

crystal occurs mainly due to the expansion of elementary layers of growth. They are 8.8 Å units high, which is equivalent to parameter a of a unit cell, and given the peculiarities of the specific structure, is also equivalent to one of the sizes of $C_{10}H_{10}N_2O_4$ molecule.

The statistical data processing reveals considerable differences in tangential dissolution rate on the two spirals, consisting of nine and four screw dislocations correspondingly. Right-screwing growth layers of the left spiral join, at some distance, the left-screwing steps of the right spiral, forming an even more complex source – a growth analogy of dislocational Frank-Read source. To calculate the tangential rates for such source, each image was overlaid by a special grid affixed to defined points – canals of dislocations. Then the position data of intersection points of grid meridian and the step contour were taken. Each two images required more than a thousand of coordinate readings. Then we schemed the step rate distribution for every image pair. The distribution came out to be bimodal, which required segregation of the results for the left and the right spirals. Each distribution was then approximated to the lognormal distribution, average tangential rates were determined as expectation value, and rate fluctuations were calculated as a mean-square deviation. The results show that for the left source steps the average tangential rate is two times greater than that for the right source ones throughout the whole experiment. On the whole, the tangential rate decreases towards the end of the experiment. The rate fluctuations for the steps of both groups also reveal an almost monotonous decrease. This means that the system is working towards equilibrium. The amount of substance in the solution is increasing and dissolution is going to give place to equilibrium. This brings forward an important inference: the left and the right part of the same growth source, located at a less than a micrometer distance from each other, have a different effect on the boundary layer of the solution. We have shown that the interface needs to have the horizontal concentration gradient of the substance which would provide a faster tangential rate of the left spiral at growth and a more active decrease at dissolution. The same assumption has been verified for bigger (100 mkm) hillocks.

This work is supported by Russian Foundation of Basic Research (11-05-00432-a), Leading Science School-7198.2010.5 and Programs of Presidium of RAS.

Keywords: AFM, crystal, growth.

MS49.P20

Acta Cryst. (2011) A67, C540

In situ observation of stress and strain evolution during surfactant-mediated growth of Ge on Si

Hidehito Asaoka,^a Tatsuya Yamazaki,^{a,b} Shin-ichi Shamoto,^a *^aQuantum Beam Science Directorate, (Japan) Atomic Energy Agency, Tokai, Ibaraki 319-1195, (Japan).* *^bEiko Engineering Co., Ltd., Hitachinaka, Ibaraki 312-0024, (Japan).* E-mail: asaoka.hidehito@jaea.go.jp

Interplay of surface stress and surface free energy determines the growth mode of heteroepitaxial layers. Therefore, surface morphology is sensitive to the presence of a third species on the surface, so-called surfactant. For instance, it is well known that the Stranski-Krastanow (SK) growth mode of Ge on Si can be suppressed with the surfactant. In such a surfactant-mediated epitaxy (SME), a flat Ge layer grows on Si while the surfactant floats up to the growth front and always covers the Ge surface. The stress in the Ge layer is relaxed by injection of dislocations into the Ge/Si interface.

Recently, Bi has attracted a lot of attention as a surfactant in Ge/Si(111) heteroepitaxy due to its ability to suppress Ge-Si intermixing and provide a chemical contrast between Ge and Si in a Scanning Tunneling Microscope (STM), allowing fabrication of self-organized

Ge/Si(111) nanostructures on the Si surface during STM measurement. In addition, low incorporation of Bi in the Ge layer allows to remove it from the flat Ge surface even after Bi-mediated growth.

We have focused on the stress and strain evolution during the initial stages of Bi adsorption on Si(111) 7x7 and Bi-mediated growth of Ge on Si(111). The stress and strain behavior and surface morphology were observed simultaneously by using real-time measurements of the substrate curvature and Reflection High Energy Electron Diffraction (RHEED). We find a clear difference in the surface stress between the clean Si(111) 7x7 surface and the Si(111) $\sqrt{3}\times\sqrt{3}$ surface covered with one monolayer of Bi (1 ML = 7.8×10^{14} atoms/cm²). Subsequent Ge deposition on Bi-adsorbed Si surface provides an increase in compress surface stress accompanied by simultaneous stress and strain oscillations. The compress stress is followed by clear stress relaxation at the critical coverage of 2 bilayers (1 BL = 2 ML) due to a variety of strain relief mechanism including injected misfit dislocation and increasing surface roughness on the Ge surface.

Keywords: epitaxy, heterostructure, stress

MS49.P21

Acta Cryst. (2011) A67, C540

Hydrothermal Synthesis of New $Er_xNi_xZn_{1-2x}O$ Nanomaterials and Their Physical Properties

Abdolali Alemi, Saeideh Jannesar Ahmadi, Aliakbar Khandar *Department of Inorganic Chemistry, Faculty of Chemistry, University of Tabriz, (Iran).* E-mail: Alemi.aa@gmail.com

Zinc oxide (ZnO) is a wide-gap semiconductor, which is transparent from the visible to infrared regions and is resistant to reductive ambient. Because of these properties, it is expected to be applicable as a material for transparent electrode and window layers of solar cells. Among the different methods of synthesis of ZnO nanostructures, the hydrothermal method is attractive for its simplicity and environment friendly conditions. In this research, New $Er_xNi_xZn_{1-2x}O$ based nanomaterials were prepared by hydrothermal method. $Zn(NO_3)_2\cdot 6H_2O$ ($x \ll 1$ mmol) and NaOH (0.6 g) were added to distilled water (60 mL), and stirred well for 20 min at room temperature. Afterwards, Ni ($NO_3)_2\cdot 6H_2O$ and Er_2O_3 with various stoichiometric ratio were added, and the mixture was transferred to a 100 mL Teflonlined autoclave. The autoclave was sealed, maintained at 180 °C for 48 h, and then cooled to room temperature. The white precipitate obtained was filtered and washed with ethanol and water. Powder XRD patterns indicate that the $Er_xNi_xZn_{1-2x}O$ crystals ($x = 0.00-0.04$) are isostructural with ZnO. SEM images show that co-doping of Er^{3+} and Ni^{2+} ions in the lattice of ZnO results in different nanorods and nanoparticle morphology. UV-Vis absorption and emission spectroscopy reveals mainly d-d electronic transitions of the Ni^{2+} ions in case of nanomaterials.

[1] R.E.I. Schrop, A. Madan, *Journal of Applied physics* **1989**, *66*, 2027-2031
[2] X. Chao, C. Lixin, S. Ge, L. Wie, Q. Xiaofei, Y. Yaqin, *Journal of alloys and compounds*, **2010**, *497*, 373-376.

Keywords: hydrothermal, semiconductor, solar cells

MS49.P22

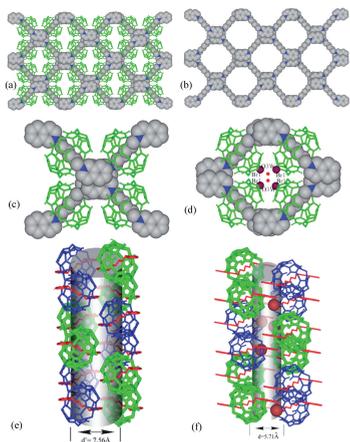
Acta Cryst. (2011) A67, C540-C541

A Novel Three Dimensional Framework Induced by $\pi\cdots\pi$ Stacking of 2,2'-(alkylene-1,6-diyl)diisoquinolinium from Q[6]-based Pseudorotaxane

Gang Wei,^a Zhi-Fang Fan,^{b,c} Xin Xiao,^b Yun-Qian Zhang,^b Sai-

Feng Xue,^b Qian-Jiang Zhu,^b Zhu Tao,^b ^aCSIRO Materials Science and Engineering, P.O. Box 218, Lindfield, NSW 2070, (Australia). ^bKey Laboratory of Macrocyclic and Supramolecular Chemistry of Guizhou Province, Guizhou University, Guiyang 550025, People's (Republic of China). ^cLiupanshui Normal College, Guizhou Province, 553004, People's (Republic of China). E-mail: gang.wei@csiro.au or sfxue@gzu.edu.cn

Q[n]-based supramolecular architectures which have intriguing structures or their potential applications in gas storage or absorption, catalysis, and optoelectronics, have been of great interest in the cucurbit(n)urils (Q[n]s) chemistry.[1-4] In present study a bromide salt of 2,2'-(hexane-1,6-diyl) diisoquinolinium (K6) was introduced to construct a novel framework induced by $\pi\cdots\pi$ stacking of 2,2'-(alkylene-1, ω -diyl)diisoquinolinium from Q[6]-based pseudo-rotaxanes. ¹H NMR spectra analysis was performed and confirmed the pseudorotaxane interaction model in which the 2,2'-(alkylene-1,6-diyl) diisoquinolinium guest threads into the cavity of Q[6] with the alkyl chain included inside the cavity and the two end isoquinolyl moieties protruding from the two opening portals. The crystal structure of the compound revealed that the assembled framework based on the Q[6]-2,2'-(alkylene-1,6-diyl) diisoquinolinium pseudo-rotaxanes have stoichiometries of $\{(K6)@(Q[6])\}^{2+}\cdot 2Br^{-}\cdot 7(H_2O)$ (1). The compound (1) has a novel three-dimensional framework constructed of two different channels – one containing stacked isoquinolyl moieties from the K6@Q[6] pseudorotaxanes and the other containing the bromide anions (See figure below). Absorption spectrophotometric and fluorescence spectroscopic analyses of the host-guest inclusion complex in aqueous solution found that the complexes were most stable at a host:guest mole ratio of 1:1. At this ratio, the complex has binding constants (K) $\sim 10^6$.



[1] J. Lagona, P. Mukhopadhyay, S. Chakrabarti, L. Isaacs, *Angew. Chem., Int. Ed.*, **2005**, *44*, 4844-4870. [2] X.-L. Ni, J.-X. Lin, Y.-Y. Zheng, W.-S. Wu, Y.-Q. Zhang, S.-F. Xue, Q.-J. Zhu, Z. Tao, A.I. Day, *Cryst. Growth Des.* **2008**, *8*, 3446-3450. [3] J.-P. Zeng, S.-M. Zhang, Y.-Q. Zhang, Z. Tao, Q.-J. Zhu, S.-F. Xue, G. Wei, *Cryst. Growth Des.* **2010**, *10*, 4509-4515. [4] H. Kim, Y. Kim, M. Yoon, S. Lim, S. M. Park, G. Seo, K. Kim, *J. Am. Chem. Soc.* **2010**, *132*, 12200-12202.

Keywords: framework, $\pi\cdots\pi$ stacking, pseudorotaxane

MS49.P23

Acta Cryst. (2011) A67, C541

Study of ZnO nanoparticles thin films deposited by spin-coating
 Alberto Alvarado-García,^a Hector Juárez,^b Mauricio Pacio,^b Godofredo García,^b Rene Perez,^b ^aCINVESTAV-IPN, programa de Nanociencias y Nanotecnología, Av. Instituto Politécnico Nacional # 2508, Col. San Pedro Zacatenco, C.P. 07360, México, D.F. (México). ^bCIDS-BUAP, Benemérita Universidad Autónoma de Puebla, Ciudad Universitaria Avenida San Claudio y 14 Sur, 72570, Puebla, (México). E-mail: jalvarado@cinvestav.mx

Thin films of zinc oxide nanoparticles, were deposited by spin coating method using glass and silicon as substrates. The obtained films were treated only at 100 °C to evaporate the solvent were the

nanoparticles stay after de deposition. The structural characterization was performed using XRD and SEM techniques in order to observe the differences of film deposition between the substrates. The ZnO thin films showed polycrystalline hexagonal structure when silicon was used as substrate and the other one shows a preferential deposition in 002 ZnO plane. UV-Vis characterization was used to analyze the optical transmittance of the thin films.

Keywords: films, ZnO nanoparticles, spin coating

MS49.P24

Acta Cryst. (2011) A67, C541-C542

Crystal Nucleation in X-ray Amorphous Natural Silicates

Yevgeny A. Golubev *Laboratory of Structural and Morphological Crystallography, Institute of Geology of Komi SC of Ural Division of RAS, Syktyvkar (Russia).* E-mail: golubev@geo.komisc.ru

The paper reports on crystal nucleation in natural x-ray amorphous silicates – noble and ordinary opals, volcanic and impact glasses. It is known, that opal is unstable and easy dehydrated for its high solubility in geological conditions. It forms crystalobalite, tridymite and quartz. The transformation occurs under widely known scheme: opal-A – opal-CT – chalcedony – quartz. Usually opals are mixture of amorphous and different degree ordered silica [1-3]. Classical suggestions about structure of noble opals – in regular intervals located spherical particles (size 100 – 500 nm) consisting of small globules in diameter of 10–50 nm, and larger globules are characteristic for opals with crystal structure elements (crystalobalite – tridymite), and in amorphous opals the sizes of primary globules are estimated in 20–50 nm. However in literature experimental data on electron-microscopic observations of internal structure of small particles in opals practically are not presented. Areas in the size of 10–30 nm with ordered atomic lattice as tridymite or crystalobalite crystals which are settling down in a disorder opal matrix in amorphous opals [4], are often observed.

We studied hypergenic noble opal from residual soil (Australia), opal of hydrothermal origin from deposit Raduzhnoe, Primorsky Region, Russia, and also ordinary opal from Spain by HRTEM, AFM and IR-spectroscopy.

By means of AFM images on regular basis packed of classical globular structure of noble opals with average diameter of globules 260 nm have been received. It is known, that opal globules are aggregated from smallest particles, however by means of AFM to us them to distinguish it was not possible. HRTEM images of the Australian opal show fragments of round particles which diameter makes from 4 up to 5 nm. These are primary globular particles which form globules observable by means of AFM.

In the Australian opal of the ordered lattice fringes to reveal it was not observed. HRTEM images show all, that the opal substance is amorphous state which shows characteristic irregular contrast. For comparison, in opal which on X-ray and IR-spectroscopic data can be carried to crystalobalite opal, HRTEM images show continuous amorphous matrix in which are included nanosized areas with crystal ordering. They are chaotically oriented from each other. Lattice distances correspond of crystalobalite. On contours it is shapeless areas though also polygonal forms in some cases are observed. In the given substance we deal mainly not with nanocrystal, and with ordered fragments of a silica network in disordered environment. It is crystal nucleation center.

The similar ordered areas in natural impact glass have been revealed. Fracture surfaces of the impact and volcanic glasses vary the sizes and the form of the isolations connected with glass nanoparticles.

The work is supported by The Program of Presidium RAS n. 17, Scientific School (7198.2010.5), RFBR 11-05-432a.