Technical research on vacuum distillation to purify magnesium to 99.99% purity

Yuan Tian\textsuperscript{1,2,3}, Xiaopan Zhang\textsuperscript{2,3}, Tao Qu\textsuperscript{1,2,3}, Fei Lyu\textsuperscript{1,2,3}, Hao Du\textsuperscript{1,2,3}, Lei Shi\textsuperscript{1,2,3}, Bin Yang\textsuperscript{1,2,3} and Yongnian Dai\textsuperscript{1,2,3}

\textsuperscript{1} State Key Laboratory of Complex Nonferrous Metal Resources Clear Utilization, Kunming University of Science and Technology, Kunming 650093, People's Republic of China
\textsuperscript{2} National Engineering Laboratory for Vacuum Metallurgy, Kunming University of Science and Technology, Kunming 650093, People's Republic of China
\textsuperscript{3} Yunnan Provincial Key Laboratory for Nonferrous Vacuum Metallurgy, Kunming University of Science and Technology, Kunming 650093, People's Republic of China

\textsuperscript{*} Authors to whom any correspondence should be addressed.
E-mail: qutao_82@126.com and shdyshilei@163.com

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Abstract

In this study, the distribution and evaporation principles of impurities in distilled magnesium metal were investigated using a low vacuum ($8 \times 10^4$ Pa) distillation purification experiment and theoretical analysis. Provided all other factors remain unchanged (distillation time, pressure, and total area of evaporation), the optimum temperature for preparing high-purity Mg (99.99%) is 750 °C. A detailed analysis of purified Mg was obtained by inductively coupled plasma mass spectrometer (ICP-MS) for 10 major impurity elements. After distillation at 750 °C, low vapor pressure impurities in condensed magnesium, including Si, Mn, Al, Fe, Cu, Ni, and Sn were significantly reduced as other impurities were slightly reduced. Our analysis confirmed a decrease in the following impurities: Fe, Si, Mn, Cu, and Al were reduced from 21.8, 78.6, 68.4, 4.4, and 39.4 ppm to 1.2, 9.3, 6.0, 1.0, and 5.4 ppm, respectively; satisfying the 99.99% Mg standard. The evaporation rate and separation coefficient were calculated under experimental conditions. To better describe the distillation process of metallic magnesium under low vacuum conditions, the mean free path is also calculated in this study under actual conditions.

1. Introduction

In today's application-oriented high-tech world, the demand for high-purity materials suitable for demanding applications is constantly increasing. High-purity materials are required to prepare well-controlled materials and equipment. Simultaneously, the purity of a metal material significantly affects the performance of the compounds made from it. Owing to its exceptionally light structure, high-purity magnesium and its alloys have many advantages, including high strength, high weight ratio, good plasticity, excellent ductility, and magnetic shielding capabilities. Furthermore, it is commonly used in the preparation of rare metals, such as uranium and zirconium, which are mainly used in aerospace technology and weapons for the military [1–9]. In addition, high-purity Mg is demonstrating great prospects in biodegradable medical implant applications due to its good combination of load bearing mechanical properties and controllable corrosion rates, coupled with its biocompatibility and biodegradability characteristics [10–17].

Many of these applications require high-purity magnesium metal (99.99% and above). Currently, the methods of purifying magnesium mainly include the solvent refining method, zone melting, and the vacuum distillation method. The solvent refining method adopts the ability of certain metals or compounds to interact with the metal impurities in magnesium to form insoluble metal compounds, and eventually precipitate in the magnesium liquid, such that the purpose of removing impurities is achieved [18]. Although the solvent refining...
method can also be used to prepare 4N magnesium, a large amount of zirconium needs to be added to the refining agent to reduce the impurities in magnesium, thus increasing the cost and causing secondary pollution instigated by certain solvents [19]. In the zone melting method, the difference between the solubility of impurities in the molten state and the solid state of the metal is used to precipitate or change the distribution of impurity elements. To carry out the zone melting process, one or more liquid regions are made to move slowly and unidirectionally along the solid ingot. Impurities are concentrated in the melting zone, and purity of the base metal in the solidification zone increases. Metal purity increases as this process is repeated. The purity of the metal obtained by zone melting is very high (5N+), but because of the slow speed, the efficiency and output are not high [20]. The vacuum distillation method is widely used to prepare high-purity metals, including some rare earth metals [21–24]. Based on the difference of the saturated vapor pressure between the metal and the impurity, the metal is distilled alone or all the impurities are distilled leaving the metal, thus realizing metal–impurity separation to achieve purification. The vacuum degree can be adjusted to control the evaporation of impurities, thus achieving a high recovery rate in a short time. The current vacuum distillation method is carried out under high vacuum conditions, because of which the cost is high [25].

Wang et al [26, 27] prepared high-purity magnesium (4N+) using a vacuum method; these studies are all realized under extremely high vacuum conditions, with the vacuum degree < 10 Pa. Because magnesium metal has no liquid phase under high vacuum conditions, it does not condense into dense metallic magnesium ingots, which limits the industrial application of this method. To overcome the above problems, this study studied the preparation of high-purity magnesium using low vacuum distillation after adding a protective gas. The whole distillation process can realize the transformation of the liquid–gas–liquid phase of magnesium metal, so that the high-purity magnesium obtained by condensation has a good crystallization effect. As a result, continuous production can be realized and dense metal magnesium ingots can be obtained. At the same time, the requirements for vacuum pumps are also reduced, thus ensuring cost efficiency.

In this study, magnesium metal was purified using a low vacuum distillation purification method, which is based on a theory. First, the impurity separation coefficient has to be calculated, and the separation coefficient $\beta$ has to be introduced as the criterion for determining the possibility of the impurities separating. Second, it is necessary to calculate the vapor pressure, $P$, the evaporation rate, $W$, and the mean free path, $\lambda$, for vacuum distillation. The parameters can be used to select appropriate evaporation conditions to remove the main impurities in the metal; thereafter, the theoretical data are combined to obtain appropriate distillation parameters for preparing high-purity metals. In this study, the mass fraction of impurities was measured by inductively coupled plasma mass spectrometry (ICP-MS), and the distribution of impurities in the distilled metal was analyzed [28, 29].

2. Theory

2.1. Distillation

The vapor pressure of any metal element is a function of temperature. The distillation process is based on the difference in the vapor pressure ($P$) of the main impurities in magnesium. The saturated vapor pressure of impurities at different temperatures can be calculated using the Clausius Clapeyron equation, as represented in equation (1):

$$\log P^*(Pa) = A/T + B\log T + CT + D$$  \hspace{1cm} (1)$$

where ($P^*$) is the pressure in pascal, $T$ is the temperature in K; and $A$, $B$, $C$, and $D$ are constants determined for each impurity [30].

The mean free path $\lambda$ is a crucial factor that affects the efficiency of the distillation process. Considering the low vacuum and argon gas conditions, mainly with the presence of magnesium vapor and argon, the mean free path of magnesium vapor in the mixed gas can be calculated using Sutherland’s formula of extension [31]:

$$\lambda_{Mg} = \frac{1}{\sqrt{2}\pi n d^2_{Mg} + \pi n d^2_{Mg-Ar} \sqrt{1 + \frac{m_n}{m_M}}}$$ \hspace{1cm} (2)$$

where $d_{Mg}$, $d_{Mg-Ar}$, $n$, and $m_{Mg}$ and $m_{Ar}$ are the diameter of the magnesium atom ($3.6 \times 10^{-8}$ cm), average diameter of two atoms ($1.817 \times 10^{-8}$ cm), gas molecule density related to the temperature and pressure, and relative atomic masses, respectively [32].

A higher purity of magnesium metal can be prepared at a lower distillation rate. To shorten the distillation time and maintain the efficiency of the removal of impurities, the distillation temperatures selected in this study were 700, 750, and 800 °C. The evaporation rate $W_0$ of Mg at the three temperatures was calculated using the Langmuir formula [31]:

$$W_0 = \frac{P_0}{\lambda_{Mg} \sqrt{2\pi n d^2_{Mg}} + \pi n d^2_{Mg-Ar} \sqrt{1 + \frac{m_n}{m_M}}}$$ \hspace{1cm} (3)$$
where $P_{Mg}, M, T,$ and $\alpha$ are the vapor pressure, molecular weight of Mg, soaking temperature, and probability of the Mg molecule remaining on the surface ($\alpha \approx 1$), respectively.

2.2. Impurity separation

To predict the separation potential of different impure elements in a metal during vacuum distillation, a separation coefficient ($\beta$) index, which is a theoretical derivation of a non-ideal mixed $i$-$j$ binary system, is considered. The $\beta$ index is expressed as [33]:

$$\beta_i = \gamma_i / \gamma_j P_i^n / P_j^n$$

where $\gamma_i$ and $\gamma_j$ are the activity coefficients of components $i$ and $j$, respectively. $P_i^n$ and $P_j^n$ are the vapor pressures of these components, respectively, which can be calculated using equation (1). If $\beta_i$ is greater than 1, it means that the element $i$ is enriched in the gas phase when separated from element $j$, whereas the inverse is verified if $\beta_i$ is smaller than one. Although separation occurred in both cases, when $\beta_i$ is equal or extremely close to one, it is difficult to separate between $i$ and $j$. When $\beta_i \leq 1$ or $\beta_i \gg 1$, $i$ and $j$ are completely separated.

3. Experimental

3.1. Purification of magnesium by distillation

A vacuum tube furnace with a maximum power of 10 kW and a frequency of 50 Hz was employed for experimental validation. Figure 1 shows a sketch of the tube furnace apparatus developed and used to purify magnesium. It consists of a pure quartz tube that can be closed at both ends and a condenser, including a movable push rod. The inner diameter and length of the quartz tube are 0.04 and 1.3 m, respectively. The condenser comprised a water-cooled tube and a copper sheet, the copper sheet is round with a diameter of 0.03 m. The copper sheet is at the forefront to collect the distillate. The movable push rod is made of iron, with a length of 1 m and an inner diameter of 0.01 m. First, the magnesium raw material to be purified (MP 650 °C, BT 1090 °C) is pretreated. Oxalic acid is used to clean the oxide film on the surface of the magnesium block. It is dried using dry paper, and then immediately put it in high-purity graphite crucible and place it on the left end of the tube furnace. The push rod at the left end and the condenser at the right end are closed, and vacuum is briefly applied to remove the air in the tube and introduce argon gas. The dynamic vacuum degree is stable at $8 \times 10^4$ Pa. When the temperature in the tube increased to the set temperature, the crucible was pushed to the heating zone with the movable push rod, and then the push rod was retracted to the left end. Magnesium began to melt and then evaporated and condensed on the copper sheet, leaving impurities with high melting point and low vapor pressure at the bottom of the crucible. After the experiment, the condensed magnesium was collected.
on the copper sheet; it was cleaned with ethanol and placed in a sealed bag to prevent oxidation, keeping sealed until testing.

3.2. Analysis of magnesium

Industrial grade magnesium ingot (Mg9995B, see ICP-MS analysis in table 1) was applied in the experiments conducted in this study. Metal impurities were analyzed by ICP-MS. Comparing the Mg9999 primary magnesium ingot standard specified in the Chinese National Standard (GB/T3499-2011) with the raw materials in this experiment (see figure 2) [34], it can be observed that the Pb, Sn, Zn, Ni, and Ti in the raw materials reached the standard, with the following high-content impurities: Si, Mn, Fe, Al, and Cu, as the main removal objects in this experiment.
| T (K) | $\beta$(Zn) | $\beta$(Pb) | $\beta$(Mn) | $\beta$(Al) | $\beta$(Sn) | $\beta$(Cu) | $\beta$(Fe) | $\beta$(Ni) | $\beta$(Si) |
|------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
| 973  | 8.35        | $8.74 \times 10^{-4}$ | $1.38 \times 10^{-6}$ | $4.25 \times 10^{-9}$ | $2.76 \times 10^{-9}$ | $3.96 \times 10^{-10}$ | $9.36 \times 10^{-13}$ | $5.47 \times 10^{-14}$ | $3.97 \times 10^{-15}$ |
| 1023 | 8.48        | $1.35 \times 10^{-3}$ | $3.53 \times 10^{-6}$ | $1.35 \times 10^{-8}$ | $8.33 \times 10^{-9}$ | $1.49 \times 10^{-9}$ | $4.84 \times 10^{-12}$ | $3.54 \times 10^{-13}$ | $2.96 \times 10^{-14}$ |
| 1073 | 6.63        | $3.01 \times 10^{-3}$ | $2.73 \times 10^{-5}$ | $1.81 \times 10^{-7}$ | $9.96 \times 10^{-8}$ | $3.04 \times 10^{-8}$ | $2.15 \times 10^{-10}$ | $2.78 \times 10^{-11}$ | $3.32 \times 10^{-12}$ |
4. Results and discussion

4.1. Metallic impurities separation
Table 1 shows that industrial grade magnesium ingot has ten impurity components. The degree of separation between each impurity and magnesium is different. We calculated the saturated vapor pressure of ten impurities at 700, 750, and 800 °C using equation (1). As shown in figure 3, the saturation vapor pressure of impurities in magnesium is considerably different. At temperatures of 700, 750, and 800 °C, the saturated vapor pressure of zinc is the largest, which is very close to magnesium, only one order of magnitude away, while the two elements are the first to evaporate. As temperature increases, lead and manganese evaporate; the remaining silicon, iron, nickel, copper, aluminum, and tin have very low vapor pressures and are not volatile, therefore, the evaporated mass of these elements is very small.

Based on the corresponding vapor pressure of each metallic element, the separation coefficients ($\beta$) of impurities in magnesium-based alloys at different temperatures can be calculated, and the results are presented in table 2. The activity coefficient $\gamma$ of each component is 1 because the industrial grade magnesium ingot can be regarded as an infinite dilute solution with ten impurities such as Zn, and Mn, as the solutes. This shows that Fe, Pb, Cu, Si, Ni, Al, Mn, and Sn have significant tendency to be separated from Mg but also different separation behaviors owing to the different $\beta$ coefficients, as discussed earlier. However, the $\beta$ coefficients of Zn are always greater than one, denoting that Zn is mainly concentrated in the gas phase. During the entire distillation process, Zn evaporates first, followed by Mg, which is enriched in the gas phase. The distillation process is shown in figure 4. As the distillation temperature increases, the magnesium raw material melts first. During the melting process, both magnesium and zinc evaporate. Because the saturated vapor pressure of zinc is greater than that of magnesium, zinc evaporates faster than magnesium. At this time, there are many impurities in the magnesium melt. High melting and boiling point impurities, such as manganese, silicon, iron, aluminum, and other impurities, are not easy to evaporate. Such impurities get concentrated in the incompletely evaporated magnesium liquid and remain at the bottom of the crucible after the distillation is completed; some impurities may evaporate and finally condense and collect in the cold end along with the magnesium and zinc vapor.

4.2. Metallic impurities–purity
The vacuum distillation results were studied for the most common impurities in magnesium. Before this study, we conducted related experiments and found that magnesium can reach the maximum evaporation when the holding time is 40 min. The impurity contents obtained under vacuum conditions ($8 \times 10^4$ Pa) after a magnesium distillation time of 40 min at 700, 750, and 800 °C, are given in table 3. A comparison between the distillation results and 4N high-purity magnesium standard content is shown in figure 5, which shows that the
Table 3. ICP-MS analysis of after low vacuum distillation industrial magnesium ingot.

| T (K) | Fe (ppm) | Si (ppm) | Pb (ppm) | Sn (ppm) | Mn (ppm) | Cu (ppm) | Ni (ppm) | Al (ppm) | Ti (ppm) | Zn (ppm) |
|-------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|
|       | 20       | 20       | 10       | 20       | 20       | 3        | 3        | 20       | 5        | 30       |
| 973   | 5.1      | 13.1     | 0.82     | 0.24     | 9.1      | 0.7      | 2.2      | 13.4     | 0.22     | 13.1     |
| 1023  | 1.2      | 9.3      | 0.32     | 0.14     | 6        | 1        | 2.5      | 5.4      | 0.58     | 12.4     |
| 1073  | 4.5      | 13.4     | 0.50     | 0.37     | 12.7     | 1.2      | 2.4      | 10.6     | 0.45     | 12.6     |
Mg9995B industrial magnesium ingot reached the 4N national standard via distillation, and the high content of Fe, Si, Mn, Cu, and Al impurities in the raw materials were all reduced below the standard line. The remaining impurities (Ni, Zn, Pb, Sn, and Ti) that reached the standard did not change significantly with the distillation temperature.

At this temperature, all high-content impurities in the raw materials were reduced to the minimum, and the separation effect was the greatest. A lower content of low vapor pressure Fe, Si, Mn, and Al impurities was achieved. Fe, Si, Mn, and Al impurities were reduced from 21.8, 78.6, 8.4, and 39.4 ppm to 1.2, 9.3, 6.0, and 5.4 ppm, respectively, in the distilled metal with the remaining impurities left in the crucible. Combining the previous theoretical calculations, it can be determined that the saturated vapor pressure of zinc is very close to that of magnesium; although the change in zinc impurity content was insignificant (from 14.7 ppm to 12.4 ppm), it still met the 4N standard. For raw materials with excessive zinc content, multiple distillations will be required to achieve the standard. Zinc evaporates before magnesium, and magnesium remains in the liquid phase to achieve purification.

Table 4 shows the direct yield of magnesium and the removal rate of impurities in magnesium after vacuum distillation. It can be observed from the table that the final removal rate of high boiling impurities is relatively high (Mn, Fe, etc), combined with the separation coefficient in table 2. The separation coefficient of this type of metal is far less than one, such that it does not readily evaporate, causing most of it to remain in the liquid phase. The higher the removal rate of impurity metals, the higher the purity of the magnesium; however, some impurities have low removal rates owing to factors such as a separation coefficient greater than one (e.g., zinc) and instrument interference. Table 3 and figure 5 show that the preparation of 4N high-purity magnesium can be achieved under the three temperature conditions; however, table 4 shows that the removal rate of zinc is the highest at 750 °C and that the magnesium is almost completely evaporated. Therefore, 750 °C was selected as the optimum temperature.
In the distillation step, magnesium was evaporated and collected on a cooled copper sheet. The distillation conditions are given in Table 5. The mean free path, $\lambda$, of the vapor mainly depends on the temperature and vacuum degree. In the distillation process, $\lambda$ is used to determine the vapor transmission process. The mean free path, $\lambda$, was $0.28 \times 10^{-4} \text{ cm}$ under the optimum experimental conditions ($750^\circ \text{C}$), while the estimated distance $L$ between the evaporator surfaces was approximately 5 cm. Because this study conducted the distillation with a low vacuum and argon protective gas, the upper part of the magnesium melt is filled with argon molecules. When magnesium molecules move up from the evaporation surface, they will continuously collide with argon molecules. From the calculated mean free path, it can be observed that two consecutive collisions between magnesium molecules and argon molecules need to move a distance of $0.28 \times 10^{-4} \text{ cm}$. This proves that magnesium and argon molecules collide frequently. After collision, some magnesium molecules will return to the melt; others will collide with the tube wall consistently, and finally condense in the condensation zone with the remaining magnesium molecules. These situations affect the evaporation rate of magnesium, the main factor causing the actual evaporation rate of magnesium to be lower than the theoretical value.

According to Table 5, it can be observed that the average distillation rate is approximately $4.386 \times 10^{-4} \text{ g cm}^{-2} \text{s}^{-1}$ under the best experimental conditions, which is approximately four orders less than the theoretically calculated value, $5.4 \text{ g cm}^{-2} \text{s}^{-1}$. In addition to the mean free path, the evaporation rate is also affected by the following factors: (i) the evaporation surface is partially occupied by other low vapor pressure impurities. The magnesium liquid in the melt is hindered when it evaporates, thus evaporation cannot be maximized; (ii) the decrease in the vapor pressure of the solvent (magnesium melt) resulting from the influence of solute (impurities); (iii) the narrow space in the tube and large number of vapor molecules gathered at the top, which hinders magnesium evaporation from the melt [35]. As the temperature rises, the distillation rate shows a downward trend, mainly because when the temperature increases, the concentration of nonvolatile impurities also increases, giving them the ability to occupy some positions on the evaporation surface and reducing the number of molecules evaporated from the surface, thus reducing the evaporation rate.

In this study, because the evaporation of magnesium was hindered by argon molecules under experimental conditions, the actual average distillation rate deviated from the theoretical calculation value. It can be observed from the mean free path that the continuous and frequent collisions of two molecules eventually led to a decrease in the average distillation rate. We used the mean free path, $\lambda$, to explain the difference between the theoretical and actual distillation rate.

### 5. Conclusions

In this study, we obtained the optimal experimental conditions required for the distillation of high-purity magnesium (approximately 4N) from starting magnesium (Mg9995B). The optimum temperature for preparing 4N magnesium is $750^\circ \text{C}$ under low vacuum ($8 \times 10^4 \text{ Pa}$) distillation and constant evaporation surface.

Owing to the huge difference between the vapor pressures of the various impurities (e.g. Fe: $4.84 \times 10^{-11} \text{ Pa}$, Si: $5.74 \times 10^{-11} \text{ Pa}$, Cu: $1.49 \times 10^{-9} \text{ Pa}$, Al: $1.35 \times 10^{-9} \text{ Pa}$, and Mn: $3.53 \times 10^{-6} \text{ Pa}$ at 1023 K), theoretical interpretations indicate the removal potential of Fe, Si, Mn, Cu, and Al from Mg—the five most common impurities in Mg—via vacuum distillation. The practical experiments on an industrial magnesium ingot (i.e., Mg9995B, including 21.8 ppm of Fe, 78.6 ppm of Si, 68.4 ppm of Mn, 4.8 ppm of Cu, and 39.4 ppm of Al) demonstrates that removal of Fe, Mn, Si, Cu, and Al agrees with the theoretical predictions. The Fe, Mn, Si, Cu, and Al contents could be reduced to 1.2, 6.0, 9.3, 1.0, and 5.4 ppm, respectively, in the condensed Mg-product. The content of other impurities also included in the 4N standard also exhibited a downward trend.

The vapor pressure of impurities and the mean free path of Mg vapor play an important role in the purity of the distillation product and the distillation efficiency. Under the optimal conditions of this experiment, the free path, $\lambda$, of evaporation is approximately $0.28 \times 10^{-4} \text{ cm}$, which is mainly caused by frequent collisions between magnesium and argon molecules. Finally, the actual distillation rate dropped to approximately...
4.386 \times 10^{-4} \text{ g cm}^{-2}\text{s}^{-1}, which deviated significantly from the theoretical value. As the temperature increased, the evaporation rate also exhibited a downward trend.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

ORCID iDs

Yuan Tian  
Fei Lyu  
Lei Shi

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