of element Sb, Sb₂O₅ and Sb₂O₃ in the raw material, and a small part exists in the form of Sb₂S₃. Pb mainly exists in the form of PbS, PbO and element Pb in the raw materials, and a small part exists in the form of PbSiO₃.

Table 4. Phase analysis of impurity elements in high-grade matte.

Elements	Phase	Content/(wt.%)	Proportion
Arsenic	As ₂ O ₃	0.003	1.28%
	As ₂ S ₃	0.160	64.00%
	FeAsO ₄	0.086	34.40%
	others	0.001	0.40%
	Total	0.250	100%
Antimony	Sb ₂ O ₃	0.008	11.11%
	Sb	0.044	61.11%
	Sb ₂ S ₃	0.003	4.17%
	Sb ₂ O ₅	0.017	23.61%
	Total	0.072	100%
Lead	Pb	0.240	17.60%
	PbS	0.660	47.82%
	PbO	0.370	26.85%
	PbSiO ₃	0.100	7.40%
	Total	1.380	100%

Figure 5a shows the microscopic morphology of sample 1 (high-grade matte) in the converting process at 2000× magnification, and Figure 5 shows the distribution of the elements around the impurity elements and the energy dispersive spectrometer analysis when the local area of the high-grade matte was amplified by 10,000× magnification. In the early stage of converting, the enrichment area of the impurity elements in the high-grade

matte was relatively dispersed, the area was small, and the accumulation area of a few impurities was generally less than 3 μm . The analysis showed that most of the impurity elements in the high-grade matte were dispersed in the melt. The distribution of elements in the sample, combined with EDS analysis, showed that the main phase of the high-grade matte was Cu_2S . In the copper-poor region, it is a substance or compound of impurity elements, including Fe, As, Sb and Pb. Table 4 lists that the content of FeAsO_4 in matte was 0.086%. Due to the low content, the FeAsO_4 phase may not be shown in Figure 5. In addition, the FeAsO_4 may decompose into other phases. The impurities arsenic and antimony were enriched at the oxygen enrichment points, and their distribution in other areas was relatively dispersed, so the main phases of arsenic and antimony may be As_2O_3 , Sb_2O_3 , and small amounts of As_2S_3 , Sb_2S_3 , and Sb. The distribution of the impurities overlapped with that of sulfur, except for slight enrichment in the oxygen enrichment area. The main phases of lead are PbS and a small amount of PbO.

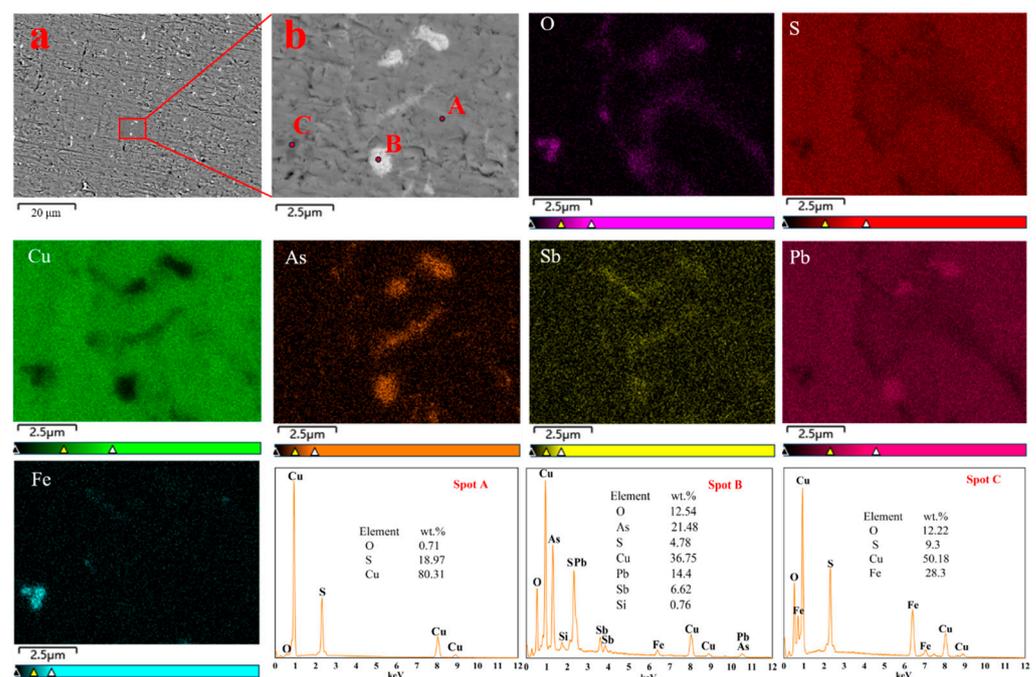


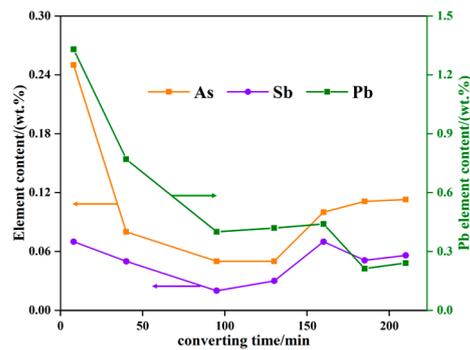
Figure 5. SEM-EDS image of the impurity distribution in the high-grade matte. (a) the microscopic morphology of the high-grade matte at 2000 \times magnification. (b) the microscopic morphology of the high-grade matte at 10,000 \times magnification. (Spot A) the high-grade matte. (Spot B) Impurity enrichment region. (Spot C) Impurity enrichment region.

3.2.2. Impurity Element Content and Distribution in Melt

Table 5 and Figure 6 show the variation in the contents of the As, Sb and Pb as the converting process of high-grade matte conversion progressed. Before 100 min of converting, the content of arsenic in the melt slowly decreased with the converting time, and after 130 min of converting, the content of arsenic gradually increased until it reached the highest value before copper production. It was found that the arsenic phase FeAsO_4 decompose and oxidized during the early stage of matte smelting, and some As_2O_3 volatilized into gas, while some As_2O_3 remained in the matte. As the sulfur in the melt was oxidized to SO_2 , the overall melt mass decreased, and the content of arsenic increased.

Table 5. As, Sb and Pb contents during the converting process.

Element	As	Sb	Pb
Sample 1	0.250	0.072	1.380
Sample 2	0.080	0.050	0.770
Sample 3	0.050	0.020	0.400
Sample 4	0.050	0.030	0.420
Sample 5	0.100	0.070	0.440
Sample 6	0.111	0.051	0.210
Sample 7	0.113	0.056	0.240

**Figure 6.** Variation in the As, Sb and Pb content during the converting process.

Before converting oxygen for 100 min, the content of Sb in the melt slowly decreased with increasing converting time. It can be seen from the continuous oxygen conversion that the antimony content in the melt gradually increased. However, after 160 min of oxygen conversion, the antimony content in the melt decreased with an increasing oxygen conversion time. In the last 30 min, the Sb content increased from 0.051% to 0.056%. The analysis showed that the elemental antimony in the matte was oxidized to Sb_2O_3 and volatilized to gas, which decreased the content of antimony in the melt with increasing converting time. As the sulfur in the melt was oxidized to SO_2 , the overall mass of the melt decreased, and the content of antimony increased. The oxygen potential of the melt increased with the continuous blowing of oxygen-enriched air during the conversion process. The increased oxygen potential led to antimony to be oxidized to Sb_2O_3 into the gas or into the slag as Sb_2O_5 . In addition, a large amount of SO_2 was produced during this period, which provides favorable volatilization conditions for Sb_2O_3 . Before the end of the converting process, the Sb content increased slightly due to the change in melt quality.

Because the lead phase in high-grade matte mainly includes PbO and Pb , some lead volatilizes into the gas during the slagging period. The quartz flux added in the middle of the converting process reacts with PbO in the melt to form $PbSiO_3$, which makes the lead content in the melt change little. When Cu_2O is present at the end of the converting process, PbS is oxidized to form volatile PbO , which enters the gas with a large amount of escaped SO_2 . The lead content in the blister copper was 0.24%, which seriously affected the subsequent smelting.

Figure 7a shows the microscopic morphology of sample 4 (white matte) in the converting process at $4000\times$ magnification, and Figure 7 shows the elemental distribution and energy dispersive spectrometer analysis of the impurities when the local area of the white matte was increased by $20,000\times$ magnification. The main phase in the melt at the end of the slagging period of high-grade matte smelting was Cu_2S . The impurity elements began to gradually accumulate in the form of oxides or compounds, and the elements Fe, As and Sb exhibited a relatively obvious aggregation phenomenon. There were impurity particles measuring approximately $5\ \mu m$, which were wrapped in the Cu_2S phase. The distribution regions of the impurity elements As and Sb were consistent and overlapped with the oxygen-enriched regions, but the distribution of As was greatly affected by Si,

while the element Sb was not. Therefore, arsenic may exist in the form of As_2O_3 or As_2O_5 , and Sb may exist in the form of Sb_2O_3 or Sb_2O_5 or form complex compounds with other impurity elements. The distribution area of Pb in the white matte overlapped more with that of S and Cu and differed greatly from that of Si. It is speculated that Pb was not oxidized at the end of the slagging period and mainly existed in the Cu_2S phase in the form of PbS. The impurity particles in the melt in the middle stage of converting were centered on SiO_2 , which was surrounded by oxides of Fe, As and Sb, while Pb was present in the Cu_2S phase in the form of PbS.

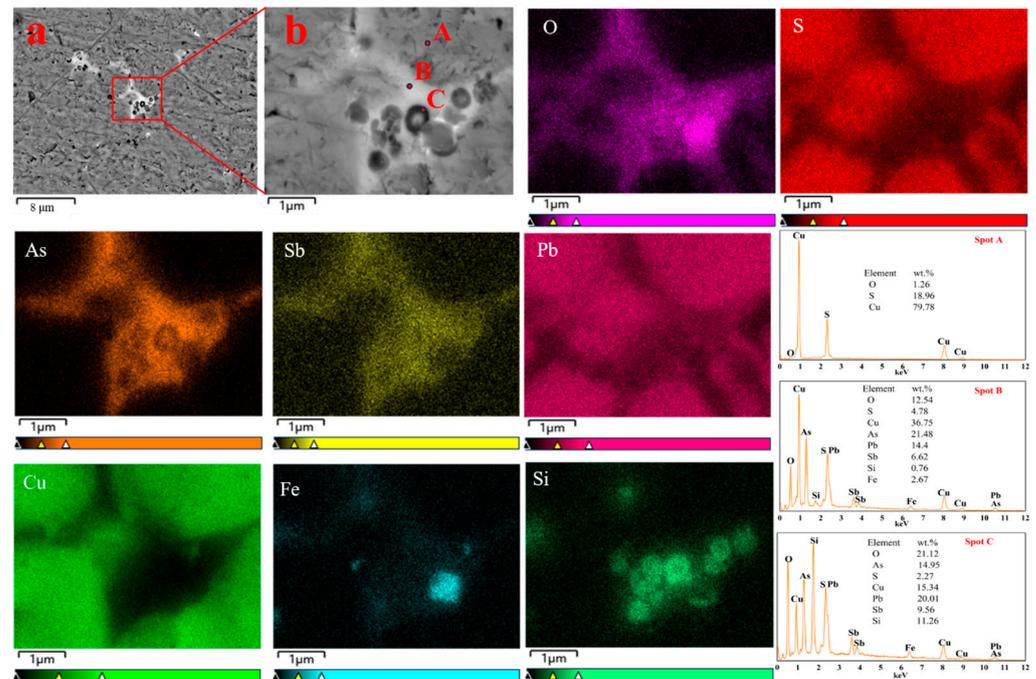


Figure 7. SEM-EDS image of the impurity distribution in the white matte. (a) The microscopic morphology of the white matte at 4000× magnification. (b) The microscopic morphology of the white matte at 20,000× magnification. (Spot A) the white matte. (Spot B) Impurity enrichment region. (Spot C) Impurity enrichment region.

Figure 8a shows the microscopic morphology of sample 6 (blister copper) in the converting process at 1000× magnification, and Figure 8 shows the distribution of elements around the impurity elements and energy dispersive spectrometer analysis when the local area of the blister copper was enlarged by 5000× magnification. The region denoted as point A is the Cu phase, point B is the matte phase, and point C is the impurity enrichment region. Before the end of the high-grade matte converting, the main phase of the product was element Cu, and the impurities As, Sb, and Pb and small amounts of Ni and Bi obviously aggregated. Most of the impurity particles were approximately 5 μm long, large particles with a radius of approximately 15 μm were also present, and impurity particles existed at the edge of the Cu_2S phase. The analysis revealed that the impurities gradually increased in abundance in the matte, and with the gradual transformation of Cu_2S to element Cu, the impurities gradually precipitated and increased in abundance. The distribution region of Pb overlapped with the distribution region of S and was not distributed in the Cu phase. It was presumed that Pb is not oxidized in matte at the end of copper converting and is not distributed in the Cu phase because PbS is oxidized after Cu_2S , which may mainly exist in the form of PbS in matte and impurity particles. The distributions of the impurities As and Sb overlapped with the distribution of oxygen, and it is speculated that As and Sb may exist in the form of oxides in blister copper.

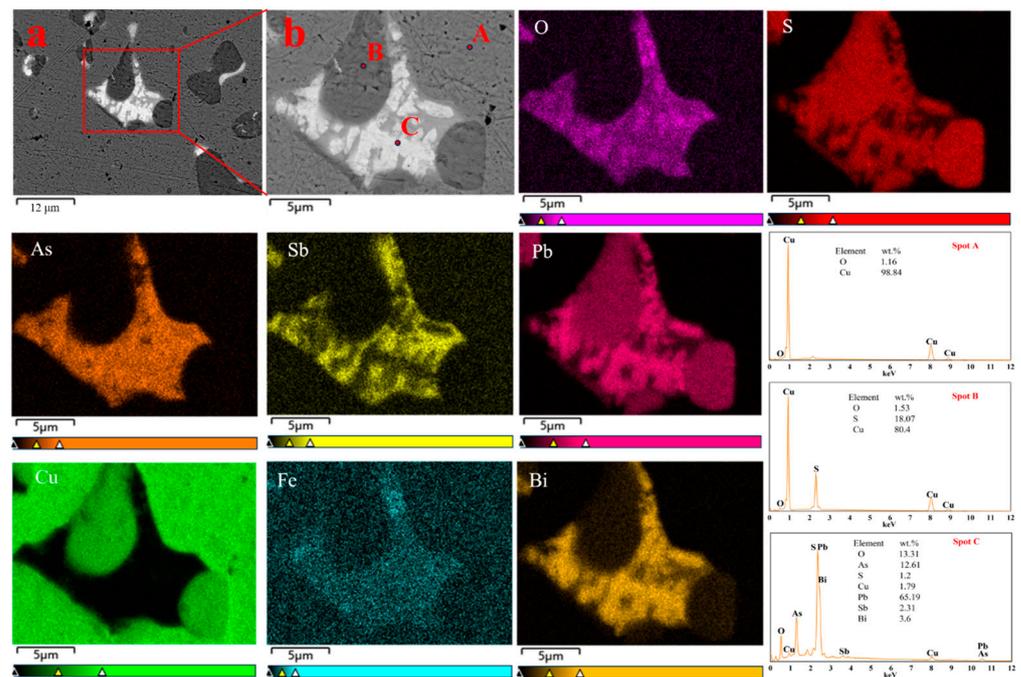


Figure 8. SEM-EDS image of the impurity distribution in the blister copper. (a) The microscopic morphology of the blister copper at 1000× magnification. (b) The microscopic morphology of the blister copper at 5000× magnification. (Spot A) the blister copper. (Spot B) the high-grade matte. (Spot C) impurity enrichment region.

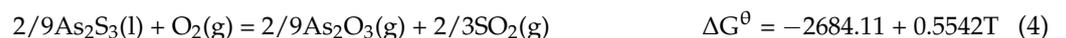
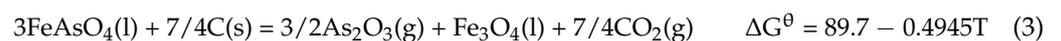
Table 6 lists the element contents of converting slag. According to the matte, converting slag quality, the quality of arsenic entering the slag phase was 1.85% of the total. The distribution ratio of lead in slag was 12.21%. Because the total amount of antimony in matte is low, antimony could not be detected in the slag.

Table 6. Chemical analysis of converting slag.

Element	Cu	Fe	S	Pb	Zn	As	SiO ₂	Ni
Content (wt.%)	16.16	38.22	0.21	1.09	1.06	0.03	18.65	0.11

3.2.3. Phase Transformation Mechanism of Impurity Elements

The phase transformation mechanism of the high-grade copper matte converting process into As is shown in Figure 9. According to the thermodynamic equation for Reaction (3), As in the form of FeAsO₄ is decomposed into As₂O₃ during the process of converting. In addition, As in the form of element As and As₂S₃ can also be oxidized to As₂O₃ (Reactions (4) and (5)).



Comprehensive analysis showed that As₂O₃ can be partially volatilized to the gas phase, and some will remain in the melt. As₂O₃ in the melt is further oxidized to As₂O₅ (Reaction (6)) when the oxygen potential is high.



As_2O_5 is an acidic oxide that can combine with other basic oxides, such as CaO , to form a stable arsenate (Reaction (7)), which easily enters the slag phase.

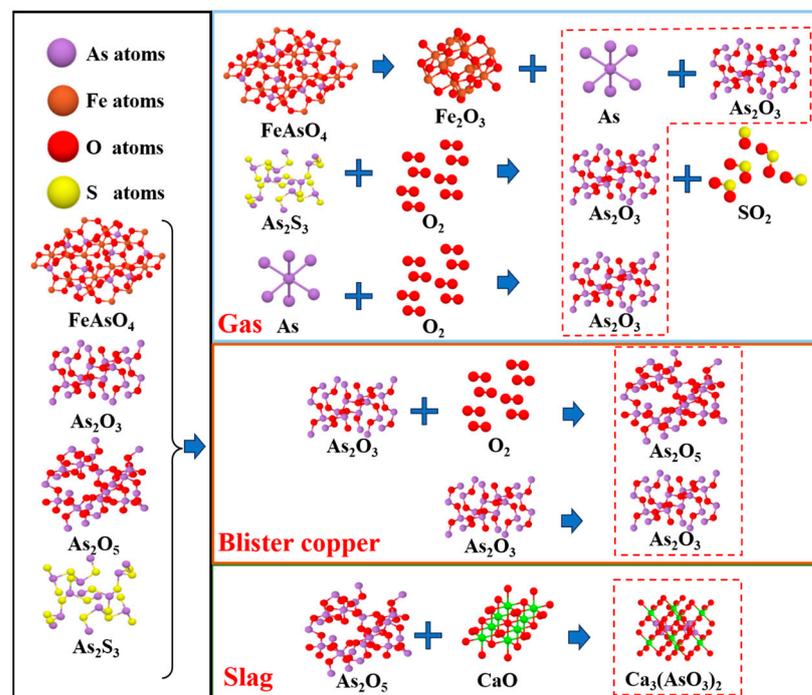
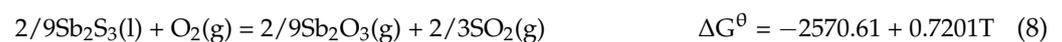
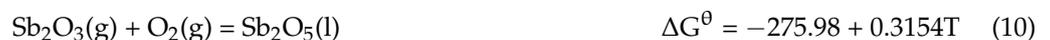


Figure 9. Migration and transformation of As during the process of conversion.

The phase transformation mechanism of the high-grade copper matte conversion process into Sb is shown in Figure 10. The antimony in the form of element Sb and Sb_2S_3 was oxidized to Sb_2O_3 (Reactions (8) and (9)) in the slagging period of converting, and Sb_2O_3 was volatile when heated.



Some unvolatilized Sb_2O_3 can be oxidized to Sb_2O_5 (Reaction (10)) in regions with high oxygen potential.



Sb_2O_5 is an acidic oxide that reacts with other basic oxides in the melt to form antimonate (Reaction (11)), which is stable, less dense, and easily enters the slag phase.



The phase transformation mechanism of the high-grade copper matte for converting Pb is shown in Figure 11. Comprehensive analysis revealed that lead in the form of PbS is oxidized to PbO (Reaction (12)) at the end of copper production. Pb, PbS and PbO in the matte can be volatilized into gas under converting conditions.



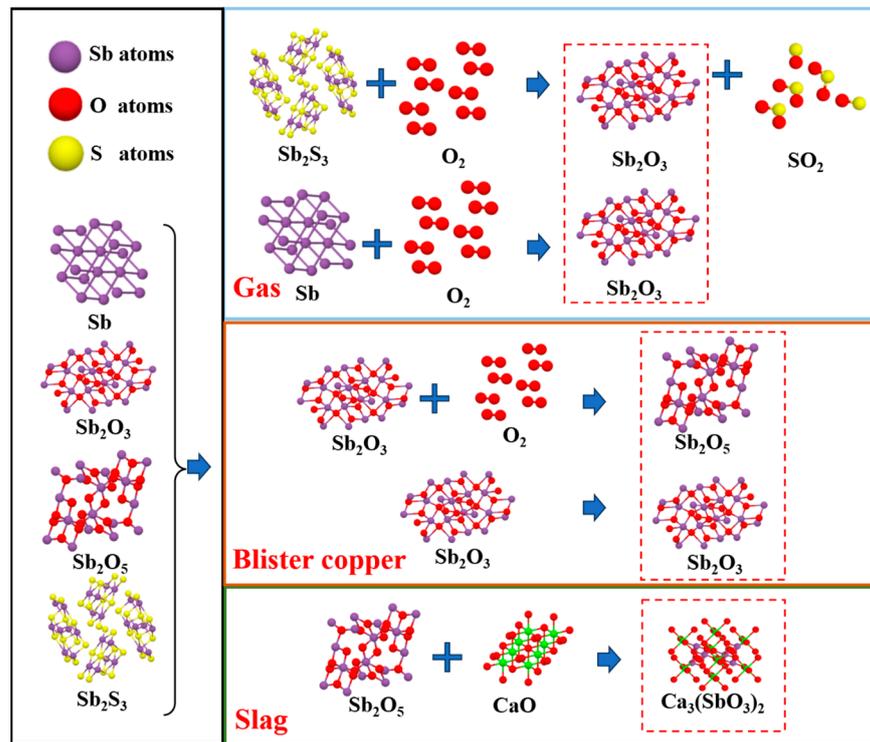


Figure 10. Migration and transformation of Sb during the process of conversion.

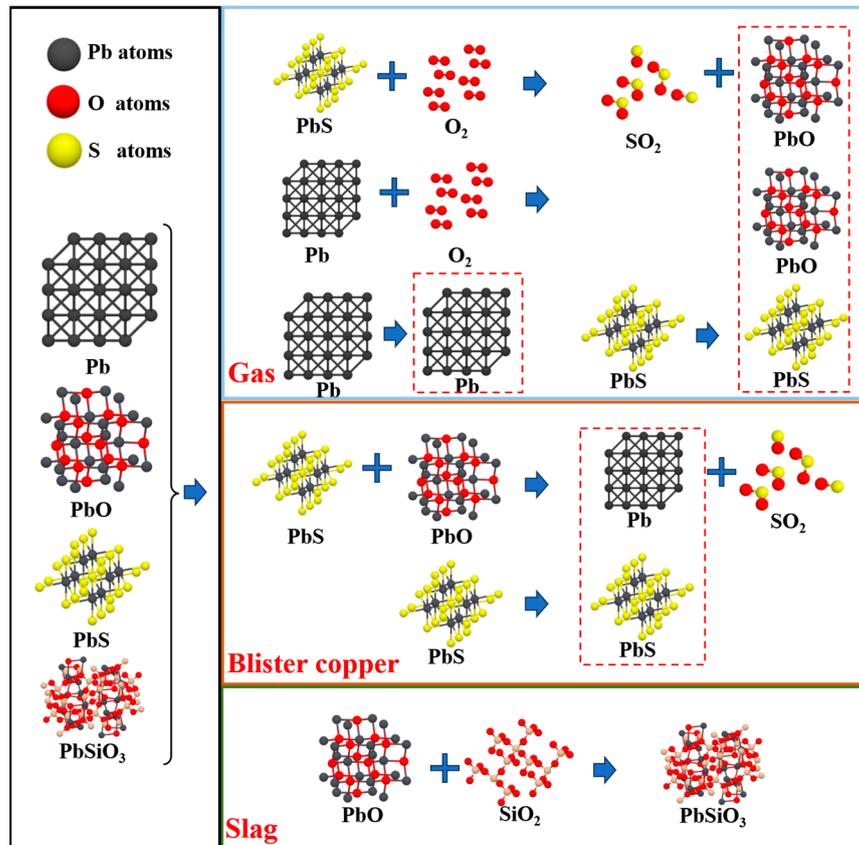


Figure 11. Migration and transformation of Pb during the process of converting.

PbO reacts with PbS to form Pb (Reaction (14)); the density of Pb is greater than that of Cu; and PbS easily remains in the blister copper to form impurities.



During the whole converting process, the unvolatilized PbO reacts with SiO₂ to form PbSiO₃ (Reaction (15)), which is the reason why the use of ferrosilicon slag in the converting process more easily removes lead.



4. Conclusions

In this paper, the changes in element content and phase in the products at each stage of the blowing process were studied. Based on the experimental results, the slagging period and the copper-making period in the process of high-grade matte converting overlap in time. Arsenic in the form of FeAsO₄ and As₂S₃ undergoes decomposed and oxidized reactions in the pre-converting stage. At the same stage, antimony in the form of Sb and Sb₂S₃ is oxidized to Sb₂O₃. In white matte, the elements As and Sb exist mainly in the form of oxides. Lead in the form of PbO leaves the melt by volatilization; in white matte it is in the form of PbS. As the converting proceeds, the impurities dispersed in the melt gradually congregate with the disappearance of the matte phase, forming particles in the blister copper phase. At the end of the converting process, the oxides of the elements As and Sb and the unoxidized PbS congregate to form complex compounds with other impurities in the blister copper. The content of Pb in the blister copper reaches 0.24 wt.%, which affects the subsequent smelting process.

Clarifying the transformation law of impurity elements in the process of converting is conducive to the targeted regulation of impurity elements, which is helpful for improving the quality of blister copper.

Author Contributions: Conceptualization, S.Z.; methodology, S.Z. and W.Q.; formal analysis, Y.W.; investigation, S.Z.; resources, B.L., Y.W. and Y.Y.; data curation, W.Q.; writing—original draft preparation, W.Q. and Y.Y.; writing—review and editing, S.Z.; visualization, W.Q.; supervision, Y.W.; project administration, W.Q.; funding acquisition, B.L. and Y.W. All authors have read and agreed to the published version of the manuscript.

Funding: This work was supported by Yunnan Fundamental Research Projects (Grant NO. 202401AT-070339) and the Yunnan Province's "Xingdian Talent Support Plan" for young talents (XDYC-QNRC-2022-0117).

Data Availability Statement: The data presented in this study are available on request from the corresponding author. The data are not publicly available due to some information that could compromise the privacy of research participants.

Acknowledgments: Thanks for the great effort of the editors and reviewers.

Conflicts of Interest: Yingbao Yang is an employee of Yimen Copper Co., Ltd. The paper reflects the views of the scientists and not the company.

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