

France. ^cLRRS-UMR 5613 CNRS - Université de Bourgogne Dijon, France. E-mail: grad19@yandex.ru

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.

[1] Ustinov A., Olikhovska L., Budarina N., Bernard F., *Diffraction analysis of the microstructure of materials*, Eds. E.J. Mittemeijer, P.Scardi, 2003, 333-359.

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

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Defects in γ Irradiated Cz-Si Annealed under High Pressure

Patrik Vagovic^a, J. Bak-Misiuk^b, A. Misiuk^c, A. Shalimov^b, K. Orlińska^b, I. Kovacevic^d, B. Pivac^d, M. Prujarczyk^c, ^a*Institute of Electrical Engineering SAS, Bratislava, Slovakia.* ^b*Institute of Physics, PAS, Warsaw, Poland.* ^c*Institute of Electron Technology, PAS Warsaw, Poland.* ^d*Rudjer Boskovic Institute, 10000 Zagreb, Croatia.* E-mail: patrik.vagovic@savba.sk

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, $c_o = 9.5 \times 10^{17} \text{ cm}^{-3}$ were irradiated by γ rays ($E=1.2 \text{ MeV}$, 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 VO 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

Artem Shalimov^a, Kirill Shcherbachev^b, Jadwiga Bak-Misiuk^a, ^a*Institute of Physics Polish Academy of Sciences, Warsaw, Poland.* ^b*Institute of Steel and Alloys, Moscow, Russian Federation.* E-mail: shalim@ifpan.edu.pl

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

Santu Chakraborty, Alok Kumar Mukherjee, *Department of Physics, Jadavpur University, Kolkata-700032, India.* E-mail: scr_ju@yahoo.co.in

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 $0.02^\circ(2\theta)$ using a Bruker D8 Advance X-ray powder diffractometer equipped with a germanium crystal primary beam monochromator ($\text{CuK}\alpha_1 = 1.5406 \text{ \AA}$). 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 [$\text{CaMg}(\text{CO}_3)_2$], (2-4% each), in addition to the main HAP phase, $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$. The whole powder pattern decomposition of the naturally occurring samples indicated sharp (001) 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 9.9-11.5%. The average crystallite 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-Al₂O₃ Composites

Apurba Kanti Deb¹, P. Chatterjee², S.P. Sen Gupta¹, ¹*Department of Materials Science, Indian association for the Cultivation of Science, Jadavpur, Kolkata-700032, India.* ²*Department of Physics, Vivekananda Mahavidyalaya, Hooghly-712405.* E-mail: msakd@iacs.res.in

Particle reinforced MMC of Al - α -Al₂O₃ 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 -Al₂O₃ 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 ($\sim 10^{10} \text{ cm}^{-2}$)