

Research Article

Ostwald Ripening of the Platinum Nanoparticles in the Framework of the Modified LSW Theory

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An analysis of the experimental data related to the mechanism of Pt particles sintering has been carried out using the modified LSW theory. The size distribution for the Pt nanoparticles at the stage of Ostwald ripening fits the generalized Lifshitz-Slyozov-Wagner model calculated with the assumption of two parallel mechanisms involved in the nanoparticles growth (dissolution): diffusion and Wagner's (controlled by the chemical reaction rate). Comparison between the experimental histograms and the curves calculated theoretically proves the governing role of the Wagner's mechanism (chemical reaction) in the Pt nanoparticles growth.

1. Introduction

Advances in technologies, especially in nanotechnologies, cause significant interest towards Ostwald ripening (OR). It is well known that the nanoscaled particles (1–100 nm) can reveal new properties different from characteristics of the bulky substances and the potential market of the nanoapplications in different branches of science and technology such as electronics, optoelectronics, information technology, medicine, and pharmacology is very wide [1]. On the other hand, OR can take place in some nanosystems because of the Gibbs-Thomson effect, and this phenomenon results in gradual growing of the bigger particles at the expense of smaller ones. Therefore, the smaller nanoclusters or nanocrystals (NC) dissolve while the bigger ones grow. This process also causes rise in the particles mean size, widening of the NC size distribution and dispersion. As a result, this process can worsen specific characteristics and performance of the nanosystem or even cause its complete degradation. Thus, OR is an unwanted effect and the investigation of its mechanism and kinetics of the NC growing/dissolution through OR can open new opportunities in controlling this process and minimization of its negative effects.

On the other hand, there are tight links between OR and the nanotechnologies. The process of a new phase formation always includes three stages regardless of the synthesis method used or the source phase (liquid, solid, or gaseous): nucleation, independent growing of the new nanoparticles, and their Ostwald ripening. The latter stage is governed by the Gibbs-Thomson effect and many useful characteristics of new nanoparticles are formed exactly during this stage [2].

The late OR takes place when oversaturation of the synthesis solution reduces to the level when new clusters do not appear any more. Then the process of the clusters size redistribution starts and the process of OR completes the final size range and dispersion [3].

A size distribution function reflecting the most complete information related to the structural composition of the nanosystem can be calculated for the given growing mechanism and nanoclusters shape using some available theoretical approaches [4–9].

The main requirements towards structural characteristics of the applicable NC can be outlined as follows. Regardless of the synthesis method used, NC should have uniform size, shape, composition, lattice structure, and other technical parameters. Besides, the size distribution should be as narrow as possible and the dispersion should be kept low.

The two separate growing mechanisms were analyzed by the primary *LSW* theory: the diffusion mechanism controlled by the bulk diffusion coefficient D_v , as introduced by Lifshitz and Slyozov [4, 5] and Wagner's mechanism controlled by the rate of the chemical bonds formation or the surface chemical reaction rate [6].

Both mechanisms are analyzed simultaneously in the framework of the modified *LSW* theory. A ratio between two subfluxes of the matter towards/outwards the NC surface, diffusion j_v and Wagner's j_i , represents a contribution of each mechanism into the resulting process of OR [9, 10]. These contributions depend on many parameters such as nature of NC, temperature, and synthesis method.

It should be mentioned that many important applications in electronics, optoelectronics, solar energy conversion, and other branches are reported [11–18] for the semiconducting nanoscaled heterosystems containing quantum dots [19–23]. The OR stage is known for such systems that were usually synthesized using the molecular-beam epitaxy in the Stranski-Krastanov mode before [24]. More advanced and less expensive methods such as dripping, liquid phase, and electrophase epitaxy are also used for this synthesis now [25–29].

As seen from [30–34], significant progress is achieved in the synthesis of the quantum dots semiconductors using various chemical and colloid methods. For instance, nanocrystals of ZnO (one of the most universal and multifunctional semiconductors) are synthesized usually from oversaturated solutions [35–45].

As reported in [46–49], the modified *LSW* theory [9, 10] proves that the growing of NC in the nanosystems in many cases involves both mechanisms (diffusion and Wagner's) [46].

This paper is devoted to the investigation of the governing role of OR in the process of Pt NC sintering that occurs in a nanocomposite material and involves both mechanisms of growing. The carbon nanotubes are used in this synthesis as reported in [47].

Evaluation of the sintering process has been performed using temporal evolution of the particles size distribution during heating from the initial temperature to 550°C and then to 800°C. The size distribution of Pt NC changed from the Gauss type to the normal logarithmic type under such heating mode [47]. Since this sintering process runs according to OR mechanism, the particles size distribution function is expected to agree with the theoretical *LSW* distribution.

As a result, the experimental histograms [47] should also be in agreement with the theoretical distributions calculated using the modified *LSW* theory and the generalized Lifshitz-Slyozov-Wagner distribution.

2. The Fundamental of the Modified LSW Theory Applied to the Pt Nanoparticles Sintering in a Nanocomposite

The initial average size of the Pt particles in the nanocomposite was 2,62 ($\pm 0,6$) nm [47]. A nanoparticle of such size is unstable thermodynamically since its specific surface is

comparatively large, which results in high free energy value. As a system goes to the equilibrium, its free energy decreases and the particles average size grows.

It should always be taken into consideration that the nanoparticles are very small and approximate amounts of their surface and body atoms are similar, which causes higher reactivity of the particles. That is why the surface and the interface processes should be considered together with the regular diffusion transportation in order to analyze growth and dissolution of nanoparticles. In other words, Wagner's (kinetic) mechanism of the nanoparticle growth/dissolution cannot be neglected in this case of OR of the Pt NC and both processes, diffusion and chemical reaction, should be taken into account for OR in the disperse phase of the nanosystems.

Two mechanisms of the nanocrystal (NC) growth are analyzed by the modified *LSW* theory [9, 10]. One of them is the diffusion mechanism (*diffusion*) introduced by Lifshitz and Slyozov [4, 5] while the second mechanism is Wagner's kinetic growth [6] controlled by the chemical reaction rate. According to this theory the flux of atoms towards/outwards NC j can be represented as a sum of the diffusion j_v and kinetic j_i subfluxes:

$$j = j_v + j_i. \quad (1)$$

This equation represents the basic idea of the modified *LSW* theory, which means two parallel mechanisms (diffusion and Wagner's) of the NC growth.

The rate of the NC growth $\dot{r} \equiv dr/dt$ should be defined to obtain a function $f(r, t)$ describing the temporal evolution of the size distribution for the NCs. It is known that the continuity equation can be used to couple $f(r, t)$ and \dot{r}

$$\frac{\partial f(r, t)}{\partial t} + \frac{\partial}{\partial r} [f(r, t) \cdot \dot{r}] = 0. \quad (2)$$

The rate of growth \dot{r} can be defined using the condition

$$\frac{d}{dt} \left(\frac{4}{3} \pi r^3 \right) = j v_m, \quad (3)$$

where j is determined by (1). After several simple transformations we can obtain from (3)

$$\frac{dr}{dt} = \frac{B^*}{r} \left(\frac{x}{1-x} \frac{r_g}{r} + 1 \right) \left(\frac{r}{r_g} \frac{r_g}{r_k} - 1 \right), \quad (4)$$

or

$$\frac{dr}{dt} = \frac{A^*}{r^2} \left(\frac{1-x}{x} \frac{r}{r_g} + 1 \right) \left(\frac{r}{r_g} \frac{r_g}{r_k} - 1 \right), \quad (5)$$

where $B^* = 2\sigma C_\infty v_m^2 \beta / kT$, $A^* = 3\sigma C_\infty v_m^2 D_v / kT$, $x = j_v / j$, $1-x = j_i / j$, $x/(1-x) = j_v / j_i$, r is radius of the particle, r_g is the maximal size of NC, r_k is critical radius of NC, which is equal to the average size $\langle r \rangle$ ($r_k \equiv \langle r \rangle$) as set by the *LSW* theory, and σ is the specific surface energy; C_∞ is the equilibrium concentration of the dissolved substance; v_m is volume of atom of the dissolved substance; β is the kinetic coefficient determining the j_i subflux; D_v is the coefficient of bulk diffusion; k is Boltzmann's constant, and T is temperature.

Equation (4) describes the rate of the Pt NC growth governed mostly by the kinetic subflux j_i with partial contribution x made by the diffusion subflux j_v . On the contrary, (5) describes the rate of the Pt NC growth governed mostly by the diffusion subflux j_v with partial contribution $(1-x)$ made by the kinetic subflux. Therefore, the rates of growth (4) and (5) can be represented in the LSW theory by the ratio between subfluxes j_v and j_i .

According to [48], $f(r, t)$ can be represented by the product of two functions: one depending on time t and another depending on the relative variable u :

$$f(r, t) = \frac{1}{r_g^4} g(u), \quad (6)$$

where r_g depends on time and

$$g(u) = Q \cdot g'(u), \quad (7)$$

$Q = 3M/4\pi\rho \int_0^1 u^3 g'(u) du$, M is total mass of the NCs in the unit of volume, and ρ is density of the NC's substance.

A definition of the relative variable in the form

$$u = \frac{r}{r_g}, \quad (8)$$

instead of the traditional form $\rho = r/r_k$, was proposed in [48], and in this way the variation interval of u became independent on the growth mechanism of the NC ($0 \leq u \leq 1$). Function $g(u)$ in form (7) (or function $g'(u)$ (with accuracy to the constant Q)) represents the generalized distribution of Lifshitz-Slyozov-Wagner (GDLSW), which involves simultaneously two mechanisms of growth diffusion and Wagner's.

The distribution $g'(u)$ can be obtained by integration of (2) with account of (4) or (5) and proceeding from differentiation by r and t to differentiation by u [9, 10]

$$g'(u) = u^2(1-u)^B(u+x^2+x)^D \exp\left(\frac{C}{1-u}\right), \quad (9)$$

where

$$\begin{aligned} B &= \frac{2x^4 + 4x^3 + 12x^2 + 10x + 5}{F}, \\ D &= -\frac{4x^4 + 8x^3 + 6x^2 + 2x + 1}{F}, \\ C &= -\frac{3x^2 + 3x + 3}{F}, \\ F &= x^4 + 2x^3 + 3x^2 + 2x + 1. \end{aligned} \quad (10)$$

Assuming $x = 0$, $B = 5$, $D = -1$, and $C = -3$, (9) transforms (with accuracy to the constant Q) into Wagner's distribution [6]

$$g'(u) = u(1-u)^{-5} \exp\left(-\frac{3}{1-u}\right). \quad (11)$$

Assuming $x = 1$, $B = 11/3$, $D = -7/3$, and $C = -1$, (9) matches the Lifshitz-Slyozov distribution [4, 5]

$$g'(u) = u^2(1-u)^{-11/3}(u+2)^{-7/3} \exp\left(-\frac{1}{1-u}\right). \quad (12)$$

The GDLSW distribution (7) can represent adequately the size distribution for $0 \leq x \leq 1$. As the average size $\langle r \rangle$ (or critical size r_k) is growing continuously during OR, the maximum size r_g grows as well, although the ratio r_g/r_k remains unchanged. According to the modified LSW theory [9, 10]

$$\frac{r_g}{r_k} = \frac{2+x}{1+x}. \quad (13)$$

Considering Wagner's mechanism of the growing and assuming $x = 0$, we obtain $r_g/r_k = 2$ and for the diffusion mechanism and $x = 1$, $r_g/r_k = 3/2$.

Since the ratio r_g/r_k is a time independent constant, the size distribution $g'(u)$ does not depend on the initial distribution. Therefore, some generally used characteristics of $g'(u)$ depending on the initial and central moments (dispersion $\langle u^2 \rangle - \langle u \rangle^2$, the mean relative radius $\langle u \rangle$, area under the curve, etc.) also do not depend on time during OR.

The function $f(r, t)$ (6) describes the temporal evolution of the size distribution. The time dependence of r_g should be determined in order to define the explicit mode of this function. The value r_g can be found from (4) and (5) by substituting r_g for r and a value of the ratio r_g/r_k taken from (13) for this ratio. Integrating (4) for the preliminary Wagner's mechanism we obtain

$$r_g^2 = B^* \frac{1}{1-x^2} t, \quad (14)$$

$$r_k^2 = B^* \frac{1+x}{(1-x)(2+x)^2} t.$$

Assuming $x = 0$, we obtain $r_g^2 = B^* t$, $r_k^2 = (1/4)B^* t$, and $r_g/r_k = 2$.

Alternatively, integrating (5) for the preliminary diffusion (diffusion) mechanism we obtain

$$r_g^3 = A^* \frac{1}{x(1+x)} t, \quad (15)$$

$$r_k^3 = A^* \frac{(1+x)^2}{x(2+x)^3} t.$$

Assuming $x = 1$, we obtain $r_g^3 = (1/2)A^* t$, $r_k^3 = (4/27)A^* t$, and $r_g/r_k = 3/2$.

Finally, the function $f(r, t)$ (6) can be defined from the dependence of r_g on t .

3. Discussion

The distribution (7) is shown in Figure 1(a) as a series of curves for various x and Wagner's distribution ($x = 0$) is shown in the embedment. The $x = 1$ curve corresponds to

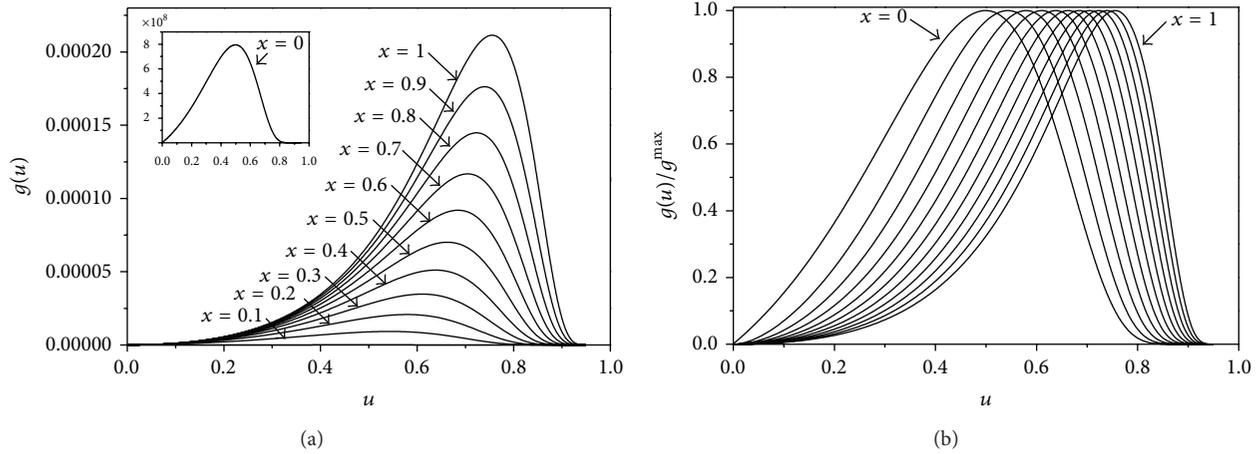


FIGURE 1: The curves corresponding to the *GDLWS* distribution calculated using the formula (7) with the iteration step $\Delta x = 0.1$ (a); same curves as in (a) normalized by their maximums (b).

the Lifshitz-Slyozov distribution. The same curves normalized by their maximums are shown in Figure 1(b). Such curves can be easily compared to the experimental data.

The experimental histogram should be presented in the same normalized mode as the theoretical curves in order to perform their comparison. The scale of the horizontal axis (it represents radii r or diameters d of NC in nm) should be transferred to the relative variable (relative diameter) $u = r/r_g = d/d_g$, where the maximal diameter d_g is taken from the histogram. The relative diameter of NC u in the histogram will be ranged between 0 and 1 after such transformation.

All values in the vertical axis corresponding to the number of particles in the unit of volume within some interval Δd are normalized by the maximum of histogram. Therefore, the histogram is normalized by the unit along all axes. The conformity or inconformity in the theoretical and experimental data can be evaluated through comparison of the experimental histograms and the normalized theoretical curves built in the same scale. The initial and central moments calculated for the experimental and theoretical distributions should be compared in order to perform the quantitative comparison.

The comparison between the normalized experimental histograms of the Pt NC [47] and the theoretical curves is shown in Figure 2. The histogram (a) shows the initial size distribution of the Pt particles and it can be noticed that this histogram and the *GDLWS* distribution (7) at $x = 0,2$ are in good agreement.

This fact confirms that the OR stage is present even in the liquid phase synthesis of the Pt NC and that the chemical reaction running on the Pt NC surface is the governing factor of OR.

It should be emphasized that the OR stage has been identified and reported using the colloid chemistry methods in some liquid phase syntheses of NC (including semiconducting NC) [32, 49–51].

Having calculated the ratio r_g/r_k (13) and the mean radius of the Pt particles ($\langle r \rangle = 2,62 (\pm 0,6)$ nm) (in the framework of

the *LSW* theory this radius corresponds to the critical value r_k), the maximal radius r_g of the Pt NC can be calculated as

$$r_g = r_k \frac{2+x}{1+x}, \quad (16)$$

where $x = 0,2$ as follows from Figure 2(a). Within the measurement error, the maximal size calculated by (16) $r_g = 4,8$ nm is in good agreement with the maximal size of Pt NC $r_g \approx 4,3$ nm measured experimentally.

A mean size of NC rises up to ($\langle r \rangle = 8,58 (\pm 2,45)$ nm) [47] and their sphericity becomes more perfect as a result of thermotreatment of nanocomposites at 550°C . The histogram (b) in Figure 2 shows size distribution for the particles at 550°C , and it is easily noticeable that it fits with the *GDLWS* distribution at $x = 0,1$, which is very close to Wagner's distribution. The maximal size r_g according to the histogram (b) is 16,5 nm, and according to the formula (16) it is about 16,4 nm. This agreement seems too good and can be a result of significant errors committed in the plotting of the experimental histograms. However, two conclusions can be drawn from the results seen in Figure 2(a): first, as noticed in [47], the shapes of the histograms (a) and (b) are quite close; second, the rate of the Pt NC growth/dissolution during their sintering is controlled by the surface chemical reaction running on the NC, that is, Wagner's mechanism. Faster diffusion processes (*diffusion*) do not control OR.

Further rise in the NC sizes occurs at rise in temperature of the nanocomposites thermotreatment and an average size of the Pt NC reaches ($\langle r \rangle = 15,89 (\pm 5,23)$ nm) at 800°C [47]. A histogram reflecting sizes distribution for the 800°C sintering of Pt NC is shown in Figure 2(c). Similar to the previous cases (a) and (b), Wagner's distribution or the *GDLWS* model at $x = 0,1$ is in good agreement with the (c) histogram. This is another proof of the governing role of the kinetically controlled Wagner's mechanism of the Pt NC sintering at 800°C . This process is one of infrequent examples of the disperse particles growth/dissolution involving OR totally controlled by the surface chemical reaction.

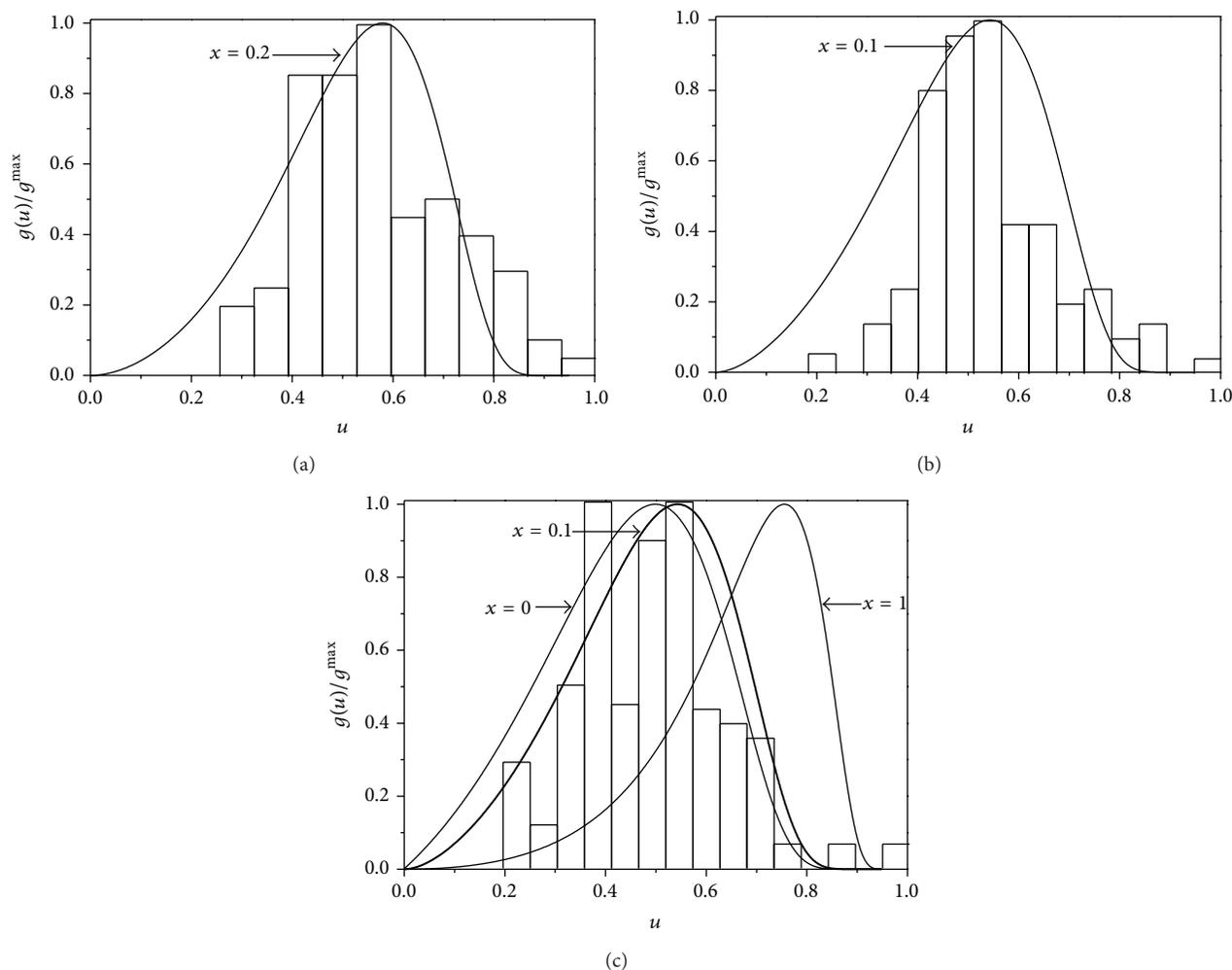


FIGURE 2: Comparison of the experimental Pt NC histograms with the theoretical *GDLSW* distributions: (a) source histogram; (b) heating to 550°C; and (c) heating to 800°C.

Good agreement between the maximal sizes of Pt NC r_g derived from the experimental histograms and calculated using the formula (16) also confirms correctness of comparison between the experimental and the theoretical size distributions. For example the maximal size calculated using (16) for 800°C is $r_g = 31,8$ nm, while the experimental value is $r_g = 33,9$ nm. Taking into account the experimental error, this difference is rather insufficient.

Therefore, the modified *LSW* theory is an adequate tool for the description of the Pt NC sintering according to the OR mechanism within the entire temperature range considered in this paper.

4. Conclusion

A theoretical analysis of the Pt NC sintering has been performed within some temperature range using the modified *LSW* theory. The theoretical *GDLSW* distribution with the x value about the unit can adequately describe the experimental size distribution for the Pt NC. Therefore, only the surface

chemical reaction on the Pt NC (Wagner's mechanism) governs the processes of growing/dissolution of the nanoparticles at the stage of OR. Moreover, the same mechanism also controls the process of the Pt NC growth/dissolution during their liquid phase synthesis. This is one of infrequent examples of OR for nanoparticles that is totally controlled by the chemical reaction according to Wagner's mechanism. This conclusion can be proven by comparison of the source experimental histograms and theoretical curves built according to the *GDLSW* approximation at $x = 0,2$.

Analysis of the high temperature sintering of Pt NC shows good agreement between the experimental and theoretical maximum sizes of the particles r_g and brings another evidence for correctness of the above theory.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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