

Phase formation under conditions of self-organization of particle growth restrictions in the reaction system

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ABSTRACT A systematic analysis of literature data concerning the influence of methods and conditions of synthesis on the possibility of self-organization of particle growth restrictions during chemical reactions of solid phase has been conducted. The prospects of using such methods to obtain nano-crystalline phases are shown. It is demonstrated that a disadvantage of such methods of synthesis is the risk of forming precursor phases instead of target products. To avoid such an outcome, several methods of synthesis are proposed. Based on the analysis of literature data, examples of the transformation of precursor nanoparticles into nanocrystals of target phases are classified and presented. A scheme that allows optimal combination of synthesis methods to obtain nano-crystalline particles of a given composition, structure, size, and shape is designed.

KEYWORDS nanocrystals, nanoparticles, self-organization, nanoreactors, particle growth restrictions

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1. Introduction

The production of crystalline particles with the smallest possible sizes can be achieved by two different mechanisms. First, it can result from thermodynamic processes related to the size of the critical phase embryo [1–4] and the crystal-chemical constraints determined by the minimum number of unit cell transformations for the formation of a stable crystalline structure of solid-phase particles [5]. Second, it can be caused by kinetic factors that condition the proportion of nucleation velocity to particle growth rate. In the latter case, it is possible to achieve minimum size particles by increasing the nucleation velocity and lowering the growth rate of new phase particles. Some aspects of increasing the rate of phase transformations were considered in [2, 6–13]. As examples of fast-flowing processes of formation of new phase crystalline particles, certain dehydration reactions under hydrothermal conditions [14–19], combustion reactions [20–30], as well as condensation of certain crystalline products from the gas phase [31–34], can be cited. It is crucial to highlight the processes whose high-speed results from the fact that the formation and growth of particles of a new phase is determined by the transfer of matter in the form of clusters with a large number of atoms [2, 35–42]. At the same time, in many cases obtaining nanoparticles in a fast-flowing process of phase formation is not possible. This is especially characteristic of the synthesis of crystalline phases of complex composition [43–56]. In these cases, it is possible to obtain nanoparticles of the synthesized phase with the smallest possible sizes by creating various kinds of restrictions for their growth. Particle growth restrictions may be thermodynamic (energetic) in nature, or they may be caused by kinetic factors. Conditions restricting the growth of particles of the new phase can be set before the start of the phase formation process or during the synthesis of a new phase. The first case involves physico-chemical processes in pre-created nanoreactors. The second case concerns self-organizing restrictions for the growth of nanoparticles during phase formation.

Different options for preliminary establishment of conditions under which mass transfer to particles of the forming phase would be restricted, that is, various types of nanoreactors were investigated in [57–68]. In these works, it was shown that the synthesis of nanoparticles in nanoreactors effectively restricts their growth and, in some cases, allows to control the morphology of the formed particles. As mentioned above, one of the options for restricting the growth of new phase particles is to conduct chemical processes in nanoreactors of diverse shapes, sizes, and compositions – such as dispersed, phase and chemical. Examples of such nanoreactors can be nanoscale and submicron pores in various materials [64, 65, 69–79], including channel pores in nanotubes [80–88], as well as various dispersed media such like microemulsions [89–91], and dispersions of nanoparticles in liquid media [92–95]. As an original nanoreactor, films

of nanoscale thickness applied, for example, by molecular layering (ML) [96–102], often called Atomic Layer Epitaxy (ALE) [103], or Atomic Layer Deposition (ALD) [104], to substrates of various compositions can be considered. Phase formation in such films or the interaction of their components with the substrate resulting in the formation of a new phase occurs with a natural restriction of the thickness of the obtained solid-phase product [105–111]. Interlayer spaces in compounds with a layered structure can also act as nanoreactors. An example of the use of interlayer spaces in hydrotalcites as nanoreactors is described in [57, 112–117]. Other heterogeneities in the structure of substances can also serve as nanoreactors – for example – cavities in polymers [118, 119]. A more complete description of various types of nanoreactors can be found in [57, 120–124], among other works.

A fundamentally different method for restricting particle growth rate is to increase the relaxation time of the process of mass transfer [6]:

$$\tau \sim D^{-1} \cdot (\Delta x)^2,$$

where D is the effective diffusion coefficient of the substance that determines the growth of particles, and Δx is the characteristic distance that the elementary components of the substance must overcome before they reach the growing particle. The latter value depends inversely on the concentration of the elementary components of the substance and the volume fraction of the particles formed in the reaction zone. Specific ways to reduce the rate of particle growth in this case may be to increase the viscosity of the reaction medium and to decrease the concentration of reagents.

Another principle of restricting the growth of particles of the synthesized phase is based on setting the conditions restricting their growth during the process of phase formation. To date, research in this area is fragmented. At the same time, the self-organization of growth restrictions of nanoparticles was observed during the formation of phases of different aggregate state, structure, chemical composition, and for nanocrystals with different morphology.

Recently, much attention has been paid to the study of the possibilities of obtaining oxide (including the production of oxyhydroxide and hydroxide) nanocrystals with a different range of variation in shape and size [11, 14, 16, 84, 125–137], including with the smallest possible crystal sizes [5]. Such interest is determined by the fact that their structure [20, 138–140] and functional properties [139, 141–147] may depend on the dimensional parameters of nanocrystals. In this regard, it is of interest to conduct a systematic analysis of the possibilities of self-organization of restrictions on the growth of particles of the forming crystalline phases. This work is devoted to this topic.

2. Nanoparticle growth restrictions occurring during the process of their formation

All types of restrictions on the growth of nanoparticles formed during their formation and presented in this paper can be conditionally divided into eight groups. The categorization depends on the physico-chemical reasons for the emergence of these restrictions as well as the methods of their formation.

The first group of restrictions on the growth of nanoparticles includes cases when the very structure of nanoparticles does not allow to increase its size continuously in certain or all directions by sequentially attaching atoms (molecules) to it. Usually, structures with a high degree of bond covalence act as such nanoparticles. The most well-known representatives of such nanostructures are carbon fullerenes, the sizes of which vary discretely depending on the number of carbon atoms in the structure from the smallest possible to the largest known to date [148–150]. It should be noted that there is a large group of similar nanostructures like buckyballs [151–153], with a very different chemical composition, including oxide buckyballs [154–160]. Analogically, the potential to increase the diameter of carbon nanotubes [161, 162] and nanotubes of other compounds [163–167] is restricted. Graphene particles, their derivatives, and morphological analogues with finite thickness are also close to the described group of nanoparticles. This variant of restricting the growth of nanoparticles can be referred to as restrictions resulting from their non-translational structure in one or more directions (Fig. 1).

The second group of restrictions on the growth of nanoparticles during their synthesis is characteristic, in particular, of nano-switches. This group is associated with the energy disadvantage of their folding following an increase in the diameter (decrease in curvature) of the nano-switch [168–173]. It is noteworthy that to date, there are only a small number of structures that differ in chemical composition and morphology (cylinders and cones) [174, 175]. The type of restrictions on the growth of nanoparticles in one or two directions that are similar in physico-chemical characteristics is associated with large differences in the values of the specific surface energy of the crystal faces. This leads to the redistribution of matter between the crystal faces during their growth and the formation of rod-shaped or lamellar morphology particles and, under certain conditions, to the formation of nanorods [19, 176–179] and nanoplates [180–183]. The described variants of self-organization of particle growth restrictions in certain directions during their formation can be classified as energy or, in generic terms, thermodynamic restrictions (Fig. 1).

Another type of nanoparticle growth restriction, which can occur during phase formation, is determined by the small magnitude of the driving force of the growth of nanoparticle size and is initiated by the high rate of nucleation [184–186]. In this case, the substance of the original composition is quickly exhausted for the almost simultaneous formation of the embryos of a new phase. The size of the forming particles in this case turns out to be close to the size of the critical embryo. Consequently, all the formed particles turn out to be similar in size. The transfer of matter from one particle to another in a system consisting of nanoparticles of almost the same size cannot be fast. This is because the driving force of the mass transfer process consists in the difference in the values of the specific surface energy, which in this specific case equals nearly zero. This type of restrictions on the growth of nanoparticles that occurred during the process of their

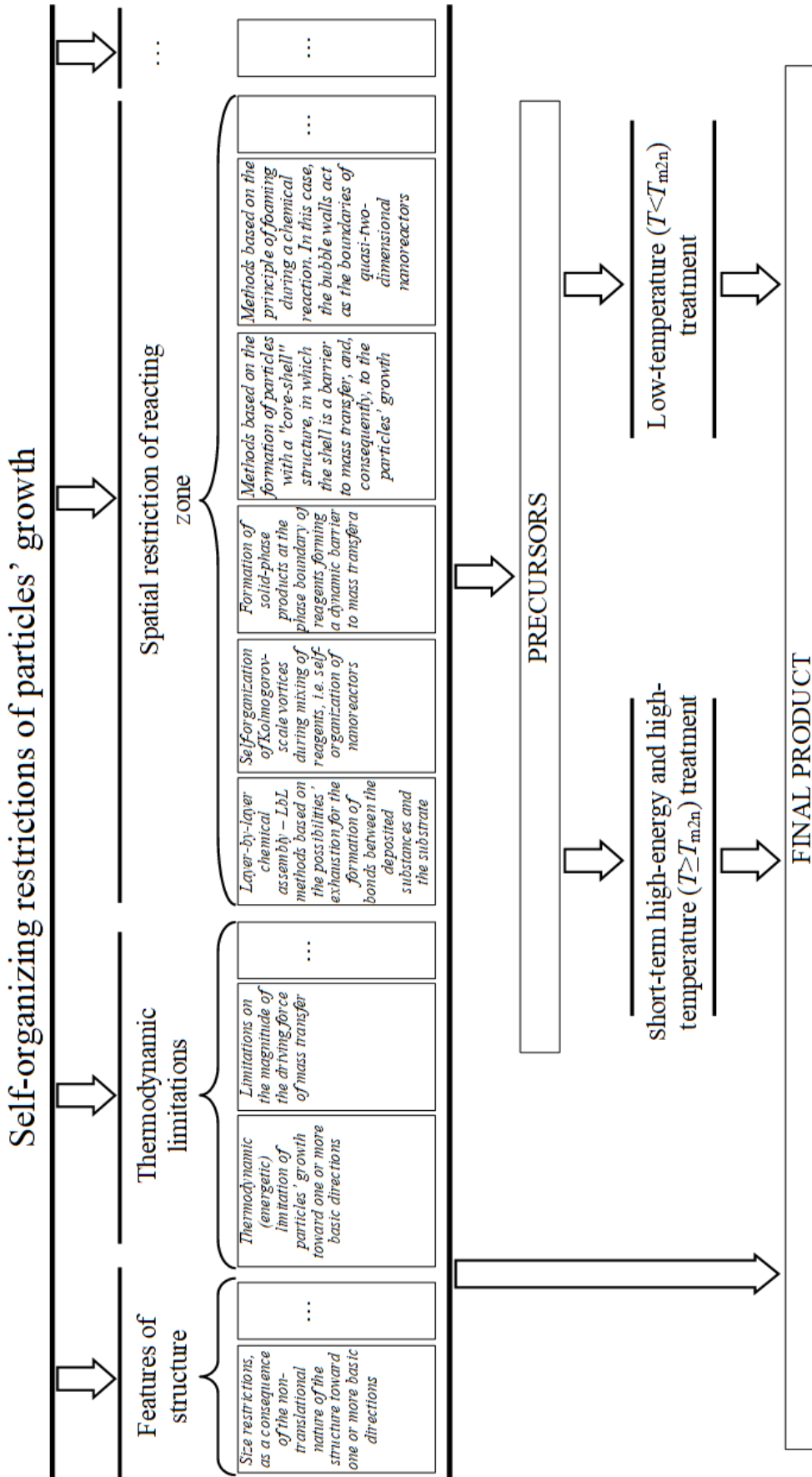


FIG. 1. Synthesis of solid phase particles over limited growth: principles and methods

formation can be classified as restrictions caused by minimizing the driving force of the mass transfer process. Some variant of this type of restriction of particle growth can be considered the case of the existence of very small particles whose specific surface energy decreases with a decrease in their size [187–193], as a result of which these particles may be in a state close to indifferent equilibrium with respect to each other. In particular, according to [194, 195], the specific surface energy of small particles is proportional to their diameter:

$$\sigma = \alpha \cdot d,$$

where $d < d_0$ is the diameter of small particles, and $\alpha = \sigma/d_0$ (Fig. 2a). In this case, in the particle size range $0 < d_i < d_0$ (Fig. 2) the expression for the value of the Gibbs energy of their formation, attributed to the unit volume, will have the following form:

$$\frac{\Delta G_i}{V_i} = \Delta g_V + \sigma_i \cdot \frac{S_i}{V_i},$$

ΔG_i is the Gibbs energy of the formation of the i -th particle, Δg_V is the volume specific Gibbs energy of the formation of the phase and, finally, S_i and V_i stand for the surface area and volume of the i -th particle. Consequently, taking into account the above dependence $\sigma(d)$, for an isometric particle, we obtain $\Delta G_i/V_i = \text{const}$ for all particles i whose size d_i lies in the interval $(0, d_0)$. In this case, none of these particles has a thermodynamic preference over other particles in the size range $(0, d_0)$.

The dependence of the Gibbs energy on the particle size can be represented as

$$\Delta G = \Delta g_V \cdot d^3 + k_f \cdot \sigma \cdot d^2 = \begin{cases} d < d_0 : & \Delta g_V \cdot d^3 + k_f \cdot \alpha \cdot d^3; \\ d \geq d_0 : & \Delta g_V \cdot d^3 + k_f \cdot \sigma_0 \cdot d^2, \end{cases}$$

where k_f is a coefficient depending on the shape of the particles.

For the case

$$k_f \cdot \alpha < |\Delta g_V|,$$

$$\sigma = \alpha \cdot d_0 : d_{cr} = \frac{2}{3} \left(\frac{k_f \cdot \alpha}{|\Delta g_V|} \right) \cdot d_0$$

the calculated value of the size of the critical nucleus is $d_{cr} < 2/3 d_0$ (Fig. 2b (I)), i.e. critical nucleus does not exist.

For the case

$$k_f \cdot \alpha > |\Delta g_V|,$$

$$\sigma = \alpha \cdot d_0 : d_{cr} = \frac{2}{3} \left(\frac{k_f \cdot \alpha}{|\Delta g_V|} \right) \cdot d_0$$

the calculated value of the size of the critical nucleus is $d_{cr} \geq d_0$ (Fig. 2b (II)).

Another type of restrictions on the growth of nanostructures is implemented within the framework of a group of methods that can be designated as methods of Layered Chemical Assembly [196]. To some extent, the term Layer-by-Layer (LbL) synthesis [196–200] is synonymous with the designation of this group of methods. Historically, the first method in this group of methods is the Molecular Layering (ML) method proposed by V.B. Aleskovsky and S.I. Koltsov [96, 99–102, 201]. In literature, this method is also referred to as Atomic Layer Epitaxy (ALE) [103] and Atomic Layer Deposition (ALD) [104]. Ideologically close to the ML is the ionic layer deposition method suggested by V.P. Tolstoy and co-authors [202]. Subsequently, this method was developed in the works of many authors [203–206]. In literature, this method is also found under terms such as Successive Ionic Layer Deposition (SILD) [207] and Successive Ionic Layer Adsorption (SILAR) [200]. The self-organization of restraining the growth of the thickness of nanolayers on the surface of a solid when using LbL methods is based on the exhaustion of the possibilities for the formation of chemical bonds between the active centers on the surface that self-organize during synthesis. These centers include landing pads and atoms (atomic groups), or ions deposited from mobile media either gaseous or liquid, with sequential (cyclic) introduction of reagents of different composition into the reaction system. The most precise synthesis of planar nanostructures by the ML method occurs in cases of formation of substances with a predominantly covalent nature of the bond. It should be noted that by changing the temperature of the ML process within a certain range, the composition, structure, and thickness of the film can be varied within this method by changing the number of active centers self-organizing in the ML process on the surface of the solid. As a consequence, the density of filling the surface layer with molecules deposited on the surface can be altered [105–108, 208]. In addition, the filling of the surface layer can also be influenced by the steric effect, which is more characteristic of organic compounds [209–211].

The described method of restricting the growth of film thickness, which is determined by the self-organization of the size of the reaction space in the direction perpendicular to the surface of the solid (Fig. 3), differs in its physico-chemical characteristics from those discussed above. The self-organization of restricting the growth of the film during its formation by LbL methods is not associated with a change in the magnitude of the driving force of the chemical interaction of reagents, as was the case in the cases described earlier. Instead, it is determined by the exhaustion of the possibility of interaction of components due to the corresponding self-organization of the reaction medium during the deposition of a substance on the surface of the solid. Thus, this type of restriction on the growth of particle sizes of the formed phases

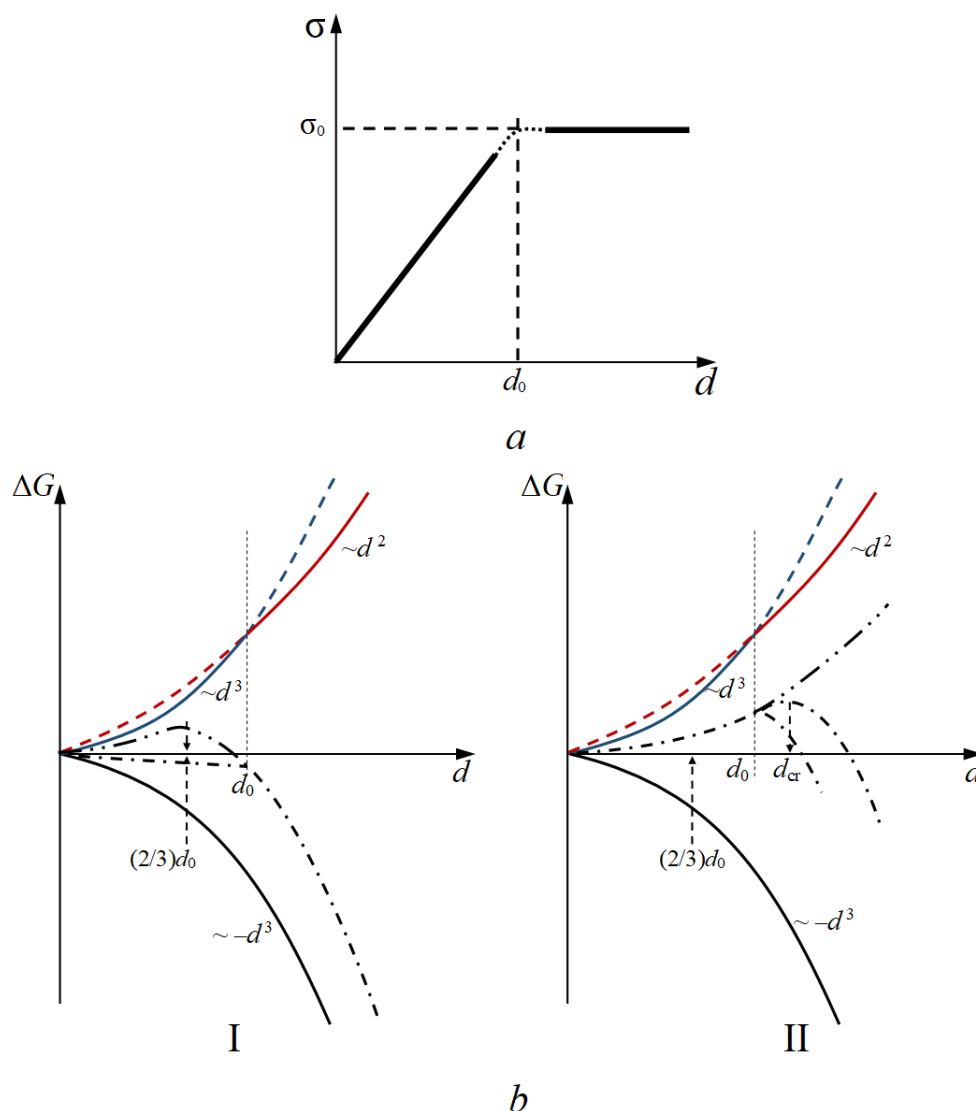
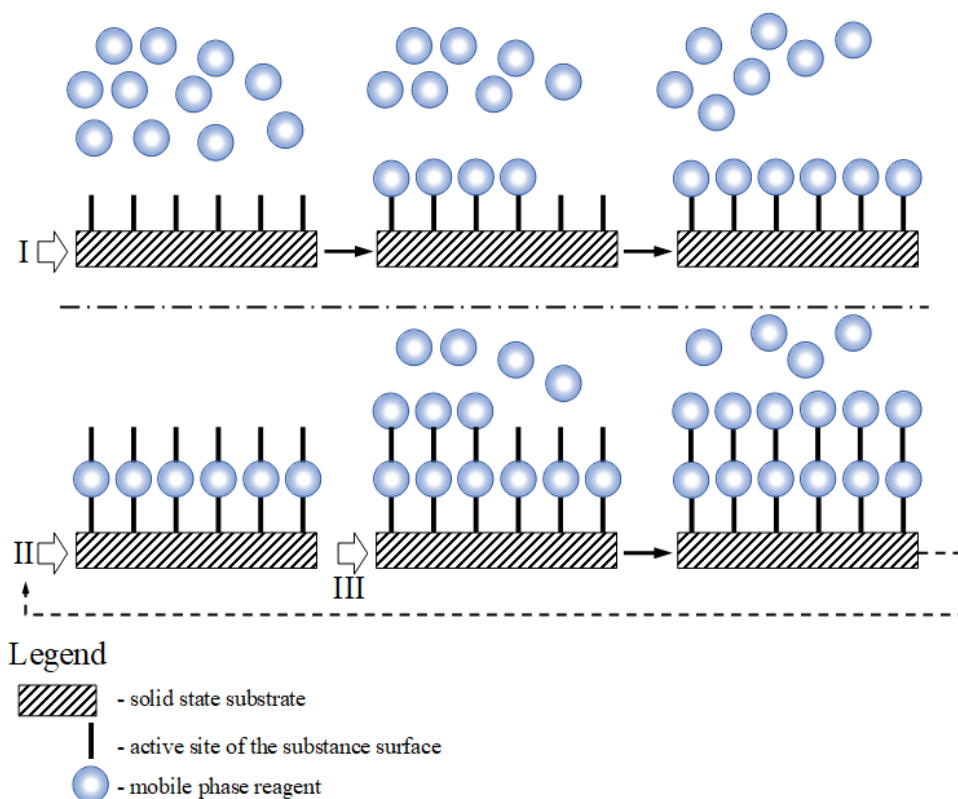


FIG. 2. Dependence of the surface energy (σ) on the particle size (d) (a); critical particle size (d_{cr}) of the formed phases (b)

introduces the next group of self-organizing constraints in the synthesis process. This class of self-organizing constraints will be based on a decrease in the possibilities for mass transfer of reagents into the reaction zone during the synthesis of nanostructures due to self-organization during the phase formation of spatially limited reaction zones (Fig. 1).

A promising direction in the organization of the synthesis of nanoparticles may be the use of intensive mixing of flows of liquid-phase reagents, in which vortices of the Kolmogorov scale are formed [212–214]. In this case, the synthesis takes place inside these vortices, and the mass transfer between the vortices is much less intense. This means that in this case, the vortices that self-organize during the mixing of reagents, in which phase formation occurs, can be considered as self-organizing nanoreactors (Fig. 1). In the works [176, 215–219] it was shown that such a method of mixing of reagents that exists, for instance, in microreactors with colliding jets [220–227], allows to synthesize nanoparticles with the smallest possible sizes under these conditions of chemical reactions. One disadvantage of the method based on the self-organization of spatial restrictions when mixing reagent solutions is, as a rule, the narrow temperature range at which solutions can be mixed. In some cases, these temperature values are not sufficient to form the final product. For example, when mixing aqueous solutions of reagents, intermediate hydroxides or amorphous forms of the substance are often created [20, 43, 142, 189, 228–232], and not the final product in the form of nanocrystalline oxide nanoparticles. That means that some nanoscale precursors are formed. In such cases, additional heat treatment is required, in which the particle size of the final product can significantly increase – as was observed during the formation of crystals of the phase with the structure of perovskite in the system $\text{Bi}_2\text{O}_3\text{--Fe}_2\text{O}_3$ [233–236].

Due to the need to achieve higher temperatures for the formation of oxide nanocrystals than is possible in the case of mixing aqueous solutions of components at atmospheric pressure, it is potentially possible to mix reagents dissolved in liquids that have higher boiling points. Another way to achieve higher temperatures is to increase the pressure in the






- Legend
-  - solid state substrate
 -  - active site of the substance surface
 -  - mobile phase reagent
- I – The cycle of mobile phase reagents' interaction with active centers on the surface of a solid-phase base;
 II – Cycle of active centers' formation on the precipitated reagent;
 III – Repeat of I-st cycle.

FIG. 3. Formation scheme of films with adjustable thickness by LbL methods

reactor to move into the field of hydrothermal synthesis. Naturally, the hydrodynamic conditions for the formation of vortices of the Kolmogorov scale change. Their boundaries acted as self-organizing spatial restrictions for mass transfer of reagents in the process of phase formation. At the same time, the transition to other reaction media in terms of chemical composition and other conditions of synthesis in terms of temperature and pressure, although it complicates in many cases the hardware design of the process, may expand the possibilities for self-organization of spatial restrictions in the reaction system.

It should be noted that when mixing reagents in the form of liquid solutions, in addition to vortex formation, other alternatives for self-organization of spatial restrictions for mass transfer of components to the particles of the resulting phase are possible. In particular, such a limitation may be the solid-phase product itself, which is formed at the contact boundary of reagent solutions (Fig. 1). The effectiveness of such a barrier for mass transfer of reagents in some cases leads to a situation where even with the usual form of introduction of one of the reagents – drops of a solution – nanocrystalline products can be obtained if two conditions are met. First, rapid nucleation is ensured at the interface of the reagents. Second, the particles formed during the new phase are quickly removed from the reaction zone, thereby ensuring the possibility of further nucleation process on the contact boundary of liquid-phase reagents [230, 234, 235, 237–240]. In the case described above, the layer of solid-phase reaction products itself acts as a self-organizing spatial restriction for the mass transfer of components from the liquid phase to the reaction zone. For the synthesis of nanoparticles with the smallest possible sizes under conditions of these spatial constraints being formed, it is important to organize a large contact surface of reagents and the rapid removal of interaction products from the reaction zone. It was shown in [241] that one of such possibilities is to carry out synthesis in apparatuses with swirling flows of reagent solutions.

A self-organizing barrier for mass transfer and, consequently, for particle growth can be surface layers that differ in their composition and structure from the volume of particles during the formation of “core-shell” structures (Fig. 1). Similar self-organizing structures during the formation of nanoparticles based on phases of variable composition were described in [242–245]. It should be noted that in these works, a noticeable decrease in the size of particles was experimentally shown precisely in the case of their formation as a “core-shell”. This is due to the difficulty of transferring matter

from one particle to another through a self-organized barrier that emerges during their formation as a surface layer of a different composition and structure – or a shell.

Another variant of self-organization of particle growth restriction is associated with the formation of foam during a chemical reaction. In cases where the layer of foam bubble walls in which phase formation occurs is thin, it can be considered as a quasi-two-dimensional microreactor or, at least, a nanoreactor. An example of such phase formation can be the synthesis of oxide nanoparticles obtained by the method of “solution combustion” [23, 25, 28, 29, 246–254] under a certain regime of this process [255, 256]. Such a “solution combustion” mode can be considered as an independent method of obtaining nanoparticles or as the method of “foam combustion” schematically depicted in Fig. 4. The effect of the self-organization of quasi-two-dimensional thin layers as quasi-two-dimensional nanoreactors upon the production of nanoparticles by the “foam combustion” method of the reduction of particle growth rate is associated with the walls (boundaries) of the layers blocking of the transfer of matter to the formed particles (Fig. 4).

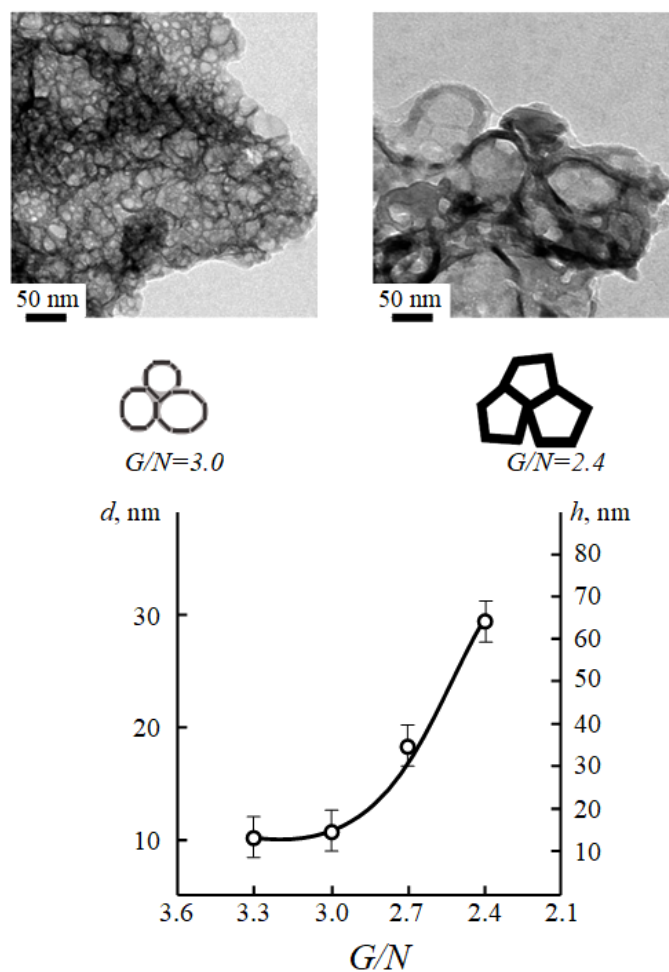


FIG. 4. Scheme of the particles growth restriction during their formation by the method of “foam combustion”. Dependence of the YFeO_3 crystallite size (d) on the thickness of the bubbles’ walls (h) formed during “foam combustion” (fuel-glycine, oxidizer-yttrium and iron (III) nitrates) at different glycine-nitrate ratios (G/N)

It should be emphasized that the effect of foaming on the course of chemical reactions was noted in different phase formation processes [20, 257–260]. Indicative in this regard is the self-oscillating process of foaming liquid during the decomposition of formic acid described in [260]. The self-oscillation of foaming is caused by an increase in the rate and reaction of decomposition of formic acid at the interface of the liquid and gaseous phases. This leads to an increase in the rate of foaming until it reaches exhaustion in the reaction zone of the reagent. Subsequently, the foam disappears, and the process starts from the initial state. Summarizing the considered cases of the influence of foaming on chemical reactions, it can be concluded that by conducting the synthesis process in a thin layer self-organizing during the chemical interaction of components at the interface of bulk phases, one can expect both an increase in the rate of chemical reactions and a decrease in the size of the particles formed due to the restricting of the mass transfer process by the boundaries of this layer (quasi-two-dimensional nanoreactor). As it was shown in [105, 106], the effects described above are also characteristic of some solid-phase reactions.

3. Multistage phase formation processes and combination of crystal growth restrictions

In some cases, one or more high-temperature operations are required to form the final product [43, 230, 234, 235, 261–263]. This is the case for example when the goal of synthesis is to obtain oxide nanoparticles and at the initial stage soft chemistry methods are used to form hydroxide nanoparticles that are stable at temperatures of soft chemical synthesis. Another similar example may be the formation of oxide nanoparticles by the “foam combustion” method in cases where the temperature-time regime of the combustion of reagents leads to the formation of an amorphous phase instead of the target product, like oxide nanocrystals of a given composition and structure, and additional processing of the precursor material is required [8, 20, 255, 264–266]. A common feature of these and other similar situations is that the conditions of synthesis with self-organizing restrictions for particle growth, primarily its temperature-time regime, do not allow the formation of the target product in one stage. Since to obtain small particles, it is necessary to reduce the influence of the mass transfer process as well as the temperature and duration of synthesis (Fig. 1), situations when additional higher temperature and longer stages are required for the formation of the target product are frequent.

An increase in the temperature of synthesis always leads to an increase in the diffusion rate, which means that it increases the growth rate of particles of the reaction product. The effect of increasing the mass transfer rate on particle growth can be reduced either by shortening the duration of heat treatment, or by spatial separation of precursor particles, or by both. Reducing the duration of heat treatment without reducing the effectiveness of obtaining the target product – nanoparticles of a given composition and structure – is possible by means of radiation and laser heating [267–273]. An example of a technology that combines short duration of heat treatment with spatial separation of precursor particles, is spray pyrolysis [274–280]. Other approaches that implement short-term high-intensity energy effects on materials are also being developed and those include processing in electron flows, radiation stimulation of processes in solid phases [281–284], processing in low-temperature plasma [285–287], and more.

The methods listed above can also be used as independent ways for obtaining nanoparticles, but their significant disadvantage is, as a rule, that they cannot be used for synthesizing nanoparticles of compounds of complex composition. Soft chemistry methods are devoid of this disadvantage. Therefore, it is promising to combine methods of soft chemistry, including in conditions of self-organization of particle growth restrictions in the reaction zone, with methods of short-term high-energy processing of precursors obtained at the first stage.

It should be noted that the high-energy effect on precursors is not the only method of transformation into the final product and, in particular, into nanocrystalline oxides from hydroxide nanoparticles. For example, methods for obtaining oxides from the corresponding hydroxides by varying the partial pressure of water in the reaction space appear to be promising in this area. The selection of the pressure (P) and temperature (T) values necessary for the dehydration of hydroxides can be carried out either according to experimental data on the stability diagrams of hydroxide phases [288, 289], or according to thermodynamic calculations [290]. When selecting (P, T)-conditions for the production of oxides within the framework of these methods, it should be taken into account that the temperature should not exceed some critical values to not critically activate mass transfer processes in the reaction system. As it was shown in [6], the melting point of non-autonomous phases (T_{m2n}) can act as such a temperature limitation [291–293]. At $T < T_{m2n}$ mass transfer cannot ensure rapid particle growth and, consequently, there are no firm restrictions on the duration of precursor treatment. In this regard, the value of T_{m2n} is a boundary that divides all phases into high-temperature $T > T_{m2n}$, processes that proceed with a high rate of transfer of matter between grains and low-temperature processes $T < T_{m2n}$ [6, 291, 294–299].

Precursors formed at the first stage of synthesis are not limited to hydroxides and, consequently, the choice of conditions for the formation of nanoparticles of the target product should be based on the analysis of the chemical stability of the corresponding precursor phases. As for hydroxides, such an analysis can be conducted on the basis of appropriate experimental data or by thermodynamic calculations.

The described approaches to the transformation of precursor nanoparticles into nanocrystalline target products can be supplemented by other methods based for example on mechanochemical effects [300–306]. However, since the systematic consideration of such methods is not the purpose of this work, it only provides a schematic representation of the possibility of combining various methods of restricting crystal growth using multistage processes of synthesis of nanocrystalline phases (Fig. 1).

4. Conclusion

Analysis of literature data has shown the prospects of synthesis methods in which particle growth restrictions are formed during the synthesis process itself. The disadvantage of many of such methods consisting in the fact that nanoparticles of precursor phases are formed instead of target products can be compensated by combining several different methods of synthesis. Based on the analysis of literature data, examples of the transformation of precursor nanoparticles into nanocrystals of target phases are classified and presented. A scheme that allows optimal combination of synthesis methods to obtain nano-crystalline particles of a given composition, structure, size, shape, is designed.

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