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# MECHANOCHEMICAL PROCESS INVESTIGATION USING OF SYNCHROTRON RADIATION: MODEL AND REAL EXPERIMENTS

B.P. Tolochko[1], M.R. Sharafutdinov[1], N.Z. Lyakhov[1], K.A. Ten[2], E.R. Pruuel[2]
*[1]Institute of Solid State Chemistry and Mechanochemistry SB RAS, Novosibirsk, Russia*
*[2]Institute of Hydrodynamics SB RAS, Novosibirsk, Russia*

The enormous collected experimental data on mechanochemical processes contains no information on the initial phase of reaction, i.e., transmission of external energy from the active elements of the activator to the crystal lattice and adaptation of this energy by the crystal. Thus, there are no data on the crystal lattice behavior under fast dynamical loading and at unloading. To correctly interpret the obtained mechanochemical data it is necessary to know the conditions they are realized in. This work implements a nanosecond resolution X-ray diffraction experiment on synchrotron radiation (SR) beams, when crystal lattice behavior under dynamical (shock) loading was studied in real time, i.e., in situ, on model systems. In an experiment modeling an elementary act of mechanochemical impact, a sample was subjected to a shock impact of the striker (usually we apply aluminum, copper or nickel plates), shot from a gas-dynamic gun. The striker accelerated under the impact of products of explosion of high explosives in the gun. An aluminum striker can accelerate up to a velocity of 3 km/s (a minimum velocity is 0.5 km/s). Upon knocking a sample, also plate-shaped, the striker produces a shock wave in the sample. X-ray photography (32 frames) is performed ~ 100 ns after the shock wave generation. A series of X-ray pictures was recorded with a time resolution of 125 ns and an exposure of 1 ns. Diffraction data obtained in this experiment allows getting information on the loading of a sample under study with a shock wave, i.e., on the energy transmission to a crystal lattice from equation of state. Relaxation processes is very important question in mechanochemistry but not enough information about available at the moment. Information on unloading processes was received in SR experiment immediately after the shock impact is of high importance. These data are also obtained from a diffraction experiment with high time resolution. We have found that "fresh" product (5-80 ns after their formation) are not resistant to a shock action (an impact on an obstacle with a velocity of 2 km/s); their crystal lattice was destroyed. Precisely the same particle but after 250 ns nucleation can withstand an impact. We have also found that this relaxation processes in product has strong temperature dependence. This result is very important for design of new more effective mechanochemistry activators. The relaxation processes in different metals (Ag, Ni, Cu) with millisecond time resolution was investigated at different temperature. The activation energy was found in air and solution environment for these metals.

# EFFECT OF MECHANICAL ACTIVATION OF THE POWDER MIXTURE 3Ti-Al ON PARAMETERS OF HIGH-TEMPERATURE SYNTHESIS AND DYNAMICS OF PHASE TRANSFORMATIONS IN CONDITIONS FAST INDUCTION HEATING

M.V. Loginova[1], A.A. Sitnikov[1], V.I. Yakovlev[1], A.A. Popova[1], B.P. Tolochko[2], M.R. Sharafutdinov[2]

[1]*Polzunov Altai State Technical University, Lenin prospect 46, Barnaul, 656038, Russia, e-mail: anicpt@rambler.ru*

[2]*Institute of Solid State Chemistry and Mechanochemistry SB RAS, Kutateladze 18, Novosibirsk, 630128, Russia*

One effective way of obtaining promising nanostructured composite materials is a combination of two technologies: pre-processing mechanical activation of the initial blend and the high-temperature synthesis (SHS). The application of mechanical activation (MA) can be influenced the structural state of the reaction mixture and the parameters of SHS, thus providing the possibility of regulating mechanisms of phase and structure of materials in the synthesis process.

MA initial reaction mixtures of 3Ti + Al was carried out in a planetary ball mill AGO-2C with water cooling. Weight balls in each drum was 200 grams, the weight of the sample – 10 g, the centrifugal acceleration balls was 400 m/s$^{-2}$ (40 g). Mechanical activation time amounted to 1, 3, 5 min. To prevent oxidation during MA drums with samples was evacuated and then filled with argon to a pressure of 0.3 MPa. To study the regularities of synthesis under dynamic conditions an experiment was conducted using an induction heater, which is adapted to the method of dynamic diffraction.

The investigations lead to the following conclusion: with increasing the time of the mechanical activation of the synthesis mixture of 3Ti + Al method of induction heating leads to an anomalous decrease the time and temperature of the beginning reaction of the components, as well as increase the rate of combustion and reduce the temperature to initiate synthesis. After three minutes of mechanical activation with the first second of reaction components has already formed as stable compounds $Ti_3Al$, $TiAl_3$, $TiAl_2$, as well as metastable phases $Ti_9Al_{23}$, $Ti_5Al_{11}$, $Ti_2Al_5$, $Ti_3Al_5$, persistence and after the synthesis. Preliminary mechanical activation treatment leads to the fact that the reaction high-temperature synthesis of a powder mixture passes without the liquid phase, in true solid combustion.

# REACTIVITY OF MECHANICALLY ACTIVATED MIXTURES IN THE PROCESS OF RADIATION-THERMAL SYNTHESIS OF FERRO-SPINELS

M.A. Mikhailenko[1], U.V. Ancharova[1], B.P. Tolochko[1], M.R. Sharafutdinov[1], N.Z. Lyakhov[1], E.A. Shtarklev[2], V.S. Eliseev[2], A.Yu. Vlasov[2], M.V. Korobeynikov[2], A.A. Bryazgin[2]

[1]*Institute of Solid State Chemistry and Mechanochemistry SB RAS, Kutateladze, 18, Novosibirsk, 630128, e-mail: mikhailenko@solid.nsc.ru*

[2]*Budker Institute of Nuclear Physics SB RAS, Lavrentieva, 11, Novosibirsk, 630090, Russia*

Electron beams allows obtaining new effects in high temperature chemistry of solid state. A stimulated influence of irradiation at comparable temperature conditions may be a subject of technological interest considering that increase of reaction rate [1]. Presumably, the use of initial oxides in nanoparticle form (obtained also by radiation-thermal method) allows additional increase of the reaction rate and may modify the ceramics properties.

Syntheses of nickel-zinc ferrites were investigated. The samples were synthesized by two different ways:

1. Radiation-thermal sintering of mechano-activated oxide mixtures;

2. Radiation-thermal synthesis of the reaction mixture prepared from nano-sized reactants powders.

The nanoparticles are synthesized from the initial oxides by radiation-thermal method.

It was shown that the co-use of electron beam and mechanochemical activation for ferro-spinels' synthesis can raise the reaction rate and reduce the sintering temperature.

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