



Theoretical research on vacuum separation of Au–Ag alloy

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Abstract: To provide an accurate prediction of the product component dependence of temperature and pressure in vacuum distillation and give convenient and efficient guidance for the designing of the process parameters of industrial production, according to the molecular interaction volume model (MIVM), the separation coefficient (β) and vapor–liquid equilibrium composition of Au–Ag alloy at different temperatures are calculated. Combined with the vapor–liquid equilibrium (VLE) theory, the VLE phase diagrams, including the temperature–composition (T – x) and pressure–composition (p – x) diagrams of Au–Ag alloy in vacuum distillation are plotted. The triple points and condensation temperatures of gold and silver vapors are calculated as well. The results show that the β decreases and the contents of gold in vapor phase increase with the distillation temperature increasing. Low pressures have positive effect on the separation of Ag and Au. The difference between the condensation temperatures of gold and silver is about 450 K in the pressure range of 1–10 Pa.

Key words: Au–Ag alloy; vacuum separation; molecular interaction volume model (MIVM); vapor–liquid equilibrium (VLE) phase diagram; condensation temperature

1 Introduction

Gold and silver, with excellent and unique physical and chemical properties, are indispensable key materials in high-tech fields such as electronics, communications, aerospace and chemical catalysis [1–4]. Due to the similar geochemical and atomic properties [1], Au–Ag alloy is produced in the cyanide gold extraction process and comprehensive utilization process of lead and copper anode by slime reduction smelting–oxidation blowing.

The methods for separating Au–Ag alloy fall

into chemical method and electrolytic method. In the chemical case, the gold and silver can be purified by aqua regia dissolving gold and nitric acid dissolving silver. However, due to the mutual encapsulation between gold and silver particles, the purification by chemical method is faced by the challenges of strict requirements for raw material composition and environmental pollution caused by nitrogen oxides. Compared to chemical method, the electrolysis method has advantages in product purity but shows poor performance in purification efficiency and economic benefit [5]. Consequently, it is urgent to develop a novel method to separate

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Au–Ag alloy.

Vacuum distillation can eliminate the shortcomings of traditional metallurgical processes. It has many advantages in the fields of alloy separation [6–8], crude metal purification [9–11], resource recovery [12–15], etc, such as high metal recovery rate, impurities recovered in metallic state, simple process, low environmental pollution, and low operating cost. KHLEBNIKOV [16] published the modern industrial experience of application of vacuum distillation of silver for separation of Au–Ag alloy, and gave the contents of Au–Ag alloy after vacuum distillation. However, the purity of residue and volatile are only about 93% and 95%, respectively, and the reason is the lack of adequate theories as guidance.

When designing a vacuum distillation experiment, reliable and accurate activity coefficient and vapor–liquid equilibrium (VLE) data play crucial roles in the selection of the appropriate distillation temperature and pressure and prediction of the separation effect. The VLE phase diagram can provide a convenient and intuitive guide for vacuum distillation. The thermodynamic data are mainly obtained by experimental and model prediction methods. The molecular interaction volume model (MIVM) based on free volume theory and lattice theory, combined with statistical thermodynamics and fluid phase equilibrium theory, has shown good reliability and consistency in the activity prediction and vapor–liquid equilibrium studies of Sn–Sb [6], Pb–Ag, Pb–Sn [7], Sn–Pb–Sb [17], Bi–In–Sn [18], and other alloy systems.

Therefore, in this work, the saturated vapor pressure of gold and silver are calculated firstly. Secondly, according to MIVM, the activity coefficients of Au–Ag alloy at different temperatures are predicted, and the separation coefficients and vapor–liquid equilibrium composition of Au–Ag alloy are calculated. Based on MIVM and VLE theory, the VLE phase diagrams, such as temperature–composition ($T-x$) and pressure–composition ($p-x$) curves of Au–Ag alloy in vacuum distillation are obtained and discussed. In addition, the triple point and condensation temperatures of gold and silver vapor are calculated. In this work, the vacuum separation theories of Au–Ag alloy have been systematically calculated for the first time. This theoretical

investigation will give accurate, efficient and reliable guidance for the separation of Au–Ag alloy by vacuum distillation.

2 Saturated vapor pressure and separation coefficient

The separation of alloys by vacuum distillation is based on the difference in saturated vapor pressures of the two components. At the same temperature, the greater the vapor pressure of a component is, the more volatile it is. The relationship between the saturated vapor pressure and temperature of a pure component can be expressed as [19]

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

where p^* is the saturated vapor pressure of the pure component, Pa; A , B , C and D are the evaporation constants for each element in the alloy, as obtained from the literature [19]. The evaporation constants of Au–Ag alloy components are given in Table 1. The relationship between the saturated vapor pressure and temperature of each component in the Au–Ag alloy is plotted in Fig. 1.

As shown in Fig. 1, $p_{\text{Ag}}^* > p_{\text{Au}}^*$, and it can be judged that under the same temperature, Ag

Table 1 Evaporation constants of Au–Ag alloy components

Component	A	B	C	D	Temperature range/K
Ag	–14400	–0.85	0	13.825	1234–2436
Au	–19280	–1.01	0	14.505	1337–3081

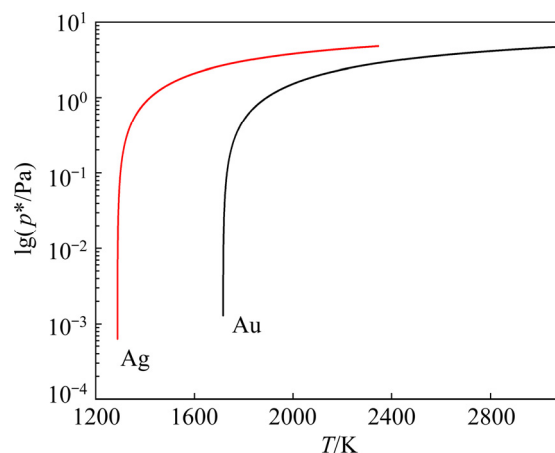


Fig. 1 $\lg p^*-T$ dependence of each component in Au–Ag alloy

volatilizes preferentially into the vapor phase than Au. In addition, when the temperature is 1373 and 1773 K, the saturated vapor pressure of Ag and Au differs by hundreds to two thousand times, and the lower the temperature, the greater the difference. Therefore, when separating gold and silver by vacuum distillation, the temperature should not be too high.

The comparison of saturated vapor pressure has certain limitations. For the actual alloy system, there are large or small interaction forces between the metal particles. These interaction forces will have a certain impact on the actual vapor pressure of the alloy components, which will affect the vacuum separation of the alloy. Figure 2 shows the Au–Ag phase diagram drawn by FactSage 7.2. It can be seen that gold and silver form an infinite solid solution, and gold can inhibit the volatilization of silver to a certain extent.

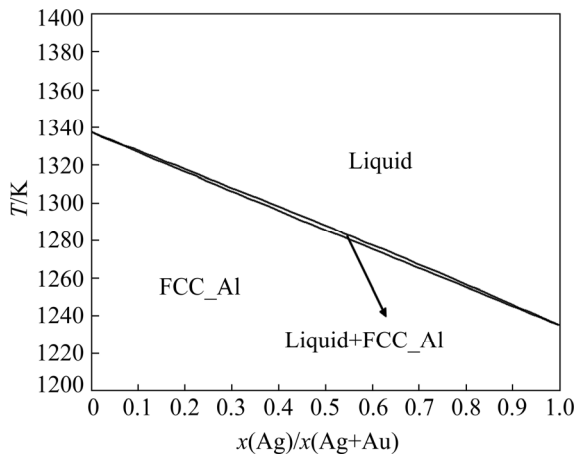


Fig. 2 Phase diagram of Au–Ag binary system at 1.013×10^5 Pa

Regarding the deviation caused by the interaction force, DAI and YANG [19] introduced the activity into the prediction of the vacuum distillation of binary alloys, and proposed using the separation coefficient as the criterion for the separation of alloy components:

$$\beta_i = \frac{\gamma_i \cdot P_i^*}{\gamma_j \cdot P_j^*} \quad (2)$$

where γ_i and γ_j are activity coefficients of i and j in i – j binary alloys, respectively. When $\beta_i > 1$ or $\beta_i < 1$, the separation of two components can be realized by vacuum distillation, and the greater the β_i is, the better the separation effect is. When $\beta_i = 1$, vacuum distillation can not separate the two components. It

can be seen from Eq. (2) that the activity coefficient is the key to calculating the separation coefficient.

According to MIVM, the activity coefficient of Au–Ag alloy components at different temperatures can be calculated. By substituting the activity coefficient and saturated vapor pressure at different temperatures into Eq. (2), separation coefficients of Au–Ag alloy at different temperatures can be obtained. The results are exhibited in Fig. 3.

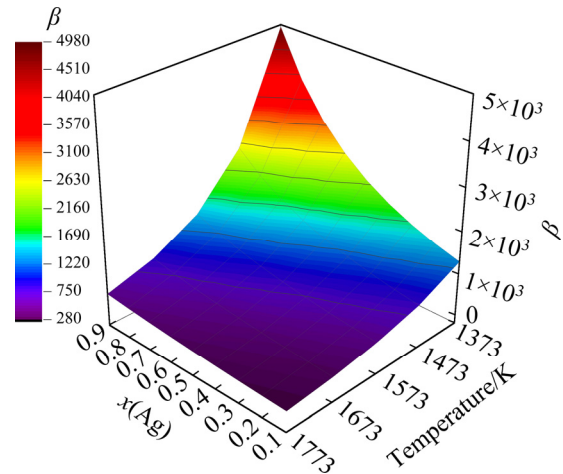


Fig. 3 Separation coefficient of Au–Ag alloy at different temperatures

It can be seen from Fig. 3 that in the temperature range of 1373–1773 K, β is much more than one, which indicates that Ag is preferentially volatilized into vapor phase and Au remains in residue. At the same temperature, with the decrease of silver content in liquid phase, that is, as the vacuum distillation goes on, the separation coefficient decreases gradually, indicating that the lower the silver content is, the more difficult it is to separate Au–Ag alloy by vacuum distillation. With the increase of distillation temperature, the separation coefficient decreases, which suggests that the temperature should not be too high. For example, β decreases from 3938 to 505 with the temperature increasing from 1337 to 1773 K when the silver in liquid is 10 at.%.

3 Vapor–liquid equilibrium composition of Au–Ag alloy

Equilibrium composition is significant to estimating the separation effect and the content of each component in vapor and liquid phase quantitatively. As for Au–Ag alloy, there are

$$w_{\text{Ag,g}} + w_{\text{Au,g}} = 1; \quad w_{\text{Ag,l}} + w_{\text{Au,l}} = 1 \quad (3)$$

where $w_{\text{Ag,g}}$ and $w_{\text{Au,g}}$ are the mass fractions of silver and gold in the vapor phase, respectively, $w_{\text{Ag,l}}$ and $w_{\text{Au,l}}$ are the mass fractions of silver and gold in the liquid phase, respectively.

When the vapor phase and the liquid phase reach the equilibrium, the relationship between the mass fraction of Ag in the vapor phase and the vapor density of Ag and Au can be expressed by Eq. (4):

$$w_{\text{Ag,g}} = \frac{\rho_{\text{Ag}}}{\rho_{\text{Ag}} + \rho_{\text{Au}}} = \frac{1}{1 + \rho_{\text{Au}}/\rho_{\text{Ag}}} \quad (4)$$

where ρ_{Ag} and ρ_{Au} are vapor densities of Ag and Au, respectively, and

$$\frac{\rho_{\text{Au}}}{\rho_{\text{Ag}}} = \beta_{\text{Au}} \frac{w_{\text{Au,l}}}{w_{\text{Ag,l}}} \quad (5)$$

Therefore, the mass fractions of silver in the vapor phase $w_{\text{Ag,g}}$ can be obtained as follows:

$$w_{\text{Ag,g}} = \left[1 + \frac{w_{\text{Au,l}}}{w_{\text{Ag,l}}} \cdot \frac{\gamma_{\text{Au}}}{\gamma_{\text{Ag}}} \cdot \frac{p_{\text{Au}}^*}{p_{\text{Ag}}^*} \right]^{-1} \quad (6)$$

Substituting the corresponding γ_{Ag} , γ_{Au} , p_{Au}^* and p_{Ag}^* at different temperatures and $w_{\text{Ag,l}}$, $w_{\text{Au,l}}$ into Eq. (6), the change of equilibrium composition of vapor and liquid phase in the temperature range of 1373–1773 K can be easily calculated, as shown in Fig. 4.

As can be seen from Fig. 4, when the silver content in the liquid phase is 80%, the silver and gold contents in the vapor phase are 99.99365% and 0.00635%, respectively, at the distillation temperature of 1373 K, and the silver and gold contents in the vapor phase are 99.95052% and 0.04948% at 1773 K, respectively. When the silver content in the liquid phase is 10%, the contents of silver and gold in the vapor phase are 99.30749% and 0.69251% at 1373 K, and the contents of silver and gold in the vapor phase are 96.92743% and 3.07257% at 1773 K, respectively.

When vacuum distillation is carried out at a higher temperature to separate Au–Ag alloy, as the vacuum distillation progresses, the silver content in the liquid phase gradually decreases, and the gold in the liquid phase also enters the vapor phase in the form of vapor, which will affect the purity of silver in the vapor phase.

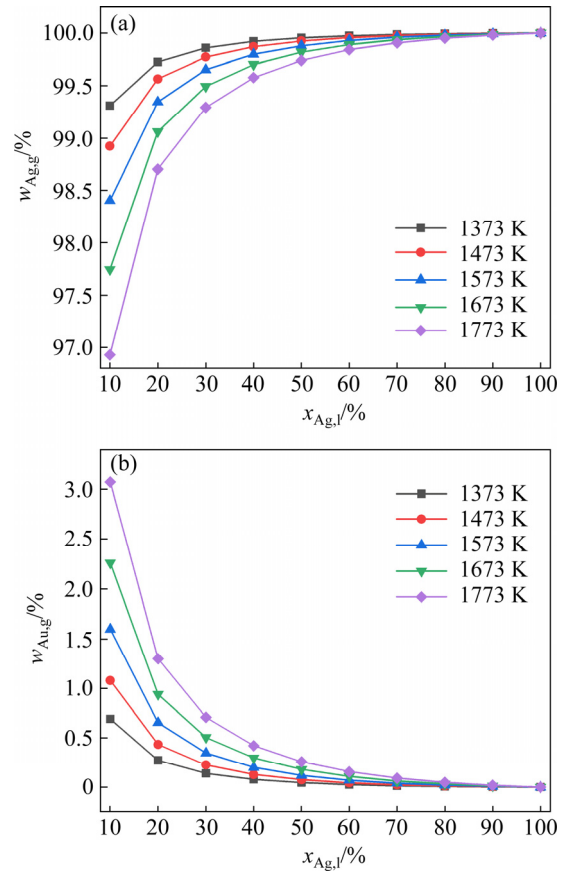


Fig. 4 Vapor–liquid equilibrium composition of Au–Ag alloy at different temperatures: (a) Ag; (b) Au

4 Vapor–liquid equilibrium phase diagram

For a binary system Au–Ag, there are

$$x_{\text{Ag}} + x_{\text{Au}} = 1; \quad y_{\text{Ag}} + y_{\text{Au}} = 1 \quad (7)$$

$$p = p_{\text{Ag}}^* \gamma_{\text{Ag}} x_{\text{Ag}} + p_{\text{Au}}^* \gamma_{\text{Au}} x_{\text{Au}} = p_{\text{Ag}}^* \gamma_{\text{Ag}} x_{\text{Ag}} + p_{\text{Au}}^* \gamma_{\text{Au}} (1 - x_{\text{Ag}}) \quad (8)$$

where x_{Ag} and x_{Au} are the mole fractions of gold and silver in the liquid phase, respectively; y_{Ag} and y_{Au} are the mole fractions of gold and silver in the vapor phase, respectively; p is the total pressure of the gold–silver binary system; p_{Ag}^* and p_{Au}^* are saturated vapor pressures of Ag and Au, respectively; γ_{Ag} and γ_{Au} are activity coefficients of Ag and Au in Ag–Au binary alloy, respectively.

According to the conditions of vapor–liquid equilibrium (VLE) of binary alloy system under vacuum and Raoult’s law [7], the expressions of the mole fractions in the liquid and vapor phases of the component Ag can be obtained:

$$x_{Ag} = \frac{p - p_{Au}^* \gamma_{Au}}{p_{Ag}^* \gamma_{Ag} - p_{Au}^* \gamma_{Au}} \quad (9)$$

$$y_{Ag} = \frac{p_{Ag}^* \gamma_{Ag} x_{Ag}}{p} \quad (10)$$

Substituting the corresponding γ_{Ag} , γ_{Au} , p_{Au}^* and p_{Ag}^* at different temperatures into Eqs. (7), (9) and (10), the T - x (temperature–composition) and p - x (pressure–composition) phase diagrams of the Au–Ag system can be obtained. The general algorithm for generating a T - x diagram is provided in Fig. 5 [6]. This is done with an iterative algorithm process by varying x_i to estimate a temperature and calculate the partial pressure of the two components until the sum of the partial pressures is equal to the total pressure on the system that we set. The T - x phase diagram of Au–Ag alloy system is shown in Fig. 6.

Figure 6 shows that the temperature range of liquid and vapor curves decreases with decreasing the system pressure. Low pressures have a positive effect on the separation of Ag and Au. For instance, if the purity of Ag we want is 0.9999, the distillation temperature at 10 Pa needs 1436.83 K, while at $p=1$ Pa, it only needs 1312.10 K.

In addition, an optimized temperature of the vacuum distillation can be obtained from the T - x phase diagrams. For example, if the purity of Ag we want is higher than 0.9999, then the distillation temperature at 1 Pa must be set to be lower than 1312.70 K. Conversely, if the purity of Au we want is higher than 0.9999, the distillation temperature at

1 Pa must be higher than 1713.52 K, and the content of Ag in the vapor phase is less than 0.03088 accordingly.

What’s more, the level rule [20] of phase diagrams can be applied to the VLE phase diagram to predict the mole fraction of volatile and residue if the temperature is specified. When assuming the distillation temperature of 1573 K and system pressure of 1 Pa, the mole fraction of Ag in the raw alloy is x_O , and a tie line of lever rule can be plotted in the T - x diagram, as shown in Fig. 6, which goes across the liquid and vapor curves to Points P and Q , respectively. When the system reaches VLE, the compositions of P and Q are x_1 and y_g , respectively. According to the lever rule, we can obtain

$$\frac{n_l}{n_g} = \frac{y_g - x_O}{x_O - x_1} = \frac{|OQ|}{|OP|} \quad (11)$$

where n_l and n_g are the amounts of substance of residues and volatiles, respectively. $|OQ|$ and $|OP|$ are the lengths of the line segment of OQ and OP , respectively.

If the total amount of substance raw alloy is n , and $n=n_l+n_g$, then the amounts of substance of residues and volatiles can be expressed as follows:

$$n_l = \frac{y_g - x_O}{y_g - x_1} n = \frac{|OQ|}{|PQ|} n \quad (12)$$

$$n_g = \frac{x_O - x_1}{y_g - x_1} n = \frac{|OP|}{|PQ|} n \quad (13)$$

where $|PO|$ is the length of the line segment of PQ .

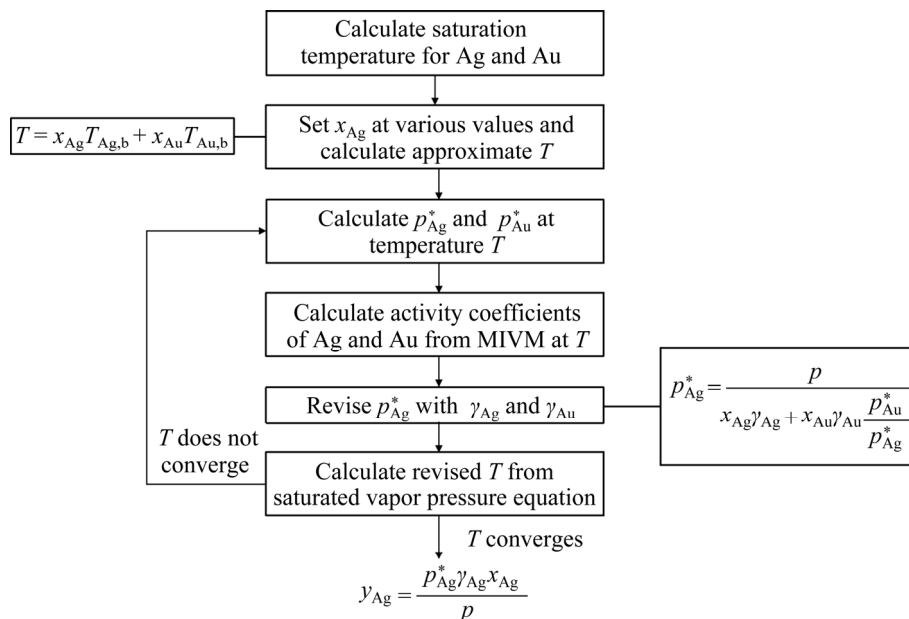


Fig. 5 Procedure of drawing T - x diagram of Au–Ag system

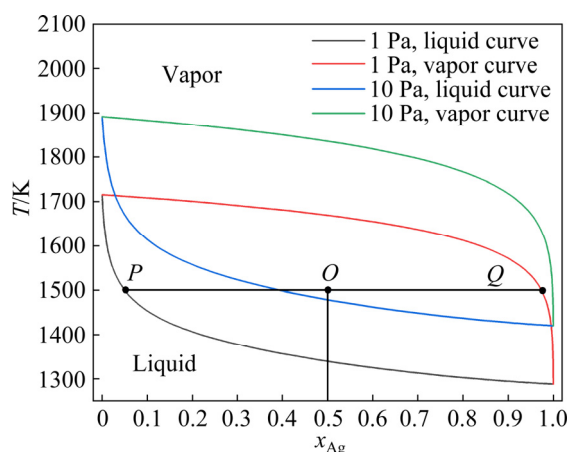


Fig. 6 T - x diagram of Au–Ag system at $p=1$ and 10 Pa

Similarly, according to Eqs. (12) and (13), the p - x phase diagram of the Au–Ag system at $T=1373$, 1573 and 1773 K can be calculated, as shown in Fig. 7. It can be seen from Fig. 7 that a lower distillation temperature requires a lower vacuum pressure. At the same temperature, with the increase of vacuum pressure, the purity of silver in the vapor phase increases, and the purity of gold in the liquid phase decreases. For instance, at the temperature of 1573 K, when the pressure ranges from 1 to 10 Pa, the content of Ag in vapor increases from 0.8984 to 0.9915, and the content of Au in liquid phase decreases from 0.9832 to 0.8347.

From the above analysis, it can be seen that when the temperature increases or the pressure decreases, the gold content in the vapor phase will increase and the silver purity will decrease. To ensure the purity of volatile silver, gold and silver vapors need to be separated by partition condensation.

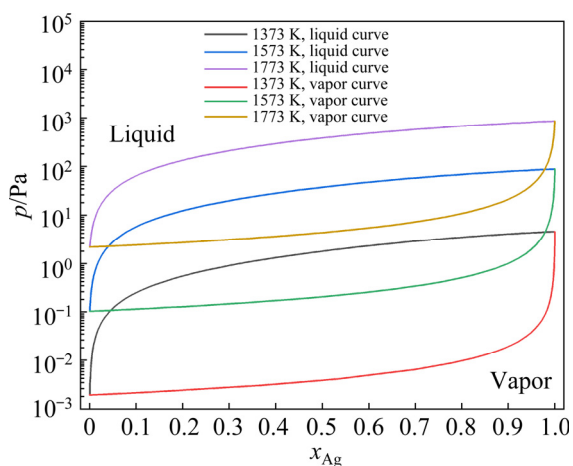


Fig. 7 p - x diagram of Au–Ag system at $T=1373$, 1573 and 1773 K

5 Triple point and condensation temperature

The behavior of condensation of metal vapor at a certain temperature and pressure can be judged by the three-phase equilibrium diagram of pure substance, as shown in Fig. 8. The temperature T_0 of the three-phase equilibrium Point O (T_0, p_0) is the melting point temperature of the pure substance, and the pressure p_0 is the saturated vapor pressure at the melting point temperature. Thus, p_0 can be calculated by Eq. (1). The temperatures and pressures of the triple point of gold and silver are given in Table 2.

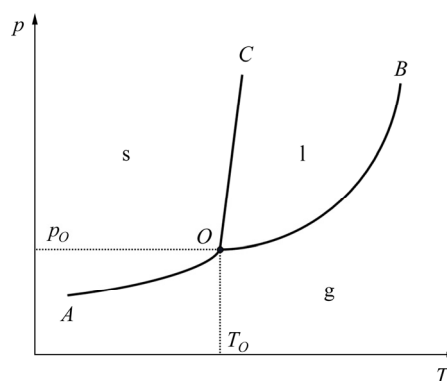


Fig. 8 Triple phase equilibrium diagram of pure substance

Table 2 Temperatures and pressures of triple-point of gold and silver

Component	Temperature/K	Pressure/Pa
Ag	1234.78	0.339
Au	1337.59	8.5×10^{-4}

It can be seen from Table 2 that the temperatures and pressures of the three-phase equilibrium point of gold and silver are 1234.78 K and 0.339 Pa, and 1337.59 K and 8.5×10^{-4} Pa, respectively. In the light of Fig. 8, when the pressures of the system are 1 and 10 Pa, with the temperature decreasing, both gold and silver vapors undergo a vapor–liquid phase transition and condense into a liquid state.

The pressure affects the condensation temperature of the metal vapor, and different pressures correspond to different condensation temperatures. Clausius equation gives the relationship between phase equilibrium pressure and temperature [20]:

$$\frac{dp}{dT} = \frac{\Delta H_m^*}{T\Delta V_m^*} \quad (14)$$

where ΔH_m^* and ΔV_m^* are the latent heat and volume change accompanying the phase change, respectively. For vapor–liquid phase transition, Eq. (14) can be expressed as

$$\frac{dp}{dT} = \frac{\Delta_{\text{vap}}H_m^*}{T(V_g - V_l)} \quad (15)$$

where V_l is the molar volume of the pure substance in the liquid state, V_g is the molar volume of the pure substance in the gas state, and $V_g \gg V_l$, so $\Delta V = V_g - V_l \approx V_g$, at the same time, by assuming that the vapor phase behaves as an ideal gas. Therefore, Eq. (15) can be expressed as

$$\frac{dp}{p} = \frac{\Delta_{\text{vap}}H_m^*}{R} \cdot \frac{dT}{T^2} \quad (16)$$

By substituting the values of $\Delta_{\text{vap}}H_m^*$ and p into Eq. (16), the condensation temperatures of gold and silver at different pressures can be obtained, as given in Table 3.

It can be seen from Table 3 that at the pressures of 1 and 10 Pa, the condensation temperatures of silver and gold vapors are 1287.90 and 1419.98 K, 1715.67–1891.91 K, respectively. In addition, the difference between the condensation temperatures of gold and silver is about 450 K, a relatively large difference. Therefore, when vacuum distillation is used to separate the Au–Ag alloy, a very small part of the volatilized gold can be separated from the silver vapor by fractional condensation with a condensing device, thereby improving the purity of the silver.

Table 3 Condensation temperatures of gold and silver at different pressures (K)

Pressure/Pa	Au	Ag
1	1715.67	1287.90
10	1891.91	1419.98

6 Conclusions

(1) At a fixed temperature, the saturated vapor pressure of silver is much higher than that of gold and the separation coefficient (β) is much more than one. The vapor–liquid equilibrium composition and VLE phase diagram of Au–Ag alloy at different

temperatures are obtained. From VLE phase diagrams, we can choose optimized conditions of the vacuum distillation according to the purity of the products.

(2) Gold and silver vapors condense into liquid state and the difference between the condensation temperatures of gold and silver is about 450 K in the system pressure range of 1–10 Pa. Therefore, the gold in vapor phase can be separated from the silver vapor by partition condensation with a condensing device, and thereby the purity of the silver is further improved.

Acknowledgments

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金银合金真空分离的理论研究

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摘要: 为准确预测金银合金真空蒸馏过程中产品成分与温度和压力的关系, 并为工业生产参数的设计提供便捷和有效的指导, 根据分子相互作用体积模型(MIVM), 计算不同温度下金银合金的分离系数(β)和气–液平衡成分。结合气液相平衡(VLE)理论, 绘制金银合金真空蒸馏的温度–成分、压力–成分相图。同时, 对金、银三相点和金、银蒸气冷凝温度进行计算。理论研究结果表明: 随着蒸馏温度的升高, 分离系数减小, 金在气相中的含量增加; 低温对分离金银具有积极效果; 在压强 1~10 Pa 范围内, 金、银冷凝温度相差约 450 K。

关键词: 金银合金; 真空分离; 分子相互作用体积模型(MIVM); 气液相平衡(VLE)相图; 冷凝温度

(Edited by Bing YANG)