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Rapid Communication

In-situ investigation on mineral phase transition during roasting of vanadium-bearing stone coal

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ABSTRACT

In this work, high temperature in situ X-ray diffraction and in situ analysis of energy dispersive spectrometer were used to find the essence of the roasting of vanadium-bearing stone coal. In the process of roasting, vanadium-bearing muscovite with quartz was converted to feldspar group minerals (albite, orthoclase and anorthite), during which Al(V)—O bonds in muscovite were broken. Meanwhile, vanadium was liberated from muscovite, which was the precondition that vanadium can be recovered. These findings were useful for proposing some measures, such as adding quartz, to recover the vanadium from stone coal more efficiently in practice.

1. Introduction

Vanadium is an important rare element which has been extensively applied in fields of steel industry [1], titanium-aluminum alloys [2,3], vanadium redox battery [4] and catalysts [5]. The gross reserve of vanadium in the stone coal accounts for more than 87% of the domestic reserve of vanadium [6]. Typical vanadium in the stone coal exists as V(III) substituting for Al(III) from the dioctahedral structure in mica group minerals [7,8], which is difficultly recovered by direct leaching. Hence, to extract this vanadium, roasting with sodium additive at high temperature is necessary [9–11], and the vanadium was liberated from the crystal structure of mica and converted to water-soluble sodium vanadate through roasting [12,13]. Subsequently, it was recovered following by the operations of water leaching, ion purification, precipitation and calcination [14,15].

The recovery of vanadium is dependent on roasting. Therefore, it will be of great significance to investigate the mechanisms of roasting. Hu et al. [13] investigated the influence of roasting temperature on the changes of mineral phase and found that the peaks of vanadium-bearing mica minerals became weaker with increasing the roasting temperature and disappeared at 900 °C. Lin and Fan [16] studied the effect of roasting additive on the changes of mineral phase. The results showed that the additive could promote the decomposition of vanadium-bearing muscovite. At present, the researchers have focused on the changes of mineral phases at different roasting temperature, time and additive to analyze their influences on vanadium recovery [17,18]. However, the mineral phase transition and the chemical reactions during roasting were ignored, which is essential to understand the roasting mechanism.

The conventional method for investigating the phase transition during roasting includes the following steps: (1) roasting the samples at required temperature and time; (2) removing the samples from the furnace and cooling them; (3) analyzing the cooled samples by X-ray diffraction (XRD). Li et al. [19] employed this method to study the phase transformation of laterite minerals. The phase changes of the pellets of the iron ore–coal during roasting were also investigated using the method mentioned above [20]. The conventional method cannot obtain the phase transition directly. During the cooling step, the phase may be changed, which could not reflect the truth.

In this work, in-situ methods were used to intuitively clarify the mineral phase transition and the chemical reactions during roasting of stone coal. It would be beneficial to shed new light on the theoretical knowledge of the roasting process and provide the theoretical guidance for the development of vanadium extraction from the stone coal in practice.

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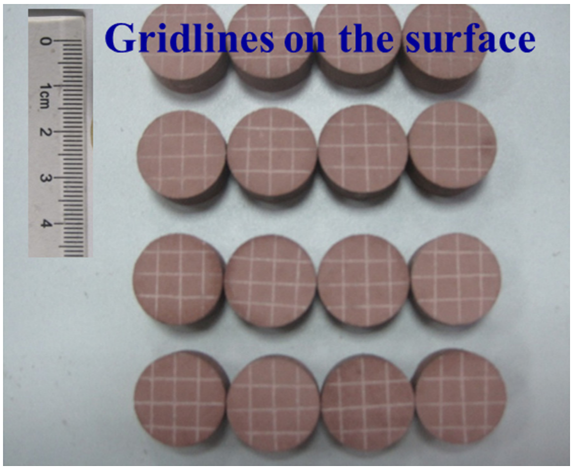

Fig. 1. Forming specimens with gridlines on the surface.

2. Experimental

2.1. Materials

The stone coal samples were obtained from Teng-da Mining and Metallurgy Co. Ltd., Hubei, PR China. The chemical and mineral compositions of the samples were introduced in our previous research [8]. It is mainly composed of 54.36% SiO_2, 10.59% Al_2O_3, 7.35% CaO, 5.93% Fe_2O_3 and 0.82% V_2O_5. The main mineral phases are quartz and muscovite. In addition, a spot of hematite and calcite also exist. The majority vanadium in the stone coal is hosted in the muscovite, which is in agreement with the vanadium occurrence in most stone coal.

The additives with 6 wt.% NaCl and 10 wt.% Na_2SO_4 were used as roasting additive in this study, which has been found be the optimal previously [21]. Both NaCl and Na_2SO_4 with analytical grade were purchased from Tianjin Kemio Chemical Regent Ltd. Before roasting, the sample was mixed with the compound additive, and then the mixtures were ground to minus 150 μm by a vibration mill (HLXZM-100).

2.2. High temperature (HT) in-situ XRD analysis

In order to dynamically observe the mineral phase transition during roasting, HT in-situ XRD analysis was conducted by Bruker D8 Advance X-ray diffractometer with Cu-Kα radiation, air flow rate 100 mL/min, voltage 45 kV, current 40 mA and scanning rate of 10°/min from 5° to 70°. The HT in-situ XRD analysis process simulated the roasting process, which was experienced three stages as follows: (1) heating up from 25 °C to 850 °C with a heating rate of 10 °C/min; (2) roasting at 850 °C for 2 h; (3) nature cooling from 850 °C to 500 °C.

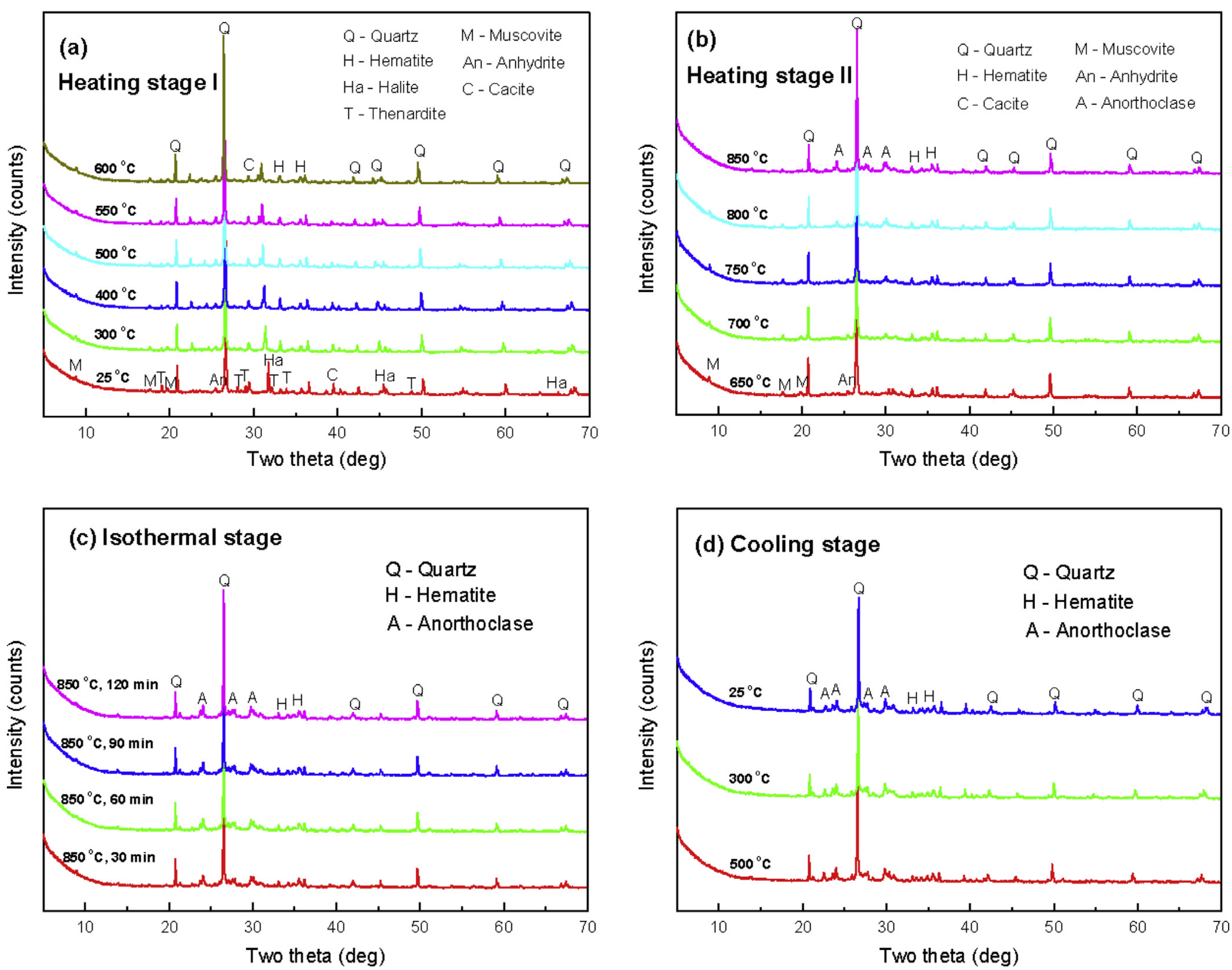

Fig. 2. HT in-situ XRD patterns of sample during roasting: heating stage I (a), heating stage II (b), isothermal stage (c), cooling stage (d).

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