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Keywords: nickel_oxide, nanocrystal, powder_diffraction

MS18.P10

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Crystallographical characterization of nanocrystals PbS doped with Ni

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The thin films of lead sulfide (PbS) doped with Ni have been successfully synthesized using chemical bath deposition (CBD) method. The films were deposited on glass substrate at temperature 80 ± ± 2 °C, using five different levels of doping Ni; 0, 2, 4, 6, 8, and 10 mLs. The structural characterization shows that the films are deposited on phase face-centered cubic and the decreases in grain size (TG) with increases of Ni concentration, from order 40 to 10 nm.

Keywords: crystallographical, nanocrystals, PbS

MS18.P11

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X-ray scattering studies of amorphous and nanocrystalline pharmaceutical materials

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The increased interest in recent years regarding the properties and applications of amorphous and nanocrystalline materials has also created the need to characterize the structures of these materials. However, due to the lack of long-range atomic ordering, the structures of nanostructured and amorphous materials are not accessible by conventional diffraction methods used to study crystalline materials. One of the most promising techniques to study nanostructures using X-ray diffraction is by using the total scattering (Bragg peaks and diffuse scattering) from the samples and the pair distribution function (PDF) analysis. The pair distribution function provides the probability of finding atoms separated by a certain distance. This function is not direction-dependent; it only looks at the absolute value of the distance between the nearest neighbors, the next nearest neighbors and so on. The method can therefore also be used to analyze non-crystalline materials. From experimental point of view a typical PDF analysis requires the use of intense high-energy X-ray radiation (E ≥ 15 KeV) and a wide 2θ range.

In this study we present PDF results obtained from several pharmaceutical materials (salbutamol sulfate, sulfamerazine and paracetamol) and discuss the applicability of the PDF analysis for structural characterization of amorphous and nanocrystalline materials with application in the pharmaceutical industry. The experimental results presented in the poster were obtained using a standard laboratory X-ray diffraction system.

This study further demonstrates that PDF analysis with a laboratory diffractometer can be a valuable tool for structural characterization of nanomaterials.

Keywords: amorphous, nanocrystal, pharmaceutical

MS18.P12

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A new method for measuring x-ray rocking curves by means of x-ray acoustooptics

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A new method for measuring angular distribution of X-ray beam diffraction intensity (method for measuring X-Ray rocking curves) is represented. Intensity distribution analysis in this method is conducted by ultrasonic modulation of a lattice parameter of X-ray acoustic crystal, used as an analyzer. The distinctive feature of this method is the possibility to lead precise and time-resolved measurements of X-ray rocking curves without using sophisticated goniometry system.

Special X-ray acoustooptics elements – X-ray acoustic resonators, consisting of a piezoelectric crystal (quartz) and X-ray optical crystal (silicone) was developed to implement the method. Piezoelectric crystal was used to create a standing acoustic wave and control effectively a tension-compression deformation in the X-ray optical crystal [1]. Developed X-ray optical scheme and optical elements allow us to create uniform (within X-ray beam footprint) time-variable deformation of crystal lattice [2] and use these X-ray acoustic resonators as analyzers of scattered X-rays beam.

Rocking curves, measured by proposed X-ray acoustic method by shape and halfwidth agrees well to curves measured according to traditional way - by rotating a crystal. Experimental results of method approbation - examples of rocking curves of (440) reflection silicon crystal and (220) paratellurite crystals measured on laboratory diffractometer using X-ray acoustic method will be presented. Fig.1. shows an X-ray optical scheme and DuMond diagram corresponding to this double-crystal scheme.

The angular and time resolution of the method is determined by speed of detecting apparatus (the minimum possible changing of phase and minimal width of stroboscopic window or number of channels of the multichannel scaler) and depends on parameters such as ultrasound frequency and amplitude of ultrasonic vibrations. Experimentally achieved resolution of the method is 0.1 arcsec. Accuracy can be increased no less than an order by using an ultra-fast multichannel scaler. Developed method and experimental schemes are totally applicable for synchrotron radiation conditions.

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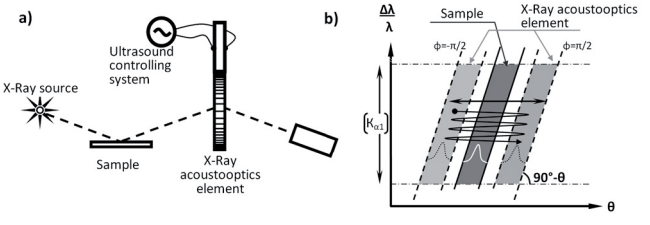

Fig.1 Experimental X-Ray optical scheme (a) and DuMond diagram (b) illustrated approach to measure rocking curves.

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Keywords: x-ray acoustooptics, method, rocking curve

MS19.P01

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Stability of the $BaMF_4$ (M = Mg, Zn, Mn) materials

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Ternary fluorides $BaMF_4$ (M = Mn, Zn, Mg, Fe, Ni, Cu, or Co) are piezoelectric at ambient conditions. Their multiferroic properties have been discussed in [1], [2]. Due to their high transparency, they can be used for optical applications [3]. Recently, $BaMgF_4$ has been suggested as a material for scintillators [4].

We studied the crystal structures of three $BaMF_4$ representatives (M = Mg, Zn, or Mn) as a function of temperature and pressure [5], [6]. Their crystal structure ($Cmc2_1$, $Z = 4$) is built of slabs of corner-sharing MF_6 octahedra which are stacked perpendicular to the crystallographic *b* axis. The Ba^{2+} ions, which are eleven-fold coordinated by fluorine in $BaMnF_4$ and $BaZnF_4$ and thirteen-fold coordinated in $BaMgF_4$, are incorporated between these layers. The hypothetical paraelectric polymorphs are not observed at ambient pressure since the estimated temperatures of the corresponding phase transitions to the centrosymmetric structures lie above the melting points of the compounds [7]. The unit-cell volumes of the $BaMF_4$ fluorides are linearly correlated with the ionic radius of the M^{2+} ion.

Our low-temperature x-ray diffraction investigations show that $BaMnF_4$, which is the compound with the largest M^{2+} ion, undergoes a phase transition to a twinned incommensurate phase at T = 245 K and atmospheric pressure [5]. The formation of the modulated phase in $BaMnF_4$ arises from the incorporation of the large Mn^{2+} cation in the octahedral sheets and the resulting increase of the cavity occupied by the Ba^{2+} ion. The other two compounds (M = Mg, Zn) do not provide any evidence for phase transitions down to 10 K [5].

Single-crystal high-pressure diffraction studies at room temperature show that $BaMgF_4$ undergoes a reversible second-order phase transition to the paraelectric phase (space group *Cmcm*, $Z = 4$) at about 6 GPa [6]. $BaZnF_4$ undergoes a reversible first-order phase transition to a monoclinic structure (space group *P11n*, $Z = 4$). Both high- and low-pressure polymorphs coexist in the pressure range 5-7 GPa [6]. $BaMnF_4$ maintains the $Cmc2_1$ structure up to pressures of 4 GPa. Above this pressure the diffraction intensities are rapidly decreasing and at 6 GPa no single-crystal diffraction could be detected in our experiment any more.

The results of our investigations indicate that considerations of geometrical parameters and of ionic radii of the M^{2+} cations are not sufficient to predict the behaviour of the $BaMF_4$ compounds at high pressures. The incorporation of a smaller M^{2+} cation is not equivalent to compressing the $BaMF_4$ materials meaning that the application of hydrostatic pressure is not exactly comparable to the exertion of chemical pressure in these fluorides.

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Keywords: fressure, fluorides, phase transitions

MS19.P02

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Novel organic-inorganic hybrid materials prepared at elevated temperatures and pressures

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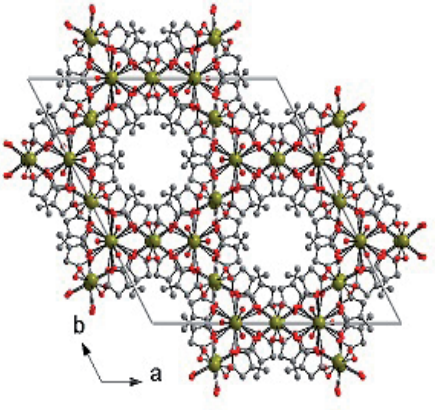
Hydro(solvo)thermal methods of synthesis constitute a convenient, clean route to novel metal carboxylates with open structures. In contrast, their synthesis in solution at room temperature, usually lead to layered structures. Depending on the nature of the carboxylic acid used as starting material, different reactions such as rearrangement, isomerization, addition, decomposition, decarboxylation, etc., may occur under hydrothermal conditions and could produce interesting derivatives, not attainable by direct synthesis. For example, the reaction of itaconic acid (methylene succinic acid) with either $CaCO_3$ or $BaCO_3$ in aqueous solution at room temperature, results in layered structures [1]. However, when the reaction is carried out with $CaCO_3$ by hydrothermal methods, a structure where the itaconate ligands undergo addition to form citramalate is obtained by slow evaporation of the filtrate. From the precipitate, a Ca-itaconate with a honeycomb-type arrangement is obtained (see figure). With $BaCO_3$, the hydrothermal reaction produces a compound where two itaconate ligands suffered an isomerization to citraconates and one ligand undergoes addition and isomerization to dimethylfumarate. In the reaction $BaCO_3$-mesaconic acid, the hydrothermal conditions lead to partial decomposition of the mesaconate moieties to produce bridging fumarate groups, in contrast with the structure obtained at room temperature [2]. These and other interesting compounds (La-aconitic acid, for example) will be presented and discussed in detail.



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Keywords: hydrothermal synthesis, metal-organic frameworks, metal carboxylates