

## INFLUENCE OF Zr-Fe GETTER FILTER ON Cd AND Zn DEEP REFINING OF INTERSTITIAL IMPURITIES

*A.I. Kondrik, G.P. Kovtun, A.P. Shcherban', D.A. Solopikhin*

*National Science Center «Kharkov Institute of Physics and Technology»,  
Kharkov, Ukraine*

*E-mail: kondrik@kipt.kharkov.ua*

The paper presents the analytical and experimental studies of the process of cadmium and zinc refinement of interstitial impurities in a vacuum using a getter filter of Zr-Fe alloy. The thermodynamic analysis of reduction reactions of cadmium, zinc, carbon oxides, and zinc nitride with getter material of Zr-Fe alloy, depending on the temperature and vacuum conditions of the metals refining process have been accomplished. The experiments showed the effectiveness to use the Zr (51)-Fe (49) wt. % alloy getter filter for Cd and Zn refinement of interstitial impurities.

### INTRODUCTION

High-purity source metals Cd and Zn are composite components of many compound semiconductor and scintillation materials. Multi-semiconductor (CdTe, CdZnTe), and scintillation single crystals (CdWO<sub>4</sub>, ZnWO<sub>4</sub>, CdMoO<sub>4</sub>, ZnMoO<sub>4</sub>, ZnSe) will be applied to the manufacture of ionizing radiation detectors [1], low-background scintillators for registration of various electromagnetic radiations, including that rare events, such as neutrinoless double beta-decay of nuclei and registration of dark matter particles [2], which at the present time have a greater interest. For the synthesis of high-quality detector materials the purity of the source zinc and cadmium should be not less than 99.9999...99.999999%, at that the concentration of each of the metallic impurity full range (up to 80 elements) is not more than  $1 \cdot 10^{-6}$  wt.%, including the interstitial impurities fraction (C, N, O) should be at the level of  $1 \cdot 10^{-5}$  wt.%. Therefore, due to the strong demands to the purity of the source components across the full range of impurity elements it is necessary to take into account all the requirements for the pure material during preparation of high-purity Cd and Zn. It requires the research and development of new approaches to the processes of the metals deep refining.

During the development of effective removal of interstitial impurities it is important to know their behavior in metals in terms of thermodynamics. This information is essential for selection of technological conditions of the purification process.

### 1. PROBLEM

The purity of the refined cadmium and zinc are mainly limited by the concentration of residual interstitial impurities [3]. Removing them from the metals is difficult due to the formation of strong bonds of these impurities with the substrate. Typical content of interstitial impurities in high-pure Cd and Zn is at least  $5 \cdot 10^{-2}$ ... $5 \cdot 10^{-3}$  wt.%, while the removal of metal impurities to the concentration  $< 1 \cdot 10^{-5}$  wt.% may be achieved by conventional distillation methods of refinement. Vacuum distillation is the best method for removal of gaseous impurities from the reactive metals, since in this case there is a decrease of oxidation and gas absorption. Oxygen and carbon, as a rule, are not removed

from the metal in the atomic state, but rather in form of compounds, however during distillation in vacuum it is necessary to combine the evaporation and condensation with reducing reactions that may be performed by using chemically active getter filters.

When selecting the getter material to clean the Cd and Zn, the following requirements to getter have been taking into account: high reactivity to gas impurities (H, N, O) and carbon-containing gases; no interaction of getter with the Zn and Cd vapors; low values of the getter material vapor pressure at the temperature of use, high sorption capacity at the operating temperature of distillation (690...810 K).

Sorption properties of a number of chemically active metals, intermetallic compounds and alloys based on zirconium and hafnium have been investigated previously in KIPT [4, 5]. These materials are suitable for the operating temperature of 500...800 K and can be repeatedly used after the relevant activation.

As follows from [4], high sorption properties inherent in the alloys of zirconium and hafnium with iron. Zr-Fe alloy was selected as the material for getter filter recommended to remove interstitial impurities from Cd and Zn, due to its high sorption characteristics, ease of production and relatively low cost.

The aim of this study is to investigate the processes of separation of Cd and Zn from interstitial impurities by means of vacuum distillation method using Zr-Fe getter filter as a reductant.

### 2. ANALYSIS AND EXPERIMENTAL STUDY OF DEEP CLEANING CADMIUM AND ZINC

At the initial purification stage the removal of carbon and oxygen occurs as volatile compounds with other impurities and depends on their proportions. With increasing the metal purity the oxygen, carbon and nitrogen, forming strong bonds with a base in the form of oxides, carbides, nitrides, are often become as residual impurities which are difficult to remove. Evaporating metal may also form compounds with atoms and molecules of the gaseous environment. If formed compounds are stable, the impurity may get into the distillate. Thus, during deep refining of metals it should be pay attention to the behavior of stable oxides, carbides and nitrides. Let us consider the behavior of interstitial impurities in Cd and Zn firstly for the example of oxygen.

To assess the effect of vacuum on the stability of oxides the standard Gibbs energy change  $\Delta G_{MeO}^0$  may be used for the reaction of metal oxide formation, adjusted by the amount that takes into account the transition to a gaseous state at a pressure P. Distillation of Cd and Zn in vacuum may be carried out at the temperature  $T \sim 0.6 \cdot T_{boil}^{MeO}$ , where  $T_{boil}^{MeO}$  is the boiling temperature of metal under atmospheric pressure.

For the case where the activity of MeO is equal to 1 as well as  $T_{boil}^{Me} < T_{boil}^{MeO}$  the oxygen potential of formation of bivalent metal solid oxide  $\Delta G_{MeO}$  is given by

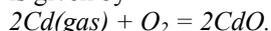
$$\Delta G_{MeO} = \Delta G_{MeO}^0 - 2RT \ln P, \quad (1)$$

where  $\Delta G_{MeO}^0$  is the standard Gibbs energy change for the reaction of the metal oxide formation; T is the temperature of process, K; P is the vapor pressure of the metal, bar; R is the universal gas constant.

The processes of vacuum evaporation of cadmium, zinc and oxygen impurity are characterized by the reactions of formation and decay of the metals oxides. Zinc forms in the condensed and gaseous states the monoxide ZnO [6], the reaction of formation of which is as follows:



Cadmium forms a stable oxide CdO, existing in a condensed and gaseous states. The formation reaction of cadmium oxide is given by



Let us estimate the stability change of the oxides with a temperature and under different vacuum condition. For the distillation of zinc at the temperatures above the boiling point (1180 K), the oxygen potential of ZnO with taking into account (1) will be written as follows:

$$\Delta G_{ZnO} = -910012 + 375.22 \cdot T - 38.3 \cdot T \lg \frac{P}{1.013 \cdot 10^5}, \quad (2)$$

where T is the temperature of process, K; P is the vapor pressure of the metal (zinc), Pa.

However, due to the strong evaporation of zinc in vacuum even at  $T = 730 \dots 870$  K, it was possible to carry out the distillation at these temperatures and extrapolate the expression (2) on this area, because here we have a Zn vapor phase also. In this temperature range ZnO exists in the solid state, which allows using the formulas (1), (2). Oxygen molecules and atoms of zinc vapors get in the getter filter and they can form there oxide, therefore it is necessary to compare the  $\Delta G_{ZnO}$  for formation of oxide ZnO with  $\Delta G_{MeO}$  for the formation of getter components oxides within operation ranges of temperature and pressure of gaseous medium. Molecules ZnO, formed in the vapor phase, can also reach to getter by means of carrying away them with intensely evaporating Zn atoms under vapor pressure of zinc  $\sim 133 \dots 13.3$  Pa. A similar physical model can be assumed to study the stability of CdO.

Oxygen potential of CdO is given by

$$\Delta G_{CdO} = -722426 + 399.24 \cdot T - 38.3 \cdot T \lg \frac{P}{1.013 \cdot 10^5}. \quad (3)$$

The Gibbs energy change applied to nitrides formation can be considered for impurity N. Zinc reacting with nitrogen and forms relatively stable chemical compound  $Zn_3N_2$  which is characteristic one for elements of group IIA [7]. According to experimental data the compound  $Zn_3N_2$  is stable up to 623 K in a vacuum and up to 773 K in air environment, and the solubility of nitrogen in zinc is low.

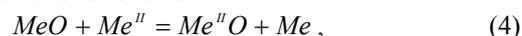
Nitrogen can form a cadmium nitride  $Cd_3N_2$  which decomposes at a temperature close to the Cd melting point, and the generated nitrogen gas can be removed from the system without getter. The solubility of nitrogen in cadmium was not discovered up to the temperature of 673 K [7].

Zinc carbide  $ZnC_2$  may be formed in systems Zn-C and Cd-C, but this compound can be obtained by chemical synthesis only. The solubility of carbon is low in cadmium and zinc at the temperatures of evaporation. Gas release from cadmium and zinc occurs in the form of CO and  $CO_2$  during heating in vacuum. Separation of cadmium and zinc from the carbon during distillation using the getter filter can be carried out by means of the reduction of the CO volatile compound to form zirconium and iron oxides. Partial pressure  $P(CO)$  and  $P(CO_2)$  are assumed to be small compared to the total pressure in the system, which is determined by the metal vapors.

Thus, let's consider from the thermodynamics aspect the Cd and Zn refining of interstitial impurities during vacuum distillation using Zr-Fe getter filter for the examples of reducing CdO, ZnO,  $Zn_3N_2$ , CO,  $CO_2$ .

Fig. 1 shows the dependences of the Gibbs energy change per 1 mole of oxygen for the formation of ZnO and other oxides vs temperature under different pressure of the gaseous environment in the system. Similar dependences are shown for the formation of CdO (Fig. 2) and  $Zn_3N_2$  (Fig. 3). It is seen from figures 1 and 2 that the stability of CdO and ZnO decreases with increasing system temperature and with decreasing pressure. To decompose ZnO under atmospheric pressure the required temperatures is in excess of 2300 K, whereas at the pressure of 1.33 Pa this oxide is instable at  $T > 1620$  K, and at pressure  $P = 1.33 \cdot 10^{-2}$  Pa that does at  $T > 1470$  K. A similar pattern is observed for CdO and differs only by the lower temperature of decomposition. It can be seen from Fig. 3 that at the zinc distillation operating temperatures (733...793 K)  $Zn_3N_2$  compound is unstable under a pressure of 133 Pa in the system.

If temperature and pressure in the system during the metal distillation is insufficient for the decay of oxide then the reduction of metals from oxides MeO using reducing agent can be used for refining. Reduction reaction scheme is as follows:



where  $Me''$  is the reducing metal.

Hard alloy Zr-Fe was selected as a metal reducing agent. Zirconium has a much greater chemical affinity for oxygen than iron. In the condensed state for the conditions under consideration it was established the existence of only one stable oxide –  $ZrO_2$ , the formation reaction of which and Gibbs energy change are as follows [6, 8]:



$$\Delta G_{\text{ZrO}_2} = -1092754 + 183.8 \cdot T - 19.15 \cdot T \lg \left( \frac{P(\text{O}_2)}{1.013 \cdot 10^5} \right). \quad (5)$$

In the formula (5)  $P(\text{O}_2)$  is the partial pressure of oxygen in the system.

Figs. 1 and 2 shows the Gibbs energy changes for the reaction of zirconium dioxide formation. The graphs show that the reduction of CdO and ZnO by zirconium may easily been performed from thermodynamics aspect within the entire investigated range of temperatures and pressures. However, the feasibility of the reaction does not guarantee its active behavior. Since in our case the distillation of cadmium and zinc is carried out at low temperatures, it may well be that the local temperature of the reactants is not enough for a quick reaction. In the steel industry to enhance metallothermic reducing the reacting system is often introduced with easily reducible oxides, that provide additional heat contributing to an increase in temperature of the reactants. For example, the easily reducible oxide  $\text{Fe}_3\text{O}_4$  is added in the charge during production of titanium by aluminothermic reduction of  $\text{TiO}_2$ , and for 100 g  $\text{TiO}_2$  it is required 83g  $\text{Fe}_3\text{O}_4$  [8]. In this regard, one can expect an increased separation efficiency of Cd and Zn from oxygen and nitrogen by carrying out the reducing reaction with zirconium in the presence of iron oxide.

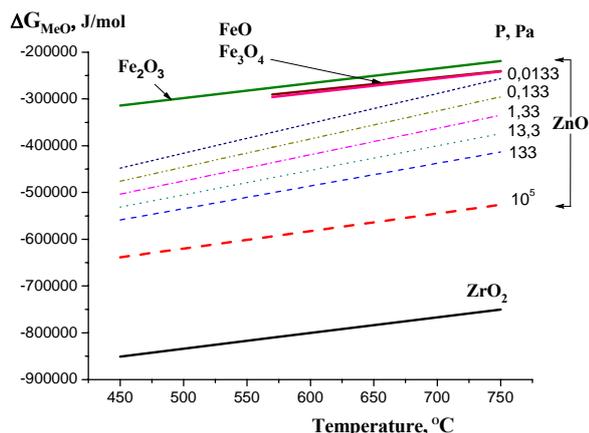
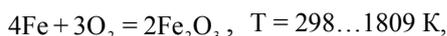


Fig. 1. Gibbs energy change for  $\text{ZrO}_2$ ,  $\text{Fe}_2\text{O}_3$  formation under oxygen partial pressure 0.00133 Pa, as well as for ZnO in depending on temperature under different pressure of metal vapor in the system:  $10^5$ ; 133; 13.3; 1.33; 0.133; 0.0133 Pa

Iron is easily oxidized in an oxidizing atmosphere to form  $\text{FeO}$ ,  $\text{Fe}_3\text{O}_4$  and  $\text{Fe}_2\text{O}_3$ . The following formation reaction of  $\text{Fe}_2\text{O}_3$  occurs [6, 8] in the conditions under consideration:



$$\Delta G_{\text{Fe}_2\text{O}_3} = -542916 + 165.9 \cdot T - 19.15 \cdot T \lg \left( \frac{133 \cdot P(\text{O}_2)}{1.013 \cdot 10^5} \right). \quad (6)$$

Our preliminary experiments showed a higher degree of Cd and Zn refining from impurities by distillation with getter of Zr-Fe, as compared with distillation by getter of pure Zr. Separation of metals from oxygen can progress as follows. Oxygen passes from Cd or Zn to Zr and Fe according to three reactions (4)–(6), at that in (4)  $\text{Me}^{\text{II}}$  and  $\text{Me}^{\text{II}}\text{O}$  correspond to Zr (Fe) and  $\text{ZrO}_2$  ( $\text{Fe}_2\text{O}_3$ ), and MeO corresponds to CdO or ZnO. In addition,

the transition of the source oxygen from iron oxide to zirconium oxide may be carried out by means of reduction of iron oxide with zirconium according to reaction scheme (4), that accompanied with heat release and increase of getter temperature, which should activate the reducing reaction of the metal from CdO and ZnO, as well as reaction (5) and (6). A similar outline can be proposed for the Zn purification of nitrogen.

The real means of Cd and Zn purification of carbon using a getter may be the reduction of C from volatile compound CO emitted from the Cd and Zn matrix by the use of filter Zr-Fe to form zirconium oxide with releasing of solid carbon on the getter surface. Condition of carbon reduction is the higher getter metal affinity to oxygen than carbon that to oxygen. Chemical affinity of zirconium and iron to oxygen is estimated in accordance with formulas (5) and (6), and affinity of carbon to oxygen – according to the change of Gibbs energy  $\Delta G_{\text{C/CO}}$  for reaction:  $2\text{C} + \text{O}_2 = 2\text{CO}$ . The formula for calculating the  $\Delta G_{\text{C/CO}}$  is:

$$\Delta G_{\text{C/CO}} = -221120 + 17975T + 383 \cdot T \lg \left( \frac{P_{\text{CO}}}{1.013 \cdot 10^5 \cdot P(\text{O}_2)} \right), \quad (7)$$

where  $P_{\text{CO}}$  is the partial pressure of CO, Pa. Formula (7) is valid for the case where the activity of carbon is equal to 1. Oxygen partial pressure in the system is assumed to be equal 0.00133 Pa. We also assume that under condition of vacuum distillation the volatile compound CO does not react with vapors of zinc, cadmium, and molecules of gaseous medium. With origin of carbon reduction at  $T > 773 \text{ K}$  it is possible to involve the volatile compound  $\text{CO}_2$  into purification process according to the reaction:  $\text{C} + \text{CO}_2 = 2\text{CO}$ .

Temperature dependence of the Gibbs energy change for the reactions of iron oxides formation are shown in Figs. 1, 2, and those for the reaction of iron nitrides formation – in Fig. 3. It follows from Fig. 2, the reduction of CdO in accordance with the above scheme, using a getter filter Zr-Fe, is possible within the whole considered intervals of temperatures and pressures.

As can be seen from Figs. 1 and 3, participation of iron in the process of ZnO reduction is possible under ultrahigh vacuum only, and that in the process of  $\text{Zn}_3\text{N}_2$  reduction – under a system pressure of not less than 0.0133 Pa at the temperature interval 753...793 K.

Fig. 3 also shows that the compound  $\text{Zn}_3\text{N}_2$  may be decomposed under high vacuum at elevated temperatures of distillation.

The compounds  $\text{Fe}_4\text{N}$  and  $\text{Fe}_8\text{N}$  are also unstable at the operating temperatures of zinc distillation 753...793 K, but the separation of zinc from nitrogen by means of vacuum distillation using a getter may be carried out according to the same outline as the removal of oxygen.

$\text{Zn}_3\text{N}_2$  decomposition and removal of releasing gaseous nitrogen from the system should prevail under conditions of elevated temperature and high vacuum.

Fig. 4 shows the temperature dependences of the Gibbs free energy change to form CO in accordance with the reaction  $2\text{C} + \text{O}_2 = 2\text{CO}$  under different CO partial pressure in the system, and to form  $\text{Fe}_2\text{O}_3$  and  $\text{ZrO}_2$  also.

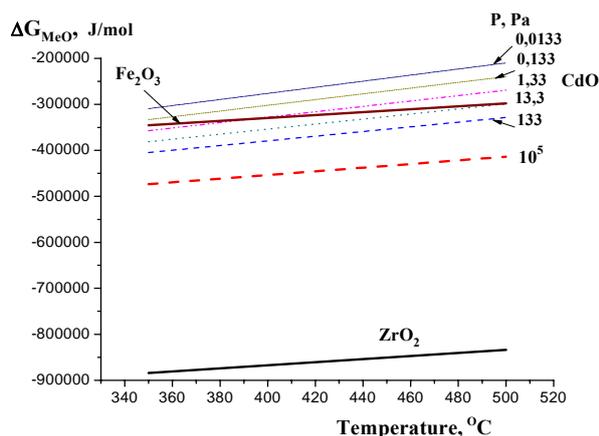


Fig. 2. Gibbs energy change for the formation of CdO depended on the temperature under different pressures of metallic vapors in the system, as well as for formation of ZrO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub> under partial oxygen pressure 0.00133 Pa

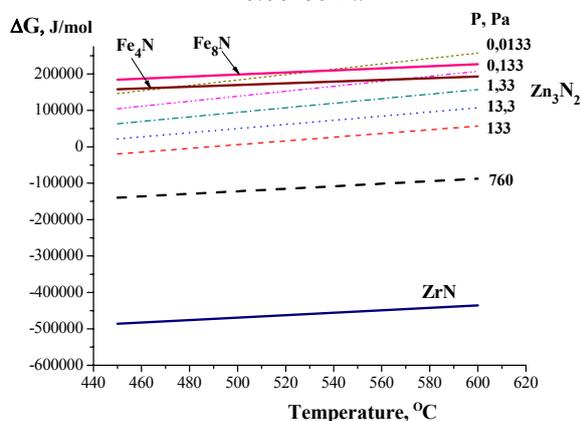


Fig. 3. Gibbs free energy change for the Zn<sub>3</sub>N<sub>2</sub> formation under different metal vapor pressure in the system 1.013·10<sup>5</sup>; 133; 13.3; 1.33; 0.133; 0.0133 Pa and for the formation of nitrides of zirconium and iron under the partial pressure of nitrogen 0,00133 Pa

The dependence of the standard Gibbs energy change for the formation of CO<sub>2</sub> is shown also for comparison. The figure shows that only zirconium can directly participate in the process of separation of distilling metal from oxygen by means of carbon reduction from volatile compound CO.

Performed thermodynamic analysis of redox reactions of the oxides of cadmium, zinc and carbon

Content of C, O, N impurities in cadmium and zinc after distillation using getter filter of Zr (51)-Fe (49) wt. %

Metal	Impurities	Impurity content, ×10 <sup>-4</sup> wt. %		
		In source metal	In refining metal	
			distillate I (no getter)	distillate II (with getter filter)
Cd	C	20	6	< 1
	N	5	1	< 1
	O	45	10	< 1
	*Σ <sub>ВНД</sub>	70	17	< 3
Zn	C	80	20	< 1
	N	20	6	< 1
	O	25	14	< 1
	*Σ <sub>ВНД</sub>	125	40	< 3

with the getter alloy Zr-Fe yielded initial data to select optimal temperature and vacuum conditions for metals deep refining of interstitial impurities.

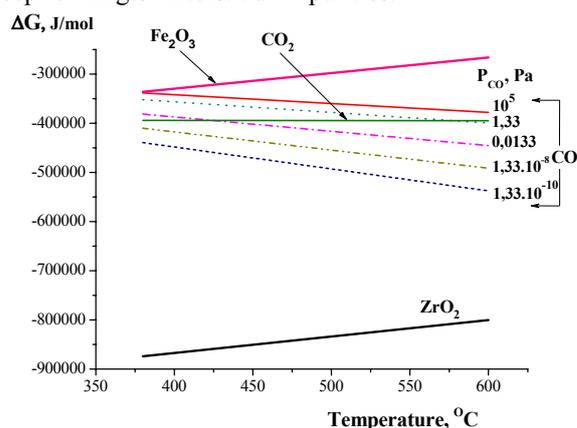


Fig. 4. Gibbs free energy change to form CO at different partial pressure of CO as well as CO<sub>2</sub> under atmospheric pressure of CO<sub>2</sub>, and for the formation of oxides ZrO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub> under a partial oxygen pressure 0,00133 Pa

Refining of cadmium and zinc using the getter filter of Zr(51)-Fe(49) wt.% alloy was performed using previously developed device and method described in [9-12]. The process of Cd and Zn refinement using the Zr-Fe filter was carried out in quasi-closed system placed in a chamber under the pressure (2.7...5.3)·10<sup>-3</sup> Pa, and at the evaporation temperatures of cadmium 653...693 K and zinc 753...793 K. The source materials for the refining were Kd0A technical purity grade cadmium (State Standard 1467-93) and brand TSV00 zinc ingots (State Standard 3640-94).

Efficiency of separation from interstitial impurities using a getter filter provided in the table. The table shows that proposed method with use of getter filter provides a more effective cadmium and zinc purification of gaseous impurities and carbon (distillates II) as compared with purification without filter. The degree of refinement using getter is more than tenfold whereas refining efficiency without getter is 3...5 fold, as compared with impurity content of the source metal.

The analysis showed at that, there was an additional (2...5 times) separation from the main metallic impurities compared with the content of these impurities in the distillates produced without a filter.

\* $\Sigma_{\text{внд}}$  is the total content of interstitial impurities.

The table also shows that the use of the getter provides a slightly more effective removal of impurities from Zn compared with Cd. It is due to the higher stability of ZnO and Zn<sub>3</sub>N<sub>2</sub> as compared with CdO and Cd<sub>3</sub>N<sub>2</sub> respectively. In other words, the more stable the compound formed with interstitial impurity, the stronger the influence getter filter for removal of the impurities.

This confirms the validity of initial assumptions about the effect of the stability of considered compounds on the concentration of residual interstitial impurities. Comparison of Figs. 1-4 with the data in the table suggests that iron plays only a role of catalyst in the zinc reduction reactions. In cadmium Fe may also directly participate in the process of CdO reduction with a subsequent transition of oxygen impurity from Fe<sub>2</sub>O<sub>3</sub> to ZrO<sub>2</sub>. Fe may participate also in separation of gas medium from molecular oxygen during the Cd distillation.

Quantitative analysis of impurities in initial and refined samples of cadmium and zinc were determined by spark mass spectrometry – SMS (Giredmet, Moscow, Russia). The error of analysis results by this method is characterized by the relative standard deviation of 0.15...0.30.

## CONCLUSIONS

The optimal conditions to carry out the processes of cadmium and zinc deep refining of the interstitial impurities are determined basing on the analysis.

Iron is only a catalyst for redox reactions involving Zr during distillation of Zn and Cd. Activating metallothermic reduction is due to the heat released in the reducing reaction: Fe<sub>2</sub>O<sub>3</sub> + Zr → ZrO<sub>2</sub> + Fe and subsequent rise of getter temperature.

A refining method using a getter filter of Zr-Fe alloy enables more efficient removal of interstitial impurities compared with distillation without filter. Their content has decreased more than tenfold as compared with the concentration in the source metals.

The more stable compound with C, O, N, the greater the effect of getter filter on removal of these impurities. An important factor affecting the concentration of residual interstitial impurities and ultimate purity of Cd and Zn is the stability of ZnO, CdO, Zn<sub>3</sub>N<sub>2</sub>, CO, CO<sub>2</sub>.

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## ВЛИЯНИЕ ГЕТТЕРНОГО ФИЛЬТРА Zr-Fe НА ГЛУБОКУЮ ОЧИСТКУ Cd И Zn ОТ ПРИМЕСЕЙ ВНЕДРЕНИЯ

*А.И. Кондрик, Г.П. Ковтун, А.П. Щербань, Д.А. Солопихин*

Представлены аналитические и экспериментальные исследования процесса рафинирования кадмия и цинка в вакууме с применением геттерных фильтров из сплава Zr-Fe от примесей внедрения (C, N, O). Выполнен термодинамический анализ восстановительных реакций оксидов кадмия, цинка, углерода и нитрида цинка с материалом геттера из сплава Zr-Fe в зависимости от температуры и вакуумных условий проведения процесса рафинирования этих металлов. Экспериментально показана эффективность применения геттерного фильтра из сплава Zr(51)-Fe(49) мас.% для очистки Cd и Zn от примесей внедрения.

## ВПЛИВ ГЕТТЕРНОГО ФІЛЬТРА Zr-Fe НА ГЛИБОКЕ ОЧИЩЕННЯ Cd ТА Zn ВІД ДОМІШОК ПРОНИКНЕННЯ

*О.І. Кондрік, Г.П. Ковтун, О.П. Щербань, Д.О. Солопихін*

Представлені аналітичні та експериментальні дослідження процесу рафінування кадмію та цинку у вакуумі із застосуванням геттерних фільтрів зі сплаву Zr-Fe від домішок проникнення (C, N, O). Виконано термодинамічний аналіз відновних реакцій оксидів кадмію, цинку, вуглецю і нітриду цинку з матеріалом геттера зі сплаву Zr-Fe в залежності від температури та вакуумних умов проведення процесу рафінування цих металів. Експериментально показана ефективність застосування геттерного фільтра зі сплаву Zr (51)-Fe (49) мас.% для очищення Cd і Zn від домішок проникнення.