Growth process of methane hydrate from D_2O ice powder was measured under pressurized CH_4 gas by in situ neutron powder diffraction. We used HRPD and HERMES [1] at JRR-3 in Japan. To avoid melting ice, the CH_4 gas was gradually applied at 240 K as an isothermal process. The diffraction peaks of methane hydrate were observed at 2 MPa. The pressure was kept at 2 MPa for several hours. After that, the pressure was increased until 6 MPa. However, the growth rate of methane hydrate did not change. Accordingly, the pressure did not affect the growth rate under the sufficient pressure for the crystal growth. Following, the temperature was changed under nearly isobaric process (6-7 MPa). When temperature was changed from 265 K to 270 K, an increase of the growth rate was observed. At 275 K, the D_2O ice was melted and the growth rate drastically increased. After 4 hours, almost all of the D_2O ice was changed to methane hydrate. As a result, temperature is more effective parameter for the crystal growth of methane hydrate. Moreover, we will discuss the size effect of ice grain.

[1] K. Ohoyama, T. Kanouchi, K. Nemoto, M. Ohashi, T. Kajitani, Y. Yamaguchi: Jpn. J. Appl. Phys. Vol37(1998)3319-3326.

Keywords: crystal growth, neutron powder diffractometry, *in situ* observations

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Abiotic growth and biological dissolution of pyrite surfaces

Katja R Etzel[1], Andreas Klingl[2], Harald Huber[2], Reinhard Rachel[2], Michael Thomm[2], Wulf Depmeier[1]

[1]Institute of Geosciences, University Kiel, Olshausenstr. 40, Kiel, Schleswig-Holstein, 24114, Germany, [2]Institute of Biochemistry, Genetics and Microbiology, University Regensburg, E-mail：ke@min.uni-kiel.de

The relevance of microbial pyrite oxidation processes has resulted in a large number of studies by which microbial oxidation of Bacteria and Archaea were experimentally examined. Because pyrite is a potential, nontoxic candidate for photoelectrochemical and photovoltaic applications, it is important to know how the surfaces of pyrite can be controllably altered. Natural pyrite crystals are often impure, thus, the synthesis of high quality pyrite single crystals is of interest. A potential technique to synthesize pyrite single crystals is the CVT- technique. With this technique it was possible to produce crystals with edge lengths of up to 8.5 mm and habits of up to five forms ({100}, {111}, {210}, {211}, and {221}). The composition and stoichiometry of the crystals were observed near to ideal FeS_2 (S : Fe = 1.98(8)) and studies of the topography of crystal surfaces indicated the high quality of the synthesized crystals. To enhance our understanding of effects of pyrite dissolution mediated by Archaea and various influence parameters (time, temperature, crystallographic orientation surfaces), etching experiments on synthetic pyrite crystals were performed. As biological oxidants two archaeal strains, namely Metallosphaera sedula and Sulfolobus metallicus, were employed. Studies with Scanning Electron Microscopy (SEM) showed cell attachment and etching effects during the whole time period. Surface alteration forms features up to several tens of microns in size, while the shape of the features varies with face-symmetry and edges of etch pits follow distinct directions. However, control experiments with abiotic oxidants showed, that the shapes of the euhedral etch pits are similar, but smaller in sizes, indicating a higher dissolution rate for microbial than abiotic etching.

Keywords: growth from gaseous phase, dissolution etch phenomena, biological activity

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Synthesis and structural characterization of ZnO deposited by chemical bath

Gabriel Juarez[1], Javier Martinez[2], Oscar Portillo-Moreno[3], Raquel Castillo[3], Reina Galeazzi[2], Constanza G Carrillo[2], Lenin Pena[2], Jose A Henao[4]

[1]SEES-CINVESTAV, Centro de Investigacion y de Estudios Avanzados del IPN, Centro de Investigacion en Dispositivos Semiconductores, Av. IPN 2508 Col. San Pedro Zacatenco, Mexico, Distrito Federal, 07360, Mexico, [2]CIDS-ICUAP, 14 sur Esq. San Claudio, Ciudad Universitaria Edif 137, Apdo. Postal 1651, 72000, Puebla, Pue. Mexico, [3]Facultad de Ciencias Quimicas Benemerita Universidad Autonoma de Puebla, Mexico, [4]Universidad Industrial de Santander-Bucaramanga Santa, Colombia, E-mail：gabriel.juarez@icbuap.buap.mx

Thin films of zinc oxide (ZnO) were deposited on glass substrates by chemical bath deposition technique (CBD), varying the contents of nitrate zinc ($Zn(NO_3)_2$) and thiourea ($(NH_2)_2CS$) in the bath solution . The deposition temperature was 80 ℃ keeping constants the pH value, volume, deposit time and mechanical stirring speed. The samples were characterized by X-ray diffraction (XRD) and transmittance. By X-ray is found that the material is polycrystalline with a Wurzita type structure and with a preferential crystallographic direction (100) for smaller amounts of $Zn(NO_3)_2$, while for larger amounts of $Zn(NO_3)_2$ the material presents a preferential crystallographic direction (002), also the diffractograms show that the presence of thiourea stimulates the reaction desired between precursors. By transmittance is found that with a larger amount of Zn $(NO_3)_2$ the material presents a tendency to increase its energy band gap.

Keywords: semiconductor thin films, zinc oxide, chemical deposition of oxides

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Influence of bath composition in structure of ZnO deposited by microwave activated chemical bath

Javier Martinez[1], Reina Galeazzi[1], Gabriel Juarez[2], Constanza G Carrillo[1], Ramon Pena-Sierra[2], Francisco Flores[1]

[1]CIDS-ICUAP, Benemerita Universidad Autonoma de Puebla, Centro de Investigación en Dispositivos Semiconductores del Instituto de Ciencias, Av. Sn Claudio esq. 14 sur, Edif. 137 Ciudad Universitaria, Col Sn Manuel, Puebla, Puebla, 72570, Mexico, [2]Depto. de Ing. Electrica-SEES, CINVESTAV-IPN, Av. IPN 2508, Apdo. Postal 14-740, 07300 Mexico 14 D. F., Mexico., E-mail：javier.martinez@icbuap.buap.mx

ZnO films were deposited on glass substrates by microwave activated chemical bath deposition technique (MW-CBD), varying the composition of the bath in the relationship 1:1, 1:2, ... 1:10 of $(Zn(NO_3)_2$:$CO(NH_2)_2$, while the reagents that makes the solution become basic are maintained constant as well as the power and time deposit. Samples were characterized by X-ray diffraction (XRD), photoluminescence and transmittance; XRD diffractograms show that material is polycrystalline, hexagonal wurzite type, and the lattice constants and the unit cell volume increases as relationship is varied from 1:1 to 1:10. Furthermore, the samples show a preferential orientation along the c-axis indicated by the intensity of 002 reflection, the same was observed in the residual powder of the reaction that was not deposited on substrate. Photoluminescence spectra show a yellow-red emission of deep-levels related to defects, but there is not significant variation due to relationship of precursors.