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Original Article

Densification mechanism of porous alumina plugs by molten steel with different oxygen levels

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ABSTRACT

To investigate the densification of porous alumina plugs, hydrogen measurements, using the Hydris probe system, were performed in molten steel with different oxygen levels at around 1600 °C. The oxygen contents in molten steel were controlled by Al and Fe-Si alloy additions to 664, 296, 92, 51 and 2.5 ppm, respectively. High oxygen levels (> = 92 ppm) in molten steel were found to favor the infiltration of steel into porous alumina plug. The infiltrating steel interacts with SiO_2-containing phases and Al_2O_3 inside the plug, forming liquid FeO_n-Al_2O_3-SiO_2 slag and $FeAl_2O_4$ (hercynite). These newly formed phases, along with the infiltrating steel fill the porous structure of the alumina plugs. As a result, severe densification was observed inside the alumina plugs in contact with molten steel containing high oxygen levels. In comparison, no densification occurred in the plugs contacting with the steel having low oxygen contents of 51 and 2.5 ppm.

1. Introduction

Porous alumina ceramic has been widely used in primary steelmaking (EAF and Converter shop) and ladle metallurgy (RH and Tundish practice) as ceramic bells for hydrogen measurements (Hydris probe system) [1–3], ceramic filters [4,5] and purging plugs [6,7], owing to its low cost and the high melting point of Al_2O_3. However, a drawback of using porous alumina ceramics is their vulnerability to steel with high oxygen levels. For example, using the Hydris system (hydrogen probe), the hydrogen content of liquid steel is measured by means of determining the hydrogen concentration in a nitrogen carrier gas. The carrier gas is injected into the steel through the probe to absorb the hydrogen until equilibrium is attained between gas and steel, and is then recollected by a porous alumina plug and then analyzed in the pneumatic unit (Fig. 1). However, it has been observed that the alumina plug in such measurement suffers from different levels of densification, thereby affecting the recirculation of carrier gas and thus the accuracy of the measured hydrogen concentration. Therefore, knowledge of the densification mechanism of alumina plugs is indispensable in improving the lifetime of porous alumina ceramic and controlling the steel quality and operation safety during ironmaking and steelmaking processes.

Interactions between dense alumina and liquid steel have been reported in a number of studies [8–12]. It is reported that Al_2O_3 is unwetted by molten steel. However, Al_2O_3 will be wetted by molten steel once the steel is contaminated with oxygen and this wettability is favored by increasing the oxygen level in the steel [8–12]. $FeAl_2O_4$ hercynite can then be generated at the Al_2O_3/steel interface by the Al_2O_3/steel interaction [8–12]. It can be therefore inferred from these results that the densification of porous alumina plug may be caused by the wetting of molten steel with oxygen, which results in the infiltration of molten steel. However, until now there are no experimental studies available confirming this conjecture.

In this work, the densifying behavior of porous alumina ceramic in molten steel is investigated. The tests were carried out by hydrogen probe measurements in molten steel in an induction furnace. To examine the influence of oxygen level on the densification behavior, the oxygen concentration in the steel was controlled by adding different amounts of Fe-Si alloy and Al during the tests. The densification mechanisms are studied by the characterization of the microstructure of the porous alumina ceramics after tests, using scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS).

2. Experimental

2.1. Experiment set-up and procedure

Hydrogen measurements (Hydris probe system) were conducted in

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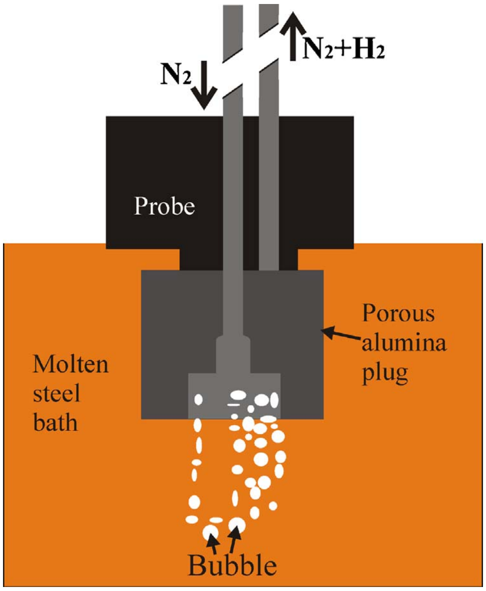

Fig. 1. Schematic of the porous alumina plug during hydrogen measurements (Hydris probe system).

an induction furnace (Inductotherm, Elphiac) at Electro-Nite (Houthalen, Belgium). Approximately 250 kg of steel was held in a refractory crucible, the inner lining of which consists mainly of MgO, and melted at around 1600 °C in the furnace. The schematic drawing of the probe for hydrogen measurement is shown in Fig. 1. A series of hydrogen probe measurements was performed by adding deoxidation agents (Fe-Si alloy and Al) to the molten steel in order to reduce the oxygen content. The first measurement was performed with an initial oxygen level of 664 ppm before the first killing. After each addition of Fe-Si alloy and Al, oxygen in the molten steel was examined by an oxygen probe and a metal sample was taken for compositional analysis with spectroscopic spark emission. After the oxygen content was found to be stable, a new hydrogen probe measurement was performed. The measured oxygen contents and steel composition are listed in Table 1. Curves of hydrogen content and recollected carrier gas pressure were recorded and displayed on the screen of the processor. From these curves one can indirectly observe the densifying extent of the alumina plug during measurements (as discussed in Section 3.2.1). After measuring for around 20s or 60s, depending on the extent of densification, the probe was removed from the molten steel and quenched in a stream of nitrogen.

2.2. Sample analysis technique

After measurements, alumina plugs were recovered for microstructural investigation. To evaluate the interaction between the alumina plug and molten steel, specimens were extracted at the upper part along the vertical direction from each recovered plug as demonstrated in Fig. 2. The specimens were embedded in a low viscosity resin

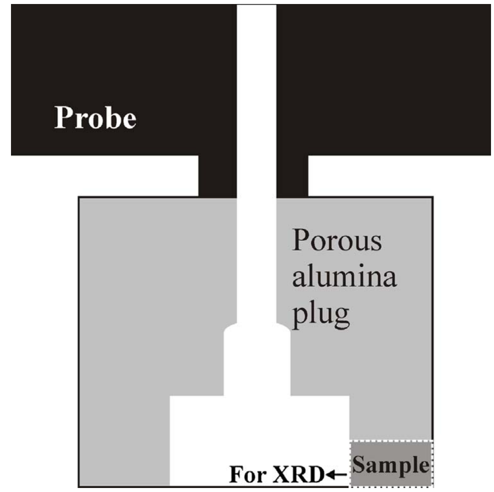

Fig. 2. Sample extraction scheme for SEM and XRD investigation.

(Epofix) by vacuum impregnation, ground with diamond plates and polished with diamond paste. The polished specimens were coated with a layer of carbon and characterized with a scanning electron microscope (SEM, XL-30 FEG, FEI), equipped with an energy dispersive spectroscope (EDS, EDAX) with an ultra-thin window. The plug surface contacting with molten steel after testing (as shown in Fig. 2) was slightly ground to keep it flat and characterized by X-ray powder diffraction (XRD, D2, Bruker) to examine phase compositions at the steel/plug interface.

2.3. Thermodynamic calculations

The thermodynamic software package FactSage 7.1 was used to support the analysis results [13]. Equilibrium calculations were performed with the equilibrium module EQUILIB, which is based on the minimization of the Gibbs free energy, and the FSstel, FT oxide and Fact PS databases. The following possible solution phases were chosen in the calculations: (1) FSstel-LIQU, (2) FToxide-SLAGA (molten oxide phase); (3) FT oxide-MeO_A (oxide solid solution); (4) FT oxide-SPINA (spinel solid solution); (5) FToxide-Mull (mullite solid solution); (6) FT oxide-CORU (corundum), and (7) FACT PS (gas phase). The steel/refractory interactions were modeled at 1600 °C by addition of one gram or two grams (x g) refractory components of Al_2O_3, mullite (71.8 wt% Al_2O_3 and 28.2 wt% SiO_2) or a SiO_2-Al_2O_3 mixture (50 wt% Al_2O_3 and 50 wt% SiO_2) to the steel [(100-x-y-z) g)] without or with 0.47wt% Si (y g). SiO_2-Al_2O_3 mixture was used to mimic the SiO_2-rich phases in the plug. The oxygen level in the molten steel ranges from 0 to 700 ppm (z g) in the calculation.

3. Results

3.1. As-delivered porous alumina plug

Fig. 3 presents the microstructure of the as-delivered alumina plug.

Table 1
Experimental parameters for the hydrogen probe measurements.

Exp. no.	Temperature (°C)	O (ppm)	Chemical composition of the steel (wt%)					
			C	Si	Ni	Cu	Fe	Others
1	1608	664	0.03	0.01	0.23	0.45	98.8	0.48
2	1626	296	0.03	0.09	0.23	0.45	98.7	0.50
3	1627	92	0.03	0.21	0.23	0.45	98.5	0.58
4	1616	51	0.03	0.31	0.22	0.45	98.3	0.49
5	1627	2.5	0.02	0.47	0.23	0.45	98.4	0.43