

INORGANIC SYNTHESIS
AND INDUSTRIAL INORGANIC CHEMISTRY

**Nanotechnology of Molecular Layering in Production
of Inorganic and Hybrid Materials for Various Functional
Purposes (a Review): I. History of the Development
of the Molecular Layering Method**

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Abstract—The history of the development of the molecular layering method is described, the fields and results of using the nanotechnology based on this method in various branches of industry in the past 20 years are analyzed, and the prospects for further development and commercialization of this method are evaluated. The first part of the review briefly describes the principal results of basic and experimental research in the field of the solid-state chemistry, including studies performed using the molecular deposition method in the former Soviet Union and in Russia, where this method was developed in the early 1960s. The main results obtained by researchers from other countries starting from the late 1970s are also discussed.

Keywords: molecular layering, nanotechnology, solids, surface, inorganic and hybrid nanomaterials, functional properties

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INTRODUCTION

The molecular layering method developed more than half-century ago by Soviet scientists S.I. Kol'tsov and V.B. Aleskovskii occupies today a prominent place in the field of nanotechnologies based on chemical methods. Up to the mid-1970s, studies in this field were developed virtually exclusively in the Soviet Union: first at the Leningrad Lensovet Institute of Technology, where possible pathways of chemical transformations of solids, based on the “core” hypothesis formulated by Aleskovskii in 1952 [1, 2], were studied, and then at the Leningrad State University, where Aleskovskii was appointed a rector in 1975 [3] and where a research team from the Institute of Technology came work with him.

Since the late 1970s—early 1980s, researchers from other countries started to publish papers in this field: Suntola et al. (Finland) [4], Damyanov et al. (Bulgaria) [5], and Öhlmann et al. (former GDR) [6]. Later, the number of papers in the field of molecular layering grew in the geometric progression, and since

the 1970s virtually all the leading countries of Europe, Southeastern Asia, and America not only pursue studies but also perform commercialization of the results obtained in the field of molecular layering.

Molecular layer deposition is the term accepted by the international scientific community. In some early papers published beyond the former Soviet Union, e.g., in the first studies by T. Suntola, the process was termed atomic layer epitaxy (ALE) [7], which does not correspond to the mechanism of the chemical transformations of solids on which the molecular layering method is based. Today, beyond Russia, the method is most commonly termed atomic layer deposition (ALD) [8–11].

In a definite step of studies in the field of molecular layering (beginning of the XXI century), the history of the development of the new technology, its sources, and dynamics of the process started to attract researchers' attention. More than 120 reviews on this subject have been published in the past 20 years. Their authors analyzed not only the general approaches and achievements of the molecular layering method

[9–21], but also the prospects for the further progress and use of the method [22–26]. Such problems were considered as chemical aspects of molecular layering [27, 28], phase formation [29], conformity [30] and molecular design of the deposited coatings [31, 32], technology of deposition of oxide [33, 34] and nitride (AlN) coatings [35], and potentialities of the method in formation of thin films on finely dispersed materials [36–42]; the use of the new technology in the production of items for microelectronics [8, 43, 44], photonics [45], and sensorics [46] of materials for alternative power engineering [47–49] and in the development of catalysts [50, 51] and of organo-inorganic hybrid materials [52–55] was discussed. Some historical aspects of the development of the new precision technology were considered in [56, 57]. Because in the Soviet period of the development of the molecular layering method the information exchange of Soviet researchers with foreign colleagues was very limited, it was commonly believed in the world that the molecular layering method was invented by Finnish physicist T. Suntola. For example, in [58] new achievements in microelectronics were attributed to the development of the atomic layer epitaxy by Finnish researchers. Aleskovskii's and Kol'tsov's disciples sent a letter to the Solid State Technology journal with the information on earlier studies on molecular layering [59] to highlight the Russian priority in this field. However, only in the past decade, with the publication of papers concerning the history of the development of the molecular layering, the world's scientific community got reliable information on the sources of the new synthetic technique [57, 60–64]. We should pay tribute to a number of foreign researchers who refined data on the pioneering studies in the field of molecular layering [65] after the previously made incorrect presentation [56].

The first part of the review deals with the history of the development of basic principles and progress of the molecular layering method in the XX century.

BASIC PRINCIPLES OF THE MOLECULAR LAYERING METHOD

Based on analysis of studies by Berthollet, Proust, Mendeleev, Shilov, and other chemists, Aleskovskii in the 1950s suggested his own concept of the structure of solids and their possible transformation pathways. This concept is based on the “core hypothesis” formulated

by Aleskovskii in 1952 in the doctoral dissertation [1]. Its principles are presented in detail in [2, 66, 67]. According to the “core hypothesis” [2, 68, 69], the chemical structure of any solid (crystalline or amorphous, inorganic or organic) consists of a core, i.e., a superpolyatomic radical consisting of structural units **A** linked into a common entity by covalent (ionic, metallic) bonds, and functional groups **B**₁, **B**₂, **B**₃, etc., enveloping it (Fig. 1).

The atoms constituting the core of a solid are its structural units. They include atoms of tri- and tetravalent elements forming very strong bonds with each other (Al, Fe, B, C, Si, etc.), often in combination with atoms of oxygen (Si–O–Si, Si–O–Al) or other elements. Functional groups **B** on the core surface are structural units that may contain both atoms of mono-, bi-, and trivalent elements (H, O, K, Na, Fe) and polyatomic groups (OH, SO₂H, NH₂, etc.). The common feature of functional groups, distinguishing them from the core structural units, is that they are not bonded with each other and are bonded only with the corresponding structural units **A** via **A–B** bonds. In accordance with the chemical model of a solid, any core is either a macroradical (e.g., of carbon in diamond) or a polyion (e.g., of an aluminum oxide core in which the chemical bonds between the structural units are ionic-covalent).

In accordance with Aleskovskii's chemical model, the formula of a solid can be presented as



where *a* is a stoichiometric coefficient characterizing the number of chemical equivalents of the core per chemical equivalent of functional groups.

The dual nature of solids, i.e., the presence of the bulk and surface, reflected in their core–functional chemical structure, predetermines two main types of their chemical transformations in which the chemical potentialities of the functional and structural units are realized:

- (1) transformations involving functional groups on the core surface;
- (2) transformations accompanied by degradation, changes in the composition, and rearrangement of the core, usually leading to changes in the chemical composition, structure, and weight of the macro body as a whole.

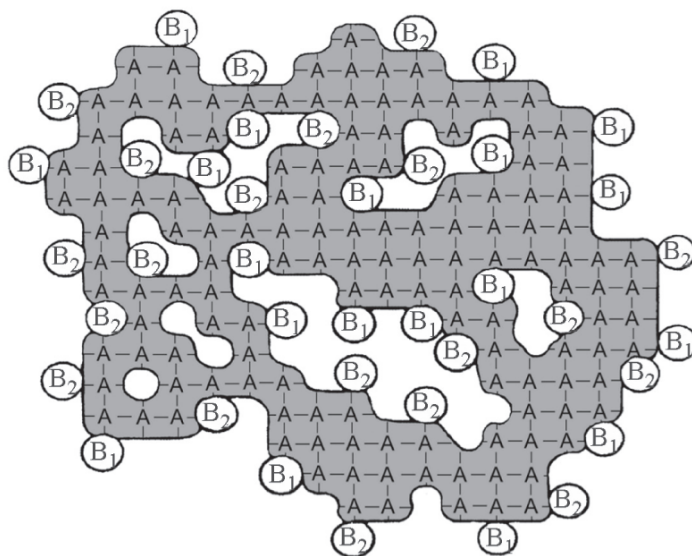


Fig. 1. Aleskovskii's model of a solid.

Transformations of the first type are reactions yielding solids similar to the starting substances in the structure, molecular mass, and composition of the core and differing only in the set of functional groups (functional or analogous transformations). Chemical transformations of solids of the second type are macromolecular transformations involving structural-chemical transformations of the core. It should be noted, however, that such transformations usually involve

not only structural units of the core but also functional groups.

The molecular layering method is based on transformations of supramolecular substances via reactions of functional groups [2, 3, 66, 67, 69–73]. Based on the core-functional model, Aleskovskii and Kol'tsov suggested the classification of various types of surface and core reactions involving supramolecular compounds; these types are presented in detail in

Table 1. Functional and core series of solids

Series of solids	Formula presentation
I. Functional series	
1. Series of analogs	$\dots - A_{n-s}(AB)_s - A_{n-s}(AC)_s - A_{n-s}(AD)_s - \dots$
2. Genetic series:	
(a) substitution	$A_{n-s}(AB)_s - \dots - A_{n-s}(AB)_{s-x}(AC)_x - \dots - A_{n-s}(AC)_s$
(b) addition	$A_{n-s}(AB)_s - \dots - A_{n-s}(AB)_{s-x}(AB \dots D)_x - \dots - A_{n-s}(AB \dots D)_s$
3. Isologous series	$A_{n-s}(AB)_s - \dots - A_{n-s}(AB)_{s-x}(A^*)_x - \dots - A_{n-s}(A^*)_s$
II. Core series	
1. Homologous series:	
(a) of macromolecules	$\dots - A_{n-2s}(AB)_s - A_{n-s}(AB)_s - A_n(AB)_s - A_{n+s}(AB)_s - \dots$
(b) of macroradicals	$\dots - A_{n-2s}(A^*)_s - A_{n-s}(A^*)_s - A_n(A^*)_s - A_{n+s}(A^*)_s - \dots$
2. Isomorphic series	$A_{n-s}(AB)_s - \dots - A_{n-s-x}M_x(AB)_{s-y}(MB)_y - \dots$
3. Polymorphic series	$A_{n-s}(AB)_s - \dots - A_{n-s-x}A'_x(AB)_{s-y}(A'B)_y - \dots - A'_{n-s}(A'B)_s$
4. Inclusion series	$A_{n-s}(AB)_s - \dots - A_{n-s}[N]_x(AB)_s - \dots$

(A) Structural units of the initial core; (A') structural units having the same composition but forming a core of different structure; (M) structural units of a new chemical composition; (B, C, D) functionals; (A*) surface radical; and ([N]) atom or molecule included in the core.

Kol'tsov's doctoral dissertation [74] and in Aleskovskii's books [2, 73].

The suggested classification allowed the products of transformations of solids to be grouped into functional and core series (Table 1) differing in the molecular mass of the core (homologous series of solids) and, in the case when the core remains unchanged, in the set of functional groups on the surface (pseudomorphoses, genetic series of solids).

In accordance with such approach, the diversity of chemical and structural changes in a solid relative to its ideal composition and chemical structure is due to polyatomic composition, core–functional structure, and diversity of transformations of a solid. The transformations can be described by stoichiometric reactions (Fig. 2). It should be emphasized that these transformations form a common system presented in the form of a cone-shaped figure (for a normal homologous series) (Fig. 3). To each member of a homologous series, there corresponds a particle of radius K_i , where K_i is the number of structural units contained in the radius of the given particle. Two adjacent homologs differ by the quantity $\Delta K = K_{i+1} - K_i = 1$, i.e., by the thickness of a monolayer of structural units. It follows from the homologous theory of structural-chemical transformations of solids, namely, from the organic relationship between the functional and core series, that functional reactions performed in a definite sequence can be used for forming on the surface of a solid a monomolecular layer of structural units linked to the core via chemical bonds.

Kol'tsov was the first who started to study reactions with functional groups of a solid. He reported the first data on the reaction of CCl_4 with porous silica in 1963 [77] and then on the reactions with silicon and titanium halides [78, 79], etc.

Studies developing and extending the views on synthetic features and potentialities of the molecular layering were performed in the 1960s under the guidance of Aleskovskii and Kol'tsov; layers of phosphorus, silicon, titanium, and germanium oxides and two-component titanium–phosphorus oxide structures were obtained [78–83]. It was demonstrated using the developed procedure of continuous monitoring of the molecular layering process with a precision nondestructive ellipsometric method (allowing determination of the thickness of the synthesized layer

with an accuracy of one monolayer) that the thickness of the oxide layer linearly increases with an increase in the number of molecular layering cycles [84, 85].

The experimental data obtained in the 1960–1970s show that molecular layering allows both synthesis of nanostructures of various chemical compositions on the surface of a solid matrix (monolayers, including multicomponent monolayers, Fig. 4c) and atom-by-atom chemical assembling of surface nano-, micro-, and macrostructures by repeated alternation of chemical reactions in accordance with the preset program (Figs. 4a, 4b). It should be emphasized that the main requirement in performing reproducible synthesis by molecular layering is carrying out various steps of reactions of appropriate agents (AC_4 , AB_4 , NB_4 , NC_4 , MC_4 , Fig. 4) with functional groups of a solid (B, C, Fig. 4) under the conditions maximum far from the equilibrium.

Thus, virtually all the synthetic potentialities of the molecular layering method were experimentally demonstrated in the period from 1963 to 1976. The following materials were used as supports in the majority of studies (published in this period in *Zhurnal Prikladnoi Khimii*/Journal of Applied Chemistry of the USSR, *Zhurnal Obshchei Khimii*/Journal of General Chemistry of the USSR, *Zhurnal Fizicheskoi Khimii*/Journal of Physical Chemistry of the USSR, *Izvestiya Akademii Nauk SSSR, Ser. Khimicheskaya*/Russian Chemical Bulletin, *Izvestiya Akademii Nauk SSSR, Ser. Neorganicheskie Materialy*/Inorganic Materials, and *Izvestiya Vuzov, Khimiya i Khimicheskaya Tekhnologiya*/ChemChemTech): samples of porous silica gels with the developed surface [78–82, 86–102], Aerosils [103], quartz fibers [104], glass spheres [105]; carbon materials: carbon black [106, 107], diamonds [108, 109], and carbon fibers [106, 110]; polymers [111, 112]; plates of semiconductor grade silicon [84, 85, 113], germanium [113, 114], silicon carbide [113, 115], and quartz [116]. The starting reagents in most cases were highly volatile liquid chlorides and oxychlorides of titanium [79, 80, 82, 84, 93, 104, 105, 110, 113, 116], vanadium [97, 98, 103], phosphorus [89, 90, 92, 111, 112], chromium [96, 115], boron (boron bromide) [99, 100], silicon (various silanes) [78, 85–88, 114], aluminum [95, 101], zinc [102], germanium [80, 81, 91], tin [94], and carbon [106–109]. Depending on the chemical composition of the layers being synthesized,

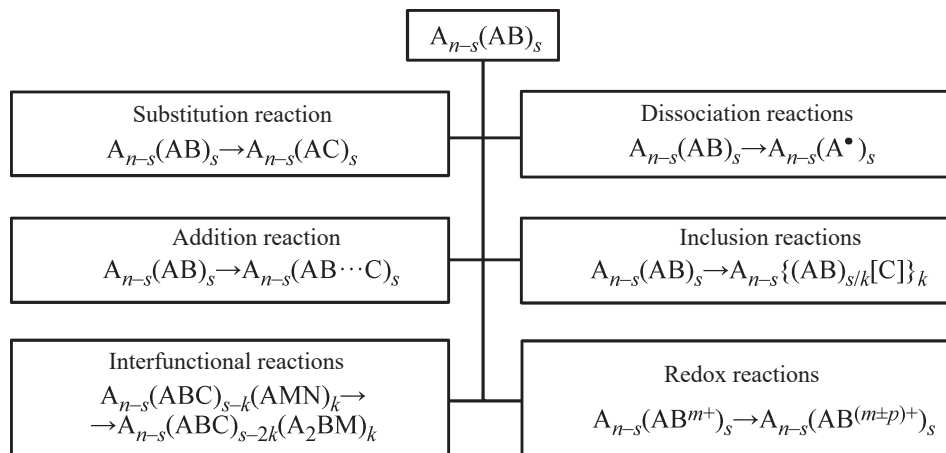


Fig. 2. Classification of functional transformations of solids [75].¹

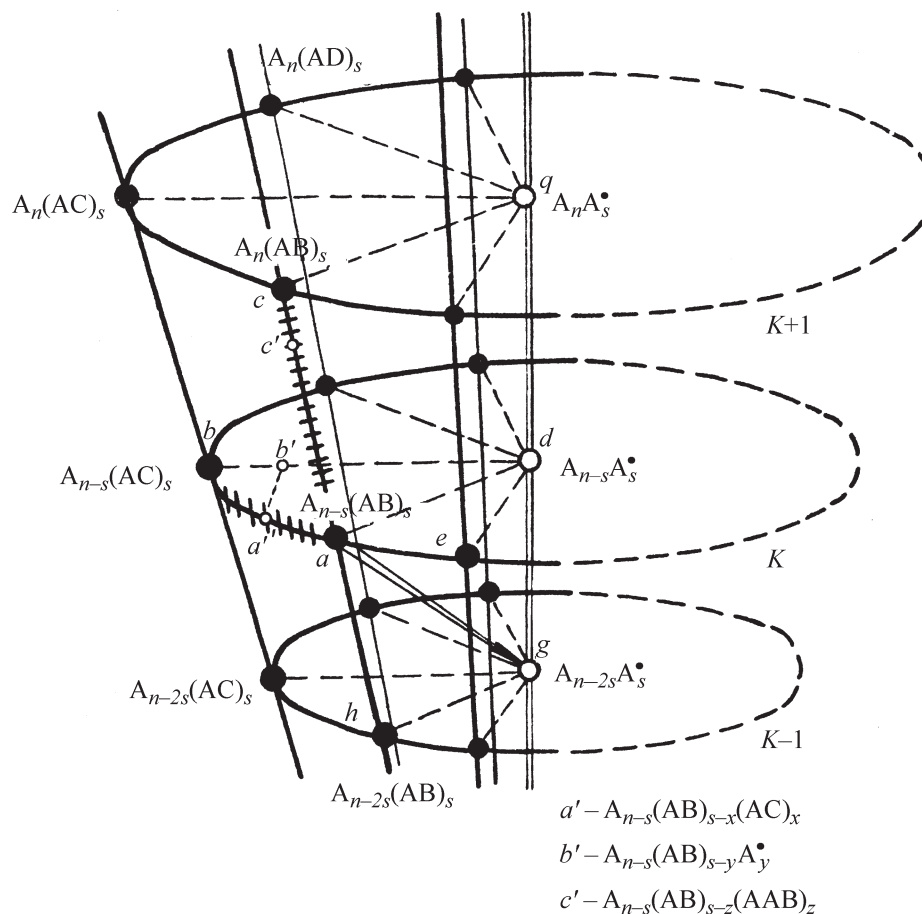


Fig. 3. Systematization of products of analogous transformations of solids [76].²

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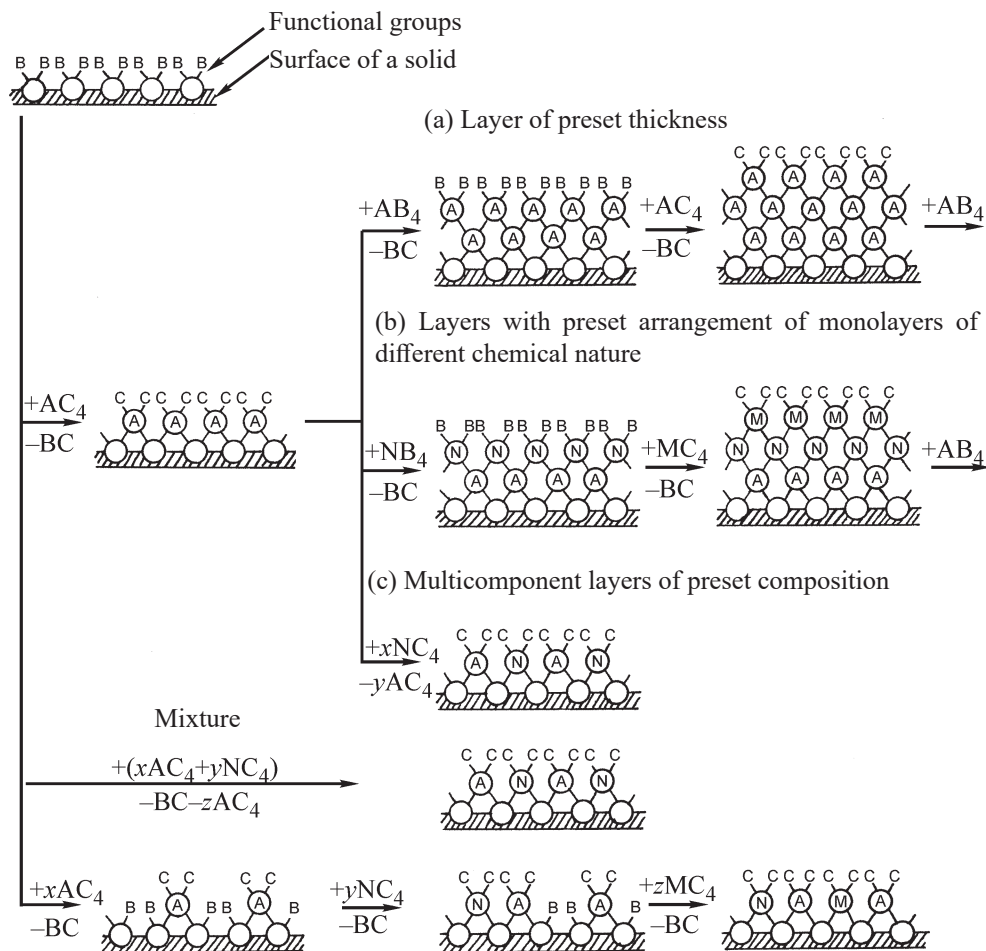


Fig. 4. Schemes of chemical assembling of nanostructures on the surface of a solid by molecular layering.

the second agent was either water vapor (to obtain oxide layers) or gaseous ammonia (to obtain nitride layers); in preparation of carbon layers, the support was alternately treated with carbon tetrachloride vapor and gaseous methane [73].

Along with electrophilic substitution of proton in functional groups, Malygin [117] performed molecular layering using redox reactions on the surface (Fig. 2). By this procedure, he prepared multicomponent oxide mono- and polylayers containing P–Ti, P–V, Ti–Cr, Cr–V, and Cr–P oxide and other structures [118–121].

In the period under consideration, all the experimental studies using dispersed and porous matrices were performed on batch flow-through laboratory installations under atmospheric conditions. The procedure was based on the scheme suggested by Kol'tsov in the

early 1960s [2]. By the beginning of the 1970s, batch vacuum installations for the synthesis on mono- and polycrystalline matrices were developed and fabricated, and in 1977 the first automated device for molecular layering at reduced pressure was constructed [56, 122].

Studies of the catalytic, sorption, electrophysical, and protective properties of products of various compositions and structures, prepared by molecular layering, revealed a number of important features (structural-size effects) related to the number of the molecular layering cycles [3, 25, 73]:

- monolayer effect, i.e., a sharp change in the properties of the material after 1–4 cycles of molecular layering;
- support coverage effect, which characterizes complete physical coverage of the matrix surface after

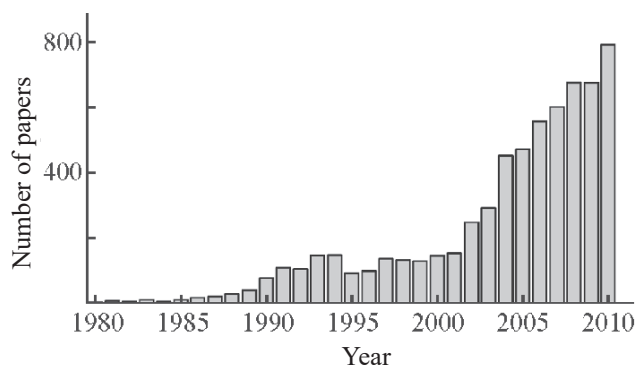


Fig. 5. Number of annually published scientific papers on molecular layering in the period from 1980 to 2010 [29].³

performing no less than 4–6 cycles of molecular layering;

- effect of multicomponent system;
- effect of mutual matching of the structure of the support and deposited layer.

Thus, in the 1960–1970s Aleskovskii, Kol'tsov, and their disciples developed in the former Soviet Union a powerful scientific and experimental base, which later served as a basis for the progress of applied studies and commercialization of the molecular layering processes.

DEVELOPMENT OF THE MOLECULAR LAYERING METHOD IN OTHER COUNTRIES

Initially studies on synthesis of solids by molecular layering were performed exclusively in the former Soviet Union at the Leningrad Lensovet Institute of Technology and Leningrad State University. Since the late 1970s, such studies were also initiated in other countries. Numerous papers were published not only in the former Soviet Union [123, 124], but also in Bulgaria [5, 125], former GDR [6, 123, 126, 127], and Finland [4]. Since the early 1980s [128], the number of papers in this field grew in the geometric progression (Fig. 5).

Specifically in that period, the range of solid matrices used in molecular layering processes was considerably expanded. Also, organometallic precursors came into use [129] along with halides, which allowed expansion of the range of elements whose compounds can be synthesized

on the surface. On the other hand, the first applied papers were published [130, 131], process equipment for implementing the molecular layering was developed [132, 133], and the first materials prepared by the new technology were developed and brought into industrial use [134].

In 1983–1984, Suntola et al. patented a procedure termed atomic layer deposition for preparing zinc sulfide layers and apparatus for its implementation [4]. In this patent, in contrast to previous patent [135] where the ZnS layer was prepared by molecular-beam epitaxy, zinc sulfide layer on a glass support was prepared by repeated alternate treatment of the support with ZnCl₂ vapor and gaseous H₂S with the removal of excess reactants and gaseous by-products after each step; i.e., the principles of molecular layering were used.

In 1983, Aidla et al. [136] described the principles of atomic layer deposition; actually, they literally repeated the principles of molecular layering presented in Kol'tsov's doctoral dissertation [74] and in more than 20 PhD dissertations and other papers long before 1983, e.g., [2, 73]. By the end of the XX century, Aleskovskii's and Kol'tsov's disciples defended about 100 PhD and about 10 doctoral dissertations dealing with molecular layering.

In addition to the previously used matrices, such materials were used as synthetic diamonds, tantalum, gallium arsenide, anodic aluminum and titanium oxides, synthetic opals, dispersed micas, photoluminophores, phenol–formaldehyde and epoxy–phenol materials, highly dispersed silicon and zinc oxides, and graphitic carbon materials [57].

The results of the previously performed basic and applied research formed the basis for setting up in the mid-1980s the production of new useful materials. The first products obtained by molecular layering were brought into industrial use in the former Soviet Union: commercial silica gels modified by the new process with phosphorus oxide layers (FS-1-3 sorbent for sorption of vapors of water, amines, and organic substances [137–139]) and with a vanadium oxide monolayer (IVS-1 indicator sorbent for water vapor [140–142]). The new sorbents are still in use; they are supplied to enterprises and used for stabilization of the gas medium in sealed items for aviation instrument making and in other fields [13, 143, 144].

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Introduction into industry of new materials prepared by molecular layering stimulated the development not only of laboratory setups but also of prototype industrial installations for implementation of this process [145]. The major attention in the development of process equipment was paid to vacuum installations used for preparing thin-film structures in electronics and adjacent fields [146].

However, in processes using friable materials when the process rate is strongly influenced by the hydrodynamic conditions in the gas–solid system, the molecular layering should be implemented in flow-through installations at atmospheric pressure. For example, the synthesis can be performed under conditions of fixed or fluidized bed.

Among studies aimed at the development of the process equipment in the early period of the development of the molecular layering method, we can distinguish studies by Drozd on the development of vacuum installations [57]. In the 1980s, Tolstoi et al. developed new flow-through installations for treatment of large items by molecular layering [147]. In the same period, Dergachev et al. developed, fabricated, and tested an automated multisection apparatus for treatment of friable materials in the fluidized bed mode [148]. In contrast to traditional schemes of processes in semicontinuous apparatuses, when all the process steps were performed in the same reaction space, the suggested multisection apparatus provides for separation of gas streams in separate synthesis steps in the own reaction unit (Fig. 6). This allows the process to be performed in the semicontinuous mode, considerably reduces the synthesis time, and eliminates the need for removing reactants and reaction products from the reaction space.

Studies performed in the last quarter of the XX century demonstrated not only broad synthetic potential of the molecular layering but also the feasibility of developing innovation materials for various purposes using this technique. Technological principles of the molecular layering method and apparatus for its implementation were developed, and the first materials prepared by the novel technique were brought into industrial use. In that period, the geography of molecular layering (ALD) was considerably expanded; studies in this field were initiated and performed on virtually all the continents in such countries as Japan, China, Republic of Korea, EU countries, the United States, Canada, Brazil, etc. [29, 33, 56, 57, 128].

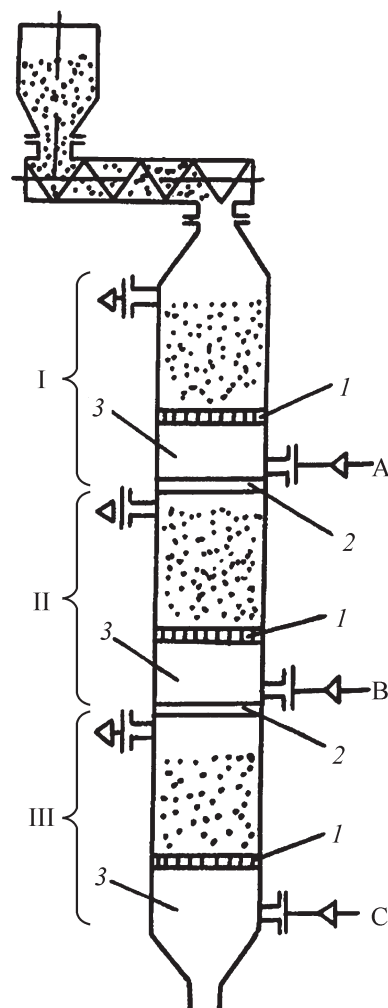


Fig. 6. Scheme of a multisection chemical reactor continuous with respect to the gas phase and semicontinuous with respect to the solid phase. (1) gas-distribution grid, (2) fall-down isolating unit, and (3) gas-distribution section.

Thus, the results obtained by the beginning of the XXI century in the field of synthesis of solid materials using nanotechnology based on molecular layering principles not only are of basic importance, but also demonstrate wide prospects for using the new precision process in various branches of industry: microelectronics and adjacent fields, sorption-catalytic materials, composites, polymers, etc.

METHODS FOR IDENTIFICATION OF MOLECULAR LAYERING PRODUCTS

Despite the facts that the molecular layering is based on principles ensuring formation of mono- and polylayers of preset composition and structure on the

surface of solids in the course of the synthesis and that the thickness of the nanocoating formed is determined not by the reactant feeding time under equal other conditions but by the product of the growth constant (which can be conventionally defined as the coating thickness after one cycle of molecular layering) and the number of molecular layering cycles, it is necessary to identify the structure and composition of the target product using physicochemical methods.

Since publication of the first papers on directional synthesis of solids by molecular layering in the 1960s, the experimental confirmation of the process occurrence and evaluation of the composition of functional groups and thin coatings formed on the matrix surface was based on chemical [82, 83] or gravimetric [149–151] analysis. The development of the theory for spectral data processing and enormous progress of the data processing rate as high-performance computers became available allowed using ellipsometry for determining the coating thickness and optical characteristics [84, 85, 152–154]. The phase composition and grain structure (size of coherent scattering domains) of a coating could be evaluated only after performing several molecular layering cycles and obtaining a layer of the new phase with the thickness exceeding its unit cell dimensions [80], which well agrees with the support coverage effect. The distribution of structures formed on the matrix surface was assumed to be homogeneous, and the presence of the amorphous constituent in the coating was estimated from changes in the coherent scattering domain size in the course of heat treatment of the modified materials [101, 113] or by performing quantitative X-ray diffraction measurements using an internal reference [155].

After the probe microscopy became available in research laboratories, atomic force microscopy (AFM) finds increasing use for analyzing the continuity and structure of a nanosized layer synthesized by molecular layering [156–167]. AFM allows identification of processes of the surface chemical modification and evaluation of the morphology, thickness, and local physicochemical properties of the conformal nanometer-thick coating formed in the course of molecular layering. AFM is applicable both to flat objects with undeveloped surface and to highly dispersed and highly porous materials except highly porous materials with irregular pore structure [168].

Diffuse reflectance spectroscopy in the UV and visible ranges is used for evaluating the sorption and catalytic properties of the synthesis products (and, correspondingly, for predicting the functional properties of chemical assembling products) since the 1970s [116, 169, 170]. The procedure for deconvolution of the diffuse reflectance spectra into components, developed at the St. Petersburg State Institute of Technology [171], allows not only qualitative and quantitative characterization of the coordination state of the surface atoms [172], but also detection of structural changes on the surface of solid materials [173, 174].

CONCLUSIONS

In the first part of the review, we considered historical aspects of the creation and development of the molecular layering technology based on Aleskovskii's "core" hypothesis in the XX century. Analysis of the papers published by authors from the former Soviet Union and other countries confirms the priority of the Soviet and then Russian research school headed by Aleskovskii and Kol'tsov, who developed and experimentally substantiated the principles of precision chemical synthesis. In the 1960–1970s, published papers on molecular layering belonged exclusively to Soviet researchers, and the first papers in this field by authors from other countries were published only in the late 1970s. However, by the beginning of the XXI century studies aimed at the development of new materials using molecular layering were performed in all the industrially developed countries.

The development of technologies and methods of physicochemical investigation in the second half of the XX century allowed control of the synthesis course and characterization of the composition and physicochemical properties of the applied coatings on the level down to single monolayers.

Thus, nanotechnologies based on molecular layering principles became an actively developing field of research by the end of the XX century, and novel products brought into use in various branches of industry have been obtained using this technique. The most promising direction of commercialization of the molecular layering technology is the development of products for micro-/nanoelectronics, alternative power engineering, sorption, and catalysis.

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CONFLICT OF INTEREST

A.A. Malygin is the Deputy Editor-in-Chief of Zhurnal Prikladnoi Khimii/Russian Journal of Applied Chemistry. The other authors declare that they have no conflict of interest.

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