KN06

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Crystalline molecular flasks

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Since our first report on a zeolite-mimic coordination network in 1994,[1] we have developed several robust coordination network complexes possessing large pores.[2-8] In some cases, the pore channels facilitate increased mobility and rapid diffusion of included guest molecules. Large organic molecules can easily enter into the pores via guest exchange. In this sense, the pore interior is a pseudo-solution state where chemical reactions may proceed as in a solution, yet can be directly analyzed by crystallography. Here, we show that single-crystal-to-single-crystal chemical reactions with large, common reagents proceed quite smoothly inside the pores of the network.[9] Taking advantage of the network's robust crystallinity, we succeeded in the acylation and ureidation of aromatic amines and imine formation from aromatic aldehydes within a single crystal. The pores of the network complexes thus serve as "crystalline molecular flasks". We also show a hemiaminal, a transient short-lived intermediate in the Schiff-base formation, can be trapped and directly observed by X-ray analysis in the crystalline molecular flasks.[10]

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Microtubule-Kinetochore Interactions

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During division the eukaryotic cell needs to accurately segregate its genetic material between daughter cells. This process involves the interaction of the microtubule mitotic spindle with special regions on chromosomes called kinetochores. Errors, which result in misplaced chromosomes, can lead to cancer or death. We have visualized the interaction of microtubules with two kinetochore components, the yeast Dam1 and the human Ndc80 complexes, using cryo-electron microscopy and image reconstruction.

The Dam1 kinetochore complex is essential for chromosome segregation in budding yeast. It is a ten-protein complex that self-assembles around microtubules, forming ring-like structures that move with depolymerizing microtubule ends, a mechanism with implications for cellular function [1], [2]. We have defined the architecture of the Dam1 complex, at about 30-Å resolution, both in its unbound and microtubule-bound states [3].

The Ndc80 complex is a key site of regulated kinetochore-microtubule attachment, conserved from yeast to humans. We have obtained a subnanometre-resolution reconstruction of the human Ndc80 complex bound to microtubules, and docked the crystal structures of the component proteins [4]. The Ndc80 complex binds the microtubule with a tubulin monomer repeat, recognizing α- and β-tubulin at both intra- and inter-tubulin dimer interfaces in a manner that is sensitive to tubulin conformation. Ndc80 complexes self-associate along protofilaments forming linear arrays.

Formation of both kinetochore complex oligomers is regulated by Aurora B phosphorylation, so that wrong attachments can be corrected. Our structures and biophysical and biochemical studies [5], [6] lead to mechanistic models of how the kinetochore machinery is capable of harnessing microtubule depolymerization for chromosome movement.

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Use of external fields in the melt growth of semiconductors

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Stationary and non-stationary magnetic fields have been widely applied for the melt growth of semiconductors in order to control the convection within the crucibles. Indeed when scaling up to industrial equipments, with melt masses above 200 Kg, the relatively large thermal gradients within the crucibles result in strong convective flows which ultimately degrade the quality of the semiconductor crystals. In some case, under very unfavorable conditions, even onset of turbulence was observed. Pronounced striations, incorporation of undesired impurities coming from the crucible/ambient and doping non-uniformity are the typical defects. In standard experimental setups, such as Czochralski or Bridgman furnaces, the magnetic fields provided by one or more electromagnetic coil(s) placed around the growth chamber, in correspondence of the crucible position, may effectively damp the convective motion. However, although capable of reducing the convection of the melt, this type of setup has hardly met the expectations of academic and industrial crystal growers due to the cost of the magnet(s) and the high energy consumption. As a matter of fact the use of magnetic field has so far been treated almost as a laboratory curiosity. Just in the case of very large melt amounts (typically CZ growth of silicon), where the elevated Rayleigh number leads to strong turbulence and negatively impacts the single crystal growth, the magnetic field found practical application.

At IKZ, in the frame of the KristMAG® consortium, an alternative approach was developed. In this case the resistive heaters of two CZ pullers and one VFG furnace were substantially modified in order to simultaneously provide heat and magnetic field to the melt [1, 2, 3]. This is possible by adopting a spiral configuration for the graphite heaters and by simultaneously feeding these coils with DC and AC currents. The DC current essentially determines the temperature set point, whereas the AC signal provides a so-called *travelling magnetic*

field (TMF). Indeed, by properly selecting the phase shift between the signals sent to three or more heaters around the crucible, the magnetic field may be made "travelling" along the crucible. In this way the normal growth parameters of the melt growth (pulling/solidification rate, temperature gradient, rotations) are enriched with new degrees of freedom, namely intensity, frequency and direction of the non-stationary magnetic field. By choosing the right field parameters, the crystal grower has the possibility of either stimulating or damping the melt convection, acquiring in this way a good control over transport phenomena in the liquid phase. This in turn provides an efficient control of the solid-liquid interface shape.
In this presentation the concept of travelling magnetic field and the necessary hardware modification will be presented. The results of TMF applied to Czochralski growth of silicon and Vertical Gradient Freeze of germanium and silicon will also be reported. These examples also show that the use of this magnet-heater ensemble provides bulk crystals of superior quality.

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Discovery of post-Perovskite at high pressure and its geophysical implications

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Recent developments in X-ray diffraction (XRD) measurements at the synchrotron radiation source, combined with laser-heated diamond-anvil cell (LH-DAC) techniques, enables the crystal structure determinations at ultrahigh-pressure and -temperature (*P-T*) conditions expected for the deep Earth. $MgSiO_3$ perovskite is known to be a primary mineral in the Earth's lower mantle below 660-km depth and the most abundant mineral inside the Earth, but its stability and possible phase transition to a denser structure in the lowermost mantle has long been a matter of debate because large seismic anomalies found in this region are not reconciled with the known properties of perovskite. Recently we discovered a novel phase transition from $MgSiO_3$ perovskite to post-perovskite through a drastic change in the XRD pattern at high *P-T* conditions near the base of the mantle around 2600-km depth [1, 2]. Crystal structure of post-perovskite was determined with the aid of computer simulations of atomic positions using the XRD pattern. Unlike perovskite, $MgSiO_3$ post-perovskite is a strongly anisotropic crystal; it has an orthorhombic symmetry (space group: *Cmcm*) with a SiO_6-octahedral sheet-stacking structure along the *b*-axis. It is isostructural with $UFeS_3$ and $CaIrO_3$, which are stable at ambient condition. The Mg^{2+} site in post-perovskite is smaller than in perovskite, resulting in a volume reduction of 1.0-1.5%. The calculated [3, 4] and measured elastic properties [5] of post-perovskite now explain the seismic-wave velocity structure in the lowermost mantle. The high positive pressure/temperature slope (Clapeyron slope) of the perovskite/post-perovskite transition boundary destabilizes the thermal boundary layer at the bottom of the mantle and remarkably enhances the mantle convection. Recent measurements of transport properties demonstrated that both electrical and thermal conductivities of post-perovskite are much higher than those of perovskite. The electronically highly conductive post-perovskite layer in the lowermost mantle enhances the electromagnetic coupling between solid mantle and liquid core, which possibly changes the Earth's rotation speed [6].

In addition, we are now able to perform XRD measurements up to 377 GPa and 5700 K, corresponding to the center of the Earth [7]. With such techniques, hcp (hexagonal-close-packed) structure has been found to a stable form of iron in the Earth's solid inner core (5100 to 6400-km depth). While the effect of impurities such as nickel and some light alloying element(s) remains to be examined, the knowledge of crystal structure of inner core material helps to predict physical properties and interpret seismic structures.

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KN10

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Exploration of the Protein Universe with High Throughput Structural Biology

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The landscape of structural biology has changed significantly over the past decade due to the overwhelming amount of novel sequence data generated from genome sequencing projects and the increased automation and robotics that facilitate protein production through crystallization to structure determination. These advances have unleashed unparalleled opportunities for re-evaluation of the size and diversity of the protein universe via explorations into new environments, such as the human microbiome and, in general, for addressing more challenging biological questions. For over a decade, the Joint Center for Structural Genomics (http://jcsg.org) has been at the forefront of developing tools and methodologies that enable the application of HTP structural biology to a broad range of biological investigations. For example, in the previous phases of the NIH PSI (http://www.nigms.nih.gov/initiatives/psi), we explored structural coverage of uncharted regions of the protein universe [1] as well asa single organism that enabled a complete structural reconstruction of the metabolic network of *Thermotoga maritima*.[2] As we embark on PSI: Biology, the JCSG is leveraging its HTP platform to take on challenging targets in stem cells and T cells that capitalize on our extensive experience to develop the best strategies to enhance chances of success. The emerging field of metagenomics has been particularly enlightening, where the human gut microbiome sequencing projects have already uncovered fascinating new families and expansions of known families for adaptation to particular environments. These high-throughput approaches can be applied to important biological problems not only in large consortia, such as the JCSG, but also in individual laboratories to tackle fundamental biological questions. Examples of the types and range of biological problems that are being tackled by PSI:Biology, as well as examples from my own laboratory on influenza virus and the search for a universal vaccine will be discussed [3]. The JCSG is located at The Scripps Research Institute, the Genomic Institute of the Novartis Research Foundation, U.C. San Diego, Sanford-Burnham Medical Research Institute, and SSRL/Stanford University, and supported by U54 GM094586, and P01 AI058113 and HHSN272200900060C.

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