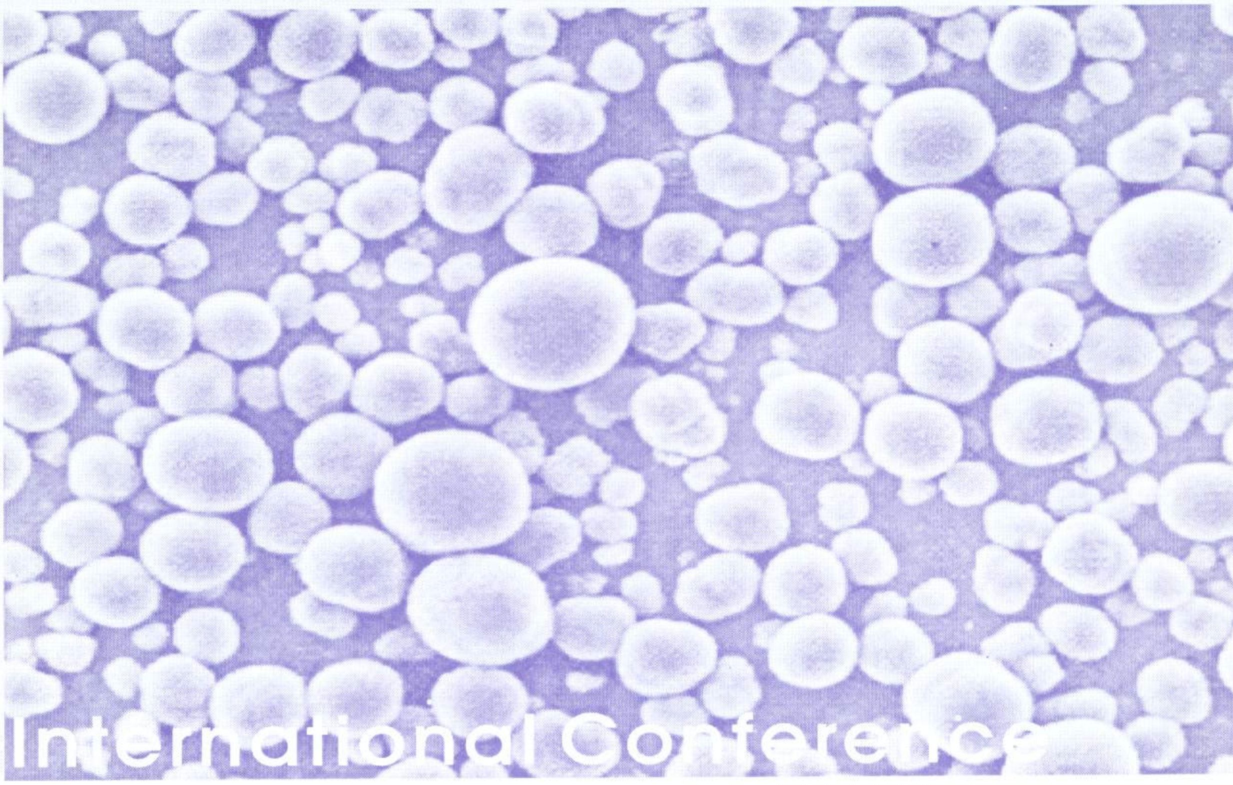
**August, 16-18, Novosibirsk**



International Conference

# "Fundamental Bases of Mechanochemical Technologies"

# BOOK OF ABSTRACTS

**2001**

INSTITUTE OF SOLID STATE CHEMISTRY AND MECHANOCHEMISTRY

SIBERIAN BRANCH OF THE RUSSIAN ACADEMY OF SCIENCES



**International Conference**

# "FUNDAMENTAL BASES OF MECHANOCHEMICAL TECHNOLOGIES"

# BOOK OF ABSTRACTS

**August 16 - 18, 2001**

# RADIATION TECHNOLOGIES OF OXIDE NANOPARTICLES PRODUCTION

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New methods for the production of oxide nanoparticles using powerful accelerated electron beam have been developed: a) decomposition of semivolatile metalorganic compound vapors in the gas phase; b) immediate evaporation (at atmospheric pressure) of natural or concentrated oxides. Commercially produced electron accelerator of the power of 100 kW has been employed.

The first method has been realized in a special reactor for carrying out experiments in a gas flow at the temperatures 700-1000 °C where electron beam is prevented from falling on the reactor surface. Nanometer length scale powders of aluminum, titanium, zirconium oxides have been obtained at the production rate of 100 g/hour.

For the production of nanoparticles by the second method a pilot installation has been used where initial solid materials are evaporated in skull conditions with the subsequent condensation of high temperature vapors in the flow as nanoparticles. As a result, oxide powders have been produced that consist of amorphous particles of the size from 50 nm to 200 nm (depending on air dilution of vapors) with the productivity of dozens kg/hour.

Physicochemical properties have been studied, and some possible application fields of the produced powders with the specific surface up to 100 $m^2/g$ have been tested. In particular, silica powder can be used in composites, lacquers, paints, etc.; magnum oxide – in electric heating elements and alloys; aluminum oxide – as abrasive materials. The obtained powders exhibit high activity when sintered, and the corresponding ceramic species are structurally homogeneous.

# THE USE OF SYNCHROTRON RADIATION FOR *IN SITU* INVESTIGATION OF THE PHYSICOCHEMICAL PROCESSES IN SOLIDS UNDER THE INFLUENCE OF DETONATION AND SHOCK WAVES

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During detonation of explosive materials (EM), very high pressure (million atmospheres) and temperature (thousands degrees) are realized. This gives a unique possibility for science to investigate the behaviour of a material under extreme conditions. Very interesting is the behaviour of the EM and other solids in detonation front.

The special instrumentation was developed for the investigation of explosion. This is an explosion chamber, detonation front sensors and position-sensitive detectors (PSD), which were synchronized with the movement of electrons in the VEPP-3. The explosion chamber now can operate with an amount of an explosive close to 50 g (the trotyl equivalent); this will be 200 g in the nearest future. It has an entrance window for the primary synchrotron radiation (SR) beam and exit windows for SAXS and WAXS [1].

Our team was the first in the world who used SR for the studies of the behaviour of the explosive material during detonation. We have obtained unique results: 1) information about the behaviour of density immediately after the front of detonations with time resolution of 250 ns was achieved; 2) new phenomenon was discovered - the appearance of the SAXS signal immediately after the detonation front; 3) SAXS signal for different explosives was explored with time resolution of 250 ns.

A set of experiments on the investigation of reactions in solids initiated by detonation (silver stearate, paraffin, teflon, Al powder) and by shock wave (copper ammonium perchlorate, nickel hexamine, paraffin, diamond powder) was carried out.

[1] B.P. Tolochko, N.Z. Lyakhov, et al. Nucl. Instr. Met. Phys. Res., 2001, v. 467-8, p. 161-164.

# THE MECHANOCHEMICAL SYNTHESIS AND COMPACTING OF THERMOELECTRIC COMPOSITE MATERIALS BASED ON BETA-$FeSi_2$

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The iron disilicide is a good candidate for the mass production of thermoelectric generators for civilian application. Efficiency of doped $FeSi_2$ leaves much to be improved. Ceramics thermoelectric properties were formed basically at the stages of doped iron disilicide synthesis, sintering and annealing. There are many ways to improve the efficiency of $FeSi_2$ based materials: 1) the particle size decreasing; 2) the synthesis of $FeSi_2$-metal oxide composite materials; 3) the superlattice (nanocomposite $FeSi_2$-semiconductor, such as SiC) preparing from iron disilicide; 4) nonconventional and multicomponent doping of iron disilicide $Fe_{1-X}D_XSi_{2-Y}D_Y$.

The purpose of this work was to synthesize composite material and ceramics based on doped iron silicide. Starting from elements, the iron disilicide powders were prepared by the method of mechanical alloying in the high-energy planetary ball mills AGO-2 and APF-4 type, ball acceleration 600 m/sec$^2$. The ceramics sintering was carried out in vacuum $10^{-5}$ torr or pure argon atmosphere in conventional heater. The blast wave compacting was carried out under different detonation velocity of explosive. Ceramics was analysed by conventional X-Ray analysis (DRON-3, $CuK_\alpha$) and by means of synchrotron radiation diffraction on VEPP-3, INP SB RAS. The phase content of samples under mechanical alloying and explosive compacting was analysed by differential dissolution method. Thermoelectromotive force of ceramics was measured within the temperature range from room temperature to 500$^0$C. Particle sizes of powder materials and grain size of ceramics were analysed by electron and optical microscopy.

The composite material was formed from initial mixtures of components by different mechanisms depending on composition and treatment condition. In case of excess of dopant elements in $FeSi_{2-X}D_X$ we observed $FeSi_2$-FeSi or $FeSi_2$-dopants phase composite. This composite type improved thermoelectric properties increasing $\sigma$ due to the metallic character of conductivity of FeSi and other silicides.

The composite $FeSi_2$-$SiO_2$ materials were formed under mechanical treatment of initial mixtures of iron disilicide and $SiO_2$ powders. In case of chemical reaction of silicon and iron under mechanical treatment in air in jars, the composite $FeSi_2$-$SiO_2$-FeSi was formed.

The differences in morphology, phase content and the distribution of doping elements between phases of ceramics were demonstrated using the samples compacted with different explosive intensity. But electrical measurements demonstrate that there are no direct inter-relations between explosive intensity and thermoelectromotive forces.

The combination of mechanochemical synthesis of $FeSi_2$-based composite powders and explosive compacting method produces the thermoelectric ceramics with nano scale grains size and nonequilibrium phase content. Under working temperature up to 450$^0$C this ceramics can be used without grain size growth process and demonstrate good thermoelectric properties.

The research was supported in part by Award No. REC-008 of the CRDF (USA).

# INVESTIGATION OF THE STRAIN RELAXATION USING SYNCROTRON RADIATION

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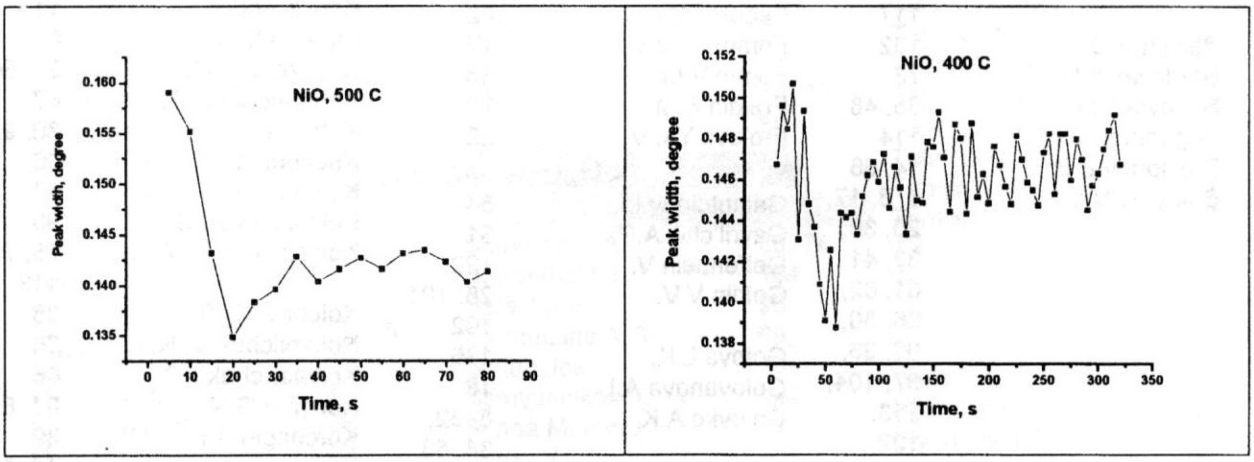
Previous works on solid-state synthesis of nickel tungstate have shown fast decrease of the width of initial powder peaks. It was decided to investigate its behavior during annealing. Synchrotron radiation, one-coordinate detector OD-3 and time-resolved diffraction technique were used for experiments on channel 5b of VEPP-3 (BINP SB RAS). The samples were mechanically activated for several time intervals. Then samples were put into furnace and the strain relaxation during annealing was investigated. Peak width was observed to decrease at the initial stage and then it increased slightly. We assumed this was related to the decreasing amount of defects (dislocations, for example) and then equilibrium state was achieved. The time of equilibrium establishment depends on temperature, so some kinetic parameters may be estimated.

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