

#### DIAMOND REFRACTIVE LENS FOR X-RAY FOCUSING

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Among other materials being used for X-ray refractive optics, diamond is one of the most attractive materials, because it has outstanding thermal properties and low absorption coupled with sufficiently high refraction index. In the present paper we report about first results on diamond refractive lenses manufacturing and experimental testing. To fabricate diamond refractive lenses a transfer molding technique has been employed, that is based on diamond growth on a pre-patterned silicon mould. Diamond films were produced by microwave plasma enhanced chemical vapor deposition (PECVD). The lenses were designed for focal length of 50 cm at the energy 9 keV. Experimental tests were performed at the ESRF ID15 (wiggler) and ID22 (undulator) beamlines using monochromatic, 'pink' and white X-ray radiation in the energy range 6-40 keV. Focusing in the order of 1-2 microns was achieved. To evaluate the effect of the diamond film microstructure (grain size, texture etc.) on the lens efficiency phase contrast imaging and diffraction techniques (SAXS and WAXS) were applied. The heatload test showed that diamond lens is capable of withstanding the full undulator beam without any damage. In view of future X-ray free electron lasers diamond refractive optics is a promising candidate to be stable under extremely powerful XFEL beams.

**Keywords:** X RAY REFRACTIVE LENSES, FOCUSING, CVD DIAMOND

#### NANOSTRUCTURED SUPERHARD BC<sub>2</sub>N MATERIAL BULKS SYNTHESIZED UNDER HIGH P-T CONDITIONS

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Newly introduced high-pressure phases of BC<sub>2</sub>N compound have extreme hardness, second only to diamond. It is expected that the superhard BC<sub>2</sub>N materials are more stable at high temperatures than diamond and will not react with ferrous metals during high-speed cutting. The synthesis of bulk samples of these superhard materials is an important practical step towards full-scale scientific characterization and engineering applications. We report here a high-pressure synthesis of well-sintered bulks of the superhard BC<sub>2</sub>N materials in samples of millimeter scale and in the form of a nanocrystalline composite. The nanostructured superhard BC<sub>2</sub>N material bulks were synthesized under high P-T conditions from amorphous phases of the ball-milled molar mixtures. Significant technological advantages of the nanostructured superhard BC<sub>2</sub>N materials include their greatly enhanced yield strength and fracture toughness.

**Keywords:** SUPERHARD B-C-N MATERIALS

#### ON-CHIP CRYSTALLIZATION BY FREE-INTERFACE AND GEL DIFFUSION

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Protein crystallization is most commonly accomplished by vapor diffusion method. In this method, at initial step one must combine protein solution and precipitant. However this step drastically changes solute composition, and in some case leads to aggregate amorphous precipitates. To circumvent this situation, it is worth taking account of slowly mixing protein solution with precipitants by, for example, dialysis or free-interface diffusion. However these methods require much protein solution in each trial and complicated manipulations.

In this study, we designed two type chips for protein crystallization. One chip utilizes free-interface diffusion. This chip contains the three grooves: Each groove has two set of 2 µl reservoirs connected with 100 µm width but different length channel. Another is gel-mediated diffusion chip. This chip contains the two grooves: Each groove has two set of reservoirs connected with a channel filled with gel. The reservoirs are 2 or 5 µl in size. These chips require only 2 µl protein solution at a minimum in each crystallization condition.

We tested and confirmed the crystallization on the chips by using hen egg white lysozyme, bovine liver catalase and *Synechococcus* PCC7942 fructose 1,6-bisphosphatase complex with its substrate 1,6-bisphosphate as model protein.

**Keywords:** BIOLOGICALMACROMOLECULES FREE-INTERFACE DIFFUSION GEL DIFFUSION

#### HIGH THROUGHPUT PROTEIN CRYSTALLIZATION IN 96 WELL MICROPLATES

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The three-dimensional structure of any protein plays a key role in understanding the exact function of macromolecules. One proven approach to structural information of proteins is based on X-ray diffraction of single crystals. In the past, automated crystallization was restricted, because a reliable hardware platform and well-suited microplates for high-throughput screening were missing.

In 1999 a collaboration between Greiner Bio-One, the Genomics Institute of the Novartis Research Foundation (GNF), San Diego (USA), the Max-Planck-Institute (MPI) and Protein Structure Factory (PSF), Berlin (Germany) has been started to solve bottlenecks in high throughput crystallography with a focus on individual requirements.

Unique 96 well plates with a standardized footprint enable high-throughput protein crystallization. The plate in use at MPI/PSF has 96 reservoirs and three corresponding crystallization wells. This allows checkerboard screening with up to 288 crystallization sets per plate to investigate optimal crystal growth, while the plate at GNF has a 1 to 1 configuration with a low profile to minimize storage space under optimized handling conditions.

Both systems allow sitting drop vapor diffusion crystallography at reduced costs. A huge storage system and a pipetting device based on solenoid ink-jet technology enables to set up a complete plate with 96 crystallization conditions in less than 3 minutes. All crystallization wells are inspected in regular intervals with an automated camera-based detection system.

This presentation will address the specific requirements for high throughput crystallization in vapor diffusion protocols. In addition an outlook will be given to new plates for crystallization under oil.

**Keywords:** HIGH THROUGHPUT CRYSTALLIZATION, SITING DROP, AUTOMATION