https://doi.org/10.46813/2026-161-035

UDC 669.725:54351

OPTIMIZATION OF BERYLLIUM PURIFICATION PROCESSES USING VACUUM DISTILLATION

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The paper presents the results of research on improving the method of vacuum distillation of beryllium during a multistage refining process. Some features of the removal of metallic impurities in the process of beryllium distillation are discussed. It is concluded that the efficiency of the beryllium refining process by vacuum distillation can be increased by selecting optimal distillation conditions under controlled environments.

INTRODUCTION

The specificity of beryllium as an object of purification lies in its high chemical activity at elevated temperatures. This high activity of beryllium negatively affects the efficiency of all existing purification methods (electro transport, vacuum remelting, zone melting, vacuum distillation, etc.) [1–3]. Until recently, it was believed that achieving a high degree of purification required combining several refining methods — for example, vacuum distillation followed by zone melting, using starting material obtained through electro transport [2, 4]. According to recent studies [5, 6] that investigated beryllium refining processes, certain patterns were identified that allow for obtaining beryllium with a purity of more than 99.999 wt.% using only vacuum distillation. To achieve such purity, at least three cycles of vacuum distillation were required, starting with beryllium of 99.8 wt.% purity.

This work examines the main factors influencing the separation of the main component and impurities, the possibilities of improving refining efficiency, and the key experimental results obtained in this area.

MATERIALS AND METHODS

The beryllium distillation process was carried out in a vacuum system with a working chamber volume of 500 l [7]. The residual gas pressure during distillation was maintained at a level of $10^{-4}…10^{-5}$ Pa using oil-free Pfeiffer Vacuum turbomolecular and forevacuum pumps. For beryllium refining, a distillation device previously developed and described in [6] was used. This device is almost entirely made (crucible, shields, substrates, heaters, etc.) of Mo and BeO. Fig. 1 shows the schematic of this distillation device, the advantages of which will be discussed later.

Evaporation and condensation temperatures were measured using thermocouples. The melt temperature was maintained in the range of 1330…1370 °C, with the evaporation rate varying between 0.16…0.35 g/(cm²·h). The specific evaporation rate was estimated by calculating the ratio of the mass of the produced condensate over time to the surface area of evaporation. The ratio of the evaporation surface area to the condensation surface area in the setup used was 1:15. Condensation of the vapor was carried out on columns made of molybdenum sheet, shaped as cylinders. A temperature gradient along the condensate was created by the heat radiation of the evaporator, and additionally, the central part of the condensation column was heated by an independent shielded molybdenum resistance furnace. This technological solution allows for the formation of separate temperature zones of condensation along the height of the column.

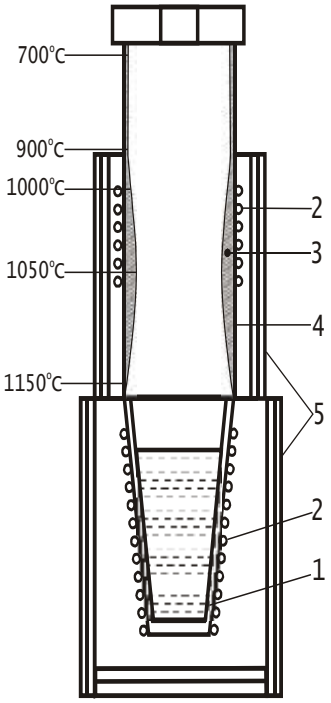

Fig. 1. Schematic drawing of the condensation column and crucible for melting Be:
1 – crucible; 2 – resistance furnace; 3 – distillate;
4 – condensation column; 5 – molybdenum screens

By installing an additional shielded heater in the middle part of the column, it became possible to expand the high-temperature zone of dense condensate formation to the middle of the column. In this high-temperature zone (1150…950 °C), the purest metal condenses. The temperature distribution along the height of the column is shown in Fig. 1. In the upper part of the column, which lacks additional heating, there is a significant temperature drop (see Fig. 1). The condensate formed in this area is porous, with a high

content of volatile impurities, and its mass does not exceed 10% of the total condensate mass.

To carry out research on optimizing the vacuum distillation processes, powdered hot-pressed beryllium was used as the starting material. The starting material was remelted into ingots in the furnace of the distillation apparatus. The remelted ingots weighed 1.2…1.7 kg and had a beryllium content of approximately 99.8 wt.% (excluding C, N, and O). Table 1 presents the average concentrations of impurity elements in the resulting ingots.

Analytical studies of the quantitative content of metallic impurities in technically pure beryllium samples, as well as in high-purity metal obtained by vacuum distillation, were carried out using the industrial laser mass spectrometer EMAL-2. The EMAL-2 mass spectrometer (manufactured by NPO "Electron", Sumy) is built on the classical Mattauch-Herzog scheme, with focusing by angles and energies, and uses a laser-plasma ion source [8]. The methodology for determining the elemental composition of beryllium samples by laser mass spectrometry is described in detail in [9].

Table 1

Average concentrations of impurity elements in the original ingots

Element	Na	Mg	Al	Si	K	Ca	Ti	Cr	Mn	Fe	Ni	Cu	Zn
Mas.%	0.001	0.0003	0.02	0.01	0.001	0.002	0.005	0.02	0.003	0.03	0.005	0.01	0.005

EXPERIMENTAL RESULTS AND DISCUSSION

To determine the optimal distillation conditions for improving the purity level of the obtained distillate, a series of experiments was conducted to study the influence of the following parameters: specific evaporation rate of the metal; temperature distribution along the condensation column; effectiveness of repeated distillation cycles [6].

Fig. 2 shows the distribution of concentrations of metallic impurities Fe, Cu, Al, Mn in the condensation zone of the highest-purity metal, depending on the specific evaporation rate (C/C_0 – the ratio of the average concentration of the corresponding element in the condensate based on two measurements to its concentration in the original metal). As can be seen from Fig. 2, the mass spectrometric studies indicate that in order to reduce the content of metallic impurities, distillation should be carried out at a specific evaporation rate in the range of 0.18…0.35 g/(cm²·h). To achieve the highest separation coefficient, summed across all impurities, the optimal evaporation rate should be within 0.22…0.27 g/(cm²·h).

Table 2 and Figs. 3 and 4 show the results of the impurity element analysis of beryllium distillate along the column height, obtained at an evaporation rate of 0.25 g/(cm²·h). Sample No. 1 is at the bottom of the column, and sample No. 4 is at the top of column.

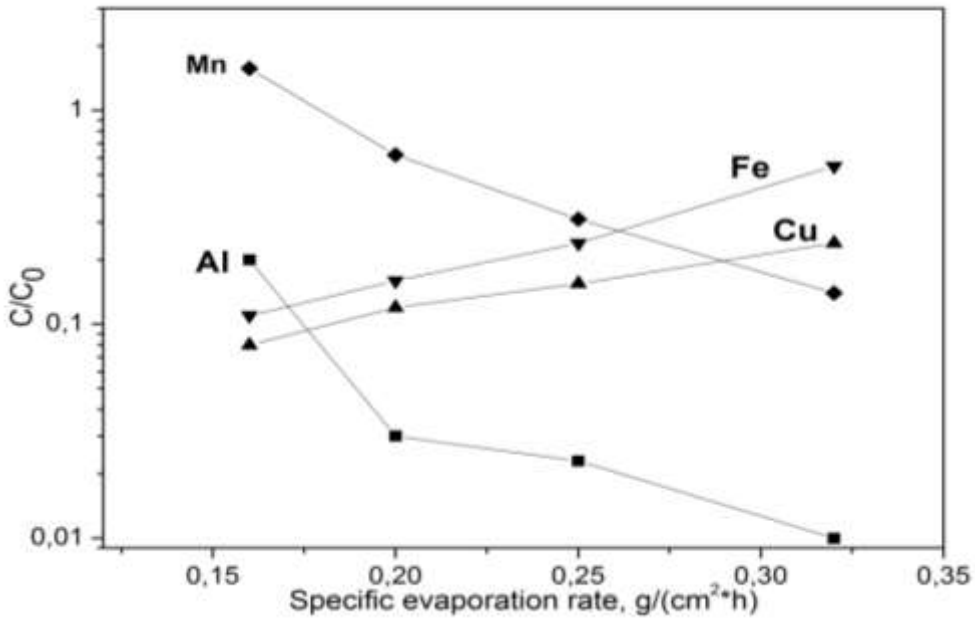

Fig. 2. Change in impurity concentrations in the condensation zone of the purest metal depending on the specific evaporation rate

Table 2

Distribution of element concentrations along the column of the primary condensate

Primary distillation	Element, ppm mass. (1 ppm mass. = 0.0001%)														
	Na	Mg	Al	Si	P	Cl	K	Ca	Ti	Cr	Mn	Fe	Ni	Cu	Zn
Sample 1	<0.04	<0.05	2.6	10	<0.06	<0.1	<0.1	2	0.5	10	10	100	2	26	<0.4
Sample 2	<0.04	<0.05	30	30	0.6	2	2	1	1	16	45	70	1	5	1
Sample 3	0.1	0.8	660	30	230	8	10	6.4	<0.2	1	230	0.8	<0.3	<0.3	0.8
Sample 4	3	10	6900	310	370	25	50	630	<0.2	<0.2	670	<0.3	<0.3	<0.3	10
Original	10	3	700	400	30	20	10	10	50	300	90	360	50	100	5

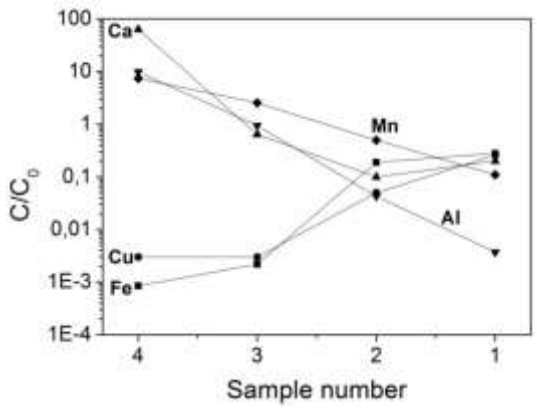

Fig. 3. Variation in the concentration of impurities Fe, Cu, Mn, Al, Ca in the primary distillate along the height of the column

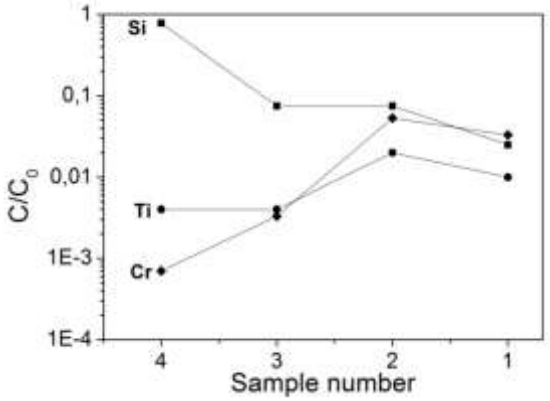

Fig. 4. Variation in the concentration of impurities Si, Ti, Cr in the primary distillate along the height of the column

As can be seen from Table 2 and Figs. 3 and 4, high concentrations of volatile impurities (Na, Mg, K, Ca, P) are observed in the upper part of the column. The concentrations of Ti, Cr, and Ni decrease by 20–50 times after one distillation cycle, and with repeated distillation cycles, these impurities do not determine the purity of the resulting distillate. The condensation zone of the purest metal (the lower half of the column – sample No. 1, sample No. 2) can be divided into two sections based on the distribution of the impurity concentrations of Fe, Cu, Al, and Mn. During deep purification of beryllium, these impurities determine the purity of the distilled metal. As can be seen in Fig. 3, in the condensation zone of the purest metal, a front is observed between low-volatile (Fe, Cu) and highly volatile (Mg, Al, Mn) impurities. As the evaporation rate increases, this front moves up the column, and conversely, as the rate decreases, the front moves down the column. This effect can be exploited to increase the efficiency of multiple distillations. Specifically, primary and secondary distillations can be performed at different evaporation rates. Primary distillation is best performed at high metal evaporation rates in the range of 0.3…0.33 g/(cm²·h). This will significantly reduce the concentration of highly volatile impurities in the primary condensate. Repeated distillation is best performed at low metal evaporation rates in the range of 0.18…0.23 g/(cm²·h), which will significantly reduce the concentration of non-volatile impurities. The feasibility of this approach is also confirmed by the distribution of silicon concentration along the column. Its concentration along the column remains virtually constant, with only its concentration being half that of the lower portion of the column.

Table 3 shows the dynamics of impurity content in beryllium following two distillation cycles conducted at different metal evaporation rates, according to the conditions described above. The impurity content calculation from Table 3 after two distillations yields beryllium with purity of 99.999% by weight, using a starting metal with a purity of 99.8% by weight (based on metallic impurities).

Improving the beryllium vacuum distillation process to reduce the number of repeated cycles can also be achieved by taking into account the significant difference in impurity content in the upper and lower portions of the condensate after the primary distillation. Since the upper portion of the condensate is enriched in highly volatile impurities, while the lower portion is enriched in less volatile ones, they should not be mixed during repeated distillations. Instead, these portions should be redistilled separately. The primary condensate, after removing the top fraction (10% of the condensate mass), is divided into two parts, the lower part being half the length of the upper part. These parts, collected after several cycles of primary distillation, are then subjected to separate redistillation.

Table 3

Impurity content after two cycles of beryllium distillation (wt.%)

Elem.	Original	1 cycle 0.33 g/(cm²·h)	2 cycle 0.2 g/(cm²·h)
Na	0.001	0.000004	< 0.000001
Mg	0.0003	0.000005	< 0.000002
Al	0.02	0.0001	0.000003
Si	0.01	0.0005	0.0001
P	0.003	0.00001	<0.000002
Cl	0.002	0.00001	<0.000003
K	0.001	0.000005	<0.000003
Ca	0.001	0.00001	<0.000003
Ti	0.005	0.0001	<0.000004
Cr	0.02	0.0006	0.00005
Mn	0.003	0.0003	0.0001
Fe	0.03	0.01	0.0005
Ni	0.005	0.0003	0.00001
Cu	0.01	0.001	0.00005
Zn	0.0005	0.0001	< 0.00001
Be	99.8	99.99	99.9991

Table 4

Impurity content after two cycles of beryllium distillation (wt.%)

Elem.	Original	1 cycle 0.25 g/(cm²·h)	2 cycle 0.32 g/(cm²·h)
Na	0.002	0.00001	< 0.000001
Mg	0.0004	0.00007	< 0.000002
Al	0.04	0.002	0.00001
Si	0.02	0.0015	0.0003
P	0.005	0.0001	< 0.000002
Cl	0.001	0.0001	< 0.000003
K	0.0007	0.0001	< 0.000003
Ca	0.001	0.0002	< 0.000003
Ti	0.006	0.0001	< 0.000004
Cr	0.007	0.0004	0.00001
Mn	0.003	0.0015	0.0001
Fe	0.03	0.002	0.0005
Ni	0.004	0.00008	< 0.000006
Cu	0.005	0.00025	0.00003
Zn	0.001	0.0001	< 0.00001
Be	99.8	99.99	99.999

Based on previous studies [6], it is reasonable to perform repeated distillations of the upper and lower parts of the primary distillate at different specific evaporation rates. The most optimal evaporation rate for the upper part is 0.3…0.32 g/(cm²·h), and for the lower part – 0.17…0.2 g/(cm²·h). At these rates, the highest separation factors for the dominant impurities are achieved. During repeated distillation of the upper part of the distillate, 90% of the charge is evaporated, while in the case of repeated distillation of the lower part, approximately 70% of the material loaded into the crucible is evaporated. Primary distillations are carried out at an evaporation rate of 0.22…0.27 g/(cm²·h), when the total separation coefficient for all impurities is highest.

Table 5

Impurity content after two cycles of beryllium distillation (wt.%)

Elem.	Original	1 cycle 0.25 g/(cm²·h)	2 cycle 0.19 g/(cm²·h)
Na	0.002	< 0.000004	< 0.000001
Mg	0.0004	< 0.000005	< 0.000002
Al	0.04	0.0002	< 0.000002
Si	0.02	0.0006	0.0002
P	0.005	< 0.000006	< 0.000002
Cl	0.001	< 0.00001	< 0.000003
K	0.0007	< 0.00001	< 0.000003
Ca	0.001	0.0002	< 0.000003
Ti	0.006	0.00007	< 0.000004
Cr	0.007	0.0003	0.00003
Mn	0.003	0.0004	0.0001
Fe	0.03	0.0075	0.0005
Ni	0.004	0.0002	< 0.000006
Cu	0.005	0.001	0.00005
Zn	0.001	0.00005	< 0.00001
Be	99.8	99.99	99.9991

Table 4 presents the elemental analysis data for the beryllium condensate, where the upper portion of the primary distillate was re-distilled at a specific evaporation rate of 0.32 g/(cm²·h). Table 5 presents the data for the lower portion of the primary distillate, where the lower portion was re-distilled at a specific evaporation rate of 0.19 g/(cm²·h).

CONCLUSIONS

The conducted research has led to the development of new technological approaches that improve the efficiency of beryllium refining by vacuum distillation, using a temperature gradient in the condensation column and a controlled specific evaporation rate. By carrying out the primary and secondary distillation within the optimal range of metal evaporation rates for each cycle or by performing separate repeated distillation of beryllium, the number of distillation cycles required to obtain beryllium with a purity of 99.999% by mass can be reduced from three to two.

REFERENCES

1. V.E. Ivanov, I.I. Papirov, G.F. Tikhinsky, V.M. Amonenko. *Pure and ultrapure metals.* M.: "Metallurgy", 1965, 263 p.
2. I.I. Papirov, G.F. Tikhinsky. *Physical metallurgy of beryllium.* M.: "Atomizdat", 1968, 450 p.
3. G.F. Tikhinsky, G.P. Kovtun, V.M. Azhazha. *Obtaining ultra-pure rare metals.* M.: "Metallurgy", 1986, 160 p.
4. I.I. Papirov, G.F. Tikhinsky, L.A. Jacobson. *Vacuum distillation of beryllium:* Review. Kharkov: NSC KIPT, 2002, 22 p.
5. N.S. Pugachev, I.V. Shpagin, A.D. Solopikhin, V.D. Virich, O.V. Kisel, K.V. Kovtun. On the Influence of Refractory Metals (Nb, Mo) on the Efficiency of Beryllium Purification by Vacuum Distillation // *Proceedings of the 9th International Symposium. High-Purity Metallic and Semiconductor Materials*. Kharkov, 2003, p. 10-14.
6. V.M. Azhazha, V.D. Virich, K.V. Kovtun, N.S. Pugachev, I.V. Shpagin. Study of the Beryllium Purification Process by Vacuum Distillation by the Method of Laser Mass Spectrometry // *Bulletin of Kharkov University. Physics Series "Nuclei, Particles, Fields"*. 2004, issue 3/25/, N 674, p. 35-41.
7. N.S. Pugachev, I.V. Shpagin, A.D. Solopikhin, V.D. Virich, O.V. Kisel, K.V. Kovtun. Improvement of the vacuum system of the beryllium distillation unit // *Proceedings of the 3rd International Conference OTTOM-3*. Kharkov, Ukraine, 2002, p. 217-218.
8. A.I. Boriskin, V.M. Eremenko, I.S. Lyalko, A.S. Bryukhanov, O.D. Smiyan, Yu.A. Bykovsky. Analytical and instrumental characteristics of the EMAL-2 device // *Control Instruments and Systems*. 1983, N 1, p. 26-29.
9. V.D. Virich, O.V. Kisel, K.V. Kovtun, I.I. Papirov, N.S. Pugachev, L.A. Yakobson, A.A. Vasiliev, V.A. Shkuropatenko. Elemental analysis of high-purity beryllium by laser mass spectrometry // *Problems of Atomic Science and Technology. Series "Vacuum, Pure Materials, Superconductors.* 2003, N 5, p. 46-50.

Article received 04.11.2025

ОПТИМІЗАЦІЯ ПРОЦЕСІВ ОЧИЩЕННЯ БЕРИЛІЮ ПРИ ВАКУУМНІЙ ДИСТИЛЯЦІЇ

В.Д. Вірич, О.В. Кисіль, К.В. Ковтун, А.Д. Солопіхін

Наведено результати досліджень щодо удосконалення методу вакуумної дистиляції берилію при багатостадійному процесі рафінування. Обговорено деякі особливості видалення металевих домішок у процесі дистиляції берилію. Зроблено висновок, що підвищення ефективності процесу рафінування берилію методом вакуумної дистиляції може бути досягнуто підбором оптимальних режимів проведення дистиляції у контрольованих умовах.