



Recycling of metals from waste Sn-based alloys by vacuum separation



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Received 25 May 2014; accepted 8 December 2014

Abstract: In order to recycle waste Sn-based alloys, the vapor–liquid phase equilibrium composition diagrams of Sn–Pb, Sn–Sb and Sn–Zn binary systems were calculated. The calculated results indicate that Pb, Sb and Zn can be separated from Sn effectively. Based on the above calculation, the industrial experiments of vacuum distillation of Sn–Pb alloy, Sn–Pb–Sb alloy, Sn–Pb–Sb–As alloy, crude Sn and Sn–Zn alloy with different contents were carried out. The experimental results show that Pb (>99% Pb) and Sn ($\leq 0.003\%$ Pb) were obtained simultaneously while Sn–Pb alloy was subjected to vacuum distillation; the crude Sn (>90% Sn, $\leq 2\%$ Pb, $\leq 6\%$ Sb) and crude Pb ($\leq 2\%$ Sn) were obtained simultaneously while a single vacuum distillation was carried out for Sn–Pb–Sb alloy; the Pb and Bi contents in the Sn ingot (99.99% Sn) achieve the grade A of GB/T 728–2010 standard, more than 50% of As and Sb was removed after vacuum distillation of crude Sn; Zn (<0.002% Sn) and Sn (about 3% Zn) were obtained while vacuum distillation of Sn–Zn alloy was conducted at 1173 K, 20–30 Pa for 8–10 h.

Key words: Sn-based alloys; activity coefficient; vacuum distillation; vapor–liquid phase equilibrium

1 Introduction

Sn is one of the earliest metals that human discovered and used and is widely used in aerospace, military, nuclear industry, electronics information, semiconductors, superconducting alloys, navigation, medicine, food industry and other fields since it has the following characteristics: low melting point, non-toxic, high atmospheric corrosion resistance, good ductility and good weldability. It is an indispensable key material in military industry and modern cutting-edge technology areas [1,2]. The Sn smelting and production level of China are in a leading position in the world. The Sn resources in China are mainly distributed in Yunnan, Guangxi, Hunan, Guangdong and Jiangxi provinces where the recoverable deposits account for 97.3% of the country's reserves [3]. The Sn production of Yunnan Tin

Group Co., Ltd. and Guangxi Tin Group Co., Ltd. accounts for about one-fifth of the total output of the world in 2011. In recent years, a large number of waste Sn resources such as Sn–Pb alloys, multi-Sn alloys, crude Sn and Zn–Sn alloys have been produced from the smelting process of Sn, Pb and Cu, the recycling process of Sn secondary resources and the production and machining process of solders, bearing babbits and Sn alloy coatings due to the development of industry and the decrease of mineral resources [4].

For a long time, many problems such as the waste of Sn, Pb, Sb and Zn resources and other resources, higher energy consumption and environmental pollution have emerged due to lack of efficient processing techniques. In order to further improve the Sn smelting level of China, the developing of more advanced technologies and equipments, therefore, has become imperative for China. A new road to industrialization

Foundation item: Project (2014HA003) supported by the Cultivating Plan Program for the Technological Leading Talents of Yunnan Province, China; Project (51474116) supported by the National Natural Science Foundation of China; Project (IRT1250) supported by the Program for Innovative Research Team in University of Ministry of Education of China; Project (20140355) supported by the Analytical Test Fund of Kunming University of Science and Technology, China; Project supported by the First-class Doctoral Dissertation Breeding Foundation of Kunming University of Science and Technology, China

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DOI: 10.1016/S1003-6326(15)63730-X

will thus be realized, an environment-friendly and resource-saving society will be achieved finally. The separation and recovery of waste Sn-based alloys by traditional methods, however, have some problems. Recently, a lot of researches including theoretical basic research and equipment upgrade have been carried out for the recovery of waste Sn-based alloys by vacuum distillation and many achievements have been achieved [5–7]. Vacuum metallurgy has many advantages such as short flow sheet and lower energy consumption. It can eliminate the disadvantages of traditional metallurgical processes. Vacuum distillation has been studied and successfully used in separation and purification of various elements from nonferrous alloys [8–10].

In this work, vacuum distillation experiments for Sn–Pb alloys, multi-Sn alloys, crude Sn and Sn–Zn alloys were carried out based on the theoretical prediction. The corresponding new technology, therefore, was developed and widely used in industries. It provides an important technique for the effective and comprehensive utilization of Sn, Pb, Sb and Zn resources.

2 Calculation and prediction for vacuum distillation of Sn alloys

2.1 Vapor pressure of components of Sn alloys

The phase transition will take place when the system pressure is less than the saturated vapor pressure of a substance, and the substance will exist in the form of gas phase. The saturated vapor pressure of the pure metals as a function of temperature was calculated using the equation and related parameters [11] given in Table 1, as shown in Fig. 1. It can be seen from Fig. 1 that the saturated vapor pressure of the components increases with the increase of temperature. The saturated vapor pressure of these components follows the sequence of $As > Zn > Sb > Bi > Pb > Al > Sn > Cu > Fe$ at the same temperature. Therefore, As, Zn, Sb, Bi, Pb and Al will exhibit a higher volatility than Sn, while Cu and Fe exhibit a low volatility.

2.2 Separation criterion and vapor–liquid phase equilibrium composition diagram for vacuum distillation of Sn–Me binary alloys

For a Sn–Me binary alloy (Me represents Pb, Sb, Zn, As, Cu, Bi, Al and Fe), according to the ideal gas state equation, the relationship between $\rho(Sn)$ and $\rho(Me)$ can be expressed as:

$$\frac{\rho(Sn)}{\rho(Me)} = \beta_{Sn/Me} \frac{w_l(Sn)}{w_l(Me)} \quad (1)$$

where $\rho(Sn)$ and $\rho(Me)$ are the vapor densities of Sn and

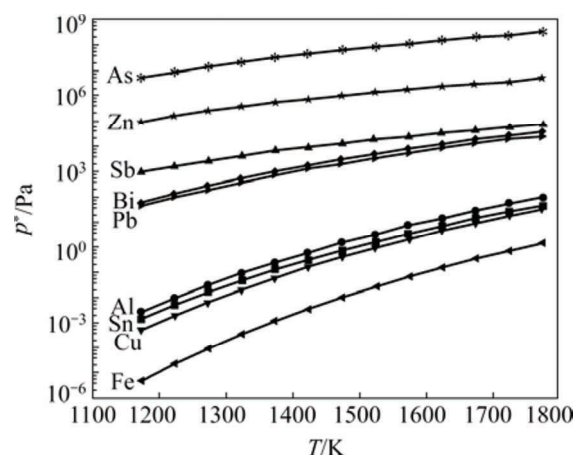


Fig. 1 Saturated vapor pressure as a function of temperature for some pure metals

Me in the vapor phase, respectively; $w_l(Sn)$ and $w_l(Me)$ are the mass fractions of Sn and Me in the liquid phase, respectively. And $\beta_{Sn/Me}$ is the separation coefficient and defined as

$$\beta_{Sn/Me} = \frac{\gamma(Sn)}{\gamma(Me)} \cdot \frac{p(Sn)^*}{p(Me)^*} \quad (2)$$

where $\gamma(Sn)$ and $\gamma(Me)$ are the activity coefficients of Sn and Me, respectively, $p(Sn)^*$ and $p(Me)^*$ are the saturated vapor pressures of pure Sn and Me, respectively. The separation of binary Sn-based alloys by vacuum distillation can be judged by $\beta_{Sn/Me}$. If $\beta_{Sn/Me} < 1$, namely, $\rho(Sn)/\rho(Sn) < w_l(Sn)/w_l(Me)$, Sn will concentrate in the liquid phase, while Me will concentrate in the vapor phase. For the same reason, Sn will concentrate in the gas phase, and Me will concentrate in the liquid phase while $\beta_{Sn/Me} > 1$. The Sn content in the liquid phase equals that in the vapor phase while $\beta_{Sn/Me} = 1$, viz., the separation of Sn and Me cannot happen.

The separation coefficient can only be used as a rough guidance in predicting the possibility of separation of binary alloys. In order to quantitatively predict the separation degree and product composition of vacuum distillation of alloys, the vapor–liquid phase equilibrium composition was introduced. Assuming $w_g(Sn)$ and $w_g(Me)$ are the mass fractions of Sn and Me in the vapor phase, respectively, then

In the gas phase

$$w_g(Sn) + w_g(Me) = 1 \quad (3)$$

In the liquid phase

$$w_l(Sn) + w_l(Me) = 1 \quad (4)$$

For gas phase,

$$w_g(Sn) = \frac{\rho(Sn)}{\rho(Sn) + \rho(Me)} = \frac{1}{1 + \rho(Me)/\rho(Sn)} \quad (5)$$

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