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***In-situ* Observation of Elementary Growth Steps on a Protein Crystal, Surface Diffusion of Protein Molecules and Dislocations inside a Protein Crystal**
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To elucidate the mechanisms of defect formation in protein crystals, one has to observe in-situ 1) behaviors of individual protein molecules in the vicinity of crystal surface, 2) consequent movements of elementary growth steps, and 3) defects formed in a crystal.

For the process 1), we have applied a single molecule imaging technique, which is popular in the field of biological physics. Using fluorescence labeled lysozyme molecules, we have succeeded in observing individual lysozyme molecules diffusing in the vicinity of tetragonal lysozyme crystals in situ, for the first time. We found that the diffusivity of lysozyme molecules close to the crystal surface is 4 orders of magnitude smaller than that in a bulk solution.

For the process 2), we have combined laser confocal microscopy (LCM) with differential interference contrast microscopy (DIM), and succeeded in observing elementary growth steps (5.6 nm in height) non-destructively [1]. Using this LCM-DIM system, now we are observing bunching processes of elementary growth steps.

For the process 3), we have applied LCM-DIM and phase contrast microscopy, and succeeded in observing dislocations normal to an incident light and inclusions in-situ during growth.

[1] Sazaki G., et al., *J. Crystal Growth*, 2004, **262**, 536.
Keywords: protein crystallization, confocal laser scanning microscopy, near-field optical microscopy

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Interface Crystallography of a Growing Interface: KDP{101} and {100}
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In crystal growth from solution, the solvent is often found to determine the morphology of the crystal or the particular polymorph that is formed. Using in situ X-ray diffraction it is possible to determine the atomic-scale structure at a solid-liquid interface [1], and thus one can try to determine the microscopic origin of the solvent effect.

Here we present our results on the solid-liquid interface structure of the {101} and {100} faces of KDP (KH_2PO_4) in aqueous growth solutions as a function of the solution pH. The morphology of KDP crystals changes as a function of the pH, the largest effect coming from a decrease in growth velocity of the {101} face for non-stoichiometric conditions. Using the high-intensity X-ray beams from the ESRF synchrotron, we find remarkably large changes in the liquid ordering as a function of pH for the {101} face, while the {100} face is largely unchanged. For high pH, we observe crystalline pre-ordering of K-ions in the solution at the {101} face.

The KDP{101} face terminates in a positive K-layer, but our results indicate that an electrochemical interpretation of the interface is less correct than a crystallographic point of view.

[1] Vlieg E., *Surf. Sci.*, 2002, **500**, 458
Keywords: crystal growth, solid-liquid interface, surface X-ray diffraction

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X-ray Diffractometer for *In-situ* and *Real-time* Monitoring of MOCVD
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X-ray diffraction is a well-established, non-destuctive, standardless tool for the analysis of epitaxial structures. We will present a new X-ray diffractometer that enables *in-situ* and *real-time* monitoring of metal-organic chemical vapour deposition (MOCVD). X-ray diffraction is a promising technique for *in-situ* monitoring of MOCVD growth because reflection high-energy electron diffraction, most frequently used for molecular beam epitaxy, cannot be applied in the MOCVD chambers due to high ambient pressure.

Our diffractometer uses a conventional X-ray source, it has no parts mounted inside the growth reactor and precise adjustment of the samples is not necessary. Therefore, it can be easily attached to a standard MOCVD reactor without the reactor chamber having to be significantly redesigned.

We will report on *in-situ* X-ray measurements on the nitride epitaxial structures during their growth in a standard AIXTRON single wafer MOCVD system. Using our diffractometer we were able to measure the growth rate and the composition in less than 20 seconds. In addition we could monitor the thermal expansion during heating and the strain relaxation in the epilayer as soon as the critical thickness was reached.
Keywords: X-ray diffraction, MOCVD, *in-situ* X-ray diffractometer

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MS47 NOVEL MATERIALS UNDER HIGH PRESSURE
Chairpersons: Colin Pulham, Nancy Ross

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Synthesis of Nano-polycrystalline Diamond and Related Hard Materials using Multianvil Apparatus
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We synthesized pure polycrystalline diamond by direct conversion of graphite in multianvil apparatus, which turned out to be made of sintered nano-grains of cubic diamond and to possess very high Knoop hardness of 130-140 GPa [1]. Two distinct fine textures were recognized in TEM observations of the present diamond; granular particles with 10-20 nm and tabular crystals with dimensions of 100-200 nm in the elongated direction. The former crystals are interpreted to be formed via a nucleation and growth mechanism, while the latter parts may be formed by the two-step martensitic transitions [2].

Some mechanical tests have been conducted on thus synthesized polycrystalline diamonds, which exhibited significantly high abrasion resistance. Attempts to make sintered bodies of polycrystalline diamond with various forms of carbon, including carbon nanotubes, flurene, glassy carbon, etc. have been made using the present technique.

[1] Irifune T., Kurio A., Sakamoto S., Inoue T., Sumiya H., *Nature*, 2003, **421**, 599. [2] Sumiya H., Irifune T., *J. Mat. Sci*., 2004, **39**, 445.
Keywords: diamond, multianvil apparatus, hard material

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Combined Diffraction and Raman Studies on Incommensurately Modulated Host-guest Structures of Elements
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Since their discovery in Ba in 1999 [1], the incommensurate host-guest structures have been found in a series of elemental metals at high pressures [2], consisting of two interpenetrating components