

**PS16.03.15 CRYSTAL GROWTH AND X-RAY STRUCTURE DETERMINATION OF A SERIES OF BIOLOGICALLY INTERESTING COMPOUNDS.** Janet M. Moloney, J. A. K. Howard, R. Cochlin, W. J. Feast. Chemistry Department, University of Durham, South Road, Durham, DH1 3LE, UK.

Crystallogenes has assumed increasing importance over the past decade. This is chiefly because many large molecules can be obtained as highly pure single crystals for diffraction studies<sup>1</sup>. Therefore, the weak intermolecular interactions which govern solid-state structure can be characterised accurately.

The crystallization of peptides and organic molecules is challenging due to the high percentage of lattice solvent, weak intermolecular interactions, pH dependency and limited solubility in aqueous systems in many cases. The synthetic methodology and background theory of crystal growth will be discussed fully.

The crystal structures of three related branched cyclopentadienyl compounds, determined from low temperature (150K) on a Siemens SMART diffractometer with area detector will be provided. The similarities of these compounds will be compared and details of solid-state packing will be provided.

#### References:

1. Crystallization of Nucleic Acids and Proteins; A Practical Approach. Ducruix, A.; Giegé, R.

**PS16.03.16 THE RESEARCH MBE SYSTEM WITH TWO MUTUALLY SYNCHRONIZED MODULATED MOLECULAR BEAMS.** Ann N. Sen'ko\*, Yu. A. Gel'man, A. A. Chernov, A. F. Sen'ko\*, Yu. M. Dymshits, Institute of Crystallography, Russian Academy of Science, Moscow, 117333, Russia, \* Grodno State University, Grodno 230023, Belarus

The 3 chamber setup to study growth processes of the A<sup>2</sup>B<sup>6</sup> compounds by MBE have been designed and built. There are 4 molecular beam sources based on effusion cells. Two sources are equipped with beam choppers. The stabilized chopping frequency varies from 10Hz to 1kHz. Reflected and reevaporated fluxes are detected by QMS and multichannel analyzer. Chopped beams are aimed at studying adsorption, sticking coefficients, surface nucleation and another MBE growth processes. Growth chamber is equipped by RHEED setup with a system to record the RHEED oscillations and Al<sub>2</sub>O<sub>3</sub> viewport which allows one to study how *in situ* sample illumination by the UV and visible light ( $\lambda=250-4500\text{nm}$ ) influences on the condensation process of thin films. The analytical chamber is equipped by RHEED, AES, QMS and duoplasmatron Ar<sup>+</sup> ion gun. The pumping system allows to achieve the residual gas pressure  $\sim 10^{-10}$  Torr.

**PS16.03.17 SYNTHESIS, STRUCTURE AND PROPERTIES OF Ba<sub>2</sub>Cu<sub>3</sub>Cl<sub>2</sub>O<sub>4</sub>.** Zhigang Zou\* and Hiroyuki Horiuchi, Mineralogical Institute, Graduate School of Science, University of Tokyo, 7-3-1 Hongo, Bunkyo-ku, Tokyo 113, Japan; Kunihiko Oka and Yoshikazu Nishihara, Electron physics section, Physical science division, Electrotechnical Laboratory, 1-1-4 Umezono, Tsukuba Ibaraki 305, Japan. \*Present address: Electron physics section, Physical science division, Electrotechnical Laboratory, 1-1-4 Umezono, Tsukuba Ibaraki 305, Japan.

Single crystals of a new barium copper oxychloride, Ba<sub>2</sub>Cu<sub>3</sub>Cl<sub>2</sub>O<sub>4</sub> were grown in a Ba(OH)<sub>2</sub>-KCl flux. Through the use of single crystal X-ray diffraction method, it was found that the compound has 4/mmm Laue symmetry. The crystal structure was refined in the space group I4/mmm, and lattice constants  $a=5.519(1)$ ,  $c=13.834(2)\text{\AA}$ ,  $Z=2$ ,  $wR=10.1\%$ . The structure is characterized by infinite layers of [CuO<sub>2</sub>] which are perpendicular to the c-axis. The closest Cu-Cu distance is  $2.7595(2)\text{\AA}$ . The Cu-O-Cu angles are  $90.64(3)$  and  $178.72(2)^\circ$ . Ba<sub>2+</sub> is coordinated to four O<sub>2</sub><sup>-</sup> ions of a square. The melting temperature of the Ba<sub>2</sub>Cu<sub>3</sub>Cl<sub>2</sub>O<sub>4</sub> is 715.0°C. This sample is an insulator. Magnetic properties of the single crystal were also measured.

## Crystal Growth IV Controlling & Predicting Crystal Morphology

**MS16.04.01 DETERMINATION OF THE CRYSTAL GROWTH UNITS BY GRAZING INCIDENCE X-RAY DIFFRACTION (GIXD) AND AFM.** David Gidalevitz<sup>†</sup>, Robert Feidenhans<sup>‡</sup> and Leslie Leiserowitz<sup>†</sup>. <sup>†</sup>Dept. of Materials & Interfaces, The Weizmann Inst. of Science, Israel; <sup>‡</sup>Dept. of Solid State Physics, Risø National Laboratory, Denmark.

Molecular crystal growth in solution takes place at the crystal-fluid interface. The local structure at this boundary plays a primary role in determining the composition, growth and habit of the crystal. In this study we apply GIXD to probe the surfaces of organic molecular crystals and their growth and dissolution interfaces.

We shall show that GIXD and AFM may be used to advantage in a complementary manner to determine close to atomic resolution the structure of the crystal-fluid interface of  $\beta$ -alanine and the  $\alpha$ -form of glycine during growth and dissolution, and so provide information on the crystal growth units. We addressed principal questions of crystal growth as to the molecular arrangement of the growing crystal surface and the structure of the "building units" which dock from solution onto the growing crystal surface.

The crystals of  $\beta$ -alanine (+H<sub>3</sub>NCH<sub>2</sub>CH<sub>2</sub>CO<sub>2</sub><sup>-</sup>) and the  $\alpha$ -form of glycine (+H<sub>3</sub>NCH<sub>2</sub>CO<sub>2</sub><sup>-</sup>) are composed of zwitterionic molecules interlinked by strong N-H...O hydrogen bonds into centrosymmetric bilayers. AFM revealed that both dissolution and growth proceed as step flow with an average minimal step height of  $7\pm 0.5\text{\AA}$  for  $\beta$ -alanine and  $5.7\pm 0.5\text{\AA}$  for  $\alpha$ -glycine, which corresponds to two molecular layers. The GIXD experiments on the (010) face of  $\beta$ -alanine on dissolution and the (010) face of  $\alpha$ -glycine on growth showed that during the X-ray data collection the surface termination of the crystals corresponded to bilayer surface truncation and did not change with time. The results described here are compatible with a model in which the molecules of  $\beta$ -alanine and glycine effectively dock or leave the crystal surface as cyclic hydrogen-bonded dimers.

**MS16.04.02 INTERDIGITATED CHIRAL ARCHITECTURES AT AIR/LIQUID INTERFACE** Ivan Kuzmenko, Ronith Buller, Meir Lahav, Leslie Leiserowitz, Dept. of Materials & Interfaces, The Weizmann Inst. of Science, Israel. K.Kjær, Dept. of Solid State Physics, Risø National Laboratory, Denmark. J. Als-Nielsen, Niels Bohr Institute, H.C. Orsted Laboratory, Denmark.

The design of supramolecular motifs requires complementarity and strong interactions between the molecules to "cement" the various building blocks. Among possible motifs, interdigitated bilayers are of importance since they constitute the walls of bio- and artificial membranes.<sup>(1)</sup>

A principal way for devising new materials involves the self-assembly of molecules at interfaces. Here we propose an approach for the design of crystalline interdigitated multilayers, composed of chiral molecules *A* and *B*, where *A* is a water-insoluble molecule composed of a long hydrocarbon chain and a hydrophilic headgroup of acid character and *B* is a water-soluble base. The acid-base complementarity should ensure alternating juxtaposition between the *A* and *B* units within each layer, whereas interdigitation of the hydrocarbon chains of the *A* molecules would promote multilayer arrangement. Molecular chirality of both components, yielding diastereomeric arrangements, is an additional variable. The phenylethylamine mandelates<sup>(2)</sup> were chosen as suitable templates satisfying the above criteria.

The chiral amphiphile, *R*-*p*-pentadecylmandelic acid, (*R*-C<sub>15</sub>-MA), when compressed over a 0.008M aqueous solution of chiral