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Research into Solid State Chemistry at the ISSC&M, SB RAS

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Abstract

The main directions of research in the field of solid state chemistry carried out at the Institute of Solid State Chemistry and Mechanochemistry, SB RAS (former Institute of Physicochemical Foundations of Mineral Raw Processing, Institute of Solid State Chemistry and Mineral Raw Processing, SB AS USSR) since 1974 till present are described.

Research into solid state chemistry started at the Institute of the Physicochemical Foundations of Mineral Raw Processing when the team of the Solid-Phase Reaction Kinetics Laboratory passed on from the Institute of Chemical Kinetics and Combustion. In the field of topochemical reactions, main attention had been paid to investigations of the nature of and reasons for autolocalization of thermal decomposition processes, or, more precisely, the nature of processes occurring at the interface between the product and the starting compounds, that is, in the reaction zone, the consequence being process heterogeneity itself [1–4].

The nature of the feedback was considered both at the level of computer simulation [5, 6] and at the level of detailed experimental investigations, starting from polymorphous transitions and finishing with decomposition processes [7–9]. The role of strain arising as a result of misfit between the molar volumes and structure in the product and starting compound has been shown [10, 11]. During recent years, molecular crystals became the subject of investigation; their behavior under the action of hydrostatic pressure and phase transitions initiated by the application of pressure [12, 13] and shear pressure [14, 15] has been studied.

Crystalline hydrates [16], complex compounds, and salts [17, 18] have been investigated. For crystalline hydrates, the nature of the anomalous dependence of the reaction rate on the partial pressure of water vapor (Toply – Smith effect) has been studied. Dehydration of the Seignette salt in ferroelectric and paraelectric phases [19], the composition of dehydration products in the reaction nuclei of different shapes, as well as topography and mechanism of incongruent melting of crystalline hydrates [20] were also investigated. An important result was the discovery of internal strain in alum by means of EPR [21]. A set-up was successfully designed, and the true value of dehydration rate at the interface has been determined [22].

The logical outcome of these works were investigations showing that the lattice is deformed on both sides of the reaction zone, and that the strain and its relaxation induced by the formation of a new surface are reasons for the positive feedback leading to heterogeneous dehydration [23]. An important result obtained recently was improvement of this model to quantitative coincidence between the experimental results and theoretical calculation [11, 24, 25].

Another direction was investigation of the kinetics and mechanism of thermal and photochemical transformations of complex compounds. Heterogeneous processes have been investigated [26–28]; the propagation of the reaction front was compared with the crystal chemical features for these processes; homogeneous reactions of the type of nitro-nitrito bond isomerization in cobalt nitropentaammoniates with different anions were also studied. To investigate the latter, modern research procedures were applied, which allowed us to compare the behavior of these compounds at different temperatures and pressures, to establish the effect of outer-spheric anions on the character of transformation, and to reveal the relationship between bond isomerization processes initiated by heating and by the action of light [29, 30].

Investigations of transfer processes in solids mainly dealt with the mechanism of anomalously high diffusion of ions in superionic crystals [31–33]. These works were initiated by the detailed investigation of the mechanism of ion diffusion in crystals and relation between the diffusion and enthalpy characteristics of the lattice. These investigations allowed the formation of a specific research area, which goes on developing at the Institute [34].

In the area of combustion research, investigation of the effect of oxide additives on DINA combustion was continued; it was shown that acceleration is associated with the evolution of additional heat due to the catalytic reaction of combustion of the gaseous products of DINA thermolysis. Subsequent investigations dealt with technological combustion, at first in binary metal–non-metal systems, and then in oxide mixtures proposed by our Institute, one oxide being reducer and the other oxidizer [35–41]. An important result obtained in this field at that time was the use of diffractometry with synchrotron radiation to investigate the combustion zone in self-propagating high temperature synthesis processes [37]. In recent years, combustion research was aimed at developing methods for the preparation of metal, oxide, and sulfide products of required morphology by combustion of complex compounds of nickel, zinc, copper, and cadmium with the temperature of the combustion zone controlled by varying the pressure of the inert gas over the sample. Another direction of recent research was investigation of conditions for a transfer of mechanochemical synthesis to self-propagating high-temperature synthesis [42, 43].

Among works on thermal decomposition of salts, two main directions of research carried out in the 1980s at our Institute can be cited.

The first direction is investigation of the mechanism of photochemical and thermal decomposition of aluminum hydride at the level of elementary stages; the results of this research allowed us to propose a new thermally developed photographic material [44–46]. A specific feature of this work is that the authors succeeded in investigating the elementary stages using a complex of physical and chemical methods they had developed themselves, and the model was brought to quantitative results, which is an extremely rare occurrence in studies of topochemical decomposition reactions.

The second direction is detailed investigation of the mechanism of thermal decomposition of copper hypophosphite [47–50], which resulted in methods governing the reaction rate and the formation of the solid reaction product – copper in a highly active catalytic form. The data obtained served as a basis for developing a new low-operation technology of metal coating of dielectrics used as a metal coating process for orifices in printed circuits in printing.

In the middle 1980s, the first investigations into intercalation chemistry were launched at the Institute. The primary task was to study selective sorption of lithium ions in the interlayer space of the hydrargillite lattice from natural brine [51, 52]. It was discovered that intercalation of lithium and chloride ions allows one to introduce a number of other inorganic and organic ions and molecules into the interlayer space and thus to use this space as a microreactor to carry out various inorganic and organic syntheses [53]. Among them, the method for the preparation of metal nanoparticles should be mentioned [54].

Research works on mechanochemistry at our Institute have already been considered in reviews dealing with the development of this research direction in the USSR [55, 56] and in Russia. This research area made special advances at the Institute of Solid State Chemistry and Mechanochemistry, since it closely approached the subjects of investigation at the Institute in the middle 1970s, when the Solid-Phase Reaction Kinetics Laboratory passed from the Institute of Chemical Kinetics and Combustion to the Institute of Physicochemical Foundations of Mineral Raw Processing (IPFMRP). This allowed us to pool the efforts of several laboratories based on this principal subject: Binding Materials Laboratory (application of mechanical activation to entrainment of ash from electric power stations for use as a construction material [57]), Rare Alkaline Metals Laboratory (use of mechanical activation to improve the processes of treatment of lithium-containing aluminosilicate raw materials lepidolite and spodumene [58]), Salts Laboratory (development of an acid-free method for converting phosphorite ores into mineral fertilizers [59]), Electrochemistry Laboratory (application of mechanochemistry to activation of galvanic couples for hydrometallurgical processing of sulfide ores [60]), and Mechanochemistry Group of the Solid State Chemistry Laboratory, which was engaged in general problems of mechanochemistry before having passed to the IPFMRP [61, 62].

Since this subject is described in more detail in another publication of *Chemistry for Sustainable Development* [63], we will restrict our consideration to the most important results.

In the field of theory of mechanochemical processes, the kinetic model proposed by V. V. Boldyrev in 1972 was elaborated further; the model took into account the pulsed and local character of mechanochemical action on a particle [64, 65]. Equations describing mechanochemical processes involving particles of different kinds have been proposed [62], and an approach to separation of the grinding and activation processes in a reactor has been suggested [66]. The role of the intermediate fluid phase in mechanochemical solid-phase reactions has been assessed [67]. The formation of linear and point defects under mechanical action in a crystal was traced by means of computer simulation [68]. The possibility of mechanochemical synthesis of icosahedral structures [69], anomalous solid solutions and intermetallides [70], and solid-phase synthesis of complex salts and ceramic materials [71–73] has been demonstrated. "Soft" mechanochemical synthesis has been developed [74]. The possibility of hydrothermal processes under conditions of a mechanochemical reactor has been shown [75].

It was shown that phosphorus contained in phosphate fertilizers can be transformed into the form assimilable by plants by means of mechanical activation; thus a new process leading to phosphate fertilizers can be developed [59]. Mechanochemical methods have been developed for obtaining materials for hydrogen accumulators [76], stomatology [77], heavy-current ion conductors [78], and nanoparticles [79].

One can also mention mechanochemistry applications aimed at simplification and cost reduction of organic synthesis, including synthesis of pharmaceuticals [80] and their modification for solubilization [81]. The outcome of these works was the development of Askopirin, a readily soluble form of aspirin.

Works in the field of radiation chemistry were a continuation of research carried out by the group headed by V. V. Boldyrev in Tomsk, but involving the new possibilities which appeared with the creation of high-power electron accelerators at the Institute of Nuclear Physics in Novosibirsk. As a result, the radiation thermal effect was discovered; at high power of radiation dose, some of the radiation-induced defects cannot be annealed off and take part in thermally initiated solid-phase chemical processes [82–84].

The achievements in the area of experimental procedures and techniques include the following:

1. Application of synchrotron radiation to investigations of heterogeneous processes. These investigations were started at the Electrochemistry Laboratory and sought to investigate cathode hydrogenation of nickel [85]; then the procedure was developed, both in Debye and Laue versions, to investigate the structure of the reaction zone in thermal decomposition and synthesis reactions [86].

2. Design of an installation for investigating the dehydration rate of crystalline hydrates [87].

3. Development of methods for investigating absorption spectra in the optical and UV regions from microvolumes of crystals [14].

4. Development of mechanical activators of different designs with several grams to half a ton charges [88–90].

5. Development of a method for estimating thermal balance during mechanical activation [91].

6. Development of a centrifugal classifier to separate finely disperse materials [92].

The results of these investigations are summarized in reviews [93–101].

An important sign of recognition for our works was participation of our researchers in international events on solid state chemistry and in international meetings in this field held in Novosibirsk.

For example, our researchers participated in International Symposia on the Reactivity of Solids in Dijon (1979), Kraków (1980), Hamburg (1996), and Kyoto (2003). Researchers from our Institute also took an active part in international conferences on mechanochemistry and mechanical activation (INCOME) and in Soviet-Japanese Symposium on Mechanochemistry organized in Novosibirsk and in Japan.

V. V. Boldyrev was President of the International Advisory Committee on the Reactivity of Solids in 1992–1996, and in 1988–1997 he was President of the International Mechanochemical Association at IUPAC.

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