



In situ observation and numerical simulation of bubble behavior in CaO-SiO₂ based slag during isothermal and nonisothermal processes



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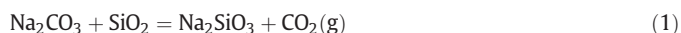
ABSTRACT

The hot thermocouple technique was used to observe the variation of bubble size in CaO-SiO₂ based molten slag at above 1273 K. Considering that the physical properties of gases change with temperature, the effect of cooling rate on the single bubble behavior was studied according to the heat and mass transfer equations. In order to investigate the distribution of pore size in solidified layer, solidification of slag was conducted in laboratory by using a water-cooled detector, and the pore size in the solidified layer was counted under the scanning electron microscope. The heat flux measured by the water-cooled detector was used as boundary conditions to establish a mathematical model that is capable of describing the characteristics of bubble size distribution. Both the calculation and the observations indicate that the time-temperature history is an important factor that could affect the bubble behavior in molten slag. For slag with no Na₂SO₄, bubbles with large size concentrate on the detector side with high cooling rate, while bubbles with small size are likely to appear on the liquid side with low cooling rate. For slags containing Na₂SO₄, bubbles with large size mainly distribute on the middle layer of the solid slag film.

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1. Introduction

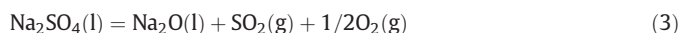
Molten slag plays an important role in metallurgical processes [1,2]. Generally, the additions of carbonates, sulphates, and fluorides could improve the fluidity of slags [3]. However, reactions among different components will generate gases with different compositions. The reaction between Na₂CO₃ and SiO₂ under 1000 K will generate CO₂, as shown in Eq. (1) [4].



If the Na₂CO₃ is excess, the surplus Na₂CO₃ may decompose into Na₂O and O₂ at higher temperature, as shown in Eq. (2) [4].



Besides carbonates, the decomposition of sodium sulphate occurs at above 1473 K, as shown in Eq. (3) [5].



In addition, the evaporation of fluoride at above 1673 K could also influence the composition and partial pressure of bubbles, as shown in

Eq. (4) [6].



Due to the chemical reactions, bubbles could easily form in molten slag during melting process. On the one hand, the growth and coalescence of large bubbles during floating are beneficial for refining process of the molten slag. On the other hand, fine bubbles with low rising velocity are easily captured by the solidified layer during cooling process, leading to the formation of porous structure in the solid slag. For a nonisothermal process, the dynamics and movement of bubbles in the liquid phase could affect the distribution of pores and physical properties of the solid phase [7]. Therefore, fundamental research about bubble behavior in molten slag is important for understanding the processes related to heat and mass transfer under high temperature.

Many experimental studies and numerical simulations were carried out to investigate the formation and dynamics of bubbles in various liquids. According to the Helmholtz free energy for the formation of critical nuclei, the nucleation rate at a given temperature for homogeneous bubble nucleation depends on the degree of supersaturation and the surface tension of bubble [8,9]. By using the thin-foil and fracture replication techniques, the mechanisms and kinetics of bubble formation could be observed under electron microscope [10]. In order to study the variation of bubble size in mass transfer process, the quasi-stationary approximation was used to predict the dissolution rates of O₂ and CO₂ bubbles in soda-lime glass melt [11]. Considering the interaction

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of gas mixtures containing SO_2 and O_2 with glass liquid, the mass transfer equations based on the diffusion mass flow on the boundary of a rising bubble were derived to calculate the bubble size as a function of time under different temperatures [12]. With the development of numerical techniques, numerous modeling studies have been conducted on the bubble behavior including bubble motion, bubble size distribution, bubble rise velocity and bubble coalescence under different ambient conditions. The Euler-Lagrange method and the Euler-Euler method are used for the phase-resolving simulation, and the motion of a single bubble can be obtained by solving its linear and angular momentum equation [13]. For the simulations of bubble diameter and velocity field, the computational fluid dynamic (CFD) technique is an effective method to study bubble behavior because this method could overcome the difficulties of determining the fluid field and concentration distribution around bubble [14]. Compared with two dimensional simulations, the volume of fluid model (VOF) has advantages in describing dynamics in bubble columns since the three dimensional VOF model show much realistic results for bubble rising [15]. Besides, the VOF model could be coupled with the level-set method to study the growth and merge of bubbles in microchannel boiling flow [16]. In addition, the direct numerical simulation (DNS) method is employed to describe the behavior of deformable bubble in bubble columns [17].

These numerical methods play an important role in determination of movement and size of bubble in different system. In most of numerical simulations, the value of temperature remains a constant during mass transfer process. However, solidification of molten slag occurs at many industrial processes, such as production of glass, coal gasification and continuous casting [24]. Due to the high temperature gradient between the liquid and mold, the maximum cooling rate of molten slag may exceed 50 K/s [18]. Because the diffusion coefficient, gas solubility, and viscosity of liquid change with decreasing temperature [19], the rate of mass transfer between gas and liquid during cooling process may be different from the bubble behavior in isothermal process. As the solidified layer evolves, the cooling rate of the liquid near the interface between the solid phase and the liquid phase decreases with the increase of the thickness of the solidified layer. Meanwhile, the cooling rate of the solid phase near the mold wall decreases with time when the heat transfer process reaches the quasi-steady state. The transient heat transfer process and the moving interface lead to the formation of a solid slag film in which pores are unevenly distributed. Therefore, it is necessary to study the effect of temperature evolution on bubble behavior in molten slag by using the heat and mass transfer coupled equations with accurate boundary conditions and thermodynamic data of chemical reactions under high temperatures.

Besides the methods used in simulations, experimental apparatus and techniques related to gas-liquid reactor are also developed to obtain the key values of parameters in calculating mass transfer rate. The high speed camera and the microscopic visualization technique have been widely used to observe the formation mechanism and kinetics of bubbles in different liquids, such as ionic liquid [20], molten slag [7], and boiling water [21]. However, it is difficult for the video techniques to record the behavior of bubble inside the vessel. In order to solve this problem, electrical resistance tomography (ERT) has been utilized to investigate the hydro-dynamics and flow patterns of bubble columns [22]. Another important reactor is gas-liquid-solid fluidized system [17]. For these three-phase processes, the direct visualization with image analysis has been developed to study dynamics of bubble in gas-liquid-solid fluidized system under high temperature and high pressure [23]. Among these techniques, the temperature of system is kept a constant, which indicates that the bubble behavior in nonisothermal process may be difficult to be observed. However, if the visualization technique could be combined with a temperature controlling system, the in situ observations of bubble behavior during fast cooling process would be achieved. Recently, the hot thermocouple technique is used to study the crystallization behavior of molten slag [25]. As a temperature-measuring device, the thermocouple could also

be used to heat slag sample, and the simultaneous measurement of temperature during heating or cooling process could be conducted [26].

From above analysis, experimental methods are conducive to in situ observation and determination of boundary conditions. Numerical simulations are important for quantification of bubble size and bubble movement. The aim of this study is to investigate the characteristics of single bubble in molten slag by numerical simulations and experimental methods, especially for the bubble behavior during nonisothermal process. In this work, the single hot thermocouple technique was used to study the effect of cooling rate and composition of slags on the behavior of single bubble during cooling process. Solidification for molten slag was conducted in laboratory by using a water-cooled detector, and the solid slag film obtained from the detector was used to analyze the bubble size distribution in the solidified layer. Meanwhile, the values of heat fluxes measured in experiments were used as the boundary condition of heat transfer. Furthermore, based on the finite element analysis, the mathematical model coupled for both heat and mass transfer was proposed to describe the bubble size distribution in slag film, and the calculated results were compared with the statistical results observed under the scanning electron microscope (SEM).

2. Experimental

The target compositions of slags used in experiment are listed in Table 1. The mass fractions of raw materials in each slag are listed in Table 2. The five slags used in experiment were synthesized by CaO , SiO_2 , Al_2O_3 , Na_2SO_4 , Na_2CO_3 , Na_2SiO_3 , CaF_2 and Li_2CO_3 . Pure chemical reagent was used as raw material of each component. The purity of each reagent exceeds 99%. In order to study the effect of carbonate and sulphate on bubble behavior in molten slag, the contents of Na_2CO_3 and Na_2SO_4 in the five slags are different. Slag 1 contains no Na_2SO_4 , and the Na_2O is prepared by adding Na_2CO_3 and Na_2SiO_3 . For slag 2, slag 3, and slag 4, the content of Na_2CO_3 in the three slags is the same, but the contents of Na_2SO_4 are different. Slag 5 contains no Na_2CO_3 , and the content of Na_2SO_4 is 4.5%. The Heat Flux Simulator developed by Wen et al. was used to measure heat fluxes through the solidified layer [27], and the schematic of the apparatus is shown in Fig. 1. The appearance of the solid slag film attached on the water-cooled detector is shown in Fig. 2.

At the beginning of the experiment, an empty crucible was heated in the MoSi_2 furnace with a heating rate of 6 K per minute. After the temperature of the furnace reaches 1673 K, 350 g of powder were added into the graphite crucible. Despite the low heating rate of the furnace, the melting process of the slag will take no > 10 min due to the high electric powder and the high temperature of the furnace. When the temperature increases to about 1200 K, gases generated from the decomposition of Na_2CO_3 begins to dissolve due to the formation of liquid slag. In the initial stage of melting, due to the high viscosity of liquid slag, the floating speed of bubble is very slow, which indicates that bubbles with small size could remain in the molten slag. The temperature of molten slag will be maintained at 1673 K for 10 min so that a homogeneous pool could form. Before the beginning of the solidification test, a locating rod will move downward automatically until the fluid level of the molten slag is detected. When the position of fluid level was determined, a water-cooled detector was immersed into the molten slag. The initial temperature of cooling water is kept at $308 \text{ K} \pm 0.5 \text{ K}$. The flow of cooling water is maintained at $0.056 \text{ kg} \cdot \text{s}^{-1}$. When the top surface of detector and the fluid level of molten slag are on the same plane, the temperatures of cooling water at inlet and outlet are recorded by the computer. As the solidification process evolves, the heat of molten slag

Table 1
The target compositions of slags used in experiment.

CaO	SiO_2	Al_2O_3	Na_2O	F	Li_2O	Total
40.0	35.6	6.7	9.0	7.8	1.0	100.0

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