Behaviours of Arsenic, Antimony and Lead in Phase Equilibria among Copper, Matte and Calcium or Barium Ferrite Slag

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Calcium or barium ferrite slag was brought into equilibrium with matte or copper-matte phases and the distribution behaviour of minor impurities such as arsenic, antimony and lead was investigated.

For equilibria involving high grade matte and slag, the impurities in slag can be considered as oxidic species, however, towards low matte grade they can be regarded as sulphidic or metallic species. This effect is due to the high mutual solubility between matte and calcium or barium ferrite slag when compared with the iron silicate slag system.

In matte-slag equilibria, arsenic and antimony were highly concentrated in calcium and barium ferrite slags differing from the case of iron silicate slag. With respect to lead distribution the opposite sequence was obtained, although lead is rather concentrated in the matte phase. These behaviours can be ascribed to the more acid character of arsenic and antimony contrary to the basic nature of lead oxide.

In copper-matte-slag equilibria, the distribution ratios of arsenic and antimony between slag and matte are extremely small, and follow the order of barium ferrite, calcium ferrite and iron silicate slag in decreasing sequence. For lead the highest distribution ratio corresponds to iron silicate slag followed by barium and finally by calcium ferrite slag.

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I. Introduction

The behaviour of minor elements such as arsenic, antimony and lead in copper smelting processes has important implications for the elimination of these elements before the electro-refining stage of crude copper.

The qualitative properties of alkaline slags with respect to the behaviours of impurities have been known for a long time(1)(2), but they are not fully understood because of the lack of thermodynamic data on these systems. However, interest in these slags is increasing rapidly because of their ability to eliminate poisonous impurities or to decrease the loss of valuable metals into slags(3)-(6). Recently Yazawa et al.(7) and Takeda et al.(8)(9) have reported the distribution ratios for arsenic, antimony and lead for metallic copper-calcium ferrite slag equilibria as a function of oxygen potential. However, very little is known about the equilibria involving matte phase, except for a report by Park et al.(10) for the distribution of lead among the copper, matte and calcium ferrite slag phases.

Equilibrium experiments for copper-matte-slag and matte-slag systems under controlled Ar-SO₂ atmospheres were carried out in this study in order to determine the distribution ratios of arsenic, antimony and lead among the copper, matte and calcium or barium ferrite slag phases.

II. Experimental Procedure

The experiments consisted of equilibrating metallic copper-matte-slag systems under Ar or Ar-SO₂ atmospheres and a matte-slag system under Ar–10% SO₂ atmosphere. The starting materials were prepared as described in a previous paper(11), and in the experimental set-up the gaseous atmosphere flowed at a rate
of approximately 200 cm³/min\(^{12}\).

The temperatures for experiments were 1573 K and 1523 K for the barium ferrite slag and calcium ferrite slag systems, respectively. These temperatures were chosen taking into consideration the minimum melting points of the BaO-Fe₂O₃\(^{13}(14)\) and CaO-Fe₂O₃\(^{15}(16)\) binaries. The specimens were fused in a magnesia crucible under the conditions given in Table 1 in the previous report\(^1{11}\). Three liquid phase equilibria were carried out under Ar or Ar-SO₂ atmosphere and two liquid phase equilibria under Ar-SO₂ atmosphere. Once equilibria were attained, the samples were rapidly cooled down and then separated into different phases. These were carefully inspected and cleaned by hand-grinder, particularly at the sample-crucible interface and the phase boundaries.

The copper content in matte was determined by spectrophotometric titration with EDTA and the arsenic content in copper, matte and slag phases by optical absorption. The lead and antimony contents in all phases were determined by atomic absorption.

The dissolution of magnesia in the ferrite slags was found to be very low about at 0.8%.

### III. Experimental Results

The distribution ratio of arsenic, antimony or lead among the coexisting phases will be presented as

\[
L_{\text{slag/matte}}^{\text{As}} = \frac{\% \text{As in slag}}{\% \text{As in matte}} = \left(\% \text{As}\right),
\]

\[
L_{\text{slag/copper}}^{\text{As}} = \frac{\% \text{As in slag}}{\% \text{As in copper}} = \left[\% \text{As}\right],
\]

and

\[
L_{\text{matte/copper}}^{\text{As}} = \frac{\% \text{As in matte}}{\% \text{As in copper}} = \left[\% \text{As}\right].
\]

The brackets ( ), [ ] and [ ] represent the slag, the matte and the copper phase, respectively, and the concentration of the element X is given in mass percent.

#### 1. Matte-slag systems

The distribution ratios of arsenic, antimony and lead between barium or calcium ferrite slag and matte are plotted in Fig. 1 as a function of copper content in matte, \(L_{\text{slag/matte}}^\text{As}\) for two liquid phase equilibria, plotted against the copper content in matte (\(p_{SO_2}=0.1\) atm.).

![Fig. 1 Distribution ratio of X between slag and matte, \(L_{\text{slag/matte}}^\text{X}\), for two liquid phase equilibria, plotted against the copper content in matte (\(p_{SO_2}=0.1\) atm.).](image)
2. Copper-matte-slag systems

The experimental results obtained for the three liquid phase equilibria may be represented by the distribution ratios between each two phases.

(1) Distribution ratio between slag and matte

The distribution ratios of arsenic, antimony and lead between slag and matte for copper-matte-slag equilibria are plotted in Fig. 2 as a function of copper content in matte. The distribution ratios for the iron silicate slag system\(^{(7,18)}\) are also included. In comparison with the cases of two liquid equilibria, shown in Fig. 1, the distribution ratios of arsenic and antimony in Fig. 2 are much lower, but those of lead are of a similar order. In the system of calcium ferrite slag, the distribution ratios of arsenic and antimony decrease slightly when the matte grade is increased up to about 70% Cu, but above this copper content in matte, the ratios increase drastically with increases in the matte grade. The same tendency is observed in the system of barium ferrite slag, however the minimum values in the distribution ratios are located at about 60% Cu grade. The distribution ratios of lead also show a similar tendency, increasing particularly in the region above 75% Cu in matte.

Comparing these results with those reported for the system of iron silicate slag, it can be inferred that the system involving barium ferrite slag presents the highest distribution ratios of arsenic and antimony and is followed by calcium ferrite slag and then by silicate slag. However, at a matte grade of about 80% Cu no significant difference is observed between barium and calcium ferrite systems. In the case of lead distribution, the highest ratio is shown by silicate slag followed by barium ferrite slag and finally by calcium ferrite slag.

(2) Distribution ratio between slag and copper

The distribution ratios of arsenic, antimony and lead between slag and copper phases are shown in Fig. 3 in relation to matte grade. The results for the iron silicate slag system\(^{(7,18)}\) are also included in Fig. 3 for comparison. As a general tendency the distribution ratios of arsenic, antimony and lead are almost constant at very low levels in the region of matte grade less than 70% Cu, but they increase drastically above this copper content in matte. The distribution ratio values of arsenic and antimony for the system of barium ferrite slag are very close to each other and slightly higher than those for the system of calcium ferrite and iron silicate slags. On the other hand, the distribution ratio of lead presents the highest value for the iron silicate, followed by barium ferrite and finally by calcium ferrite slag systems. The general trends in Fig. 3 are similar to those in Fig. 2.

![Fig. 2 Distribution ratio of X between slag and matte, \(L_{\text{X}}^{\text{slag}}\) for three liquid phase equilibria, plotted against the copper content in matte.](image-url)
(3) **Distribution ratio between matte and copper**

In Figs. 4, the distribution ratios of arsenic, antimony and lead between the matte and the copper phases are plotted against the copper content in matte, suggesting a general trend of concentration of these elements in copper metal. For arsenic and antimony, the highest distribution ratios are observed in iron silicate slag followed by calcium and then by barium ferrite slags. On the other hand, for lead, similar distribution ratios are obtained for both iron silicate and calcium ferrite slag followed by barium slag in decreasing order. A slight increase in distribution ratio with matte grade is observed in ferrite slag equilibria.

However in the iron silicate system an almost constant value is recognized.

**IV. Discussion**

1. **Matte-slag two liquid systems**

In the matte-slag equilibrium system, it is accepted that arsenic, antimony and a considerable fraction of lead are existing in matte in a metallic state. Thus, it is possible to describe the behaviour of a minor element, X, based on the following oxidation reaction:

\[
[X] + \frac{y}{2} O_2(g) = (X O_y). \tag{4}
\]

The equilibrium constant, \( K \), for this reaction is

\[
K = \left( \frac{a_{XO_y}}{a_X} \right) \times \frac{P_{O_2}^{2/2}}, \tag{5}
\]

where \( 2v \) is the valence of the element X. The distribution ratio, \( L^m_{X/Cu} \), can be rewritten as a function of equilibrium constant, \( K \), activity
coefficients of X in matte and slag, $\gamma_X$ and $\gamma_{XO}$, total number of moles in hundred grams of each phase, $nT$, and oxygen partial pressure, $p_{O_2}$, as follows:

$$L^m_{X} = \frac{K (n_T) \left( \frac{\gamma_X}{\gamma_{XO}} \right) p_{O_2}^{1/2}}{n_T \times (\gamma_{XO})}, \quad (6)$$

where $n_T$ is estimated at 1.48, 1.22 and 1.54 for slag, matte and metallic copper phases, respectively. By replacing the values of $n_T$, the equilibrium constant (as given in Table 1) and $\nu$, which is 1.5 for arsenic or antimony and 1.0 for lead, the eq. (6) may be rewritten:

$$L^m_{Sb} = 1.72 \cdot 10^2 \cdot \frac{\gamma_{Sb}}{\gamma_{SbO_1.5}} \cdot p_{O_2}^{0.75}, \quad (7)$$

$$L^m_{As} = 2.44 \cdot 10^2 \cdot \frac{\gamma_{As}}{\gamma_{AsO_1.5}} \cdot p_{O_2}^{0.75}, \quad (8)$$

$$L^m_{Pb} = 3.19 \cdot 10^2 \cdot \frac{\gamma_{Pb}}{\gamma_{PbO}} \cdot p_{O_2}^{0.5}, \quad (9)$$

The values of activity coefficients of oxide species in calcium ferrite slags, $\gamma_{XO}$, were reported by Takeda et al. taking account of the dependence on $p_{O_2}$ and $T$, and they are listed in Table 2. The activity coefficients in the matte phase, $\gamma_X$, for arsenic and antimony, were obtained from the work of Itagaki et al. and for lead from Watanabe et al. These values, together with those for oxygen potentials reported by Yazawa, are reproduced in Fig. 5. The data reported by Itagaki et al. were preferable to those of Roine et al., because they cover the high matte range. Substituting these values of activity coefficients and those of oxygen potential for eq. (7) to (9), the distribution ratios were evaluated as shown in Fig. 6, with solid lines for the region above 65% Cu.

For a matte grade higher than around 65% Cu, the general trends of both calculated and experimental results are the same. Accordingly, over this matte grade it is estimated that

![Figure 5](image-url)

**Fig. 5** Relationship between the copper content in matte, activity coefficient of X, $\gamma_X^{12(22)}$, and oxygen potential, $p_{O_2}$, at 1573 K and $p_{O_2}=0.1$ atm.

![Figure 6](image-url)

**Fig. 6** Calculated and experimental values for the distribution ratio of X between calcium ferrite slag and matte, $L^m_{X}$ plotted against the copper content in matte for two liquid phase equilibria ($p_{O_2}=0.1$ atm.). Dashed lines: experimental values. Solid lines: theoretical values.

Table 1 Equilibrium constant, $K$, for oxidation of arsenic, antimony and lead.

| Reaction                  | $K$ (1573 K) |
|---------------------------|--------------|
| Sb$_{(3)}$ + 3/4O$_2$ = SbO$_1.5$ | 1.42 $\times 10^5$ |
| As$_{(3)}$ + 3/4O$_2$ = AsO$_1.5$ | 2.01 $\times 10^5$ |
| Pb$_{(3)}$ + 1/2O$_2$ = PbO | 2.63 $\times 10^5$ |

Table 2 Thermodynamic data for activity coefficients, $\gamma_{XO}$, in calcium ferrite slag (referred to 1573 K).

| Matte grade [%Cu] | Activity coefficient, $\gamma_{XO}$ |
|-------------------|-------------------------------------|
|                   | AsO$_{1.5}$ | SbO$_{1.5}$ | PbO |
| 50                | 0.028       | 0.33        | 2.99 |
| 62                | 0.032       | 0.37        | 2.95 |
| 70                | 0.034       | 0.38        | 2.95 |
| 80                | 0.068       | 0.57        | 2.94 |
Behaviours of Impurities among Copper, Matte and Ferrite Slag

most of the arsenic, antimony and lead is dissolved as oxide in the slag phase and as metallic in the matte phase in accordance with the hypothesis postulated in eq. (4). At a matte grade above 65% Cu, the behaviour of lead is governed only by an increase in the oxygen potential, $p_{O_2}$, while, for arsenic and antimony, a drastic decrease in the activity coefficients, $\gamma_{As}$ and $\gamma_{Sb}$, towards higher grade matte results in overcompensation for the increment in oxygen potential.

For lower matte grades, below 65% Cu, the curves calculated on the assumption of oxidic dissolution and the ones fitted to the experimental values are in disagreement with each other. For these experimental curves a decrease in $L_{x/m}$ with decrease in copper content in matte is observed in ferrite slag systems. This discrepancy in behaviour can be discussed based upon the mutual solubility between matte and ferrite slags.

For matte grades less than 70% Cu, the mutual dissolution between matte and ferrite slags increases more drastically than with silicate slag systems, and the matte and the slag tend to form a homogeneous single phase below 40% and 48% Cu in matte for calcium(10) and barium(11) ferrite slag systems, respectively, where the distribution ratios $L_{x/m}$ can be assumed as unity. Thus, the behaviour of distribution ratios obtained for matte grade from 50% to 65% Cu may be explained qualitatively by the effect of mutual solubility between matte and slag. If we assume equilibrating slag and matte to consist only of oxide, $\{MOv\}$, and sulphide, $\{MSv\}$, respectively, and the distribution ratio of a minor element X, $L_{x/m}$, is (%s in slag)/ (%m in matte), we may consider that there is no mutual dissolution between oxide and sulphide phases. However, when the contributions of dissolving matte in slag, (%MSv), and dissolving slag in matte phase, (%MOv), are taken into account, the distribution ratio may be written as a first approximation:

$$L_{x/m} = \frac{(%MOv) \cdot s + (%MSv) \cdot m}{(%MOv) \cdot s + (%MSv) \cdot m}. \quad (10)$$

As shown in Fig. 8 and Fig. 9 in the previous report(11), it is clear that when the matte grade decreases, for example at 50% Cu, around 30-40% of sulphides dissolve in ferrite slag and about 35% of oxide + sulphide, except those of CuS$_{0.5}$, dissolve into matte in ferrite slag systems. Based on these data the sulphide content in the present slag was estimated(11), and the oxide content in the matte phase was calculated in consideration of the fact that barium, calcium and iron in matte were present mostly as sulphide, as shown in Fig. 7. Similar tendencies for the dissolution of oxides in matte were found on the assumption that the ratio of iron sulphide to iron oxide is a function of the matte grade(25) or by estimation of the content of iron oxide species in matte(26).

Selecting the maximum values of $L_{x/m}$ for arsenic and antimony and the minimum value of $L_{Pb/m}$ evaluated at 65% Cu matte by using eq. (7) to (9) and substituting these for eq. (10), together with those for (%MSv) and (%MOv) from Fig. 7, the values for "s" and "m" were calculated for a given value of %s + %m. Thus, the distribution ratios of As, Sb and Pb at lower copper contents in matte were evaluated as shown in Fig. 6. Although these results cannot be very precise quantitatively, it is clear that the calculated distribution ratios from eq. (10) agree, in their general tendencies, with the experimental results.

Although the metallic dissolution of arsenic and antimony in matte was postulated in the above discussion, the existence of arsenic and antimony as sulphide(27), even in despite of
their low affinity for sulphur\(^{(28)}\), cannot be fully denied.

2. Copper-matte-slag three liquid systems

In respect to copper-matte-slag equilibria, the distribution ratio between slag and matte may be also derived from eq. (6) as follows:

\[
L_s^{m} = 1.21 \cdot K \cdot \frac{[y_s]}{[y_s^{0}]} p_0^{\gamma / 2} \tag{11}
\]

By replacing the values of activity coefficients in matte\(^{(21)}\) and slag\(^{(8)}\) phases, oxygen potentials as reported by Yazawa\(^{(23)}\) for silicate slag system, and the equilibrium constant from Table 1, the data in Table 3 were calculated. From this table almost constant and low values for the term \(L_s^{m} \) are observed in the range 50-70% copper in matte, being similar to those found in the present study. The abrupt change in distribution ratio at about 70% matte grade may be explained by the oxidizing conditions.

The distribution ratio between slag and copper phase in three liquid phase equilibria can be analyzed by considering eq. (4) where "X" corresponds to dissolution in metallic phase. Taking into account the values for \((n_T)\) and \([\eta_T]\) explained in relation with eq. (6) we obtain

\[
L_{c/u}^{s} = 0.96 \cdot K \cdot \frac{[y_s]}{[y_s^{0}]} p_0^{\gamma / 2} \tag{12}
\]

The activity coefficients of minor elements in slag and in copper phase, quoted by Takeda et al.\(^{(8)}\), the equilibrium constant given in Table 1 and the oxygen potentials\(^{(23)}\) for these equilibria were replaced in eq. (11) and Table 4 was compiled. From these data it is observed that up to a matte grade at around 70% in copper almost constant and low values for the distribution ratios are obtained. However, above this matte grade an abrupt change similar to that found in the present study (see Fig. 3) occurred.

The distribution ratio between matte and copper can be represented by the ratio of eq. (12) to eq. (11) which may be written as

\[
L_{c/u}^{m} = \frac{L_s^{m/cu}}{L_s^{m}} = 0.79 \cdot \frac{[y_s]}{[y_s^{0}]} \tag{13}
\]

By using the data given in Table 3 and Table 4 the distribution ratio between matte and copper was calculated as given in Table 5. In the case of lead a constant value is observed. However, at very high matte grade a sudden change for the distribution ratio of arsenic and antimony occurred but at lower matte grades an almost constant value is recognized.

It can be easily shown that the general tendency agrees with the experimental results displayed in Fig. 4. Because no dependence of \(L_{c/u}^{m} \) on \(p_0\) is expected from eq. (12), the slight increase in distribution ratios with copper content in matte may be due to the change in activity coefficients, especially in the case of arsenic and antimony. However, at low copper contents in matte, especially at those where matte and slags form a homogeneous single

| Matte grade [%Cu] | Distribution ratio, \(L_s^{m/cu}\) |
|------------------|--------------------------|
| 50               | 2.88 \times 10^{-4}      |
| 60               | 4.92 \times 10^{-4}      |
| 70               | 8.65 \times 10^{-4}      |
| 80               | 2.90 \times 10^{-1}      |

| Matte grade [%Cu] | Distribution ratio, \(L_s^{m/cu}\) |
|------------------|--------------------------|
| 50               | 7.84 \times 10^{-4}      |
| 60               | 8.49 \times 10^{-4}      |
| 70               | 13.91 \times 10^{-4}     |
| 80               | 7.51 \times 10^{-2}      |
phase, the distribution ratios between slag and copper and between matte and copper should tend towards the same value because of the high mutual solubility between matte and ferrite slags.

As described above, general trends shown in Tables 3 to 5 agree with Figs. 2 to 4, but the values calculated in Tables 3 to 5 are generally around one order lower than those obtained experimentally. This may be ascribed to the fact that the concept of mutual dissolution is not considered in eqs. (11), (12) and (13).

Moreover, the estimation of the activity coefficients and the postulation of trivalent behaviour for arsenic and antimony might not necessarily be correct at the extremely low oxygen potential in the three liquid phase equilibria. Accordingly, the absolute values in Tables 3 to 5 cannot be so precise.

V. Summary

The behaviour of arsenic, antimony and lead in ferrite slag-matte and ferrite slag-matte-copper systems can be summarized as follows:

1. Matte-slag equilibria

(1) For matte-slag equilibria, arsenic and antimony are highly concentrated in the ferrite slag phase, but the contents in the silicate slag are lower than in the matte. Although lead is generally concentrated in the matte, ferrite slags reject lead much more than silicate slags. These effects are due to the acidic character of arsenic and antimony in contrast to the basic character of lead, or in other words basic ferrite slags are liable to take up acidic impurities but not so liable to take up basic ones, when compared with acidic silicate slag.

(2) For matte grades above 65% Cu, arsenic, antimony and lead exist mainly as oxic species in slags due to the higher oxygen potential, while below this matte grade they may occur as metallic or sulphidic species, partly because of the high mutual solubility between ferrite slags and matte.

2. Copper-matte-slag equilibria

(1) The distribution ratios for arsenic, antimony and lead show that they are highly concentrated in the copper phase, followed by the matte and finally by the slag phases in decreasing order. The distributions of arsenic and antimony are a little higher in ferrite slags than in silicate slag. Just the opposite tendency is observed for lead.

(2) Over about 75% Cu content in matte, As, Sb and Pb occur mainly as oxidic species in slag. On the low matte grade side these elements may be considered as metallic or sulphidic species in slag, mainly because of the rather low and constant oxygen potentials as well as the high dissolution between matte and basic slags, specially when compared with silicate slag base systems.

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