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IMPURITY DISTRIBUTIONS IN A MAGNESIUM SUBLIMATES

I.I. Papirov, A.I. Kravchenko, A.I. Mazin, A.V. Shiyan, V.D. Virich
National Science Center "Kharkov Institute of Physics and Technology", Kharkov, Ukraine
E-mail: papirov@kipt.kharkov.ua; alex@krawa.net

The dependence of character of impurities distributions in thickness of vacuum sublimation magnesium condensate from its purity is ascertained. The condensate with summary impurities concentration ~ 10^{-3} mas.% has more uniform impurities distribution and bigger part of impurities with monotonic distribution than the condensate with summary impurities concentration ~ 10^{-2} mas.%. The impurity concentrations on the surface of rest exceed the impurity concentration inside the rest on moving off 2·μm from surface in 2–600 times (at initial summary impurity concentration ~ 10^{-1} mas.%).

The vacuum sublimation is effective method of fine cleaning of magnesium as perspective material of medical stents [1]. Earlier the impurities distribution in thickness of vacuum sublimation magnesium condensate with summary impurity concentration ~ 10^{-2} mas.% was studied and complicated character of impurities distribution in condensate was fixed (with the exception of Mn and Ca impurities with distributions on distillation equation [2, 3]). Meanwhile, the multiple sublimation allows obtaining more pure condensate [1].

The features of forming of impurity distributions in sublimation condensates with difference purity are not studied. The impurity distributions in sublimation rests is not studied too – though it is known that in processes which are concerned with diffusion in liquid (crystallization, electrolysis) the diffusion layers with thickness ~ 0.01...0.1 mm are formed [4, 5] and it is waited the forming of diffusion layers in solid states at sublimation too.

The objectives of this work were the investigation of impurity distributions in magnesium sublimates (as in condensates with difference purity and in rest).

The sublimation of magnesium and the control of impurities in it are like in previous studies [1, 2]. The same material with initial summary impurity concentration ~ 10^{-1} mas.% is used (with level of impurities concentration Al, Si, Mn ~ 10^{-2} mas.%; S, Cl, Ca, Fe, Zn ~ 10^{-3} mas.%; P, K, Ti, Cr, Ni, Cu ~ 10^{-4} mas.%). Basic parameters of processes was following: a temperature of evaporation was 700…800 K, a temperature of condensation was on 50…70 K smaller than a temperature of evaporation, a final degree of distillation was 80%. In first process, the initial material had shape of ingot. The condensate of previous process used as initial material in each following process at multiple sublimation. The impurity distributions in condensates was studied in most tick of condensates (on section on axis line of cylinder sublimation arrangement at thickness of condensate near 4 cm). The rest of initial process (the ingot with thickness near 4 cm) was analyzed on surface and in side on section. The elemental composition of the materials was determined by laser ionization mass spectrometry using an EMAL-2 analyzer (sensitivity, ~ 10^{-6} mas.%, measurement accuracy, 30%).

The results of investigation are given in Tables 1 and 2. At estimation of diffusion layer δ in a rest it was considered that thickness of layer that is sputtering at one pass of EMAL-2 laser is < 0.5 μm [6]; 4 passes of laser was performing.

Follow summaries are made:

1. The character of impurity distributions in condensates (with summary concentration of impurities ~ 10^{-2} and ~ 10^{-3} mas.%) in thickness of condensate dependences from a pure of condensate. In clearer condensate:

– the impurity distributions are more uniform (the bigger and the least values of summary concentration of impurities differ in 3 times; while in a less pure condensate they differ in 4 times);

– the distributions of most impurities (except Fe and Cu) and the distributions of summary concentration are monotonous while nearly all impurities in less pure condensate show a non-monotonic distribution;

– there are not impurities with distributions according distillation equation (as Mn and Ca in less pure condensate [2]).

2. Near the surface of the rest the barrier layer wit thickness δ < 0.002 mm is revealed, and the impurity concentration on the surface exceeds the impurity concentration inside the rest in 2–600 times. At the same time, the impurity concentration inside the rest out of the barrier layer is close to the initial concentration.

3. Complicated character of impurity distributions in magnesium condensates and dependence of character of impurity distributions from purity of condensates against the background of high concentration of impurities in the barrier layer on the surface of the rest, apparently demonstrates the important role of chemical interaction in magnesium-impurities system during sublimation.

Table 1

Impurity concentration (C) in condensate after 3- and 1-multiple (in brackets) sublimation and the summary impurity concentration (Σ) at different distances *x* from the substrate

Im-purity	C (or Σ), 10^{-3} mas.%, at different *x*, mm				
	x=3	*x*=11	*x*=18	*x*=23	*x*=33
Al	0.01 (0.41)	0.02 (0.38)	0.01 (0.16)	0.01 (0.22)	0.01 (0.17)
Si	0.03 (0.40)	0.02 (0.55)	0.02 (0.40)	0.01 (1.60)	0.01 (1.00)
P	<0.003 (0.02)	<0.003 (0.02)	<0.003 (0.02)	<0.003 (0.02)	<0.003 (0.02)
S	0.3 (1.5)	0.5 (0.7)	0.6 (3.6)	1.1 (6.9)	1.3 (4.8)
Cl	0.11 (0.64)	0.15 (0.51)	0.20 (1.10)	0.17 (1.70)	0.21 (0.97)
K	0.07 (0.07)	0.06 (1.10)	0.05 (0.50)	0.03 (0.29)	0.04 (1.10)
Ca	0.06 (0.15)	0.06 (0.30)	0.05 (0.33)	0.04 (0.47)	0.05 (0.18)
Ti	<0.1 (<0.2)	<0.1 (<0.2)	<0.1 (<0.2)	<0.1 (<0.2)	<0.1 (<0.2)
Cr	0.08 (0.06)	0.04 (0.09)	0.04 (0.05)	0.04 (0.04)	0.04 (<0.03)
Mn	<0.01 (0.45)	<0.01 (0.45)	<0.01 (0.45)	<0.01 (0.45)	<0.01 (0.45)
Fe	0.21 (0.40)	0.16 (0.50)	0.05 (0.20)	0.09 (0.13)	0.12 (0.10)
Ni	0.05 (<0.04)	<0.01 (<0.04)	<0.01 (<0.04)	<0.01 (<0.04)	<0.01 (<0.04)
Cu	<0.003 (0.1)	0.19 (1.1)	0.42 (7.4)	0.18 (7.0)	0.92 (6.4)
Zn	0.3 (2.5)	0.5 (3.2)	0.6 (4.3)	0.6 (7.8)	0.8 (3.6)
Σ	1.2 (6.7)	1.7 (10.0)	2.1 (18.8)	2.2 (26.9)	3.5 (18.5)

Table 2

The ratio of impurity concentration on surface of rest of magnesium ingot (C') to concentration of this impurity inside of rest

(C_r – at distance from surface X ≥ 0.002 mm > δ)

Im-purity	C'/C_r	Im-purity	C'/C_r	Im-purity	C'/C_r
K	$6·10^2$	Fe	3·10	Zn	5
S	$3·10^2$	P	2·10	Si	4
Cl	7·10	Cu	1·10	Cr	2
Ca	5·10	Al	8	Mn	2

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РАСПРЕДЕЛЕНИЯ ПРИМЕСЕЙ В СУБЛИМАТАХ МАГНИЯ

И.И. Папиров, А.И. Кравченко, А.И. Мазин, А.В. Шиян, В.Д. Вирич

Установлена зависимость характера распределения примесей по толщине конденсата магния, полученного вакуумной сублимацией, от чистоты конденсата. Конденсат с суммарным содержанием примесей ~ 10^{-3} мас.% имеет более равномерное распределение примесей и большее число примесей с монотонным распределением, нежели менее чистый конденсат с суммарным содержанием примесей ~ 10^{-2} мас.%. Содержание примесей на поверхности остатка в 2–600 раз превышает исходное содержание примесей внутри остатка на удалении от поверхности более 2 мкм (при исходном суммарном содержании ~ 10^{-1} мас.%).

РОЗПОДІЛ ДОМІШОК У СУБЛІМАТАХ МАГНІЮ

І.І. Папіров, О.І. Кравченко, О.І. Мазін, О.В. Шиян, В.Д. Вірич

Встановлена залежність характеру розподілу домішок по товщині у конденсаті магнію, що одержано вакуумною сублімацією, від чистоти конденсату. Конденсат з сумарним вмістом домішок ~ 10^{-3} мас.% має більш рівномірний розподіл домішок та більше число домішок с монотонним розподілом, ніж менш чистий конденсат з сумарним вмістом домішок ~ 10^{-2} мас.%. Вміст домішок на поверхні залишку в 2–600 раз перевищує вміст домішок всередині залишку на відстані від поверхні > 2 мкм (при вихідному сумарному вмісту ~ 10^{-1} мас.%).