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Densification of growing coatings by ion beams

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Abstract. A theoretical model to describe the evolution of microstructure of thin films and densification by ion assistance is proposed. In the model kinetics of point defects, including their interaction with voids and with the growing film surface, is considered. To solve the set of equations a computer code was developed. Film densification by ion bombardment is investigated as a function of ion energy and the ratio of ion-to-vapour current densities. It is shown that there are optimal ion energy and ratio of ion-to-vapour fluxes for obtaining dense coating with high growth rate.

1. Introduction

The growth of films and coatings by means of molecular deposition and condensation upon substrates is one of the widespread methods of thin film and coating production [1–4]. Films deposited from vapour at low temperatures have a low packing density, being lower than the density of a bulk material by 5–15%, and, as a result, have poor properties. Density decreasing occurs due to formation of vacancy clusters and microvoids because of low mobility of deposited atoms [5–7]. In order to obtain dense films, ion beams of low energy (<1 keV) are used during deposition of atoms and molecules [7–11]. The mechanisms that are responsible for film densification are known only in general. Many papers have been devoted to the theoretical consideration and computer simulation of kinetic processes in ion-assisted deposition of films [12–17], but details of the densification process are still unclear.

When a material is bombarded by low energy ions, point defects are formed. Generation of Frenkel pairs does not lead to essential change of the film density, but vacancies reduce the density. Thus, only implanted ions are capable to increase the film density. However, the total number of implanted ions is not sufficient to explain experimental results [11, 13, 14]. In [13], [14] and [18] the following explanation of this phenomenon was proposed. At low ion energy the majority of vacancies is formed near the surface and can be refilled by vapour atoms. Interstitials are formed deeper in the bulk and, despite a high mobility, not all of them reach the surface. A fraction of them diffuses deep into the film where they are absorbed by vacancy clusters and voids. Just these surplus interstitials are the cause of film densification.

In the present paper we develop our previous model (see [18–20]) by taking into account kinetics of voids.

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2. Film growth kinetics with bombardment by self-ions. Basic equations

Let us consider processes occurring in a growing film during deposition of metal atoms assisted by the simultaneous irradiation with ions of the same atoms. We assume that the film occupies an infinite half-space $x > x_0(t)$, where $x_0(t)$ is the coordinate of the plane surface. Fluxes of neutral atoms $J_n(t)$ and ions $J_{ion}(t)$ fall onto the film surface. The ions generate vacancies and interstitials in the film and stop within it. The stopped ions will be also considered also as interstitials. The point defect generation per atom is described by functions

$$Q_v(x,t) = J_{ion}(t)\Omega p_v(x,t) \quad (1)$$

$$Q_i(x,t) = J_{ion}(t)\Omega p_i(x,t) + J_{ion}(t)\Omega p_{imp}(x,t) \quad (2)$$

where $p_{v,i}(x,t)$ is the profile of generation of vacancies and interstitials per ion with the energy E, $p_{imp}(x,t)$ is the profile of implanted atoms, Ω is the atom's volume. Generated point defects react with each other, with voids and with the surface. These reactions are of two kinds: diffusion-controlled and athermal ones. First we shall consider the following reactions:

- vacancy–interstitial annihilation: $\mathrm{v} + \mathrm{i} \to 0$;
- vacancy absorption by a void $\mathrm{S} : \mathrm{v} + \mathrm{S} \to \mathrm{S}^+$;
- interstitial absorption by a void $\mathrm{S} : \mathrm{i} + \mathrm{S} \to \mathrm{S}^-$.

Vacancy–interstitial annihilation is described by the rate constant

$$\alpha_{iv} = \frac{4\pi R_{iv}}{\Omega}(D_i + D_v) \quad (3)$$

where R_{iv} is the radius of spontaneous annihilation, $D_{i,v} = D^0_{i,v}\exp(-E^m_{i,v}/k_B T)$ are the diffusion coefficients of interstitials and vacancies, $E^m_{i,v}$ are the migration energies, k_B is the Boltzman constant, T is temperature.

Due to the shadow effect, a porous structure may be formed in the film during low temperature deposition [6, 7]. Though the voids have an elongated form, for the sake of simplicity we assume that they have the form of a sphere. It should be underlined that formation of this structure is not bound up with diffusion of vacancies because of low temperature. Here we do not consider the reason for void formation. It is important for us that porous structure is generated without diffusion and irradiation. Our goal is to describe the evolution of this structure under ion irradiation.

The rate constant for the processes of interaction of point defects with a void is

$$D_{i,v} S_p \quad (4)$$

where S_p is the averaged void sink strength. For a single void with radius R [21]

$$s_p = 4\pi R. \quad (5)$$

Let the size distribution function of voids $f(R,x,t)$ be normalized to the number of voids per unit volume $n(x,t)$

$$\int_0^\infty f(R,x,t)\,\mathrm{d}R = n(x,t). \quad (6)$$

Then the void sink strength has the following form

$$S_p(x,t) = \int_0^\infty s_p f(R,x,t)\,\mathrm{d}R = 4\pi \int_0^\infty R f(R,x,t)\,\mathrm{d}R. \quad (7)$$

The athermal reactions are spontaneous absorption of point defects by the film surface, by voids, spontaneous recombination of vacancies with interstitials in cascades and with vapour atoms near the surface. Here we consider only the spontaneous recombination of vacancies

with vapour atoms and interstitials near the surface, as the most important of all athermal reactions.

Let us consider more in detail processes occurring in a narrow, about 1–2 lattice constants, layer near the surface. At a low ion energy most of the vacancies are formed in this layer, and knocked out atoms either leave a growing coating, or penetrate deep into it. The vapour atoms refill vacancies located at small depth and as a result the density increases because of atoms displaced from surface layers deep into a coating. This mechanism was proposed for the first time by Muller. The parameter describing this reaction is the depth δ of a subsurface layer; that is defined as a depth where vapour atoms can penetrate. It is natural to suppose this value has to be about one or two distances between high packed planes, or one or two lattice constants [13].

To describe the kinetics of point defects in the film we use the following rate equations:

$$\frac{\partial}{\partial t}C_v(x,t) = -\nabla q_v - \alpha_{iv}C_iC_v - D_vS_pC_v + Q_v^{eff}(x,t) \tag{8}$$

$$\frac{\partial}{\partial t}C_i(x,t) = -\nabla q_i - \alpha_{iv}C_iC_v - D_iS_pC_i + Q_i^{eff}(x,t) \tag{9}$$

where $C_{v,i}$ are the vacancy and interstitial concentrations per atom,

$$q_{v,i}(x,t) = -D_{v,i}\nabla C_{v,i}(x,t) \tag{10}$$

are the vacancy and interstitial fluxes, $Q_{v,t}^{eff}$ are the effective point defect generation rates per atom, derived from equations (1), (2) taking into account refilling of vacancies by vapour atoms and interstitials:

$$Q_v^{eff}(x,t) = \begin{cases} 0 & x \leqslant x_0 + \delta \\ Q_v(x,t) & x > x_0 + \delta \end{cases} \tag{11}$$

$$Q_i^{eff}(x,t) = \begin{cases} \max(Q_i(x,t) - Q_v(x,t), 0) & x \leqslant x_0 + \delta \\ Q_i(x,t) & x > x_0 + \delta. \end{cases} \tag{12}$$

It should be underlined that equation (11) is correct if the number of vapour atoms and interstitials refilling the vacancies in the subsurface layer is more than the number of generated vacancies.

The surface is assumed to be an ideal sink, so that the boundary condition can be written as:

$$C_{i,v}(x_0,t) = 0. \tag{13}$$

The growth rate depends on the vapour flux J_n, the fluxes of point defects at the surface $q_{i,v}(x_0,t)$ and the dimensionless density on the surface $\rho(x_0,t)$:

$$\frac{\mathrm{d}x_0}{\mathrm{d}t} = -(J_n\Omega + q_i(x_0,t) - q_v(x_0,t))/\rho(x_0,t). \tag{14}$$

It is necessary to add an equation for the void sink strength S_p (or for the size distribution function of voids $f(R,x,t)$) to equations (8), (9) and (14). Under irradiation, voids change their size according to the equation:

$$\frac{\partial}{\partial t}f(R,x,t) + \frac{\partial}{\partial R}(v_R f(R,x,t)) = Q_p(R,x,t) \tag{15}$$

where $v_R = \mathrm{d}R/\mathrm{d}t$ is the rate in the size space, Q_p is the rate of void generation.

The growth or dissolution rate of a void in the effective medium approximation is given by the following equation

$$\frac{\mathrm{d}R}{\mathrm{d}t} = \frac{1}{R}[D_vC_v(x,t) - D_iC_i(x,t)]. \tag{16}$$

We assume that at the initial moment of time the void distribution is homogeneous

$$f(R, x, 0) = f_0(R) \tag{17}$$

where function $f_0(R)$ depends on temperature and vapour flux.

Besides, we assume that new voids are not formed during irradiation because of vacancy immobility at low temperature, i.e.

$$Q_p = 0. \tag{18}$$

Last we assume that the void distribution on the surface of a growing film is the same as in absence of irradiation, i.e. is not dependent on time:

$$f(R, x_0, t) = f_0(R). \tag{19}$$

Our goal is to find the density far from the film surface, i.e. at $x \to \infty$. The expression for the dimensionless density $\rho(x, t)$ has the following form:

$$\rho(x, t) = 1 - C_v(x, t) + C_i(x, t) - \frac{4\pi}{3} \int_0^\infty R^3 f(R, x, t)\, \mathrm{d}R. \tag{20}$$

From equations (8), (9) and (15), (16), (20) the equation for the film density follows

$$\frac{\partial \rho}{\partial t} = \nabla(q_v - q_i) - Q_v^{eff} + Q_i^{eff}. \tag{21}$$

To solve this equation it is necessary to know fluxes of point defects $q_{i,v}$, i.e. the solution of the diffusion equations (8), (9).

If the atom and ion fluxes do not depend on time, after transients the film grows steadily. In this case all functions depend on a combination $x + Vt$, where $V = -\mathrm{d}x_0/\mathrm{d}t$ is the growth rate. The complete set of equations describing the film growth in the coordinate system moving with the surface can be written as follows:

$$-D_v \frac{\mathrm{d}^2}{\mathrm{d}x^2} C_v + V \frac{\mathrm{d}}{\mathrm{d}x} C_v + \alpha_{iv} C_i C_v + D_v S_p C_v = Q_v^{eff}(x) \tag{22}$$

$$-D_i \frac{\mathrm{d}^2}{\mathrm{d}x^2} C_i + V \frac{\mathrm{d}}{\mathrm{d}x} C_i + \alpha_{iv} C_i C_v + D_i S_p C_i = Q_i^{eff}(x) \tag{23}$$

$$S_p(x) = 4\pi \int_0^\infty f(R, x) R\, \mathrm{d}R \tag{24}$$

$$\frac{\partial}{\partial x} f(R, x) + \frac{\partial}{\partial R} \left(f(R, x) \frac{\mathrm{d}R}{\mathrm{d}x} \right) = 0 \tag{25}$$

$$\frac{\mathrm{d}R}{\mathrm{d}x} = \frac{1}{VR} [D_v C_v(x) - D_i C_i(x)] \tag{26}$$

$$V = \frac{1}{\rho(0)} \left[J_n \Omega - D_v \frac{\mathrm{d}}{\mathrm{d}x} C_v(0) + D_i \frac{\mathrm{d}}{\mathrm{d}x} C_i(0) \right] \tag{27}$$

where the coordinate $x = 0$ corresponds to the surface. The boundary conditions for concentrations of point defects are

$$C_{i,v}(0) = 0 \tag{28}$$

$$\frac{\mathrm{d}}{\mathrm{d}x} C_{i,v}(\infty) = 0. \tag{29}$$

The initial size distribution, or equivalently, the boundary condition for the size distribution function of voids (19) takes the form

$$f(R, 0) = f_0(R). \tag{30}$$

It is easy to see that equations (25), (26) subject to boundary condition (30) have the solution

$$f(R,x) = f_0(\sqrt{R^2 - R_m^2(x)})\frac{R}{\sqrt{R^2 - R_m^2(x)}} \tag{31}$$

where

$$R_m^2(x) = \frac{2}{V}\int_0^x [D_v C_v(x') - D_i C_i(x')]\,\mathrm{d}x'. \tag{32}$$

The density in the bulk of the film is given by the expression that follows from equation (21)

$$\rho(\infty) = \rho(0) + \frac{1}{V}\left[D_v \frac{\mathrm{d}}{\mathrm{d}x}C_v(0) - D_i \frac{\mathrm{d}}{\mathrm{d}x}C_i(0)\right] - \frac{1}{V}\int_0^\infty [Q_v^{eff} - Q_i^{eff}]\,\mathrm{d}x. \tag{33}$$

In the case of immobile point defects (i.e. at $D_{i,v} = 0$) this formula reduce to the expression for the film density derived in [13].

3. Growth kinetics of film bombarded by self-ions. Results

Using the model described above let us consider the growth of chromium film that is subjected to irradiation by chromium ions.

We choose the initial size distribution function of voids in the Gaussian form:

$$f_0(R) = \frac{2}{\sqrt{\pi}R_0}\exp\left(-\frac{R^2}{R_0^2}\right)n_0 \tag{34}$$

where R_0 is the characteristic radius of voids, n_0 is the number of voids per unit volume. For comparison some calculations were performed with the initial size distribution function of voids in the form of a δ-function:

$$f_0(R) = \delta(R - R_0)n_0. \tag{35}$$

All calculations were carried out for the temperature $T = 300$ K, the total current density $J_n + J_{ion} = 3.5 \times 10^{16}$ cm^{-2} s^{-1}, $R_0 = 4.0$ Å and the initial (boundary) density $\rho(0) = 0.9$. Numerical values of the parameters used in calculations are given in table 1.

Profiles of generation of vacancies, interstitials and implanted atoms $p_{v,i,imp}(x)$ were obtained by the PC version of code PERST [25]. As an example these profiles are presented in figure 1 for ion energy $E = 300$ eV. The vacancy profile is shown with the minus sign. The sum of profiles $\sum(x) = p_i(x) + p_{imp}(x) - p_v(x)$ is negative near the film surface because of

Table 1. List of parameters.

Vacancy diffusivity pre-exponential D_v^0 (cm^2 s^{-1}) [22]	0.30	Interstitial diffusivity pre-exponential D_i^0 (cm^2 s^{-1}) [22]	0.015
Vacancy migration energy E_v^m (eV) [23]	1.10	Interstitial migration energy E_i^m (eV) [23]	0.15
Volume per Cr atom Ω (Å^3)	12.0	Temperature T (K)	300
Recombination radius R_{iv} (Å)	5.8	Thickness of δ-layer (Å)	2.05, 4.1, 6.15
Total flux $J_n + J_{ion}$ (cm^{-2} s^{-1})	3.5×10^{16}	Ratio of ion-to-vapour fluxes J_{ion}/J_n	0–1
Initial density $\rho(0)$	0.90	Initial void R_0 (Å)	4.0
Ion energy E (eV)	50–500	Displacement energy E_d (eV) [22]	28
Sublimation energy U_s (eV) [24]	4.12	Stop energy E_f (eV)	4

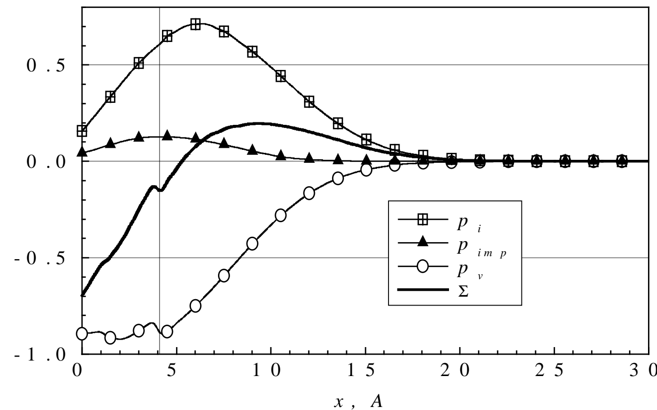

Figure 1. Profiles of point defects generated by $E = 300$ eV chromium ions in the chromium film.

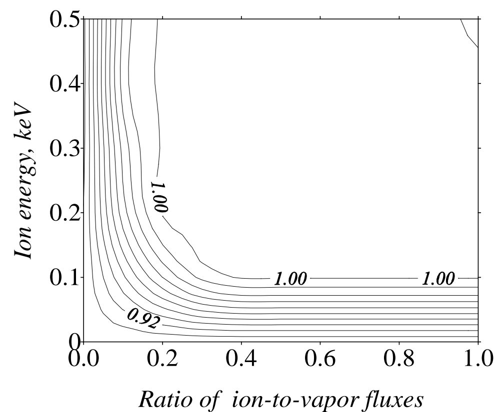

Figure 2. The calculated dimensionless density of Cr coating versus the ion energy and ratio of ion-to-vapour current densities. Total flux $J_n + J_{ion} = 3.5 \times 10^{16}$ cm^{-2} s^{-1}, thickness of δ-layer $\delta = 4.1$ Å, $R_0 = 4.0$ Å.

the surface depletion. The vertical line separates the region in which all vacancies disappear due to recombination with vapour atoms, implanted atoms and interstitials.

Results of calculations of the coating density and the rate of growth obtained for different ion fluxes and ion energies are shown in figure 2 and figure 3 correspondingly. In these calculations we chose the thickness of the δ-layer to be equal to double the distance between close packed planes.

From figure 2 one can see that a dense coating can be obtained if the ion energy is higher than 100 eV and if the ratio of ion-to-vapour fluxes is higher than 0.2. However figure 3 shows that at ion energies higher than 200 eV the growth rate decreases because of sputtering. The growth rate reduces with increasing ion energy and/or ratio of ion-to-vapour fluxes. Therefore there exists an optimal range of ion energy and ion-to-vapour ratio for obtaining dense films or coatings with maximum rate of growth. In our case these parameters obey the relations $E = 100$–150 eV and $J_{ion}/J_n > 0.25$.

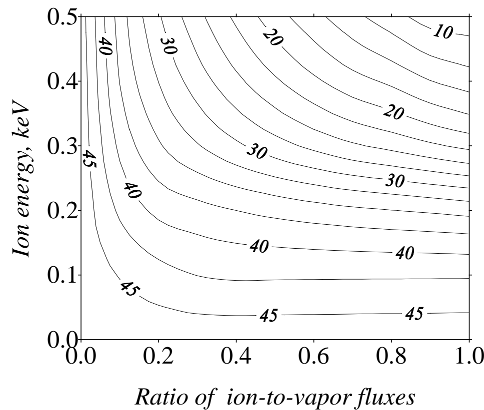

Figure 3. The calculated growth rate (Å s^{-1}) of Cr coating versus the ion energy and ratio of ion-to-vapour current densities. Total flux $J_n + J_{ion} = 3.5 \times 10^{16}$ cm^{-2} s^{-1}, thickness of δ-layer $\delta = 4.1$ Å, $R_0 = 4.0$ Å.

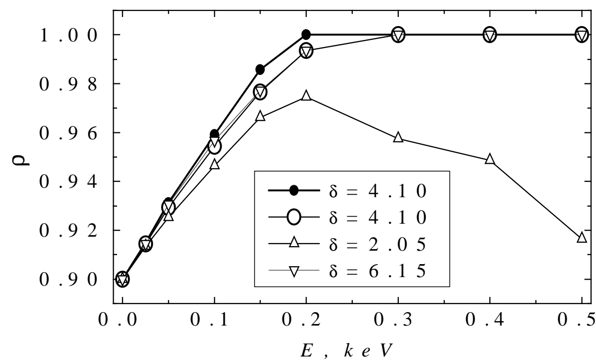

Figure 4. Dependence of the dimensionless density on the ion energy for different thickness of δ-layer. Values of δ are given in ångströms. Total flux $J_n/J_{ion} = 3.5 \times 10^{16}$ cm^{-2} s^{-1}, $J_{ion}/J_n = 0.15$.

We also investigated the sensitivity of results with respect to different thicknesses of the refilling layer and the form of initial size distribution function. Figure 4 and figure 5 show the dimensionless density versus ion energy at $J_{ion}/J_n = 0.15$ and $J_{ion}/J_n = 0.25$ correspondingly. Open symbols represent calculations for initial size distribution in the Gaussian form (34), and solid circles display calculations for initial size distribution in the form of a δ-function (35). It can be seen that the difference between cases of two-layer ($\delta = 4.10$ Å) and three-layer ($\delta = 6.15$ Å) refilling depth is small. At shallow refilling depth ($\delta = 2.05$ Å) the density decreases at high ion energy. Obviously, that this value is not correct, because the contribution of the sputtering process increases with ion energy increasing.

In the case when all initial voids are of the same size (35) the densification process is more intensive as compared to the case of the Gaussian size distribution (34). The reason is that in case (34) there are a few large voids which have significant volume and which are not sufficient as a sink for interstitials. In spite of this the difference between calculations with these two distribution functions is small.

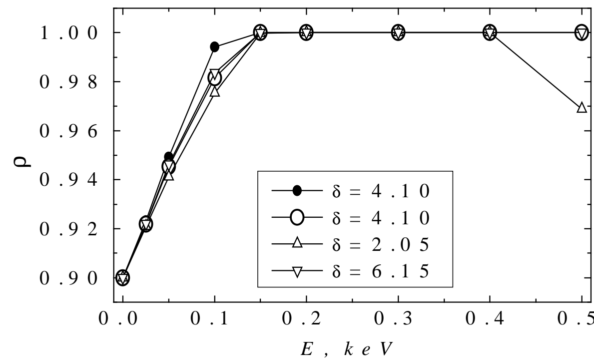

Figure 5. Dependence of the dimensionless density on the ion energy for different thicknesses of δ-layer. Values of δ are given in ångströms. Total flux $J_n/J_{ion} = 3.5 \times 10^{16}$ cm^{-2} s^{-1}, $J_{ion}/J_n = 0.25$.

4. Conclusions

(1) A physical model of low temperature densification of films produced by vacuum deposition of low energy atomic-ion fluxes of metal was formulated. The diffusive mobility of point defects was shown to play an important role in film densification even at rather low temperatures of deposition ($T < 400$ K).

(2) There exist the optimal range of ion energy and ratio ion-to-vapour fluxes for obtaining dense films or coatings with maximum rate of growth. For chromium films and coatings there are $E = 100$–150 eV and $J_{ion}/J_n > 0.25$. Application of ions with higher energies leads to sputtering and reduces the rate of growth.

(3) Approximation of average void size results in a higher density of film as compared to the case of the Gaussian initial size distribution, but the discrepancy is not large.

(4) On the basis of the proposed model a package of computer programs was developed. This package allows us to calculate the density and the growth rate of films (coatings) as a function of technological parameters of the process of deposition, such as ion energy, ion and atom fluxes, substrate temperature etc.

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Appendix. Short description of the code PERST

Like well known codes TRIM [26], ACAT [27] etc, this code is based on the concept of binary collisions of ions with initially motionless atoms of a target. A film is considered as a homogeneous amorphous medium with density ρ. The mean free path of a particle is supposed to be rectilinear. The elastic scattering of an incident particle on atoms of a film is considered in the classical approach of binary collisions [28]. The atom–atom interaction is described by a universal potential with shielding by Firsov [29]. A moving particle loses energy in a continuous way. A particle is considered stopped if its kinetic energy becomes lower than the

threshold stop energy E_f. A motionless atom of film after interaction with an incident particle begins to move if it receives energy higher than the threshold energy of movement E_m. In simulations of sputtering the bulk binding energy E_b is assumed to be equal to zero, and the energy of a stop E_m is put equal to the sublimation energy U_s [24] and the surface binding energy is chosen equal to U_s. When modelling the Frenkel pair generation, E_m is chosen equal to the threshold energy of defect production E_d. There are technical difficulties in using of two various values of energy during simultaneous modelling of these two processes. This difficulty is excluded by a preliminary choice of appropriate recombination radius of point defects $R^s_{iv}(E_d)$. In particular, for chromium $E_d = 28$ eV, $R^s_{iv} \cong 0.88l$, where $l = \rho^{1/3}$ is the mean free path.

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