Rare Metal Materials and Engineering
Volume 46, Issue 5, May 2017
Online English edition of the Chinese language journal

Available online at www.sciencedirect.com
ScienceDirect

Cite this article as: Rare Metal Materials and Engineering, 2017, 46(5): 1176-1182.

ARTICLE

Effect of Electrical Conductivity and Porosity of Cathode on Electro-Deoxidation Process of Ilmenite Concentrate

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Abstract: The performance of the cathode is always closely related to the electro-deoxidation process and its current efficiency. In this work, the effect of sintering temperature on the electrical resistivity of an ilmenite concentrate cathode was measured. The results show that the sintering temperature has a significant effect on the electrical conductivity of the cathode. The electrical resistivity of the ilmenite concentrate cathode decreases with increasing of the sintering temperature and increasing of the contact area. Besides, the influence of the cathode porosity on electrochemical process during preparation of Ti-Fe alloy in molten salt was studied. The results also reveal that the porosity of ilmenite concentrate cathode directly affects the electro-deoxidation process. The increasing of porosity is beneficial to the formation of intermediate product ($CaTiO_3$) and improvement of the current efficiency.

Key words: electrical resistivity; porosity; current efficiency; electro-deoxidation; ilmenite concentrate

FFC Cambridge process is a new technique by which pure metals and alloys can be directly prepared from their respective oxides [1-6]. However, the FFC technology has not been industrialized due to its low current efficiency to achieve a low O content in the produced metal. In recent years, researches about improving current efficiency have been become a hot spot. Schwandt et al. [7] proposed that a graphite pseudo-reference electrode combines with additions of calcium oxide to improve potential control. The speed and current efficiency of the experiment were significantly increased. Chen et al. [8] attributed the overall rate of O from metal oxide to the lower metal/electrolyte interface. The reduction of TiO to Ti could be improved by increasing the pellet porosity. Fray et al. [9] also compared the current efficiency by using either a graphite anode or an oxide based anode. Actually, current efficiency is affected by many factors in electro-deoxidation process, such as reaction process, the molten salt, property of the cathode and the anode. Among these factors, the cathode plays an important role in determining the current efficiency during electro-deoxidation process. However, the cathode performance is mainly influenced by the cathode conductivity and cathode porosity.

Although the importance of cathode has been realized, very few studies have examined the effect of the cathode resistivity and cathode porosity on electro-deoxidation process. Marques[10] studied the effect of temperature on resistivity of two ceramic cathode materials ($LSCFe_3$ and $LSCFe_7$). Similarly, Zhao et al.[11] indicated that the electronic conductivity of $La_{2-x}Sr_xCuO_{4-\delta}$ cathode increased with the temperature decreasing. In regard to the study of cathode porosity, Fray[12] used highly porous titanium oxide as precursors and studied the effect of porosity on the rate of reduction. Chen[8] reported that the porosity of the TiO_2 cathode affects directly the morphology of the Ti produced by electro-reduction.

The above electrochemical reduction was studied using various cathode materials and mainly dominated by the oxide cathodes. The present work is focused on reducing ilmenite concentrate to prepare Fe-Ti alloy directly by the FFC-Cambridge process. The ilmenite concentrate cathode contains ilmenite phase, magnetite phase and few geikielite phase, which is more complicated than the pure oxide cathode. A high performance cathode is an important part to obtain high electrolysis efficiency.

Thus, the purpose of the present work is to study the effect

Received date: May 9, 2016
Foundation item: National Natural Science Foundation of China (51404044); Fundamental Research Funds for the Central Universities (106112015CDJZR135502)
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of the sintering temperature on the electrical resistivity of the cathode, and the influence of the cathode porosity on the electrochemical process. It is expected that the conductivity of the cathode is improved by controlling the sintering parameters. Besides, the increasing of cathode porosity is beneficial to the electrolysis process. The results will provide a guiding significance for improving the cathode performance and increase the electro-deoxidation rate.

1 Experiment

Ilmenite concentrate from Pan Zhihua area in China was used as the raw material. The main composition of ilmenite concentrate cathode was $FeTiO_3$. The ilmenite concentrate was mixed with a certain amount of binder and the mixture was compacted into cylindrical pellets under a load of 15 MPa. The pellets were 10 mm in diameter and 15 mm in thickness. After initial drying at room temperature for 12 h, the green pellets were sintered in a tube furnace to predetermined temperatures and durations in air atmosphere. The pellets were sintered under three different conditions: (i) 300 ^{o} C for 2 h, (ii) 600 ^{o}C for 1 h, and (iii) aim temperature for 3 h.

After sintering, the samples were polished by emery prior to measurement of resistivity of the cathode. The experimental apparatus is shown in Fig.1. Two copper sheets were put on the surface respectively to guarantee good contact with sample. A multimeter, connected with copper sheet, was used to measure the resistance of samples. To ensure sufficient contact between the sample and the copper sheet, the clamping device was introduced between the upper and lower surfaces of the sample and copper sheet. The electrical resistivity fluctuated in the beginning, while 30 min was necessary for stabilizing the values measured and obtaining the electrical resistance of sample. Meanwhile, the height and cross-section area of sample were measured. The dependence is expressed as follows:

$$\rho = RS/L \tag{1}$$

where, ρ is electrical resistivity, R represents resistance of the cathode after sintering; S represents the cross-section area of the cathode after sintering; L represents height of cathode after sintering. The mass of cathode is 10 g.

Porosity (θ) of the sintered ilmenite concentrate pellets was measured by the following mass-volume relationship.

$$\theta = \left(1 - \frac{M}{V\rho_s}\right) \times 100\% \tag{2}$$

where, M, V and ρ_s represent mass, volume, and density of the sintered pellet, respectively.

The electro-deoxidization experiment included both the pre-treatment to dry and purify the electrolyte and the electrolysis to decompose the oxide of the cathode. The pre-treatment process involved the thermal drying of the solid $CaCl_2$ and the pre-electrolysis of the molten $CaCl_2$. The solid $CaCl_2$ was dried in an oven at about 100 °C for 2 h and at 300 °C for 3 h to remove physical water and crystal water, respectively. Electrochemical reduction experiments were carried out in graphite crucible, which was dried at 150 °C before the experiments. An ilmenite concentrate pellet was used as the working electrode and a graphite rod as the anode. The interior of reactor was purged with dry argon continuously. Pre-electrolysis was carried out at 850 °C for 2 h with 2 V to remove water and some impurities further. Then the ilmenite concentrate cathode was electrolyzed in $CaCl_2$ molten salt to prepare Fe-Ti alloy at 900 °C and the voltage was increased to 3.1 V. After electrolysis, the pellet and the graphite rod were removed from the electrolyte and cooled to room temperature at top in the furnace. The pellet sample was put into the distilled water for 12 h and then cleaned by Q2200E ultrasonic cleaner to remove the electrolyte which entered into the sample. The morphology and elements distribution were observed by SEM (scanning electron microscopy) and phase composition was analyzed by XRD (Rigak D/Max-2500). The schematic diagram of electrolytic apparatus is shown in Fig.2.

2 Results and Discussion

2.1 Electrical conductivity and porosity of cathode

The electrical resistivity of ilmenite concentrate cathode is presented as a function of the sintering temperature in Fig.3. The electrical resistivity of ilmenite concentrate cathode decreases remarkably with the sintering temperature increasing. In order to illustrate the influence of temperature

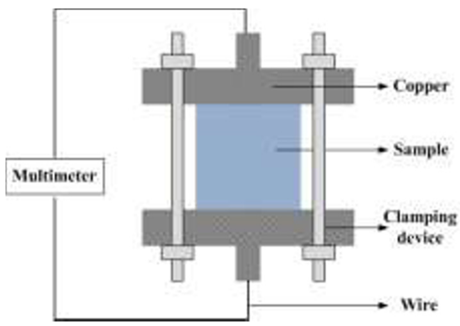

Fig.1 Apparatus for the measurement of electrical resistivity of sample

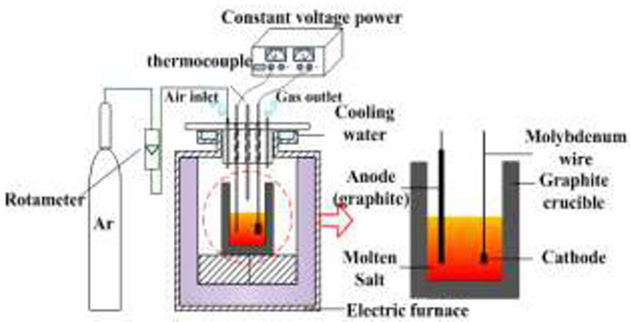

Fig.2 Schematic diagram of electrolytic apparatus

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