Self-organization of quasi-equilibrium stationary condensation in accumulative ion-plasma devices

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Abstract

We consider both theoretically and experimentally self-organization process of quasi-equilibrium steady-state condensation of sputtered substance in accumulative ion-plasma devices. The self-organization effect is shown to be caused by self-consistent variations of the condensate temperature and the supersaturation of depositing atoms. On the basis of the phase-plane method, we find two different types of the self-organization process to be possible. Experimental data related to aluminum condensates are discussed to confirm self-organization nature of quasi-equilibrium steady-state condensation process.

Key words: Self-organization; Stationary condensation; Supersaturation

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1 Introduction

Formation of various nanosystems in the course of the condensation process is known to be achieved due to widespread technologies such as the molecular-beam epitaxy, the metal-organic vapour-phase epitaxy, the liquid-phase epitaxy, the electrolytic deposition etc. [1,2]. Characteristic peculiarities of these technologies are both quasi-equilibrium and steady-state to be as follows:

- the condensation process occurs at sufficiently low supersaturation to provide a proximity to phase equilibrium between condensate and depositing substance;
- this supersaturation remains stable in the course of the time to guarantee steady-state conditions of the condensation process.
Due to above conditions adsorbed atoms can be coupled with growing condensate surface only if the strongest chemical bonds are realized to minimize the free energy [3,4].

The proximity to the phase equilibrium is known to be governed by decrease of the supersaturation

$$\xi = \frac{n - n_e}{n_e}$$

(1)

where \(n\) and \(n_e\) are current and equilibrium concentrations of atoms above the growth surface. In the case of vapour-condensate systems, the latter of this concentrations is described with the empirical relation

$$n_e = \frac{A(T)}{k_B T} \exp \left( - \frac{E_d}{k_B T} \right)$$

(2)

where \(E_d\) is the desorption energy, \(T\) is the growth surface temperature, \(k_B\) is the Boltzmann constant; the temperature dependent parameter \(A(T) = \exp(\alpha + \beta T + \gamma / T)\) is determined with a set of constants \(\alpha, \beta\) and \(\gamma\) being characteristics for a given substance [5].

In the case of volatile substances, the desorption energy is so small \((E_d \ll k_B T)\) to expand large value of the equilibrium concentration (2) into a series. As a result, the supersaturation becomes less sensitive to temperature change that simplifies implementation of quasi-equilibrium steady-state condensation at relatively high deposition fluxes.

For the vapour-condensate systems related to the molecular-beam epitaxy, low supersaturations are obtained only due to increased growth surface temperature at relatively weak deposition fluxes. That is why the molecular-beam epitaxy can be implemented mainly for those substances which have relatively high volatility.

In chemically active medium-condensate systems, being the basis of metal-organic vapour-phase epitaxy, liquid-phase epitaxy and electrolytic deposition, the proximity to the phase equilibrium is stimulated additionally by reversible chemical processes. The latters induce reverse transitions of adsorbed atoms which are weakly coupled with growth surface into environment. In Eqs. (1) and (2) this is expressed in decreasing value \(E_d\) of the desorption energy down to an effective value that increases the equilibrium concentration \(n_e\) stimulating proximity to equilibrium. From this point of view, above chemical methods make it possible to reach low supersaturations at condensation of weakly volatile substances that gains its advantage over the molecular-beam epitaxy.

The present work is devoted to consideration of the system low temperature plasma-condensate which enables to reach a proximity to phase equilibrium by means of heating growth surface with the help of plasma stream, both momentum and energy transfer from plasma particles to adatoms, and their partial
Due to the latter two mechanisms the desorption energy $E_d$ gets an effective value [3,6,7]

$$E = E_d - \delta E$$

with a stochastic addition $\delta E$ characterized by the mean value $\bar{E}$ and the dispersion $\sigma^2 \equiv \left( \delta E - \bar{E} \right)^2$ (the overline means averaging over plasma particle collisions). Thus, in analogy with the chemical systems the low-temperature plasma-condensate systems make it possible to stimulate proximity to equilibrium at condensation of weakly volatile substances. However, making use of the plasma arrives at the problem to fix steady-state conditions during the condensation process.

For all mentioned technologies, there arises a common range of problems to fix steady-state regime of the condensation process at vanishing supersaturations. The main point is that such crucial technological parameters as the growth surface temperature $T$ (hence, the equilibrium concentration $n_e = n_e(T)$) and the depositing flux $J$ are mutually independent and are regulated by different power sources. Also, under quasi-equilibrium conditions the condensing flux $J_c$ is obeyed the inequality $J_c \ll J$. Hence, even slight relative variations $\Delta J/J$ of the depositing flux can arrive at considerable changes in condensation kinetics at condition $\Delta J \sim J_c$. (The same effect can cause variation of the equilibrium concentration $n_e(T)$ at slight fluctuations of the growth surface temperature $T$.) In the absence of self-organization, these problems are solved by artificially created feedback between condensation kinetics and depositing flux by means of control systems that arrives at considerable rise in the cost of technologies.

Therefore, the aim of this Letter is two-fold: i) elaborate a technological device, whose operation within self-organization regime ensures extremely low steady-state supersaturations; and ii) build up a theoretical model to describe above self-organized process. In correspondence with these aims, the outline of the paper is as follows. In Section 2, we state physical backgrounds of operation of the accumulative ion-plasma devices. On the basis of these conceptions, we consider theoretically the self-organization of extremely low stationary supersaturations in Section 3. Experimental validation of the self-organization scheme is given in Section 4, and Section 5 concludes our consideration.

### 2 Physical backgrounds of operation of the accumulative ion-plasma device

Principle components of our sputtering device are the magnetron sputterer in combination with the hollow cathode which operate under high pressure of highly refined argon ($P_{Ar} = 10 \div 30 Pa$) (see Fig.1). Such a construction allows...
to stabilize and increase the discharge current due to both hollow cathode and the magnetron effects [8,9], the last of which localizes the erosion zone in addition. It is worthwhile to stress that almost all atoms of inert gas and sputtered substance become ionized when get inside the hollow cathode [8,9]. Under increased pressure, plasma particles collide so frequently and intensively to average their energy effectively. These collisions decrease the dispersion $\sigma$ of the effective desorption energy (3) that, in turn, stabilizes condensation process.

The self-organization process starts with accumulation of a sputtered substance near the growth surface. According to Ref. [10], atoms sputtered by magnetron lose their energy (in other word, thermalize) due to collisions of particles of high-pressure plasma, so that their further movement becomes diffusive. Related thermalization length is estimated as $l \propto (T_{Ar} + \Delta T)/T_{Ar} P_{Ar}$ with $T_{Ar}$ and $\Delta T$ being the temperature of unheated working gas and its growth under discharge. Hence, the thermalization length is reduced with increasing the pressure $P_{Ar}$ and decreasing discharge power that reduces the temperature growth $\Delta T$. As show estimations [9,10], the flux of non-thermalized atoms in direction of the anode can be considered as negligible at the pres-
sure $P_{Ar} = 20 \div 40$ Pa and the discharge power less than 100 W, when the thermalization length is subject to the condition $l \leq L/4$ with respect to the target-anode distance $L$ depicted in Fig.1.

As it is seen from this figure, the flux $J_s$ of sputtered substance condenses mainly on the surface adjacent to the erosion zone with the flux $J_q$ and diffuses partially into the hollow cathode with the flux $J_d$. (It should be noted that at the initial time of the device operation, there is only inert gas plasma inside the hollow cathode.) With power up, the concentration of sputtered atoms $n_0$ at the hollow cathode entry is determined by the fluxes $J_e$ and $J_P$, the first one being drift flux due to the presence of electrical field near the inlet, and the second one being caused by pressure difference between interior and entry of the hollow cathode. As a result, variation of the atom concentration $n_0$ at the hollow cathode inlet is governed by the equation

$$\dot{n}_0 \propto (J_s - J_d - J_q) - (J_e - J_P)$$

(4)

where and hereinafter the point above a symbol denotes the time derivative. As the hollow cathode volume is rather small, the drift components $J_e$ and $J_P$ become equal quickly and then sputtered substance penetrates into the hollow cathode due to diffusion only. Because both components $J_s$ and $J_q$ are much more than the diffusive flux $J_d$, one can suppose its variation affects slightly on the concentration $n_0$. At the steady-state condition $J_d = J_s - J_q$, diffusion process determines accumulation of thermalized atoms in the interior of the hollow cathode to increase their concentration over a thermodynamic threshold $n_e$ needed to start condensation of the sputtered substance on the substrate.

To understand the lowering of steady-state supersaturation in the course of the condensation process, let us assume the growing surface has reached such a temperature, when inequality $n \geq n_e$ starts to be satisfied due to accumulation of sputtered substance. Then, the supersaturation (1) takes a positive value to initiate the barrier nucleation process that increases the growth surface temperature due to the atom thermal accommodation. On the other hand, the flux of depositing atoms decreases due to rupture of circular fluxes that, in turn, reduces the supersaturation to minimal values providing condensation process. In the following section, we shall show that self-consistent variations of the surface temperature and the supersaturation is a cornerstone of self-organization process ensuring extremely low steady-state supersaturations.

1 It is worthwhile to note, under plasma influence, main part of deposited atoms are reevaporated to be next ionized again and returned onto this surface under electric field [8]. Such a circular mechanism of mass transfer accumulates additionally depositing atoms in immediate vicinity of the growth surface.
3 Theoretical consideration of self-organization of steady-state quasi-equilibrium condensation

Let us start with consideration of main reasons for variation of the surface temperature in the course of the ion-plasma device operation. First, each of plasma ions transfers to the surface the average energy \( k_B (T_2 - T_1) \) determined by the temperature difference of plasma ions, \( T_2 \), and adsorbed atoms, \( T_1 \), respectively (see the temperature distribution depicted in the left panel of Fig.1). Then, the energy delivered per unit time from the plasma to the unit surface can be written in the form

\[
\dot{E}_1 = \chi \theta (T_2 - T) .
\]  

(5)

Here, \( \chi \) is a parameter being the production of the Boltzmann constant and the plasma flux falling down the growth surface, \( \theta \equiv (T_2 - T_1)/(T_2 - T) \approx 1 - T_1/T_2 = \text{const} \) is thermal accommodation coefficient of adsorbed atoms rewritten with accounting the condition \( T \ll T_2 \). Second, the rate of energy transfer to the growth surface due to thermal accommodation of condensed atoms

\[
\dot{E}_2 = k_B (T_2 - T) J_c
\]  

is determined by the temperature difference of plasma and condensate accompanied with the condensing flux

\[
J_c = \frac{\delta}{\tau} (n - n_e).
\]  

(7)

Here, \( \delta \) and \( \tau \) are characteristic length and time of the circle motion of condensing atoms near the growth surface. Finally, the energy removal to the chiller per unit time by means of thermal conductivity is determined by the obvious expression

\[
\dot{E}_3 \approx \frac{\eta}{d} (T_0 - T)
\]  

(8)

where \( T_0 \) stands for the chiller temperature, \( d \) and \( \eta \) are the total thickness and effective thermal conductivity of two-layer system consisting of condensate and substrate.

Combination of the equations (5) – (8) with obvious definition of the variation rate of the surface temperature

\[
c \dot{T} = \dot{E}_1 + \dot{E}_2 + \dot{E}_3
\]  

(9)

arrives at the differential equation

\[
c \dot{T} = \left[ \left( \chi \theta T_2 + \frac{\eta}{d} T_0 \right) + \frac{k_B n_e \delta}{\tau} T_2 \xi \right] - \left[ \left( \chi \theta + \frac{\eta}{d} \right) + \frac{k_B n_e \delta}{\tau} \xi \right] T
\]  

(10)
with \( c \) being the surface heat capacity of the growth surface. The equation (10) shows the time dependence of the surface temperature is determined by the following set of values: plasma parameters \( \chi, \delta \text{ and } \tau \); condensate-substrate parameters \( \eta, c \text{ and } d \); temperatures characterising condensate \((T_1, T_2, \theta \approx 1 - T_1/T_2)\) and chiller \((T_0)\); equilibrium concentration \( n_e \) defined with Eq.(2); and finally, supersaturation \( \xi \) given by Eq.(1). Remarkably, both square brackets in Eq.(10) depend on the temperature \( T \) only through the equilibrium concentration (2), whereas the last term in right-hand side contains the factor \( T \) itself. Moreover, the last terms in both square brackets are proportional to the supersaturation \( \xi \), whose time dependence must be found next.

At finding the equation for the concentration \( n \), one should take into account that condensation occurs onto the inner surface of the hollow cathode with large area \( S \), whereas the diffusive flux penetrates toward the cathode interior throughout the inlet hole with small area \( s \). Then, the rate of the concentration \( n \) variation is determined by the difference between incoming diffusive and outgoing condensing fluxes so that, with accounting that accumulation of substance takes place inside the region with thickness \( \delta \) near the growth surface, one obtains

\[
\dot{n} = \frac{1}{\delta} \left( \frac{s}{S} J_d - J_c \right). \tag{11}
\]

According to Eq.(11) the steady-state regime is reached under the condition

\[
s J_d = S J_c \tag{12}
\]

which arrives at the relation \( J_c < J_d \) due to the non-equality \( s < S \). The condensing flux is determined by Eq.(7), as the diffusive component is given by the Onsager-type expression

\[
J_d \simeq -\frac{D}{\lambda} (n - n_0) \tag{13}
\]

with effective length \( \lambda \), along which the concentration varies from \( n_0 \) to \( n \), and the temperature dependent diffusion coefficient \( D \sim T_2^{3/2} \) [10]. Taking into account Eqs. (2), (7), (11) and (13), we yield the final form of the equation of motion for the supersaturation (1):

\[
\dot{\xi} + B(T) (1 + \xi) \dot{T} = \frac{s}{S} \frac{D}{\lambda \delta} \xi_0 - \left( \frac{1}{\tau} + \frac{s}{S} \frac{D}{\lambda \delta} \right) \xi \tag{14}
\]

where \( B(T) = \beta - \frac{1}{T} + \frac{(E_d/kB) - \gamma}{T_2} \) and a magnitude \( \xi_0 = (n_0 - n_e) / n_e \) relates to the inlet hole of the hollow cathode. Similarly to the differential equation (10), the first term in right-hand side of Eq. (14) depends on the temperature only through the equilibrium concentration (2), whereas the second term is proportional to the supersaturation with a constant factor inside the parentheses.
The system of the ordinary differential equations (10) and (14) describes self-consistent variations of the surface temperature $T$ and the supersaturation $\xi$ during the self-organization process of extremely low steady-state supersaturations. The form of possible solutions of these equations is seen from the phase portraits depicted in Fig.2 where the set of parameters is selected applicable to the aluminum. Analysis of the phase portraits arrives at the conclusion that regime of the self-organization is governed specifically by the ratio $s/S$. In the case of high values $s/S$ (Fig.2a), with the temperature growth the representative phase path 1 goes through high supersaturations, whereas in the opposite case (Fig.2b) negative values $\xi$ precede to the steady state related to the node. From the physical point of view, such a behaviour is explained by that accumulation of the sputtered substance is in advance of heating of the growth surface, in the first case, whereas at small ratios $s/S$ slight accumulation rate enables positive supersaturations $\xi$ only near the steady state. From the technological point of view, the second regime is much more preferable in case of little deposition time.

Under the steady-state conditions $\dot{T} = \dot{\xi} = 0$, the system of Eqs. (10) and (14) shows that the stationary supersaturation increases monotonically with growing effective desorption energy $E$, so that extremely low supersaturations $\xi \ll 1$ are achieved only when $E < 0.1 \text{ eV}$. Usually, the bare desorption energy for metals is estimated as $E_d \sim 0.4 \text{ eV}$ [5], and the proximity to the phase equilibrium is provided by the plasma influence that reduces the bare value $E_d$ to the effective energy $E$ in accordance with Eq.(3).

## 4 Experimental validation of self-organization

We have considered above the self-organization process of quasi-equilibrium steady-state condensation of sputtered substance in accumulative ion-plasma devices. At the same time, both numerical simulations [11,12,13,14] and experimental investigations [3,14,15,16,17,18] display a secondary self-organization process. In the course of such a process, three-dimensional micro- and nanostructures are arranged on the substrate to possess complicated architecture with narrow distributions over sizes and forms of structural elements. In contrast to the self-organization of the system plasma-condensate, the secondary self-organization involves in the regime of the self-organized criticality driven by fluctuations [19]. Really, the result of the primary process is that two phase system plasma-condensate is staying near stationary equilibrium, but strongly above a critical point, so that a phase transition is thermodynamically impossible. However, appearance of active centers, playing the role of space distributed fluctuations, initiate the condensation process according to the following sce-
Fig. 2. (Colour online) Phase portraits presenting solutions of Eqs. (10) and (14) at $s/S = 1/4$ (a) and $s/S = 1/10$ (b) (the rest of parameters is as follows: $D = 10^5 cm^2s^{-1}$, $E = 2.674 \cdot 10^{-20} J$, $d = 0.1 cm$, $\lambda = 1 cm$, $\delta = 0.1 cm$, $\tau = 10^{-6} s$, $n_0 = 10^8 cm^{-3}$, $\theta = 0.95$, $\chi = 7.4 \cdot 10^{-3} J^{-1}cm^{-2}s^{-1}$, $T_2 = 6000 K$, $T_0 = 300 K$, $c = 3 \cdot 10^{-3} J^{-1}$, $\eta = 6 \cdot 10^{-3} W cm^{-1}K^{-1}$ (for glass). The node corresponds to $T = 898 K$, $\xi = 1.1 \cdot 10^{-4}$, $J_c = 1.1 \cdot 10^8 cm^{-2}s^{-1}$ (a) and $T = 898 K$, $\xi = 7.4 \cdot 10^{-5}$, $J_c = 7.4 \cdot 10^7 cm^{-2}s^{-1}$ (b).

The condensation mechanism is beyond the scope of the equilibrium thermodynamics to be defined by both kinetics \[4\] and synergetics \[19\].
In the course of the deposition process, adsorbed substance is embedded atom-by-atom into the growing surface on active centers with the highest energies of chemical bonds. Among such centers, one can point out the mono-step bends on the growing crystal surface, inhomogeneities on the atomic-rough surface, regions of crystallite joining and so on [5]. Due to the reduced surface density of the active centers, the chemical bonds spectrum should be appreciably discrete. That allows for one to separate those centers whose energies of chemical bonds $E_i$, $i = 1, 2, \ldots$ is lower than a critical threshold $E_c$ fixed by external conditions. On these centers, the atom-by-atom formation of the condensate becomes hardly probable because of subcritical energies of chemical bonds $E_i < E_c$. By definition, the threshold $E_c$ relates to the desorption energy $E_d$ in the equilibrium concentration (2) at zero supersaturation (1) and given concentration $n$ and temperature $T$. According to Eqs. (1) and (2), the critical energy of chemical bonds

$$E_c = k_B T \ln \left[ \frac{A(T)}{nk_BT} \right]$$

increases with tending to the phase equilibrium due to concentration decrease and temperature growth.

In our experiment, we investigate the structure of the aluminium condensates, produced in proximity to equilibrium in the plasma-condensate system. The scanning electron microscopy images of the ex-situ grown condensates are shown in Fig.3. These condensates were obtained on glass substrates by means of the accumulative ion-plasma system described in Section 2 at the discharge power 1.8W during the deposition time $t = 9h$ and at both pressures 20 $Pa$ and 15 $Pa$ of the highly refined argon. It is the argon pressure as technological parameter, that controls supersaturation, or degree of proximity to equilibrium, at stable discharge power. Thus, if argon pressure decreases from 20 to 15 $Pa$, plasma particle concentration decreases as logical result, and, therefore, their average energy increases. This reduces the effective desorption energy and accordingly to Eqs. (1) and (2) reduces supersaturation as well.

According to Fig.3a, at increased pressure 20 $Pa$, there are condensed weakly bound crystals whose habitus is fixed by crystallography planes akin to (210) because of the the conditions $E_{(531)} < E_c < E_{(210)}$ inherent in energies $E_{(hkl)}$ of atoms in half-crystal position on the $(hkl)$-planes of fcc-metals [20]. It is worthwhile to stress the structure depicted in Fig.3a can be statistically homogeneous up to tens of micrometers in thickness. Such a picture of the condensate formation is caused with the sequence of repeated nucleation on the active centers of the growth surface being usually regions of crystallite joining.

With reduction of the working gas pressure down to 15 $Pa$, at remaining sta-
Fig. 3. (Colour online) Scanning electron microscopy images of *ex-situ* grown aluminium condensates at layer-by-layer (a) and normal (b) crystal growth (the arrows on the lower panel point out the single crystals retaining faceting of the previous structure). There are used the glass substrates at the argon pressures $20\text{ Pa}$ (a) and $15\text{ Pa}$ (b), the discharge power $1.8\text{ W}$, the deposition time $9\text{ h}$, the area ratio $S/s \sim 4$ (the condensate thickness is of order $12 \div 14\mu\text{m}$).
ble of the rest of technological parameters, the supersaturation decreases approaching the phase equilibrium of the system plasma-condensate. This means increasing the critical energy of chemical bonds $E_c$ which becomes higher the energy $E_{(210)}$ of atoms on the plane (210), but lower the energy $E_r$ of chemical bonds on atomically rough surface. As a result, the conditions $E_{(210)} < E_c < E_r$ are fulfilled to change the mechanism of atom attaching to growth surface. Above assumption explains the transition from tangential to normal growth of round shaped crystals with atomically rough surface shown in Fig.3. Proximity of normal and tangential mechanisms of the crystal growth is confirmed by the fact that in Fig.3b several crystals labeled with arrows have faceting appropriate to tangential growth along the (210) plane. It should be stressed as well that, due to quasi-equilibrium conditions of the condensation process, there is no secondary nucleation in the regions of crystallite joining what governs formation of the columnar structures [21].

Thus, the comparison of the electron microscopy images depicted in Figs. 3a and 3b allows for one to conclude that the microstructures related are obtained as result of different mechanisms of crystal growth. Obviously, above transition from layer-by-layer to normal crystal growth may be possible at two-fold conditions: first, the system plasma-condensate should be extremely near the phase equilibrium (to increase the critical energy $E_c$); second, condensation process should be strongly steady-state (to fix a location of the threshold $E_c$ in the chemical bond spectrum). As is shown in Section 3, both conditions pointed out are provided due to the self-organization of quasi-equilibrium steady-state condensation within device presented in Section 2.

5 Conclusion

In contrast to widespread technologies, we propose here original method to realize quasi-equilibrium steady-state conditions of condensation. Character peculiarity of this method is that condensate surface grows atom-by-atom on active centers having the strongest chemical bonds. We show such regime is ensured by the accumulative ion-plasma device whose advantage in comparison with already existing systems consists in that it does not require cumbersome control system. The self-organized regime of the device operation is confirmed both theoretically and experimentally.

Above regime is shown to be achieved by means of two principle components of the device to be the magnetron sputterer and the hollow cathode. Due to diffusion, accumulation of condensed atoms occurs in the interior of the hollow cathode up to quasi-equilibrium concentration whose value remains constant during condensation process. Heating growth surface with the help of both plasma stream and decreasing the effective desorption energy causes
extremely low supersaturation near condensate overall.

The physical reason to reach the quasi-equilibrium steady-state conditions is that condensation process evolves under scenario of the self-organization. On the basis of the phase-plane method, it is shown the self-organization is governed by self-consistent variations of the surface temperature and the supersaturation.

To confirm that quasi-equilibrium steady-state condensation process is evolved under scenario of the self-organization, we investigate the structure of the aluminium condensates obtained at different technological conditions. Related scanning electron microscopy images show the microstructures obtained as result of the transforming layer-by-layer crystal growth into normal one. That, in turn, may be possible if the system plasma-condensate is near the phase equilibrium and condensation process is strongly steady-state. Just that very conditions are provided due to the self-organization of low steady-state supersaturations within device proposed.

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