



Separation and enrichment of PbS and Sb₂S₃ from jamesonite by vacuum distillation



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ABSTRACT

The formula for jamesonite is Pb₄FeSb₆S₁₄ (or 4PbS·FeS·3Sb₂S₃). This paper proposes a novel method for the separation and enrichment of PbS and Sb₂S₃ from jamesonite by vacuum distillation. The experiments were carried out at temperatures ranging from 1073 K to 1473 K and at pressures less than 10,000 Pa. The amount of Sb, Pb, and Fe in the volatiles and residues was determined by chemical titration analysis and the enrichment ratios of Sb, Pb, and Fe were calculated. We discuss the influence of the distillation temperature and holding time on the enrichment ratios of each element. The results of the XRD and EDS analyses of the residues and volatiles indicates that FeS remains in the residues as the major compound, Sb exists in the form of Sb₂S₃, and Pb exists in the form of PbS in different condensers. Experimental results showed that Sb₂S₃ condensed at about 725 K PbS condensed at about 984 K in different condenser.

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1. Introduction

Jamesonite contains many valuable elements such as Sb, Pb, Ag, and Zn. It is one of the most important sulfide minerals for smelting Sb, and accounts for 30%–40% of the antimony resources in China [1]. The formula for jamesonite is Pb₄FeSb₆S₁₄, which is considered to be in the form of a lead sulfide and antimony sulfide solid solution. Therefore, the formula Pb₄FeSb₆S₁₄ can also be written as 4PbS·FeS·3Sb₂S₃. The key to using jamesonite effectively is to separate its lead and antimony components from each other. Smelting is the only way to separate PbS from Sb₂S₃ and FeS, as they cannot be separated by physical milling [2]. The first step of the current process is obtaining a Pb–Sb crude alloy by reduction smelting of jamesonite. Pb and Sb can then be obtained by redox chemistry of the crude alloy. However, the process is associated with many problems such as long run times, a complicated procedure for the separation of Pb and Sb, a low yield, and SO₂ pollution [3]; moreover, only Grade 2 antimony can be produced by

traditional pyro-metallurgical methods. Other promising processes such as chlorination hydrolysis, chlorination carbonization, and slurry electrolysis have recently been developed to overcome these problems [4–6]. However, there are many shortcomings with these processes such as high amounts of alkali consumption, long production periods, high initial costs, high energy consumption, and low profitability [7–10].

Vacuum distillation as an effective purification method has been used in the preparation of lead-tin alloys, high-purity bismuth, and metallurgical-grade silicon [11–16], but the separation and enrichment of PbS and Sb₂S₃ from jamesonite by vacuum distillation has not been previously reported. The possibility of separation of PbS and Sb₂S₃ with FeS by vacuum distillation is discussed theoretically in this paper. Experimental results show that PbS and Sb₂S₃ can be extracted from jamesonite separately by vacuum distillation. Sb₂S₃ directly used for the production of Sb₂O₃ and PbS can be used for the production of PbO, which is used to achieve the separation of lead and antimony in the source mineral, and improves the utilization of jamesonite while reducing energy consumption. This paper provides a new method for the separation of lead and antimony in jamesonite. It is generally known, most of minerals smelting lead and antimony are sulfide minerals which are considered to be in the form of a lead sulfide and antimony

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sulfide and other sulfide solid solution. So The research provide a reference to the separation of lead sulfide, antimony sulfide and iron sulfide in other sulfide minerals.

2. Method

2.1. Saturated vapour pressure

Different sulfides can be separated and enriched from jamesonite by vacuum distillation, depending on the different properties of each sulfide during vaporising and condensing. Differences between the saturated vapour pressures of different sulfides in the pure state influence the process of jamesonite vacuum distillation at different temperatures. Using formulas and related thermodynamic data previously derived [8], the relationship between saturated vapour pressure of each sulfide and the process temperature can be calculated from Eq. (1),

$$\lg p^{\theta} = AT^{-1} + B\lg T + CT + D \quad (1)$$

where p^{θ} is the saturated vapour pressure of the pure substance in Pascals (Pa); T is the absolute temperature in Kelvin (K), and the evaporation constants A , B , C and D for the different sulfides can be found elsewhere [8]. Using the values of A , B , C and D , the saturated vapour pressure can be calculated from Eq. (1). Each sulfide has its own temperature range using Eq. (1) to calculate the saturated vapour pressure.

As shown in Fig. 1, a comparison of the saturated vapour pressures of different sulfides at the same temperature can be used as a rough guide for determining which sulfide might exhibit preferential volatilization. One with a higher vapour pressure can be expected to volatilize preferentially, while others with lower vapour pressures will remain as residues after vacuum distillation [13].

Fig. 1 shows that the saturated vapour pressures of Sb_2S_3 and PbS are much higher than that of FeS , and at proper temperatures Sb_2S_3 and PbS can be expected to volatilize from jamesonite during distillation, resulting in the concentration of FeS in the residues. Therefore, Sb_2S_3 and PbS can be volatilized and condensed selectively at proper temperatures and pressure since the saturated vapour pressure of Sb_2S_3 is far greater than that of PbS .

2.2. Experimental

The raw material used in this study was a type of jamesonite

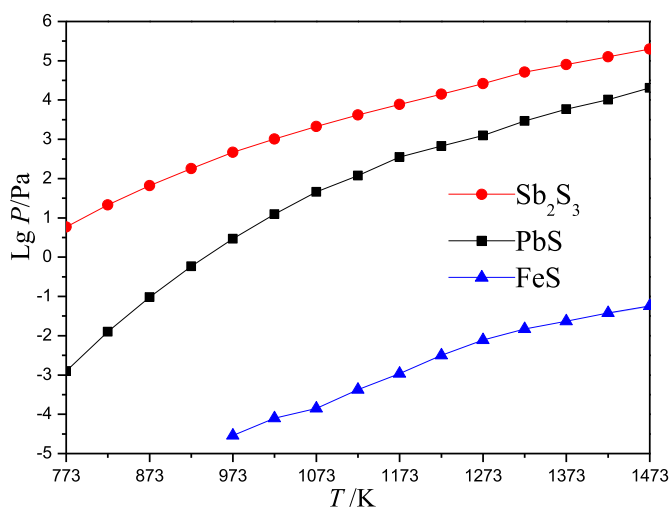


Fig. 1. Lgp-T diagram of different sulfides being in jamesonite.

that was provided by a smelter in Guangxi, China, the chemical composition of which was 24.43% Pb, 16.5% Sb and 14.52% Fe (by mass fraction). Fig. 2 is the XRD spectrum of this raw material, which shows that the main components of the mineral are $Pb_4FeSb_6S_{14}$ with some ZnS . We found that ZnS has been enriched on the first graphite condenser. Because zinc sulfide has nothing to do with the experimental research, the behavior of zinc sulfide is not discussed in this paper.

The experiment was carried out in a self-designed HZSL-18-type vacuum distillation furnace. The vacuum distillation furnace and heating & multi-stage condensing apparatus put in the vacuum distillation furnace is shown in Fig. 3. The apparatus consists of a backflow plate and three graphite condensers. Each graphite condenser is 10 cm tall, and the condenser cooling surface is 80 cm². The internal structure of the multi-stage condensing apparatus is shown in Fig. 4. Jamesonite was decomposed into PbS and Sb_2S_3 and both were volatilized into the gas phase. The sulfides were then volatilized through different graphite condensers from the bottom up and condensed at different points due to their different condensation temperatures and the temperature of each graphite condenser increases as the temperature of the graphite crucible putting in the heating area of vacuum furnace as increases.

500 g jamesonite was weighed out after drying, and placed in a graphite crucible in the vacuum furnace heating zone. A multi-stage self-condensing device was then placed at the top of the crucible. The furnace was evacuated after sealing until the pre-determined pressure was achieved, and the crucible was heated to a predetermined temperature, where it was held for a period of time before cooling. Volatiles and residues were recovered from different graphite condensers and the crucible. During the experiment, the position of the graphite condensers remained unchanged and the temperature remained fixed at a pre-determined temperature. The parameters of distillation temperature and holding time were determined by testing the contents of different metals in the volatiles and residues. The elemental contents of the samples were determined by chemical titration and atomic absorption spectroscopy, and the structure of the residues and volatiles were studied using XRD and EDS.

The mass fraction of each element in the volatiles (X_i) was calculated using Eq. (2), while the enrichment ratio (Y_i) of each element was calculated using Eq. (3)

$$X_i = (m_1 \cdot \omega_{i1} - m_2 \cdot \omega_{i2}) / (m_1 \cdot \omega_{i1}) \quad (2)$$

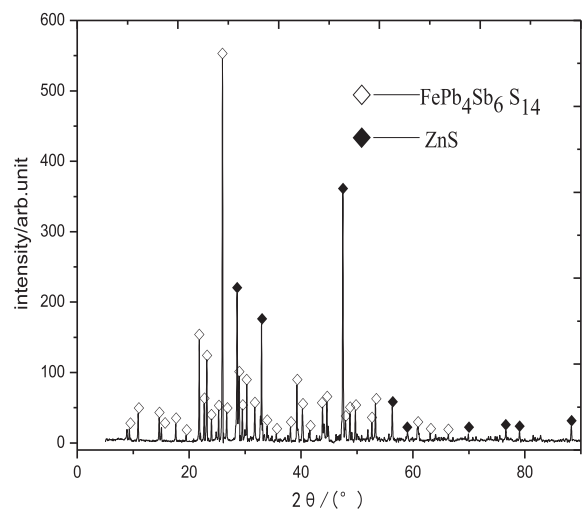


Fig. 2. The XRD spectrum of this raw materials.

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