Sulfur Distribution Ratio in Iron and Steelmaking Slags

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ABSTRACT

The sulfur distribution ratio (Ls) is an expression of the amount of sulfur in slag to the amount of sulfur in molten metal. The sulfide capacities calculated from Reddy-Blander (RB) model were used to calculate the sulfur distribution ratio (Ls). A new Reddy model for sulfur distribution ratio (Ls) was developed for modelling Ls for ironmaking, steelmaking, and secondary steelmaking conditions. Besides calcium oxide, the major component in the ladle furnace (LF) slags is alumina. The Ls was calculated for typical LF slags as a function of temperature and composition. The model calculated Ls values are in good agreement with the experimental and industrial slags data. The extremely low oxygen potential in these furnaces after fully-killed steel is favourable for desulfurization.

For any given slag's composition and temperature, its sulfide capacity can be calculated *a priori*. The equilibrium sulfur distribution ratio between this slag and liquid metal in a particular vessel can also be calculated *a priori*. The Ls for ladle furnace (LF) slags is about twice those for BF slags and ten times those of BOF slags. The desulfurization of pig iron in BF and desulfurization of steel in LF during secondary steelmaking are recommended. Steel desulfurization in steelmaking furnaces is not effective. Since the model used to calculate the sulfur distribution ratio (Ls) is *a priori*, it is a useful tool for the iron and steel makers in improving the product quality and optimization of the industrial furnace operations.

INTRODUCTION

The sulfur distribution ratio (Ls) can be expressed as the amount of sulfur in slag to the amount of sulfur in molten metal. Sulfide capacity is of crucial importance to iron and steelmakers who are continuously trying to improve product quality. Sulfur drastically decreases the ductility and strength of steel, and it is undesirable except for free-cutting steels. Sulfur also causes hot-shortness and helps propagate hydrogen-induced cracks. In certain grades of clean steel, e.g. armor plates, plates for offshore oil installations and pipelines, the sulfur content should be less than 20 ppm (0.002 wt. % S) to prevent hydrogen-induced cracking. The sulfide capacities calculated from the Reddy-Blander (RB) model were used to calculate the sulfur distribution ratio.

Reddy-Blander (RB) Model for Sulfide capacity:

Reddy and Blander (RB) developed a model (Reddy and Blander, 1987, Reddy and Blander, 1989, Chen, et.al. 1989, Reddy, et.al., 1992, Reddy, 2003), for calculating sulfide capacities of binary and multicomponent aluminate and silicate slags. They showed that sulfide capacities could be calculated *a priori* based on the knowledge of the chemical and solution properties of oxides and sulfides. In another publication, Zhao and Reddy, (1995) extended the model to binary aluminate melts. Derin, et.al., (2010) applied the model to ternary silicate melts containing FeO. RB model was extended to other slag systems, titania slags (Derin, et. Al., 2004), lead industrial slags (Derin, et. Al., 2005 and 2006), multicomponent aluminosilicates (Yahya and Reddy 2011, Pelton, et.al., 1993,) and phosphates (Yang, et.al., 2014, Pelton, 2000) and sulfates (Pelton, 1999), arsenates (Reddy and Font, 2003), antimonate (Font and Reddy, 2005), and prediction of sulfur and oxygen partial pressures in copper flash smelting slags (Derin and Reddy, 2003). In all systems, excellent agreement between the model and experimental data was observed. The RB model calculated the sulfide capacities of binary, ternary and higher order aluminate slags of the system CaO-FeO-MgO-MnO-Al_2O_3-SiO_2. In this work, the RB model predicted sulfide capacities were incorporated in developing a new Reddy model for sulfur distribution ratios in iron and steelmaking slags.

Basic oxides like CaO, FeO, MgO and MnO dissociate into cations and oxygen ions and on the other hand, acidic oxides such as SiO_2 and Al_2O_3 consume oxygen ions and form complex silicate and aluminate ions. Using CaO-$AlO_{1.5}$ as an example, the binary system is divided into two regions; one basic and one acidic. The model equations used to calculate the sulfide capacity are:

Basic Binary: $0 \leq X_{AlO_{1.5}} \leq 0.33$

$$C_S = 100 \bullet M_S \bullet K_{Ca} \bullet a_{CaO} \bullet \left(\frac{1 - 2X_{AlO_{1.5}}}{\overline{M}} \right) \qquad\qquad 1$$

Where M_S is the atomic weight of sulfur and K_{Ca} is the equilibrium constant for the reaction, a_{CaO} is the activity of the calcium oxide in the binary system, and $\overline{M}$ is the average molecular weight of solution. For a given slag's temperature and composition, K_{Ca} and a_{CaO} were obtained from thermodynamic data. (Bale, et.al., 2002, Gokcen 1996).

Acidic Binary ($0.33 \leq X_{AlO_{1.5}} < 1$):

Acidic binary ($0.33 \leq X_{AlO_{1.5}} \leq 0.5$):

Acidic binaries are divided into two regions. For intermediate acidic binary (0.33 ≤ $X_{AlO1.5}$ ≤ 0.5), the solution is a mixture of polymeric species and monomer ions (Flory, 1953). The model equation for this region is:

$$C_S = 100 \bullet M_S \bullet K_{Ca} \bullet a_{CaO} \bullet exp\left(-1.25\left(1 - \frac{1}{m}\right)\right) \bullet \frac{X_{AlO_{1.5}}}{\overline{M}} \qquad 2$$

Where m is the polymer chain length given by:

$$\frac{1}{m} = (1 - a_{CaO}) \bullet \left(\frac{1}{X_{AlO_{1.5}}} - 2\right) \qquad 3$$

Acidic binary (0.5 ≤ $X_{AlO1.5}$ < 1):

For the remaining portion of the acidic binary, the solution is mostly polymeric. The model equation for this region is:

$$C_S = 100 \bullet M_S \bullet K_{Ca} \bullet a_{CaO} \bullet exp(-1.25) \bullet \frac{X_{AlO_{1.5}}}{\overline{M}} \qquad 4$$

The most complicated multicomponent system investigated in this work has six components. In this system, four oxides are basic; CaO, FeO, MgO and MnO, while two oxides are acidic; SiO_2 and Al_2O_3. To calculate the sulfide capacity of this system, it is broken down into four ternaries: CaO-SiO_2-Al_2O_3, FeO-SiO_2-Al_2O_3, MgO-SiO_2-Al_2O_3 and MnO-SiO_2-Al_2O_3. Equations 1-4 were used to calculate the sulfide capacities of each ternary system. The activity of the basic component was obtained using the same mole fractions of the acidic components as in the multicomponent system. The average molecular weight of solution is obtained for the ternary system. Instead of using $X_{AlO_{1.5}}$, $X_{AlO_{1.5}} + X_{SiO_2}$ was used, where these acidic mole fractions were the same as in the multicomponent system. Once the sulfide capacity of each ternary was calculated, the following equation was used to calculate the sulfide capacity of the multicomponent system:

$$\log C_S = y_{Ca} \log C_{S(Ca)} + y_{Fe} \log C_{S(Fe)} + y_{Mg} \log C_{S(Mg)} + y_{Mn} \log C_{S(Mn)} \qquad 5$$

Where y_i is the equivalent cationic fraction of species i. The comparison of Cs for available experimental data to RB model results of CaO-FeO-MgO-MnO-$AlO_{1.5}$-SiO_2 and several other multicomponent slag systems at various temperatures (Reddy, 2003A) were discussed. An excellent agreement was obtained between the experimental sulfide capacities to that of RB model *a priori* calculated sulfide capacities of the metallurgical process important slag systems.

Reddy Model for Sulfur Distribution Ratio

At normal iron and steelmaking conditions, sulfur is present in molten metal in a dissolved form. The gas/metal reaction is thus simply:

$$\frac{1}{2}S_{2(gas)} = [S] in\ iron \qquad \Delta G_6^\circ = -135,060 + 23.43T (J/mol) \qquad 6$$

The equilibrium constant of reaction (6) is written as:

$$K_6 = exp\left(\frac{-\Delta G_6^\circ}{RT}\right) = \frac{a_S}{p_{S_2}^{1/2}} \qquad 7$$

The sulfur in equilibrium between slag and molten metal is written as the reaction:

$$[S] + (O^{2-}) = \frac{1}{2} O_{2(gas)} + (S^{2-}) \qquad\qquad 8$$

The equilibrium constant for the reaction (8) is written as:

$$K_8 = \left(\frac{p_{O_2}^{1/2}}{a_{[S]}} \right) \bullet \left(\frac{a_{S^{2-}}}{a_{O^{2-}}} \right) \qquad\qquad 9$$

The gas/slag reaction for sulfur can then be written as:

$$\frac{1}{2} S_{2(gas)} + (O^{2-}) = \frac{1}{2} O_{2(gas)} + (S^{2-}) \qquad\qquad 10$$

The equilibrium constant for this reaction is:

$$K_{10} = \left(\frac{a_{S^{2-}}}{a_{O^{2-}}} \right) \bullet \left(\frac{p_{O_2}}{p_{S_2}} \right)^{\frac{1}{2}} \qquad\qquad 11$$

The expression for sulfide capacity (Fincham, and Richardson, 1954) is:

$$C_S = (\text{wt.\% S in slag}) \bullet \left(\frac{p_{O_2}}{p_{S_2}} \right)^{\frac{1}{2}} \qquad\qquad 12$$

Combining equations (11) and (12):

$$K_{10} = \left(\frac{a_{S^{2-}}}{a_{O^{2-}}} \right) \bullet \frac{C_S}{(wt.\% \text{ S in slag})} \qquad\qquad 13$$

Since sulfur is present in molten metal in small amounts (< 0.2 wt.%), dilute solution approximation can be used. Henry's law can then be applied to obtain the following expression for the activity of sulfur dissolved in liquid metal:

$$a_{[S]} = f_S \bullet [wt.\% \, S \text{ in metal}] \qquad\qquad 14$$

The value of the activity coefficient (f_S) can be calculated from the molten metal composition and interaction parameters. Using equations [6], [8], [10], [12] and [14] and rearranging them, an expression for the distribution ratio is obtained:

$$L_S = \frac{(wt.\% \, S \text{ in slag})}{[wt.\% \, S \text{ in metal}]} = \frac{(S)}{[S]} = \frac{f_S \bullet C_S}{K_6 \bullet p_{O_2}^{1/2}} \qquad\qquad 15$$

For a given slag composition and temperature, the Cs can be calculated using the RB model as described above. The oxygen partial pressure depends on the process and operating conditions. It can be determined by considering each process separately.

RESULTS AND DISCUSSION

1.Sulfur Distribution Ratio in Ironmaking

In the process of ironmaking, iron oxides are reduced by coke to produce pig iron. The oxygen partial pressure in such reducing conditions is low and can be determined by the following reaction:

$$\text{C (graphite)} + \frac{1}{2} O_2 \text{ (gas)} = \text{CO (gas)} \qquad \Delta G_{16}^{\circ} = -117,989 - 84.35T \text{(J/mol)} \qquad 16$$

The equilibrium constant of reaction (16) is:

$$K_{16} = \frac{p_{CO}}{a_C \bullet p_{O_2}^{1/2}} = exp\left(\frac{-\Delta G_{16}^{\circ}}{RT} \right) \qquad\qquad 17$$

The partial pressure of carbon monoxide can be calculated considering the different gases present in the blast furnace. The gases such as hydrocarbons (i. e. methane) and air, liquids (i. e. oils), and solids (i. e pulverized coal) are also injected through the tuyeres in the BF. Some plants may use certain hydrocarbons such as methane, but most gas input into the furnace is air. Air contains about 0.79 and 0.21 mole fractions of nitrogen and oxygen, respectively. Assuming that all the oxygen input into the furnace is consumed by reaction (16), two moles of carbon monoxide will be produced for each mole of oxygen. Assuming that the total furnace pressure is 1 atm, the partial pressure of

carbon monoxide can be calculated as: $p_{CO} = X_{CO} \bullet p_{Tot.} = 0.35 \bullet 1 = 0.35$ atm. For activity of carbon is unity, substituting equation (17) into equation (15), the Ls is given as:

$$L_S = \frac{K_{16} \bullet f_S \bullet C_S}{0.35 \bullet K_6} \qquad\qquad 18$$

The activity coefficient of sulfur in liquid iron (f_S) is determined from the metal composition using the following equation:

$$log(f_S) = \sum e_S^j \bullet [wt.\% j]_{in\ iron} \qquad\qquad 19$$

The term e is called interaction coefficient. Interaction coefficients slightly change with changes in temperature (Deo and Boom,1993, 1993A). A typical pig iron composition in weight percentages would be: 3 C, 1 Mn, 1 Si, 0.1 P and 0.1 S. Using equation (19), the activity coefficient of sulfur is calculated as : f_S = 2.54. The equation (18) then becomes:

$$L_S = \frac{2.54 \bullet K_{16} \bullet C_S}{0.35 \bullet K_6} \qquad\qquad 20$$

Equation (20) was used to calculate the sulfur distribution ratio of blast furnace slags in equilibrium with molten iron. The model calculated distribution ratios for liquidus slag/metal at 1400 and 1500°C are plotted against the experimental data (Filer and Darken, 1952, Hatch and Chipman, 1949) in Figure 1.
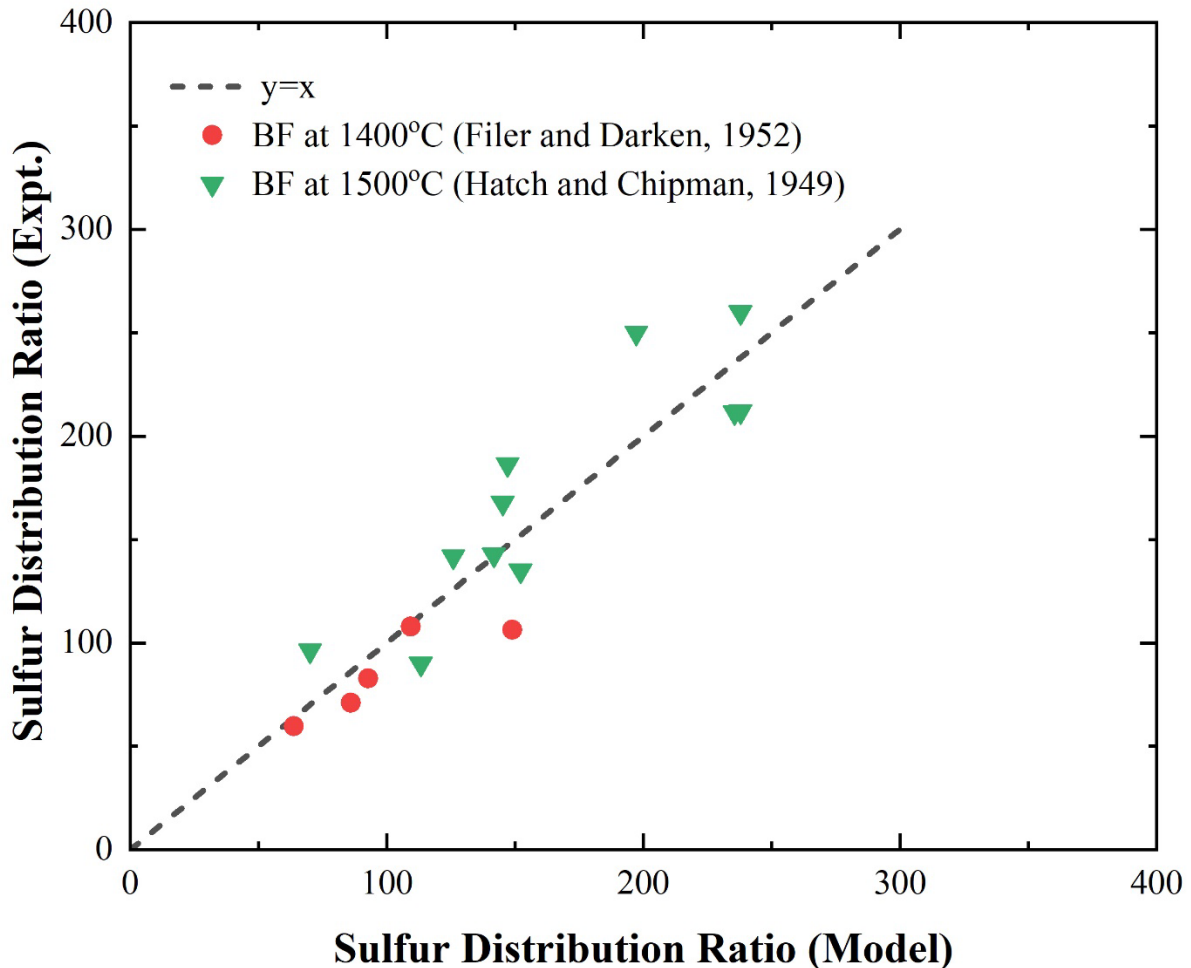

Figure 1. Comparison of experimental data and model calculated data for sulfur distribution ratio (Ls) of BF ironmaking slags and iron.

The model calculated sulfur distribution ratio data are compared with the experimental data in the BF slags and liquid iron system over a wide compositions and two temperatures summarized in Figure 1. The present model reproduces most of the experimental data within experimental uncertainties, the average of relative value for this model was found to be 14.5%. There are some

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points where the difference is noticeable. The method used here assumes equilibrium between slag and metal. It also assumes good mixing of each phase. These conditions are difficult to control and verify in laboratory experiments. Turkdogan (1996) reported that distribution ratios at turndown are about one-third to one-half of the slag/metal equilibrium values. There are two main observations that can be made from Figure 1. Increasing the temperature from 1400 to 1500°C nearly doubled the sulfur distribution ratio. The other observation is the large values of the distribution ratios, which were up to 300 at 1500°C.

2.Sulfur Distribution Ratio in Steelmaking

Steelmaking is an oxidation process to remove the oxidizable elements from hot metal and scrap to the furnace slag as the steel is decarburized with oxygen blowing. Aluminum, silicon, phosphorous and manganese are all readily oxidized into the slag phase. The BOF charge normally contains only small amounts of Al, P and Mn. The oxidized silicon makes an important component of steelmaking slags. The oxidizing conditions needed to decarburize the steel will also oxidize some of the iron. Lime is universally used as a fluxing agent to form steelmaking slags. Magnesia saturation of slag is needed to protect the furnace lining. The iron oxide goes into the slag phase and becomes an important component of slag. It follows that the main components of steelmaking slags are CaO, SiO_2, FeO and MgO. For a typical steel and steelmaking slag compositions used to determine the sulfur distribution ratio are Steel (wt.%): 0.5 C, 0.3 Mn, 0.01 Si, 0.06 S and 0.01 P; and Slag (wt.%): 47 CaO, 24 SiO_2, 15 FeO, 9 MgO, 3 MnO and 2 Al_2O_3. At or near turndown, among all other oxidation reactions, the iron oxidation reaction is what determines the oxygen potential:

$$Fe \text{ (l)} + ½ O_2 \text{ (g)} = \text{(FeO)} \qquad \Delta G_{21}^\circ = -238,070 + 49.45T \text{(J/mol)} \qquad 21$$
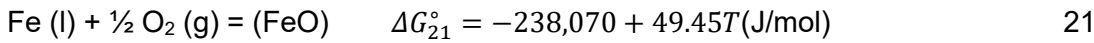
Since the calculations performed here are for slag/metal equilibrium, reaction (21) is used to calculate the oxygen partial pressure. The equilibrium constant for reaction (21) is then obtained from the equation:

$$K_{21} = \frac{a_{FeO}}{a_{Fe} \cdot p_{O_2}^{1/2}} = exp\left(\frac{-\Delta G_{21}^\circ}{RT}\right) \qquad 22$$

The equation (22) for the pressure of oxygen, activity of Fe equal to 1 and substituting into equation (15), the equation (23) can be obtained:

$$L_S = \frac{f_S \cdot C_S \cdot K_{21}}{K_6 \cdot a_{FeO}} \qquad 23$$

All parameters in equation (23) can be calculated from the temperature and compositions of slag and metal. The calculation will be performed here at 1600°C for the above slag and metal compositions. The sulfur activity coefficient was calculated as: f_S = 1.122. The activity of iron oxide was obtained (Bale, et.al., 2002) and interaction parameters (Ghosh, 2001A) and Gibbs energy (Ghosh, 2001, Gokcen 1996). The sulfide capacity of the slag was calculated using the RB model as described above. The sulfur distribution ratio was calculated as:

$$L_S = \frac{f_S \cdot C_S \cdot K_{21}}{K_6 \cdot a_{FeO}} = \frac{(1.122) \cdot (5.3430 \times 10^{-2}) \cdot (1.1383 \times 10^4)}{(3.4887 \times 10^2) \cdot (2.4105 \times 10^{-1})} = 8.11 \qquad 24$$

The equilibrium sulfur distribution value for steelmaking is 8.11. The distribution ratio reported (Turkdogan, 1983) for similar system is 9.2. The model calculated data is in good agreement with the experimental data.

3.Sulfur Distribution Ratio in Secondary Steelmaking

In secondary steelmaking, the steel produced at the BOF or EAF is treated for impurity reduction and composition control in the ladle furnace (LF). The first step taken is usually to deoxidize the steel. When the aluminum bars with steel core are added at the top of the furnace, they sink to the bottom as their density increases with aluminum melting. This process achieves very low contents of dissolved oxygen, and the product is called fully-killed steel.

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The added aluminum will dissolve in steel and react with dissolved oxygen. This oxygen partial pressure is determined by the reaction:

[Al] + ¾ O₂ (g) = (AlO$_{1.5}$) (liquid slag) ΔG_{25}° = −722,358 + 165.795T(J/mol)   25
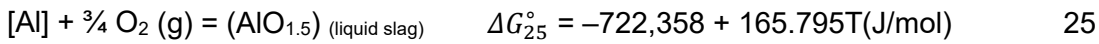
From reaction (25), The equilibrium constant is written as

$$K_{25} = \frac{a_{AlO_{1.5}}}{a_{[Al]} \cdot p_{O_2}^{3/4}}$$ 26

Rearranging equation (26) for the partial pressure of oxygen and substituting into equation (15):

$$L_S = \left(\frac{f_S \cdot C_S}{K_6}\right) \cdot \left(\frac{K_{25} \cdot a_{[Al]}}{a_{AlO_{1.5}}}\right)^{\frac{2}{3}}$$ 27

The activity of AlO$_{1.5}$ can be obtained for a given slag composition and temperature. The activity of aluminum dissolved in steel can be calculated as:

$$a_{[Al]} = f_{Al} \cdot [\%Al]$$ 28

The activity coefficient is calculated form steel composition using:

$$log(f_{Al}) = \sum e_{Al}^{j} \cdot [wt.\% j]_{in\ iron}$$ 29

A typical of steel composition (Wt%) in the ladle furnace is: 0.4 C, 0.1 Mn, 0.1 Al, 0.01 Si, 0.01 P and 0.05 S. Substituting the interaction coefficients and the composition, the activity coefficient of aluminum f_{Al} = 1.1 and activity coefficient of sulfur f_S = 1.2 were calculated. A typical LF slag composition in (wt %) is: 47 CaO, 1 FeO, 9 MgO, 1 MnO, 32 Al$_2$O$_3$ and 10 SiO$_2$. By Knowing the activity of AlO$_{1.5}$ in slag and Cs for slag was calculated using RB model at the temperature of steel in the ladle furnace, the distribution ratio calculated using equation (27) as follows:

$$L_S = \left(\frac{f_S\, C_S}{K_6}\right) \cdot \left(\frac{K_{25} \cdot f_{Al}\,[\%Al]}{a_{AlO_{1.5}}}\right)^{\frac{2}{3}} = \left(\frac{1.2\ x\ 2.5247 \times 10^{-3}}{2.7844 \times 10^{2}}\right) \cdot \left(\frac{9.1268 \times 10^{10}\ x\ 1.1\ x\ 0.1}{2.8731 \times 10^{-2}}\right)^{\frac{2}{3}} = 670$$ 30

The equilibrium sulfur distribution value for secondary steelmaking is 670. The experimental distribution ratio reported by Inoue and Suito (1994) for similar system is 663. The experimental and model data are in good agreement.

The large Ls value for LF indicates that steel desulfurization in the ladle furnace is very effective. Fully-killed steel can be desulfurized in the ladle furnace with lime-alumina slags to achieve very low sulfur contents (below 10 ppm). The sulfur distribution ratios for LF are about twice those for BF and about ten times those of BOF. It is clear that steel desulfurization in steelmaking furnaces is not effective.

In industrial operations, many factors influence the sulfur distribution ratio. These factors include slag volume, gas bubbling practice, slag skimming practice, sampling procedure, analysis techniques, sample exposure to the environment and the location and time at which the sample was taken. The values calculated using Reddy model here are to give a general idea about the actual distribution ratio. The true distribution ratio can be higher or lower, but for the most part it should be close to the model calculated value. Calculation of Ls for all three systems for an extended compositions and temperatures are in progress. Since the method used here is *a priori*, it is a useful tool for the furnace operators. They can adjust the slag composition towards one with high distribution ratio and thus have high desulfurizing power.

CONCLUSIONS

The sulfide capacities predicted using the Reddy-Blander model were used in calculation of sulfur distribution ratios for ironmaking, steelmaking and secondary steelmaking processes. The Reddy model calculated data shows good agreement with the experimental data. For a given slag's composition and temperature, its sulfide capacity can be calculated *a priori* using the RB model. Further, the sulfur distribution ratio between this slag and liquid steel in a particular vessel can also be calculated *a priori* using the Reddy model. The sulfur distribution ratios for LF are about twice those for BF and about ten times those of BOF. The desulfurization of pig iron in BF or desulfurization of steel in LF during secondary steelmaking are recommended. Since the model used to calculate

the sulfur distribution ratio (Ls) is *a priori*, it is a useful tool for the iron and steel makers in optimization of the industrial furnace operations.

ACKNOWLEDGEMENTS

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