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## GROWTH OF CATALYTICALLY ACTIVE NANOSTRUCTURES IN THE NONEQUILIBRIUM EPITAXY REGIME

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*The dynamics of metal atom deposition on a flat metal substrate in the diffusion mode has been studied. Conditions for growing up the periodic structures with a developed surface morphology are found. The applicability of the results obtained to the manufacture of catalysts is analyzed. In particular, when platinum atoms are deposited on a gold substrate, which is expedient by cost reasons, a system of nanopillars confined by (111) facets can be formed under special conditions. This structure possesses an ultrahigh catalytic activity and is promising for applications in chemical industry.*

*Keywords:* catalysts manufacturers, nanostructures, epitaxy.

### 1. Introduction

Nanoscale morphology of the surface has been intensively studied, especially in the case where the surface growing due to the adsorption of atoms, ions, or molecules, simultaneously changes its structure [1–7]. The research of the mechanisms of structure growth on surfaces used as catalysts and the conditions of formation of crystallographic facets with enhanced activity (e.g., facets with the Miller indices (111) in Pt structures) are of great importance due to a considerable number of applications. Recently [8–10], it has been demonstrated experimentally that, in the course of surface growth, the clusters and nanopillars including structures with a large fraction of facet (111) area can be formed.

The obtained results stimulated the corresponding theoretical researches in two directions. First, the calculation models of many-particle systems that, on the one hand, would correspond to real physical one as much as possible and, on the other hand, could be tackled with the use of available computational resources had to be developed for this class of problems. The second direction consists in the application of this tool to solve the following problems: (i) the choice of substrates that would be the best for growing up catalytically active morphological structures,

(ii) the optimization of the amount of a deposited substance in order to provide the largest active area, and (iii) the determination of key factors (the temperature and the concentration regimes) that would provide a controllable growth of nanostructures in a nonequilibrium state avoiding their transition into the state of chaotic dynamics.

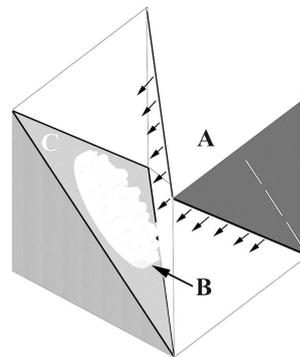
Hence, it is important to elucidate how the surface evolves under such conditions and whether it is possible to provide the formation of required (desirably, homogeneous) morphologies. This task demands the application of a general modeling that would involve several competitive dynamical processes: mass transfer, surface relaxation, evaporation, and deposition. In this work, the properties of formed structures (i.e. the chemistry and physics of their catalytic activity) are not studied from the viewpoint of their further application. The main efforts are aimed at determining the mechanisms of synthesis of such surface structures, whose morphology is favorable for catalysis. The object of our researches is a platinum (Pt) structure, a typical catalyst with catalytically active (111) facets. The model is based on the general approach used in atomistic modeling, i.e. the kinetics of particles, atoms, ions, or molecules (we will refer to them as atoms). This model was recently developed and used in researches dealing with the growth of nanoparticles with a fixed shape both on a substrate [1] and in the bulk

[11]. In the present work, the model is adapted to consider the growth of deposits on the surface of type (111).

The dynamics of the surface modification process is substantially governed by the diffusion of free atoms over the surface, which is accompanied by a surface diffusion of the hopping type. Some atoms can escape from the surface, and then come back again. The motion of atoms over the surface is regulated by a thermal-like rule, which will be considered below. The rate of surface diffusion is responsible for the possibility to establish a local equilibrium. It is evident that, if the surface diffusion is not taken into account, the adsorption of free atoms gives rise to a fractal structure [12–14]. If the external layer relaxes within the time interval  $\tau_r$  that is much shorter than the time  $\tau_d$  of the formation of an additional layer as a result of the atomic flux to the surface, a stationary regime of growth takes place. In this regime ( $\tau_r \ll \tau_d$ ), the isolated equilibrium nanoparticles obtain the Wulff shape [15–19]. We are not interested in this regime of surface layer growing. Evidently, the case  $\tau_r \gg \tau_d$  with the uncontrollable surface growth also does not correspond to the aim of this work. Hence, we intend to study a moderately nonequilibrium regime with  $\tau_r \sim \tau_d$ .

A controllable synthesis of nanoparticles in the moderately nonequilibrium regime (without a breakdown into chaotic dynamics) was recently studied in work [14]. On the basis of a proposed physical model, it was shown that the nanoparticles with various faceted shapes can be grown up for a metal of the same crystal class. The results obtained were confirmed by experimental data for a body-centered lattice [19]. Therefore, we may hope that the results of our work [14] obtained with the use of the same model are reliable. In parallel, the problem of lowering the cost of catalysis is solved. Instead of a platinum substrate, we propose to use a gold one with the orientation (111) (the lattice parameters of gold and platinum are very close to each other). Hence, platinum atoms are deposited on the gold (111) surface.

We suppose the following scenario. If the density of a diffusion flux corresponds to the moderately equilibrium regime, there emerge the periodic cluster structures on the substrate surface. In the course of time, every of those clusters is transformed into a nanopyramid, with a considerable part of its surface being



**Fig. 1.** Configuration of the calculation region: (A) emitter, the lower surface of which is a source of free atoms, (B) fragment of one of the structures obtained in numerical experiments, (C) substrate plane

formed by the (111) facets of platinum (the geometry of a calculation region and the example of a deposited atom configuration are shown in Fig. 1). The final structure is a layer of platinum with a developed catalytically active surface. For the described regime to be realized and the usage of the given platinum mass to be maximally effective, the corresponding conditions have to be provided.

Note that the orientation of a substrate with a face-centered lattice is important for the creation of the desirable surface structure. In work [1], the facet (100) was used in a similar problem. In that case, nanorods grew on the substrate; the nanorode tops were presented by the platinum (111) facets, whereas the lateral surface by the facets (100) and (110), which were perpendicular to the substrate plane and are characteristic of the equilibrium Wulff configuration. Such a general configuration of a nanorod is associated with a bad choice of the initial geometry, because it is difficult to provide a stable uniform growth of nanorods under such conditions. Should one of the rods dominate by height, it would attract the diffusion flux and slow down the growth of its neighbors. As will be shown below, this shortcoming does not arise for the substrate orientation (111). Nanopyramids which are formed on the substrate are confined by (111) facets both from above and, to a great extent, from aside (at an acute angle to the substrate surface), which substantially reduces the possibility of the advanced growth for them.

The structure of the paper is as follows. In Section 2, the details of a numerical model are discussed,

and the physical interpretation of dynamic laws forming the basis of the model is presented. In Section 3, the results of calculations are presented and analyzed. Section 4 contains our conclusions.

## 2. Model Description

The geometry of a calculation region is depicted in Fig. 1. In a cube with an edge of  $480a$  ( $a$  is the distance between neighbor (100) atomic layers, and  $2a$  is the size of an elementary cell in the face-centered cubic (fcc) crystal), two planes are marked. They are perpendicular to cube's diagonal, pass through cube's vertices, and divide the diagonal into three equal parts. One of the planes (C) is a substrate, and the other (A) is an emitter of free atoms. The distance between the planes  $d \sim 280a$ . In a layer  $10a$  in thickness which adjoins the emitter, a constant concentration of free atoms,  $n_0$ , is maintained. Below, the dimensionless distances normalized by  $a$  will be used. The time will be reckoned in units of  $\tau$ , the time interval between the jumps of free or adsorbed atoms. Free atoms move chaotically, which is simulated by jumps in random directions. The hopping distance per time unit equals 1. Then, the diffusion coefficient  $D$  and the density  $\Phi$  of diffusion flux perpendicularly to the substrate equal

$$D = \frac{1}{6}, \quad \Phi \sim \frac{Dn_0}{d}, \quad (1)$$

where  $n_0$  is the number of atoms in the volume  $a^3$ , and  $d = 280$  is the distance between the planes. The zones under the substrate and above the emitter are supposed to be unreachable for atoms. The cube edges elastically repulse atoms that try to leave the calculation region. A characteristic structure formed on the substrate of adsorbed atoms is shown in Fig. 1 in the circle.

When a free atom reaches the substrate surface or the surface of already formed cluster structure, it occupies the nearest vacancy. After the atom is captured, it occupies the volume  $a^2$  with the center at the crystal lattice site.

Adsorbed atoms can move into neighbor vacancies or even escape from the surface of a formed cluster. In the fcc crystal, every atom can interact with its 12 nearest neighbors. If this number,  $m_0$ , is less than 12, the probability, per time unit, that the atom

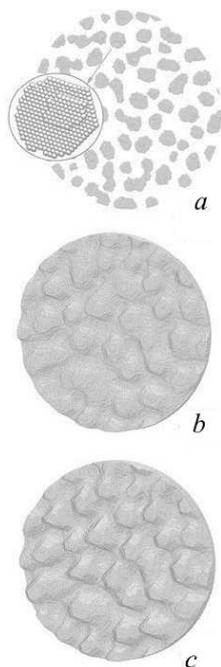
will try to change its localization place equals  $p^{m_0}$ , where  $p < 1$  is the problem parameter ( $p \sim 0.5 \div 0.8$  [14]). The direction of probable motion is random. It is connected with the number of neighbors in the new position and is realized with a probability proportional to  $e^{m_i|\varepsilon|/kT}$ , where  $\varepsilon < 0$  is the binding energy of the atomic pair, and  $m_i$  is the number of the nearest neighbors in a new position (the value  $m_i = 0$  corresponds to the probable evaporation of the atom from the cluster surface). In our calculations, we used the parameter  $\alpha = \exp \frac{\varepsilon}{kT} \approx 2$  [14]. Within one step of Monte-Carlo (MC) algorithm, which was selected to be equal to the time unit, the displacements of both free and adsorbed atoms occurred randomly. The total number of the atoms involved in the dynamical process reached  $10^6$ , and the number of MC steps was  $(20 \div 60) \times 10^6$ .

## 3. Results of Numerical Simulation and Their Analysis

### 3.1. Typical size of nanoformations

It is natural that, at the initial stage of free atom adsorption on the substrate surface, only isolated clusters are formed (Fig. 2,a). A further evolution can go in two ways. The first way corresponds to an equilibrium regime, where the separate clusters form a continuous (filled) first layer, and this process is repeated periodically. The second way is nonequilibrium and the most important. Instead of the dense filling of the first layer, the formed clusters start to grow upward, and the second, third, and so on layers are formed on them (see the inset in Fig. 2,a).

We can evaluate the size  $l$  of a cluster, for which the tendency to form a multilayer structure prevails over the tendency to expand over the substrate. For the formation of the following layer of atoms to start, it is necessary that the deposition time  $(\Phi l_2)^{-1}$  of a free atom onto a cluster with the characteristic size  $l$  should be of an order of the time  $l^2/(2D_s)$  needed for an adsorbed atom to roll down (diffusively) from this cluster. Here,  $D_s$  is the diffusion coefficient of a separate atom on the filled (111) surface:  $D_s = 0.43p_0^3$  (we took into account that this atom is connected with three atoms located below and can jump in six equiprobable directions each  $\sqrt{2}$  in length, and the jump time (in dimensionless units) equals to 1; we recall that the dimensionless units for the length and time are used).



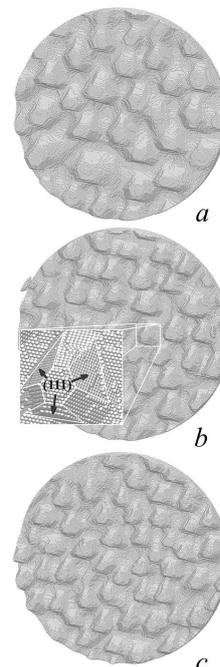
**Fig. 2.** Evolution of the surface of type (111) during the diffusion growth. The flux of free atoms onto the surface  $\Phi = \Phi_0 \approx 1.77 \times 10^6$ , the parameter  $\alpha = 2$ . The time:  $10^5$  (a),  $3.5 \times 10^6$  (b), and  $6 \times 10^6$  MC steps (c)

For the parameters indicated above, we obtain

$$l = \left( \frac{2D_s}{\Phi} \right)^{1/4} \approx 20, \quad (2)$$

which quantitatively agrees with the data in Fig. 2, a. The parameter  $l$  characterizes the size of a region on the substrate, where the equilibrium regime of growth locally dominates. Therefore, the shape of clusters that grow on the substrate upward has a tendency to be transformed into the Wulff configuration in the case of an isolated nanoparticle. In this case (Figs. 2 and 3), at the late stages of epitaxy, which is nonequilibrium for the substrate as a whole, the surface becomes covered with a system of rather ordered nanopyramids. The latter, owing to their physical origin, are confined by, mainly, planes (111).

Note that, for the nanopyramids to preserve their equilibrium shape (at a length scale of about  $l$ ) when growing further, they need to accumulate the mass not only at their apices, but also on the lateral faces that are inclined with respect to the substrate. Therefore, if the balance  $\tau_r \sim \tau_d$  is insignificantly violated, i.e.  $\tau_r \geq \tau_d$ , the development of pyra-

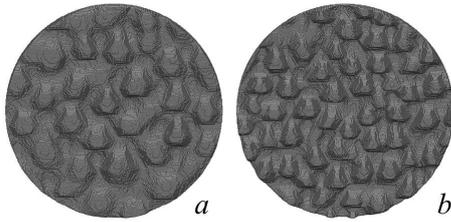


**Fig. 3.** Final configuration of the surface structure in the  $2\Phi_0$  growth regime with the flux of free atoms  $\Phi = \Phi_0$  (a),  $2\Phi_0$  (b), and  $3\Phi_0$  (c).  $\alpha = 2.5$ . The time is fixed at moments, when the numbers of atoms are approximately the same in all cases. The inset: Schematic image of facets of nanopyramids grown up on the surface. Arrows mark facets (111)

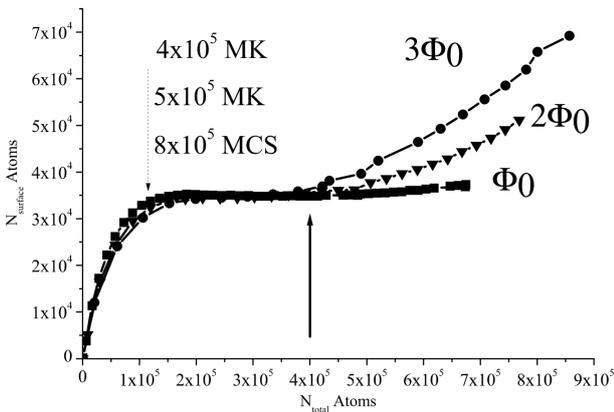
mid instability along their height is less probable than in the case of the substrate (100) orientation [11]. It will be recalled that, in the case of such an orientation, the lateral faces of nanorods are perpendicular to the substrate plane and parallel to the general direction of atomic transport, which gives rise to the elongation of nanorods keeping their cross-section constant. The diffusion flux of free atoms is so redistributed in the space that it is completely concentrated at the nanorod tops.

In view of the mechanism of stationary growth of nanopyramids in our case (if the optimum regimes for the fabrication of catalytic structures are determined), the epitaxy has to be terminated in due time. Otherwise, the transport of free atoms would be spent only on additional deposition beneath the system of nanopyramids rather than a modification of the surface morphology.

In Fig. 3, the characteristic size of pyramids  $l$  monotonously diminishes, as the flux grows from  $\Phi = \Phi_0 \approx 1.77 \times 10^6$  (panel a) to  $\Phi = 3\Phi_0$  (panel c).



**Fig. 4.** Final configuration of the surface structure. The concentration and the flow of free atoms  $\Phi = 3\Phi_0$  are identical for both panels. The parameter  $\alpha = 2$  (a) and 2.5 (b)



**Fig. 5.** Dependence of the number of catalytically active surface atoms ( $y$ -axis) on the total number of atoms in the system ( $x$ -axis) for fluxes  $\Phi = \Phi_0$  (squares),  $2\Phi_0$  (triangles), and  $3\Phi_0$  (circles).  $\alpha = 2.5$ . The dashed arrow marks the time (in Monte-Carlo steps), when the initial substrate becomes filled. The solid arrow marks the number of adsorbed atoms, at which the catalytically active area is almost identical irrespective of the growth regime



**Fig. 6.** Growth of two-dimensional clusters on the lateral faces of nanopyramids.  $\alpha = 2.6$ , a time moment of  $3.7 \times 10^6$  MC steps

As a result, the number of nanopyramids in Fig. 3,  $c$  is one and a half times as large as in Fig. 3,  $a$ . This effect resulting from the increase of  $\Phi$  is close to the estimate  $l_{3\Phi_0}^2/l_{\Phi_0}^2 = \sqrt{3}$ , which follows from Eq. (2). The time in Fig. 3 was so selected that the total number of adsorbed atoms in each panel is the same.

Another, not less important, parameter in formula (2) is the surface diffusion coefficient  $D_s$ . A temperature reduction ( $T \sim 1/\alpha$ ) reduces the mobility of surface atoms, since the difference between the probabilities for states with different numbers of nearest neighbors increases. Figure 4 shows the final surface for the parameter  $\alpha = 2$  (panel a) and 2.5 (panel b), with all other parameters remaining identical (in particular,  $\Phi = 3\Phi_0$ ). One can see that the scale of epitaxy instability decreases.

In Fig. 5, the dependences of the number of surface atoms, for which the number of nearest neighbors varies from 9 to 11 (the catalytically active atoms, CAAs), on the number of all atoms adsorbed on the substrate are depicted for three regimes of growth:  $\Phi = \Phi_0$  (squares),  $2\Phi_0$  (triangles), and  $3\Phi_0$  (circles). In all cases,  $\alpha = 2.5$ . The most effective usage of the deposited material was reached at  $\Phi = 3\Phi_0$ . Every point in the plot corresponds to a specific time. The difference between the regimes consists not only in the rate of deposition on the substrate. The plot (Fig. 5) testifies that the adsorption of  $4 \times 10^5$  atoms gives rise to an identical number of CAAs in different regimes (marked by a solid arrow in Fig. 5). If  $7 \times 10^5$  atoms are spent in all three variants, the areas occupied by CAAs differ by approximately a factor of two. Let us consider two limiting cases. (i)  $\Phi = \Phi_0$ . After long deposition ( $6 \times 10^6$  MC trials), the mass increases by 215%, and the number of CAAs by 6%. The main part of the spent material uniformly covers the substrate forming a thick layer without high nanopyramids or other inhomogeneities. (ii)  $\Phi = 3\Phi_0$ . The increase of the spent material by 173% gives rise to the increase in the number of CAAs by 51%. Such a difference between the useful areas of CAAs is determined by the velocity of the nanopyramid apex growth with respect to the surface bottom, which also grows intensively. The pyramid apex grows slowly with respect to the bottom in the moderate regime and rapidly, if the flux is intense.

If the nanopyramid grows further, the area of its lateral faces becomes larger than the critical size  $l$ ,

and the lateral faces start to play the role of a substrate: first, the two-dimensional clusters grow on them, as it occurred at the beginning of the growth (see Fig. 2, *a*), and, later on, three-dimensional ones (Fig. 6). Those results lie beyond the scope of our research. Studying Fig. 3 attentively, the shape of nanopramids in the case  $\Phi = 3\Phi_0$  can be regarded as tending to the tetrahedral one. In more details, the features of morphology evolution are considered in the next section.

### 3.2. Morphology

The shape dynamics of new formations has specific features. In particular, as the growth begins (Fig. 2, *a*), the two-dimensional clusters, “rags”, are formed on the substrate. They have a blurred hexagonal shape resulting from the circumstance that every adsorbed atom on the (111) surface forms a vacancy in the directions of hexagon vertices. Therefore, diffusing atoms create “hexagonal” surface clusters with energy-gained vacancies located at the cluster perimeter. From a certain moment, the scenario is repeated already on the cluster surface, so that the cluster becomes two-layer, three-layer, and so on, and the transition from the two-dimensional to three-dimensional growth takes place.

Later on, there emerge the hexagonal nanopramids (see Fig. 2 and the inset in Fig. 3) with the lateral faces (111). They are constructed from energetically equidistant planes of the equilibrium configuration of nanoparticles with the fcc symmetry (the Wulff shape) localized in a confined region with side  $l$ , in which the local equilibrium is established. Numerous experiments demonstrate the shape evolution from a truncated hexagonal regular pyramid to a tetrahedron (Figs. 3 and 4), although a regular tetrahedral shape was not reached. The tendency of nanopramids to have a tetrahedral shape is associated with different energy structures of the (111) and (100) facets. The facet (111) is catalytically active; it is characterized by a higher diffusion coefficient of adsorbed atoms,  $D_s$ , and a higher work function than that for the (100) one [1]. Therefore, the average times of growth for a single layer on the (111) and (100) surfaces are different. In particular, in Fig. 2 of work [1], the faces of the type (100) in a macroscopic nanoparticle completely disappear. This is a result of the growth from an initial grain at the following pa-

rameters:  $\alpha = 1$ , the concentration  $n_0 = 1.8 \times 10^{-3}$ , and an exposure time of  $3.5 \times 10^6$  MC steps. A similar phenomenon is observed on the nanopramids that are the local humps of the considered structure.

### 4. Conclusions

The deposition of metal atoms on a flat substrate can take place in a quasiequilibrium regime, when a homogeneous layer of adsorbed atoms over the whole surface is unstable, but, in local zones, the equilibrium surface configurations are created, the form of which correlates with the equilibrium Wulff configurations for isolated nanoparticles. In the fcc case, if the crystallographic facet (111) is the substrate plane, the system of nanopramids confined by the same facets (111) from both above and aside can be formed. The gain in the surface area increase is more efficient at high densities of the free atom flux onto the substrate.

The unstable regime of atomic epitaxy can be applied to the fabrication of highly active Pt catalysts. In this case, gold can be used as a substrate by the cost reasons. The substrate surface can be either smooth or preliminary prepared. The latter case implies that the gold nanopramids are formed on a gold substrate fabricated in the unstable regime of “first layer” sputtering. Those pyramids serve as centers of the platinum deposition. If the corresponding regime of “second-level” epitaxy is selected, it is quite possible that platinum will form nanoparticle chains oriented perpendicularly, which are close to the Wulff configuration by their shape. Such surface structures with a developed morphology are characterized by an ultrahigh catalytic activity. The data presented in Fig. 6 testify that the described scenario can be realized, which will be the theme of our further researches.

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ВИРОЩУВАННЯ  
КАТАЛІТИЧНО АКТИВНИХ НАНОСТРУКТУР  
У НЕРІВНОВАЖНОМУ РЕЖИМІ ЕПІТАКСІЇ

Резюме

Досліджено динаміку нашарувань, які утворюються атомами металів, що осаджені в дифузійному режимі на металеву плоску підкладку. Знайдено умови, за яких можна вирощувати періодичні структури з розвинутою морфологією. Проаналізовано можливість використання одержаних результатів під час виробництва каталізаторів: при осадженні атомів платини на золоту підкладку (що доцільно з міркувань вартості) при відповідних умовах може утворюватись система нанопірамід, обмежених гранями (111). Така структура має надвисоку каталітичну активність і є перспективною для широкого використання в хімічній промисловості.