

Phase Equilibrium Studies of Nonferrous Smelting Slags

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Pyrometallurgy is the primary technique for the production of many nonferrous metals such as copper, lead, and zinc. The phase equilibrium information of smelting slags plays an important role in the efficient extraction of metals and energy consumption. The experimental technologies used in phase equilibrium studies are compared. The presentation and applications of the pseudo-ternary and pseudo-binary phase diagrams are demonstrated in the Fe–Si–Ca–Zn–Mg–Al–Cu–S–O system.

Keywords: phase equilibrium ; nonferrous smelting slags ; Fe–Cu–Si–Ca–Zn–Mg–Al–S–O system ; liquidus temperature ; FactSage

1. Introduction

Most of the copper, lead, and zinc exist in the Earth's crust as sulfide minerals. Pyrometallurgy is the primary technique used to produce primary copper, lead, and zinc from the sulfide concentrates ^{[1][2]}. Cathode copper can be obtained from copper concentrates through the smelting, converting, fire refining, and electrorefining of blister copper ^[3]. Major smelting technologies include flash smelting and bath smelting, depending on the difference in the reaction medium and spatial distribution within the furnace ^{[4][5]}. Flash smelting predominantly involves gas–solid reactions. By injecting dried and finely ground copper concentrates, along with oxygen and flux, into a flash furnace through a concentrate burner, rapid reactions occur to form molten matte and slag ^[6]. Flash smelting has a high production capacity due to the rapid oxidation of the fine sulfide concentrate ^[7]. The flash smelting technology is developing continuously through the optimization of burner operation and slag chemistry, resulting in reduced dust generation, increased oxygen concentration, and enhanced processing capacity ^{[8][9][10]}. Bath smelting primarily involves liquid–gas reactions. Within the furnace, copper concentrates are vigorously mixed and stirred by high-pressure gas to achieve fast oxidation. Depending on the location of the tuyeres, bath smelting technologies include side-blowing, top-blowing, and bottom-blowing processes ^[11]. Bottom-blowing smelting technology offers advantages such as feed adaptability, higher gas pressure, long lance life, autogenous smelting, and low-temperature smelting. Its technical superiority has become more prominent since Fangyuan Copper Co., Ltd. developed a two-step copper smelting process ^{[11][12]}. Crude lead is obtained from lead concentrates through an oxidation process to remove sulfur and a reduction process to remove oxygen ^[13]. Similar to the smelting technology used in copper production, flash smelting ^[14] and bath smelting ^[15] technologies are the main oxidation methods employed for lead concentrates. Bath technology has replaced the sinter plant–blast furnace process due to its high thermal efficiency, less feed preparation, and efficient SO₂ utilization ^[16]. Although oxygen pressure leaching has been developed for producing zinc in recent years, pyrometallurgy is still utilized to extract zinc from lead and zinc mixed concentrate ^[17].

Significant reactions in the production of copper, lead, and zinc occur related to the slags. The properties of slag, such as liquidus temperature, solid proportion, and composition, determine the operating temperature, flux addition, refractory, and efficient recovery during the metal production process ^{[18][19]}. Phase equilibrium information of the slag is crucial for optimizing operations and developing new smelting processes. Accurate and reliable phase equilibrium data also serve as the foundation for the development of thermodynamic databases. The chemical compositions of the slags in copper, lead, and zinc production can be represented by the Fe–Cu–Si–Ca–Zn–Mg–Al–S–O system.

2. Phase Equilibrium Studies at Iron Saturation

The quenching technique is commonly employed to investigate phase equilibria of silicate-based slags, which are readily converted into glass upon rapid cooling. The compositions of the glasses can be easily measured at room temperature. The “FeO”–SiO₂–CaO system serves as the fundamental slag in non-ferrous smelting processes. Bowen et al. ^{[20][21]} and Allen et al. ^[22] utilized the quenching technique to investigate the “FeO”–SiO₂–CaO system at metallic iron saturation. In

earlier studies [20][21], a master slag was prepared by mixing and melting SiO_2 , FeC_2O_4 (or Fe_2O_3), and CaCO_3 powders in air. Small samples of the master slag were equilibrated in Fe crucibles under a stream of N_2 and then quenched. A petrographic microscope was used to identify the phases present in the quenched samples. The liquidus temperature was determined between the temperature at which the mixture was all liquid and some lower temperatures at which crystals were present in the sample.

Impurities such as MgO and Al_2O_3 are also introduced into the slag in the smelting process from gangue and refractory. Many researchers [23][24][25] have studied phase equilibria in the “FeO”– SiO_2 – CaO – Al_2O_3 and “FeO”– SiO_2 – CaO – MgO systems in equilibrium at iron saturation. The liquidus line in the “FeO”– SiO_2 –Anorthite, “FeO”– Al_2O_3 –Anorthite, “FeO”– CaSiO_3 –Anorthite, “FeO”–Gehlenite–Anorthite, and “FeO”– CaSiO_3 –Gehlenite systems at iron saturation has been studied by Schairer et al. [23][24], which reported three univariant lines in the “FeO”– SiO_2 – CaO – MgO system at iron saturation, i.e., liquid + merwinite + olivine + magnesio-wüstite, liquid + merwinite + olivine + melilite, and liquid + pseudowollastonite + olivine + melilite.

An improved methodology to accurately measure the compositions of the phases present in the quenched sample was developed with the wide application of electron probe microanalysis (EPMA) [26][27]. In these studies, the initial mixture was prepared with high-purity chemicals including excess Fe powder to ensure that the slag was equilibrated with iron at 1031–1280 °C. A small amount of mixture (0.1–0.3 g) was pelletized and equilibrated in Fe envelopes under a stream of N_2 at the target temperature. A thermocouple was placed adjacent to the sample to accurately measure the temperature. After equilibration, the sample was dropped into water to achieve fast cooling. It is easy for the small sample used in the experiments to attain equilibrium and rapid cooling. The microstructure and compositions of the phases presented in the quenched sample can be determined by EPMA.

ZnO is commonly present in nonferrous slags. However, phase equilibrium studies for ZnO -containing slag are difficult due to the vaporization of zinc under reducing conditions. Accurate phase equilibria studies for the slag systems containing zinc depend on the appropriate experimental procedure [28][29][30][31][32][33][34][35][36][37]. ZnO -containing master slags were prepared at high temperature in air. The quenched master slag was ground and mixed with metallic iron powders to obtain the final mixtures. The pellet was placed in a sealed envelope made of Fe foil or Pt foil. The mixtures were equilibrated at the target temperature under an N_2 gas atmosphere and then water-quenched to prepare the sample for EPMA analysis. The reaction between ZnO and Fe provided “FeO” for the slag. Pre-“fixed” ZnO in the matte and slag, sealed envelope, and controlled equilibration time enabled sufficient ZnO to remain in the final slag.

Al_2O_3 is commonly present in nonferrous smelting slags and comes from ores, coal ash, and refractory. Zhao et al. [30][31][32][33][34] systematically investigated the phase equilibria in the “FeO”– SiO_2 – CaO – Al_2O_3 – ZnO system at iron saturation. The effects of CaO/SiO_2 and $(\text{CaO} + \text{SiO}_2)/\text{Al}_2\text{O}_3$ ratios on primary phase fields and liquidus temperatures were discussed. It was found that the presence of Al_2O_3 in the slag results in the formation of a spinel phase, which was consistent with the observations of industrial slags.

Effects of minor elements such as Na_2O , K_2O , MgO , and sulfur on liquidus temperatures of the “FeO”– SiO_2 – CaO – Al_2O_3 – ZnO slag at iron saturation were reported by Zhao et al. [35][36][37]. The presence of up to 1 wt% of Na_2O and K_2O did not introduce new primary phases in the composition range investigated. The size of the wüstite primary phase field was extended and the size of the spinel primary phase field was reduced with the addition of Na_2O or K_2O . Liquidus temperatures are slightly increased in the wüstite primary phase field and decreased in the spinel primary phase field by the addition of 1.0 wt% of Na_2O or K_2O [35]. In contrast, the presence of MgO in the slag can increase the liquidus temperatures significantly in both wüstite and spinel primary phase fields.

3. Phase Equilibrium Studies under Controlled Po_2

It is relatively easy to conduct high-temperature experiments at iron saturation. Iron can be used as a container and to control the oxygen partial pressure. However, Po_2 at iron saturation is around 10^{-10} atm, which is much lower than that in the process of nonferrous smelting. The mixture of CO and CO_2 was commonly used to control the Po_2 of the slag system through gas–liquid reactions [38][39][40][41][42][43][44]. The CO/CO_2 ratio at a given temperature and Po_2 was calculated by FactSage. The gas flow rate was accurately controlled by the flow meters and confirmed by a DS-type oxygen probe. As gas is involved in the reactions, it takes longer for the slag samples to attain equilibrium at high temperature. Inert material such as Pt was usually used as the container for the experiments. Nikolic et al. [42][43][44] studied the phase equilibrium of the “FeO”– SiO_2 – CaO system at 1150–1350 °C under Po_2 from 10^{-5} to 10^{-9} atm. The primary phase fields determined in the composition range investigated included tridymite, wollastonite, pseudo-wollastonite, olivine, and wüstite. Wüstite will convert to spinel at high Po_2 .

The effect of oxygen partial pressure on the 1300 °C isotherms of the “FeO”–SiO₂–CaO–MgO and “FeO”–SiO₂–CaO–Al₂O₃ slags was investigated by Zhao et al. [45] at $P_{O_2} = 10^{-5}$ – 10^{-9} atm. Wüstite, spinel, and tridymite were found to be the primary phase fields in the composition range investigated. Henao et al. [46] reported the phase equilibria of the “FeO”–SiO₂–CaO–MgO–Al₂O₃ system at $P_{O_2} = 10^{-8}$ atm and 1250 to 1350 °C. This study focused on the tridymite primary phase field at a fixed amount of 3.3 wt% of Al₂O₃ or 3.3 + 3.3 wt% of MgO + Al₂O₃. Xie et al. [47] studied 1200 °C isotherms of the “FeO”–SiO₂–CaO–MgO–Al₂O₃ system at a fixed P_{O_2} of 10^{-8} atm. Effects of up to 6 wt% of MgO and/or Al₂O₃ on the isotherm positions were discussed. The experimental results were also compared with the FactSage 8.2 predictions.

Phase equilibrium studies of the ZnO-contained slag systems under controlled P_{O_2} have been conducted by Liu et al. [48] [49] [50] [51] [52]. The P_{O_2} was fixed at 10^{-8} atm which is related to the copper smelting slags. The systems investigated included “FeO”–SiO₂–ZnO [48], ZnO–“FeO”–SiO₂–Al₂O₃ [49], ZnO–“FeO”–SiO₂–CaO [50] and ZnO–“FeO”–SiO₂–MgO [51].

4. Phase Equilibrium Studies under Controlled P_{O_2} and P_{SO_2}

In the sulfide smelting process, sulfur is in equilibrium between the slag and gas phases. It is more accurate to investigate the phase equilibria by involving sulfur. In the case of the copper smelting process, liquid matte is also in equilibrium with slag and gas. The experimental data close to the operating conditions can be obtained by equilibrium experiments under controlled matte grades and P_{O_2} and P_{SO_2} . The experimental procedure was also more complicated [53] [54] [55]. The oxide mixture was prepared by mixing the required SiO₂, CaO, MgO, Al₂O₃, and pre-conditioned Fe₃O₄, which was prepared from Fe foil at the same P_{O_2} and temperature as the equilibrium experiments. The required matte was prepared by mixing and melting the Cu₂S and FeS mixture under Ar atmosphere. The open substrate was made from the primary phase of silica or spinel. FactSage was used to calculate the SO₂/CO/CO₂ ratios to obtain the target P_{O_2} and P_{SO_2} at the required temperature. The oxide pellet and matte were placed on the substrate to react at the required temperature for at least 16 h under the flow of SO₂/CO/CO₂. After quenching, the compositions of all phases present in the samples including liquid slag, oxide crystals, and matte were measured by EPMA.

Phase equilibria of copper smelting and converting slags have been extensively studied involving slag, matte, and gas [53] [54] [55] [56]. The correlations of matte grade with P_{O_2} , P_{SO_2} , and temperature were reported by Chen et al. [53]. The experiments were carried out using a spinel substrate. The databases selected in FactSage 7.2 were “FactPS”, “FToxide”, and “FTmisc”. The matte grade increases significantly with increasing P_{O_2} at a fixed temperature. The increment in the matte grade is more sensitive at high temperature. However, at a fixed P_{O_2} , the matte grade decreases with increasing temperature and P_{SO_2} . When the matte grade and P_{SO_2} are stable, the liquidus temperatures in the spinel primary phase field increase with increasing P_{O_2} .

The effect of CaO and MgO on gas/slag/matte/solid phase equilibria in the Fe–Cu–Si–O–S system was investigated at fixed P_{SO_2} in the spinel primary phase by Sun et al. [57] [58] [59] [60], and in the tridymite primary phase field by Fallah-Mehrjardi et al. [61] and Sineva et al. [62].

The effect of Al₂O₃ on the matte/slag/spinel/gas equilibria in the Fe–Cu–Si–Al–S–O system in the spinel primary phase field has been investigated by Chen et al. [63] at 1250 °C and $P_{SO_2} = 0.25$ atm, and by Sineva et al. [64] at 1200 °C and $P_{SO_2} = 0.25$ atm. The presence of Al₂O₃ in the slag decreased the iron, sulfur, and copper concentrations in the slag. The effect of Al₂O₃ showed the same trends as that of MgO and CaO [64]. The combined effects of CaO, MgO, and Al₂O₃ on gas/slag/matte/solid equilibria were studied in the Cu–Fe–O–S–Si–Al–Ca–Mg system at a fixed P_{SO_2} of 0.25 atm and 1200 °C [65] and 1300 °C [66].

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