



Phase change and kinetics of vacuum decomposition of molybdenite concentrate



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ARTICLE INFO

Article history:

Received 3 February 2015

Received in revised form

8 March 2015

Accepted 9 March 2015

Available online 17 March 2015

Keywords:

Vacuum decomposition

Phase change

Kinetics

Molybdenite concentrate

ABSTRACT

Vacuum decomposition process of molybdenite (MoS_2) concentrate was investigated under pressure of 10–200 Pa in the temperature range 1423 K–1773 K. The phase evolution analysis demonstrated that molybdenite concentrate decomposed step by step which meaning that MoS_2 decomposed to Mo_2S_3 firstly and then Mo_2S_3 decomposed to Mo. Thermodynamic calculation results shown that the decomposition of MoS_2 was easily to be achieved when the temperature was higher than 1773 K and the pressure was lower than 200 Pa. Temperature and pressure can significantly influence the decomposition reaction, and higher temperature and lower pressure helped to accelerate the decomposition reaction rate. The Mo content of the molybdenum metal product was 97.69%, and the sulfur content of the obtained sulfur could reach 98.6% and more than 98% sulfur can be recovered. Kinetic study of the process indicated that the decomposition reaction followed the shrinking unreacted core model and controlled by chemical reaction step: $1 - (1 - \alpha)^{1/3} = kt$, and the apparent activation energy of the decomposition reaction was 348.955 kJ/mol.

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1. Introduction

Molybdenite is the major industrial mineral to produce molybdenum. Because of excellent flotability of molybdenite, molybdenite concentrate can be easily produced by flotation method. At present, molybdenite concentrate is converted to molybdenum oxide by oxidizing roasting firstly, and then the resultant oxidized concentrate is purified by conversion of the molybdenum oxide to ammonium molybdate, finally commercial molybdenum metal can be obtained by reduction of molybdenum oxide with H_2 in two or more stages [1]. However, there are some problems in this process: SO_2 produced in the oxidizing roasting step is extremely harmful to the environment, and recycling SO_2 by acid-making method is difficult just because the concentration of SO_2 in the roasting gas is too low; and also the process is so complex that the overall recovery of molybdenum is low [2].

In order to avoid pollution in the conventional process, some new production methods have been proposed, such as reduction

with carbon (or hydrogen) in presence of lime, chloridizing roasting, hydrometallurgy, and bioleaching. But these processes don't have good industrial prospect. Padilla et al. [3] investigated the carbothermic reduction of molybdenite concentrate in presence of lime and the mixtures of Mo, Mo_2C and CaS were produced. But how to remove CaS and refine the metallic molybdenum product is a problem. Zhao et al. [4] reported a direct hydrometallurgy process for molybdenum extraction from Ni–Mo ore, which included the leaching of molybdenum by air oxidation in alkaline solution, purifying the leach solution, enriching molybdenum by ion exchange and crystallizing ammonium molybdate tetrahydrate. But the process is complicated and the extraction efficiency needs to be improved, and also the residues and waste solution need to be treated [5]. The process of bioleaching is low energy consumption, high selectivity and no pollution, but its slow leaching speed, low efficiency and poor adaptability limit its further industrial application [6,7]. The vacuum decomposition process of molybdenite concentrate was put forward very early, and the Climax Company in the United States had also focused on this process for many years, but there were no further application production [8,9]. In order to reduce the production cost and develop environmental friendly process, vacuum decomposition process was put forward again, of which the main step is the decomposition of molybdenite concentrate in a vacuum furnace, and metallic molybdenum and

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sulfur can be obtained directly after one step decomposition [10]. This process offers possibility of no emission of SO_2 , and it also makes molybdenum extraction easier.

The main purpose of this research is to investigate the morphology and phase evolution in the vacuum decomposition process, and also to obtain some key kinetic parameters which affecting the vacuum decomposition reaction.

2. Experimental methods and materials

2.1. Experimental methods

The molybdenite concentrate was mixed with binder and agglomerated to pellets of which the diameter was about 1 cm. Then the pellets were dried in an oven at 423 K for about 2 h. The decomposition experiments were carried out in a vacuum furnace with the temperature from 1423 K to 1773 K and the pressure from 10 Pa to 200 Pa. The pellets were charged into the vacuum furnace when the temperature reached the target value, and then the chamber was evacuated to the desired pressure level by a vacuum pump. The weight change was recorded in the decomposition process by a quartz spring balance, and the sample was put on a corundum plate which was hung from the quartz spring balance. In each experiment, about 20 g sample were put on the plate. The obtained sulfur vapor can be recovered by condensation, and the resultant metallic molybdenum product can be used for steel-making or made from other products after further refining. The process flow diagram was shown in Fig. 1.

The phase analyses were performed by an X'per Pro powder diffraction from Dutch PANalytical company, using $\text{CoK}\alpha$ radiation, scanning 2θ range from 20° to 120° . Morphology was analyzed by SEM from Japan Hitachi Ltd.

The decomposition fraction (α) is defined as follow:

$$\alpha = \left(\frac{w_0 - w}{w_0} \right) / \left(\frac{M_{\text{S}_2}}{M_{\text{MoS}_2}} \right) \quad (1)$$

where w_0 is the initial weight of molybdenite concentrate pellet; w is the weight of pellet at t ; M_{S_2} is the molecular weight of S_2 ; M_{MoS_2} is the molecular weight of MoS_2 .

2.2. Composition analysis of molybdenite concentrate

The composition of molybdenite concentrate used in this study is exhibited in Table 1, and the XRD pattern of molybdenite concentrate is shown in Fig. 2. It was evident that the content of

Table 1
Chemical composition of molybdenite concentrates from an enterprise.

Mo	S	SiO_2	CaO	Sn	P	Cu	Pb	Bi	As
57.20	39.2	2.0	0.50	0.01	0.01	0.10	0.10	0.02	0.001

molybdenum was 57%, and the major phase was MoS_2 . The percentage of MoS_2 in the concentrate can be estimated to be 95.4 wt pct, and the main impurities in the molybdenite concentrate were SiO_2 and CaO.

3. Results

3.1. Phase evolution

The phase evolution in the vacuum decomposition process of molybdenite concentrate is shown in Fig. 3. The experiments were carried out under pressure of 20 Pa. Fig. 3(a) exhibits the initial state of pellets at room temperature. As shown in Fig. 3(b), the concentrate pellet had no apparent change after it was heated at 1423 K for 2 h. Fig. 3(c) presents the morphology of pellet which was treated at 1523 K for 2 h, and there were two layers formed in the diameter direction. In Fig. 3(d), when the concentrate pellet decomposed at 1598 K for 2 h, three layers were formed in the pellet. Fig. 3(e) shows that the pellet obtained at 1623 K was composed by two layers, and the MoS_2 core had disappeared. In Fig. 3(f), the pellets produced at 1748 K had visible metallic luster. It is explicit that with the temperature rising from 1423 K to 1748 K molybdenite concentrate pellets decomposed layer by layer from outer to inner.

It was observed in Fig. 3 that there were three layers formed in the pellet at 1598 K. Phase analyses were carried out for different layer parts of pellet in Fig. 3(d) and the results are exhibited in Fig. 4. As were shown in Fig. 4, the central part of the pellet was unreacted MoS_2 , the middle part was Mo_2S_3 , and the outer layer was Mo metal. So, it is definite that MoS_2 decomposed step by step under pressure of 20 Pa at the temperature range 1423 K–1773 K, which meaning that MoS_2 decomposed to Mo_2S_3 firstly and then Mo_2S_3 decomposed to Mo.

In order to further understand the phase change in the vacuum decomposition process, thermodynamic calculation of decomposition reactions were carried out. In Mo–S system, there are two kinds of stable compounds, MoS_2 and Mo_2S_3 . The possible reactions in the vacuum decomposition process are listed as follow [11]:

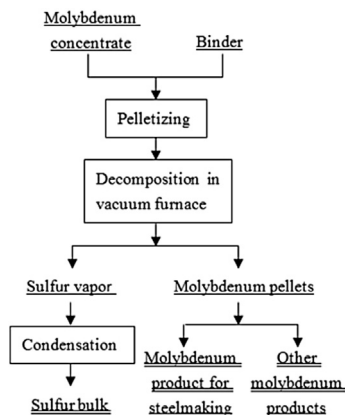


Fig. 1. Vacuum decomposition process of molybdenite concentrate.

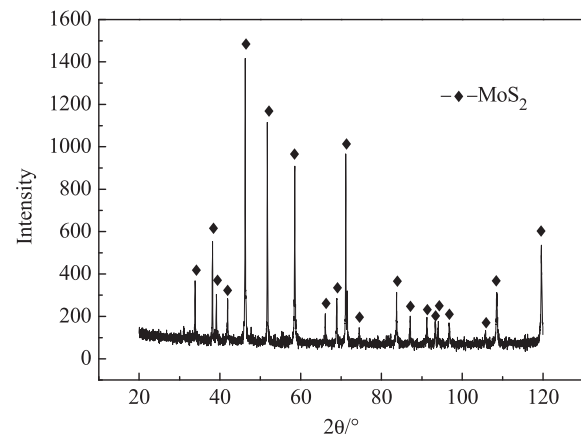


Fig. 2. X-ray diffraction pattern of molybdenite concentrate.

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