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Reductive kinetics of Panzhihua ilmenite with hydrogen

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Abstract: The hydrogen reduction of Panzhihua ilmenite concentrate in the temperature range of 900−1050 °C was systematically investigated by thermogravimetric analysis (TG), X-ray diffraction (XRD) and scanning electron microscopy (SEM) methods. It was shown that the products of the Panzhihua ilmenite reduced at 900 °C were metallic iron and rutile. Above 1000 °C, ferrous pseudobrookite solid solution was generated. During the reduction process, element Mg gradually concentrated to form Mg-rich zone which can influence the metallization process. The reduction reaction proceeded topochemically and its related reduction kinetics were also discussed. The kinetics of the reduction indicated that the rate-controlling step was the diffusion process. The apparent activation energy of the hydrogen reduction of Panzhihua ilmenite was calculated to be 117.56 kJ/mol, which was larger than that of synthetic ilmenite under the same reduction condition.

Key words: Panzhihua ilmenite; synthetic ilmenite; hydrogen reduction; kinetics; rate-controlling step; magnesium migration

1 Introduction

The mineral ilmenite (nominally $FeTiO_3$) is one of the primary mineral resources for producing titanium dioxide and Ti [1−3]. Panzhihua ilmenite in Sichuan, southwest of China, is one of the largest ilmenite reserves over the world with an estimated ilmenite reserve of about 8.7×10^8 t, which accounts for more than 90% of the total titanium resource of China and over 35% of the world [4−6]. Panzhihua ilmenite concentrate is a rock-type mineral that contains low-grade titania and high content of impurities (especially high content of MgO), which make it difficult to upgrade the ilmenite ore [7]. Due to its high impurity content, the reducibility of ilmenite is low and Panzhihua ilmenite is unsuitable for the chlorination process to produce TiO_2 pigment [8,9]. In a traditional method, ilmenite ore is smelted with carbon in electric furnace for preparing titanium-bearing slag. The smelting process always requires a long time and a high temperature, and the slag-forming reagents added to produce a fluid titania-rich slag will dilute the concentration of titanium dioxide in the slag and have deleterious effects on the subsequent processes of extracting titania [10,11]. Therefore, it would be beneficial to develop a direct reduction process that produces solid titania-rich slag and metallic iron, and the metallic iron can be removed from the reduced products by either leaching or mechanical separation.

In recent decades, there has a rising interesting in the direct reduction of ilmenite ores, and hydrogen has been investigated as the predominant reductant for the direct reduction of ilmenite ores [12−15]. Furthermore, many studies on the reduction of synthetic ilmenite ores by hydrogen were reported. ZHAO and SHADMAN [16] as well as VIJAY et al [17] examined the reduction process of synthetic ilmenite by hydrogen. It was suggested that intrinsic chemical reaction and diffusion of gaseous species through product layer were the rate-controlling factors during the reduction process. The temporal profiles of conversion had a sigmoid shape and presented three different stages, i.e., original induction

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stage, medium acceleration stage, and final deceleration stage. VRIES et al [18,19] employed a pressurized thermogravimetric microbalance to investigate the reduction of synthetic ilmenite pellets in the temperature range of 550−900 °C and pressure range of (1.2−10)×10^5 Pa. However, these isothermal experiments of reduction of Bama ilmenite using H_2−Ar gas mixtures were carried out by WANG et al [20].

However, the kinetics of reduction of Panzhihua ilmenite by hydrogen is still unclear because of high content of impurities in the ilmenite. In the present work, the influences of temperature and hydrogen content on the reduction rate and degree were investigated, and the reduction kinetics was also discussed. The activation energy of reduction reaction was calculated, and the hydrogen reduction mechanism of ilmenite concentrate was discussed based on the experimental results.

2 Experimental

2.1 Materials

The chemical composition of Panzhihua ilmenite concentrate used in the present work is shown in Table 1. For comparison, synthetic ilmenite was prepared from the mixture of Fe, Fe_2O_3, and TiO_2 with an appropriate molar ratio at 1200 °C by roasting for 30 h. Figure 1 further indicates the crystalline phases of the synthetic ilmenite and Panzhihua ilmenite. As shown in Fig. 1, the main crystalline phases of the natural ilmenite are magnesian ilmenite ((Fe,Mg)TiO_3) with a small amount of titanomagnetite (Fe_2TiO_4−Fe_3O_4). The natural ilmenite and synthetic ilmenite powders were ground by planetary ball mill and screened to obtain similar particle size fractions in the range of 26−104 μm. Then, approximately 1.2 g ilmenite powder was pressed into a cylindrical pellet sized 8 mm in diameter and 5 mm in thickness.

Table 1 Chemical composition of Panzhihua ilmenite (mass fraction, %)

TiO_2	FeO	Fe_2O_3	MgO	SiO_2
45.48	32.11	7.41	6.85	3.34

Al_2O_3	CaO	MnO_2	V_2O_5	S	P
2.18	1.67	0.68	0.09	0.04	0.15

2.2 Experimental procedure

2.2.1 Thermogravimetric (TG) analysis

The schematic diagram of the thermogravimetric apparatus for the hydrogen reduction experiments is shown in Fig. 2. It consists of a vertical furnace and a computer monitor system used for recording the mass variations during the process. In a typical experiment, an alumina crucible was hung on the central section of furnace chamber by a sapphire extension wire. The sample was put into the alumina crucible and then preheated to a specific temperature in argon atmosphere. Then, in the isothermal period, the reactant gases (H_2−Ar mixture) were blown into the reaction area. Both of hydrogen and argon gases were measured and controlled by high-accuracy mass flow-meters. At the end of the experiment, pure argon gas was purged into the crucible. Finally, the sample was cooled in inert atmosphere.

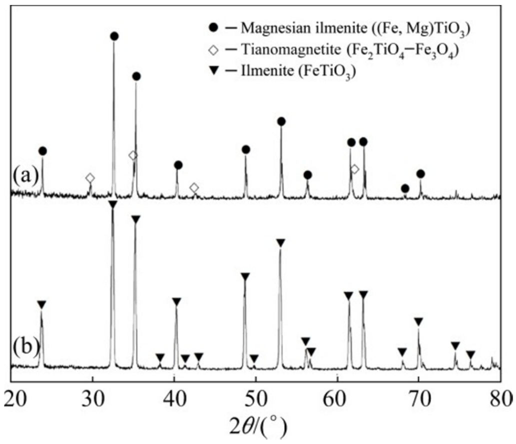

Fig. 1 XRD patterns of Panzhihua natural ilmenite (a) and synthetic ilmenite (b)

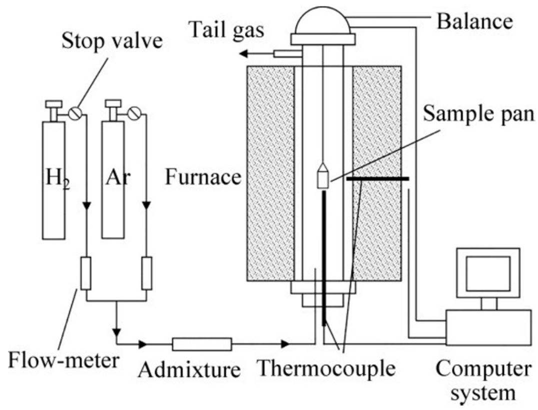

Fig. 2 Schematic diagram of TG apparatus

2.2.2 Characterization of reduced ilmenite

The morphology of the reduced pellet was examined using a JEOL JSM−6700F scanning electron microscope (SEM). The elemental composition was analyzed by energy-dispersive X-ray (EDX) spectroscopy attached to the SEM and also by inductively coupled plasma (ICP). The phase constitution was determined by Rigaku D/Max−2550 X-ray diffractometer (XRD).

3 Results and discussion

As the gas product during the hydrogen reduction process is only water vapor, the reduction degree can be

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