Manganites with perovskite structure are the class of compounds referred as the rare earth manganites that is characterized by extremely interesting structural and physical properties and phenomena, including Colossal Magnetoresistance (CMR) [1].

The structure of the $RE_{1-x}M_xMnO$ (RE= rare earth) oxides is close to that of the cubic type perovskite (CaTiO3), but the structure of manganites generally corresponds to a lower rhombohedral symmetry (LiNbO3) or orthorhombic (GdFeO3) structure [2] such as LaSrMnO3, S.G. Pnma.

We present preliminary studies and results of the $RESrMnO_3$ system where substitutions RE=Dy,Yb for La in perovskite type structure were carried out. Samples were prepared by the solid-state reaction method in air at ambient pressure. Process of synthesis was followed by thermal analysis (TGA and DTA) and X-ray powder diffraction (XRD). Morphology of resultant samples have been observed by Scanning Electron Microscopy (SEM) and the stoichiometry has been analyzed by Electron Dispersive X-Ray Spectroscopy (EDX).

[1] T.V. Ramakrishnan, H.R. Krishnamurthy, S.R. Hassan, G. Venketeswara. *Proc. Indian Acad. Sci.* (*Chem. Sci.*), **2003**, *115,* 767–774. [2] A.M. Haghiri-Gosnet, J.P. Renard, *J. Phys. D: Appl. Phys.* **2003**, *36,* R127.

Keywords: perovskite, manganite, rare-earth.

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Crystallization in lipidic cubic phase. The impact of additives on phase behaviour

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To solve the structure of a membrane protein to atomic resolution is still a challenging task. Apart from conventional crystallization techniques the use of lipidic cubic phase (LCP) is of growing interest and was successfully applied to crystallize a number of G protein coupled receptors [1-4]. The LCP three-dimensional networks consist of a continuous bilayer defined by two distinct water channels, referred to as bicontinuous networks. Most commonly monoolein (MO) is used, forming cubic phases at temperatures suitable for crystallization.

A versatile tool to study this phase behaviour is x-ray diffraction analyzing the data in the small angle regime. Fitting scattering models to the experimental data allows to identify structural changes in the aqueous and the lipid bilayer subcompartments.

To make use of the cubic phase and to facilitate the crystallization of membrane proteins, detergents, precipitants and other additives need to be added. These chemicals tend to destabilize the network structure. Therefore it is crucial to understand their impact on the phase behaviour.

These effects are studied by addition of detergents and precipitants, known to facilitate the crystallization of membrane proteins, according to a ranking from the Membrane Protein Data Bank. In preceding experiments, depending on the type of detergent or additive and the concentration clear differences in the phase boundaries could be detected.

[1] S.G.F. Rasmussen, H.-J. Choi, J.J. Fung, E. Pardon, P. Casarosa, P.S. Chae, B.T. DeVree, D.M. Rosenbaum, F.S. Thian, T.S. Kobilka, A. Schnapp, I. Konetzki, R.K. Sunahara, S.H. Gellman, A. Pautsch, J. Steyaert, W.I. Weis, B. K. Kobilka *Nature* **2011**, *469*, 175-180. [2] D.M. Rosenbaum, C. Zhang, J.A. Lyons, R. Holl, D. Aragao, D.H. Arlow, S.G.F. Rasmussen, H.-J. Choi, B.T. DeVree, R.K. Sunahara, P.S. Chae, S.H. Gellman, R.O. Dror, D.E. Shaw, W.I. Weis, M. Caffrey, P. Gmeiner, B.K. Kobilka *Nature* **2011**, *469*, 236-240. [3] B. Wu, E.Y.T. Chien, C.D. Mol, G. Fenalti, W. Liu, V. Katritch, R. Abagyan, A. Brooun, P. Wells, F.C. Bi, D.J. Hamel, P. Kuhn, T.M. Handel, V. Cherezov, R.C. Stevens *Science* **2010**, *330*, 1066-1071. [4] E.Y.T. Chien, W. Liu, Q. Zhao, V. Katritch, G.W. Han, M.A. Hanson, L. Shi, A. Hauck Newman, J.A. Javitch, V. Cherezov, R.C. Stevens *Science* **2010**, *330*, 1091-1095.

Keywords: X-ray_diffraction, membrane_protein, biocrystallography

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Crystal growth and characterization of 4-(dimethylamino) benzaldehyde doped TGS Crystals

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Single crystals of 4-(dimethylamino) benzaldehyde doped ferroelectric Tri Glycine Sulphate (DBTGS) were grown by slow evaporation from its aqueous solution at ambient temperature, using solution growth method. Solution grown DBTGS crystals were characterized by dielectric and pyroelectric measurements. The capacitance of DBTGS was measured using HP 4194A impedance/ gain phase analyzer at 10 kHz at a cooling rate of 1°C/min using Metler hot stage FP90 and the pyroelectric current was measured using Keithley 610C electrometer over the temperature range of 30 to 60°C in the ferroelectric direction. The direct method of Byer and Roundy was used for the measurement of pyroelectric current. The sample preparation and the measurement technique used in the measurement of capacitance are discussed in detail. Pure TGS crystals exhibits ferroelectric phase transition at 48.5°C with ε'_{peak} 8800 . However for doped DBTGS crystals phase transitions is observed at 51°C with decreased ε'_{peak} (1930). Higher Pyroelectric coefficient was observed for the doped DBTGS crystal. The doped crystal was irradiated with graded dosages from 5 kGy to 80 kGy electron beam from 8MeV Microtron (Energy-8MeV, pulse current-50mA (max), pulse duration-2.5μsec, pulse repetition rate-250 Hz (max)) at room temperature and radiation effects on the dielectric and pyroelectric properties of the crystals were investigated. The dielectric study shows that there is a gradual reduction in dielectric constant at T_C and shifting of Curie temperature towards lower temperature region with increase in electron radiation dose. The material figure of merit (Fv) was found to be higher for the irradiated crystals.

Keywords: crystal growth, TGS crystals, ferroelectric

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Generation of high quality crystal surfaces of small soft organic crystals

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In a time resolved micro-crystallography experiment the X-ray beam is focused to a size of a few microns. This allows probing a small crystal volume very close beneath the surface, where high populations of the excited state can be generated (visible pump light has penetration depths of a few microns only at the absorption maximum) [1]. Such experiments require well defined and low-roughness crystal surfaces. Traditional crystal polishing methods can not be applied for many

organic crystals due to their softness causing them to crack.

Therefore we have developed and tested different methods to obtain the required quality and low roughness surfaces of soft organic crystals. All polishing methods are based on a high speed air bearing which can be equipped with different tools. The crystal is mounted on a piezo driven x-,y-,z- stage and can be moved with respect to the polishing device.

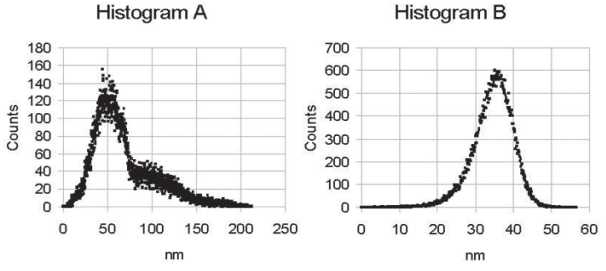
Beside different conventional polishing heads containing diamond grains of different sizes a custom made dimaond cutting tool was used. It consists of a monocrystalline diamond knife which is rotating perpendicular to the crystal surface.

The crystal surfaces generated with the different methods were characterized by atomic force microscopy (AFM). We used tapping mode imaging to reduce the forces on the crystal during the scanning process. The best quality surfaces possess a root-mean-square (rms) roughness of 5.3 nm for a 5 μm x 5 μm area. The rms roughness for a 80 μm x 80 μm area which corresponds to about 75 % of the whole crystal surface is about 14.8 nm.

We also cutted crystals using a commercial microtome suitable for electron microscopy sample preparation. Surfaces with a rms roughness of 4.7 nm for a 50 μm x 50 μm area and 2.9 nm for a 5 μm x 5 μm area could be achieved.

One big advantage of the air bearing cutting device compared to the microtome is the fast and easy processing of a large amount of crystals with high quality surfaces suitable for crystallographic and related experiments.

The figure below shows the histograms of height levels of a crystal polished by a conventional polishing head (A) and a crystal polished by the diamond knife tool (B).



[1] A. Meents et al, *J. Appl. Cryst.* **2009**, *42*, 901-905.

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Compositional distribution of isomorphic crystals during spontaneous crystallization

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A non-trivial bimodal distribution of crystals in the isomorphic composition was revealed at spontaneous mass precipitation in aqeuos solutions. Crystal ensembles of the series $(Pb,Ba)(NO_3)_2$, $(Co,Ni)(NH_4)_2(SO_4)\cdot 6H_2O$, $(Co,Ni)K_2(SO_4)\cdot 6H_2O$, $(NH_4,K)H_2PO_4$, $(Al,Cr)K(SO_4)_2\cdot 12H_2O$, and K(Cl,Br) were precipitated during 10–15 s in solutions of various isomorphic ratios at the supercoolings 10 °C and 20 °C. Precipitates were dried by filter paper after the solution decantation.

The isomorphic ratios in individuals of each the series were determined for random samplings of 100–120 individuals and some control samplings up to 400–500 crystals. X-ray fluorescence was used for $(Co,Ni)(NH_4)_2(SO_4)_2\cdot 6H_2O$ and $(Al,Cr)K(SO_4)_2\cdot 12H_2O$. Synchrotron-based XRF (ANKA FLT beamline, Karlsruhe) was used for the $(Pb,Ba)(NO_3)_2$ and $(Co,Ni)(NH_4)_2(SO_4)_2\cdot 6H_2O$ samplings [4]. Microprobe analyser ABT-55 Link AN 100000/85S was used for $(Co,Ni)(NH_4)_2(SO_4)_2\cdot 6H_2O$, $(Co,Ni)K_2(SO_4)_2\cdot 6H_2O$, and $(Al,Cr)K(SO_4)_2\cdot 12H_2O$ [4]. XRPD was used for the probes of K(Cl,Br), sieved to the fractions <10, 10–30, 30–50, 50–100 and >100 microns. Isomorphic compositions of crystal ensembles $(NH_4,K)H_2PO_4$ and K(Cl,Br) were also estimated by means of X-ray micro-tomography (SkyScan 1172).

The compositional distributions are asymmetric with two modes. However the bimodality is not quite clear sometimes due to such samplings were selected without the finest fractions. The distributions are sensitive to the probe total composition and supercooling: the peaks move regularly to one of the end-member. The XRD measurements for the series $(Pb,Ba)(NO_3)_2$ and K(Cl,Br) displayed a nonlinear function of the composition on crystals sizes [3]. The compositional bimodality is coordinated with that of the surface nuclei observed by AFM on a crystal surface of the mixed potassium-rubidium diphthalate [1]. This is in accordance with a high dispersion of crystal composition measured on a surface of grown $(Pb,Ba)(NO_3)_2$-crystals [5].

The compositional bimodality can be understood by the developed concept of mixed crystal formation [1, 2]. It establishes nucleation of two kinds caused by kinetic factor and displayed by modified concentration diagrams.

Results obtained by different analytical methods show that spontaneous mass crystallisation as well as monocrystal growth of solid solutions lead to formation of crystals with rather complex compositional distributions. These data confirm substantially the concept of mixed crystal formation [1].

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[1] A.E. Glikin *Polymineral-metasomatic crystallogenesis* **2009**, *Springer NY*, 312. [2] L. Yu. Kryuchkova, A.E. Glikin, S.N. Kovalev, A.E. Voloshin *Proceedings of Russian Mineralogical Society* **2002**, *3*, 62-77. [3] A.E. Glikin, L. Yu. Kryuchkova, Yu. V. Plotkina, M. Yu. Sinai, P. Gille, J. Schneider, R. Stark *Proceedings of Russian Mineralogical Society* **2007**, *spessial issue "Crystallogenesis and Mineralogy", 7-35*. [4] L. Yu. Kryuchkova, A.E. Glikin, M. Yu. Sinai, A.A. Shiryaev *Proceedings of the16th BIWIC* **2009**, 232-238. [5] V.D. Franke, A.E. Glikin, L.Yu. Kryuchkova, E.V. Tabuns *Geology of Ore Deposits* **2007**, *49*, 7, 641-647.

Keywords: mass crystallization, isomorphism

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A comparison towards counter-diffusion and sitting drop-vapor diffusion technique in improving resolution data of a novel organic solvent tolerant lipase

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Quality of diffraction data is important in order to generate a better electron density map which provides valuable, accurate information and enzyme functions in structure analysis afterward. To achieve the above objective, one way is to applied different kinds of crystallization techniques. In this study, we discussed about the improvement of crystal quality by counter-diffusion technique and sitting-drop-vapor diffusion technique.