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By HREM it was shown that the nanostructure states of Cu-powder prepared by ball milling is characterized by the presence of a high density of dislocations and twin faults. For the determination of microstructure parameters of Cu-powder the method of analysis of the X-ray powder diffraction pattern was developed [1].

The microstructure evolution of Cu-nanostructured powders depending on the ball milling conditions (namely, frequency of shocks, kinetic energy of shocks, the ratio between normal and tangential components of shock under constant time of the milling) was investigated. It is shown that the frequency of shocks in the range of 6-17 Hz does not affect on microstructure characteristics. The dislocation and twin densities are functions of the kinetic energy of shocks and the dislocation and twin densities change in non monotonous way when the energy of shocks increases. The ratio between normal and tangential components of shock is dominant factor that determines the ratio between edge and screw of dislocations.

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Keywords: nanostructured materials, ball-milling, X-ray powder diffraction pattern

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Defects in γ Irradiated Cz-Si Annealed under High Pressure

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The effect of uniform stress on creation of oxygen - related defects in annealed Czochralski grown silicon, with point defects introduced by γ irradiation, is investigated. The (111) oriented Cz-Si samples with interstitial oxygen concentration, c_o = 9.5x10^{17} cm^{-3} were irradiated by γ rays (E=1.2 MeV, dose 1000 Mrad). Next the samples were treated for 5 h or 10 h at 920, 1270 and 1400 K under hydrostatic Ar pressure (HP) equal to 1.1 GPa. The kind, dimension and concentration of defects were estimated from X-ray diffuse scattering data. While the concentrations of point defects were similar for irradiated and non irradiated Cz-Si, X-Ray diffuse scattering was stronger after the HP treatment of γ irradiated samples. The temperature dependent effect of HP at annealing is specific for γ irradiated Cz-Si. The changes in diffuse scattering intensity are accompanied by the changed concentration and dimension of point defects conglomerates; the average defect dimension is below 100 nm. HP affects oxygen precipitation in γ irradiated Cz-Si mostly through its effect on the creation and transformation of *V*O and V_mO_n complexes, the last ones acting as the nuclei for oxygen precipitation.

Keywords: Cz-Si, γ irradiation, annealing stress

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X-ray Diffuse Scattering on the First Type Defects in Semiconductors

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X-ray diffraction techniques give powerful tool for the investigation of the defect structure in crystals. For the case of I type defects (as interstitials, vacancies, dislocation loops etc.) in the lattice, main of these methods [1,2] are based on the analysis of the Huang and Stoks-Wilson scattered ranges, which contain information about the distortion field around the defect.

From our observations we conclude that described experimental procedures for determination of defects size and their concentration are trustworthy at the special case and can not be used for general one.

In this work we analyzed more basic case of the defect structure which include the simultaneously presence of defects giving lattice deformation of opposite signs. The effects following from the assumption that defect concentration should be described as a function of defects size are considered in details.

All diffraction measurements presented in this work were made using the triple-axes diffractometer Philips X'Pert MRD.

[1] Larson B.C., Schmatz W., *Phys. Rev. B.*, 1974, **10(6)**, 2307. [2] Patel J.R., *J. Appl. Cryst.*, **8**, 186.

Keywords: diffuse scattering, point defects, high-resolution X-ray diffraction

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Structural and Microstructural Studies of Synthetic and Naturally Occurring Hydroxyapatites using Powder Diffraction

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Hydroxyapatite (HAP), the main constituent of mammalian hard tissue, is an important mineral reservoir for the metabolic activity of the organism. Structural and microstructural characterization of several naturally occurring HAP samples extracted from human tooth, goat bone, rabbit bone and synthetic HAP have been carried out using X-ray powder diffraction studies. Diffraction data were collected with a step scan mode at an interval of 0.02°(2θ) using a Bruker D8 Advance X-ray powder diffractometer equipped with a germanium crystal primary beam monochromator (CuK$\alpha_{1=}$ 1.5406 Å). Preliminary phase identification of the naturally occurring HAP samples using the ICDD data base indicated presence of small amounts of Calcite ($CaCO_3$) and Dolomite [$CaMg(CO_3)$], (2-4% each), in addition to the main HAP phase, $Ca_{10}(PO_4)_6(OH)_2$.The whole powder pattern decomposition of the naturally occurring samples indicated sharp (00l) reflections, is in agreement with the earlier reports of preferred orientation in HAP crystals along the c-axis. Rietveld analysis carried out incorporating the structural parameters of different constituents phases using the program TOPAS showed final R_{wp} values ranging between 9.9-111.5%. The average crystallize sizes in the samples vary between 500 to 400 nm. The refined P-O distances in the synthetic HAP sample differ significantly compared to these observed in the naturally occurring samples.

Keywords: hydroxyapatite, microstructure analysis, Rietveld refinement

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An X-ray Diffraction Study on Dislocation Microstructure of as-prepared Al-Al_2O_3 Composites

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Particle reinforced MMC of Al - α-Al_2O_3 were prepared in the powder metallurgy route with 20, 50 and 75wt% of Al powder. The line profile analysis of the composites was done in the Whole-Pattern Fitting procedure based on the Rietveld structure refinement codes. It also incorporates the microstructure refinement codes based on either phenomenological parameters [1] or physically based model [2]. Here the microstructures of α-Al_2O_3 containing Al-based composites were studied in terms of ab-initio quasi-composite model of dislocation cell structure [3] and ellipsoidal log-normal distribution of crystallite size.

Postproduction plastic deformation of the Al grains and hence stress relaxation of the composites have taken place during cooling from the sintering temperature (500°C). It was also noticed that in the composites with lower concentration Al, each reflection of Al could be fitted with two peaks indicating dissimilar fragments with different dislocation density and arrangements. The region of high (~10^{10} cm^{-2})

and low (~10^9 cm^{-2}) dislocation density has been characterized as cell walls and cell interiors respectively with compressive and tensile stresses in accordance with the quasi-composite model. The results are in gross agreement with earlier TEM studies.

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Keywords: composites, X-ray diffraction, dislocation structure

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Strain-free Oxide Nanopowders, Facts and ex-Oxalate MgO Case

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The microstructural characterization of nanocrystalline materials is of particular importance in the study of chemical fragmentation processes and oxides prepared from solution routes. Only a few examples of strain-free (i.e. with negligible microdistortion) oxides have been reported. Their preparation is often not trivial, since the chemical nature of the precursor used and the experimental conditions can influence the microstructural properties. Representative examples of strain-free oxides are ZnO [1], CeO_2 [2] and Y_2O_3 [3]. A new example, i.e. nanocrystalline MgO obtained from the thermal decomposition of the oxalate precursor is investigated in detail. The study is based on line broadening analysis carried out with the Voigt/Langford integral breadth and Fourier methods combined with the pattern decomposition technique. The whole pattern refinement method is also applied. A good agreement between the results obtained from the varied approaches is found. Ex-oxalate MgO is strain free. The crystallites are, on average, isotropic with sizes increasing from 130 Å to 640 Å in the annealing temperature range 500-1200°C and crystallite growth varies exponentially. The results obtained from the different methods are discussed and are also compared to those observed with MEB and BET techniques.

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Keywords: line profile analysis, microstructure, nanoparticles

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Geterostructures of Bacterial Cellulose Acetobacter Xylinum Intercalated by Drug Materials

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Organic-inorganic hybrid materials based on bacterial cellulose with metal nanoparticles are interesting for medical applications. High-crystal cellulose matrix obtained at static growth of Acetobacter Xylinum (AX) in the process of intercalation of drugs was investigated by methods of small and wide-angle X-ray scattering, electron diffraction, transmission electron microscopy and atomic force microscopy.

The investigation of interaction of polyvinilpyrrolydone (PVP) and germicide preparation Poviargol and Catapol (Ag° and Se° nanoparticles stabilized by PVP) with gel-films AX by diffraction methods has shown that besides reflections associated with crystal-state cellulose, in both cases reflections from PVP phase are present. In the case of gel-films AX with Poviargol the reflections are observed from silver. Small-angle X-ray scattering experiments with composite samples allowed us to estimate the relative amount of intercalated PVP, Ag and Se in the process of their desorption by substituion with water, and to determine the size distributions of the nanoparticles. The distributions show relatively large amount of small particles (5-20nm) and minority of larger ones (up to 100nm).

Keywords: electron and X-ray diffraction, bacterial cellulose, organic-inorganic hybrid materials

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Honeycomb Diffuse Intensities in $NaREF_4$ Upconversion Materials

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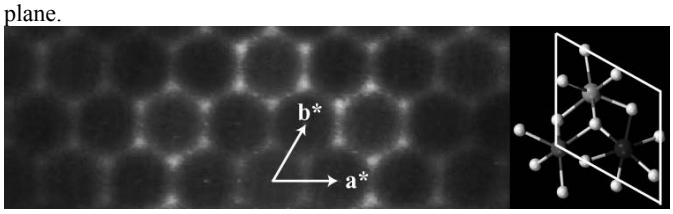
Hexagonal sodium rare-earth (RE) fluorides, e.g. NaYF4: Yb, Er, are very efficient upconversion materials which emit visible light upon infrared excitation. The efficiency of the upconversion process depends mainly on the doping ratio, the phase purity and the Na:RE ratio. These phases have hexagonal structures with a disordered cation distribution. Here we report a detailed investigation of $Na_{1.5}La_{1.5}F_6$. The reconstructed layers of reciprocal space contain either sharp Bragg reflections for integer values of *l*, or planes with honeycomb like diffuse intensities for half-integer values of *l* (Fig left: *h k* 1.5). The Bragg reflections indicate a hexagonal metric and the average structure could be refined with space group symmetry *P*-6. It shows three different columns of cations with Na, La or a 1:1 ratio of both (Fig right). The diffuse intensities are well reproduced if it is assumed that Na and La alternate regularly in the disordered columns along **c** and that Na and La alternate with a probability less than one in the **a**,**b** plane.



Keywords: disordered materials, diffuse scattering, phosphors

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Stacking Faults and Internal Strains in DHCP Phase of La

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High-resolution synchrotron X-ray diffraction measurements of La have been carried out in the range of temperatures from 80 K to 800 K at ambient pressure. The powder diffraction patterns contained characteristic features of DHCP crystals with stacking faults. Additional peak broadening was attributed to a lattice strain. A considerable amount of lattice strain along a stacking direction can consistently account for an upward jump of the close-packed interplanar spacing observed in course of the DHCP-to-FCC phase transition at about 500 K, contrasting with the downward atomic volume jump at this phase transition.

Quantitative analysis of basic parameters of the planar defects along with strain calculations is presented. The temperature evolution of stacking faults concentration and lattice strain in DHCP structure of La indicates on an equilibrium nature of the observed lattice imperfections rather then on a non-equilibrium one. Such an equilibrium microstructure can arise due to an additional gain in electronic energy, which stabilizes the DHCP lattice, at cost of elastic distortive energy [1]. The analysis of X-ray measurements complemented by TEM study shows that a partial relaxation of the induced short-range elastic stresses can proceed via incretion of stacking faults in DHCP structure of La.

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Keywords: metals, stacking faults, phase transitions and structure