important resource for the international research community. Currently there are two operational undulator beamlines: 24ID-C - fully tunable in the energy range from 6 to 22keV (cover most element edges for phasing) and 24ID-E - fixed energy at ~12.66keV (optimized for Se SAD experiments). These operational beamlines are currently open to NE-CAT members and general APS users.

Both undulator beamlines are fully equipped with state-of-the-art instrumentation for its users. MD2 microdiffractometers installed at both the beamlines provide very clean beams from 5 microns to 100 microns in diameter and have exceptional sample visualization systems capable of visualizing micron-sized crystals with extreme clarity. Large-area CCD-based ADSC Quantum 315 detectors at both beamlines not only provide the best diffraction data, but also make it possible to resolve large unit cell dimensions. Both beamlines are equipped with ALS style robotic sample mounting systems, thereby making screening of large numbers of crystals much faster and less effort intensive. A new software suite RAPD provides data collection strategies and quasi-real time data integration and scaling through 128 core computing cluster. A simple automated MR/SAD pipeline for rapid structure solution is implemented. Users of the beamlines are supported by experienced resident crystallographers and have access to a full suite of data processing and structure analysis. A fully equipped chemistry laboratory and cold-room are also available for users.

NE-CAT facility is used to focus on NE-CAT research on structural studies involving technically challenging crystallographic projects. In order to meet these needs several novel hardware and software ideas are implemented. A summary of beamline capabilities, technology, scientific highlights and details of availability will be presented.

NE-CAT maintains a website at http://necat.chem.cornell.edu/.

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Keywords: macromolecular crystallography, microdiffraction, synchrotron X-ray instrumentation

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Recent developments in modeling single molecule imaging by x-ray free electron lasers

G. Faigel, G. Bortel, Z. Jurek, M. Tegze, *Research Institute for Solid State Physics and Optics, H-1525 Budapest, POB 49. (Hungary).* E-mail: gf@szfki.hu

We study the possibility of imaging small cluster of atoms or single molecules [1] by intense and short x-ray free electron laser pulses. Lately, the first hard x-ray free electron laser (LCLS) came into operation. Experiments on this line have been started. In the first experiments 20-50 nm resolution is the best expectation, but ultimately atomic resolution is the goal. In the nm scale the radiation damage is not a problem within the time window of the probe pulse. However, going into the few Å resolution range, the motion of atoms in the samples during the x-ray pulse has to be taken into account. The other difficulty in single molecule imaging is that the 3D diffraction pattern has to be compiled from the measured 2D patterns with unknown orientation. However, the determination of the orientation of 2D patterns is hindered by the low statistics of individual measurements. This can be circumvented by sorting the patterns into classes, in which every particle has the same orientation. We studied both of the above problems. Using our special molecular dynamics tool we model the motion of atoms in biological specimens, and we give limits to pulse parameters [2,3]. Concerning the classification problem, we worked out a special quality threshold approach, which allows the classification of realistic large data sets within a practical time scale [4].

[1] R. Neutze et al., *Nature*, **2000**, *406*, 752. [2] Z. Jurek, G. Faigel, M. Tegze, *European. Physics Journal. D*, **2004**, *29*, 217. [3] Z. Jurek, G. Faigel, *European Physics Letters*, **2009**, *86*, 68003. [4] G. Bortel, G. Faigel, M. Tegze, *Journal of Structural Biology*, **2009**, *166*, 226.

Keywords: XFEL, imaging

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Bringing microfocus beam and improved sample environment to MX users at Diamond

David R. Hall, Jun Aishima, Lucia Alianelli, David Butler, Graham Duller, Ralf Flaig, Richard Fearn, Paul Gibbons, Martin Gilbert, Mic Harding, Lee Hudson, Katherine McAuley, Ronaldo Mercado, Jerry Nash, James Nicholson, Brian Nutter, James O'Hea, Geoff Preece, Adam Prescott, Pierpaolo Romano, Juan Sanchez-Weatherby, James Sandy, Kawal Sawhney, Thomas Sorensen, Adam Taylor, Tim Whitewood, Mark Williams, *Diamond Light Source, Diamond House, Chilton, Oxon, OX11 0DE (UK).* E-mail: david.hall@diamond.ac.uk

The phase I macromolecular cystallography (MX) beamlines at Diamond Light Source [1] are continually undergoing improvement. To bring together many of the developments by Diamonds scientific and technical staff a major upgrade project has been undertaken to incorporate the best of the original sample environment, goniometry and end-station alongside recent developments and to introduce microfocus optics to the phase I MX beamlines. To this end the end-station has been redesigned to accommodate all the new features and provide a stable, adaptable platform for the future.

One of the biggest changes to the beamlines is the addition of tools to facilitate mini- and micro-focus beams. An aperture based system has been incorporated which allows masking of the incoming beam to 5 - 20 microns. In addition, beam microfocusing at discrete energy ranges has been added by the use of compound refractive lenses. This gives a focussed beam of approximately 10 x 5 microns (h x v) with a flux density of ~5 x 10^{15} ph/mm^2/sec at 12.7 keV. These two upgrades individually and in combination bring a new dimension to the experiment types possible for users of Diamonds MX phase I beamlines.

For the sample environment we have retained the excellent goniometer and shutter which combined with the stable beam provided by the machine has to date given superb data quality yet added improved sample centering stages to give more accurate and quicker sample centering. In the near future *in-situ* plate screening and data collection will also be possible in this set-up. The goniometer and sample environment have been reconfigured and designed to accommodate the recently installed Pilatus 6M detector on I03 and the mini-kappa crystal positioning system with no collisions when fully open through full rotation when using standard SPINE pins. The end-station will continue to be combined with the fast and reliable ACTOR robot system with rapid sample pre-centering via an industrial vision system incorporated into the end-station. Sample visualisation is achieved with an improved on-axis viewing system which has a fast zoom, a stable mounting system and the possibility to upgrade cameras readily.

To assist investigations of sample environment on room temperature crystals a new support has been developed which allows the easy exchange by users from the cryojet, used for traditional low temperature data collection, to the humidity controller I (HCI) for investigations of crystal quality and data collection at room temperature and the effect of dehydration on diffraction quality.

First results from these advances on the recently commissioned set-up on I04 will be presented. Beamlines I03 and I02 will upgraded this year with the same developments.

[1] http://www.diamond.ac.uk/Home/Beamlines/MX.html

Keywords: microbeam, macromolecular crystallography, beamline

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The new neutron single crystal diffractometer "BioDiff" for proteins at FRM II

Andreas Ostermann,[a] Tobias. E. Schrader,[b] Michael Monkenbusch,[c] Bernhard Laatsch,[d] Philipp Jüttner,[a] Winfried Petry,[a] Dieter Richter,[c,b] [a]*Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II), Technische Universität München, D-85747 Garching.* [b]*Forschungszentrum Jülich GmbH, Jülich Centre for Neutron Science – FRM II, D-85747 Garching.* [c]*Forschungszentrum Jülich GmbH, Institute for Complex Systems, D-52425 Jülich.* [d]*Forschungszentrum Jülich GmbH, Zentralabteilung Technologie, D-52425 Jülich*. E-mail: Andreas.Ostermann@frm2.tum.de

Hydrogen atoms play an important role in many biological processes. Especially hydrogen atoms in polarized bonds are often involved in enzymatic catalysis. These hydrogen atoms take part in the substrate binding process and are essential for proton transfer reactions during the catalysis. Therefore the knowledge about the protonation states of amino acid residues in the active centre of proteins is crucial for the understanding of their reaction mechanisms. However, hydrogen atoms, especially rather flexible ones, are often barely detectable in X-ray structure determinations of proteins. On the other hand, hydrogen atoms are clearly visible in neutron crystallography experiments even at moderate resolutions (d_{min}<2.0Å).

The new neutron single crystal diffractometer "BioDiff" has finished its final construction phase. The instrument is a joint project of the Forschungszentrum Jülich (FZJ/JCNS) and the Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II). "BioDiff" is especially designed to collect data from crystals with large unit cells. The main field of application is the structure analysis of proteins, especially the determination of hydrogen atom positions.

By using a highly orientated pyrolytic graphite monochromator the diffractometer is able to operate in the wavelength range of 2.4Å to about 5.6Å. Higher order wavelength contaminations are removed by a neutron velocity selector. To cover a large solid angle and thus to minimize the data collection time the main detector of "BioDiff" consists of a neutron imaging plate system in a cylindrical geometry. A Li/ZnS scintillator CCD camera is available for additional detection abilities. The main advantage of this instrument is the possibility to adapt the wavelength to the size of the unit cell of the sample crystal while operating with a clean monochromatic beam that keeps the background level low. First user operation of the instrument is anticipated to start around autumn 2011.

Keywords: neutron, diffractometer, protein

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XRD analysis of human dental tissues using synchrotron radiation

M. V. Colaço,[a] R.C. Barroso,[a] I.M. Porto,[c] R.F. Gerlach,[d] F.N. Costa,[b] D. Braz,[b] R. Droppa Jr.,[e] [a]*Physics Institute, State University of Rio de Janeiro, Brazil,* [b]*Nuclear Instrumentation Laboratory/ COPPE,* [c]*Department of Morphology, State University of Campinas,* [d]*Department of Morphology, Stomatology and Physiology, University of São Paulo,* [e]*Natural and Human Sciences Center, Federal University of the ABC Region.* E-mail: mvcolaco@gmail.com

The mineral phase of human tooth enamel and dentin was identified as a calcium phosphate with an apatite structure as early as 1926 using X-ray diffraction, namely as hydroxylapatite (HAp - $Ca_{10}(PO_4)_6(OH)_2$). The discussions on details of the apatite and tooth problems have induced continuous studies employing new and more refined instruments in an endeavor to find specific phases and to answer specific questions on the apatite problem [1-3]. In the present work, the crystallinity and average crystallite size of enamel, dentin and circumpulpal dentin from five healthy human third molar teeth were analysed using synchrotron X-ray powder diffraction. All the measurements were carried out at D12A-XRD1 beamline in the National Synchrotron Light Laboratory (LNLS), Campinas, Brazil. This study was approved by the Committee of Ethics in Research (FORP/USP 2003.1.1329.58.2), according to the Resolution 196/96 of the National Commission of Ethics in Research.

The powder diffraction patterns were collected over an angular range from 20º to 52º in 2θ with statistical uncertainty smaller than 2% for the scattering count. In order to allow an estimation of the wavelength and zero shift of each experiment, SRM 676a (alumina powder) reference sample was also run. The diffraction patterns were analysed using the Rietveld method, in which the structural parameters describing the dominant crystalline phase, HAp, were refined (ICSD CIF 9011092). The Rietveld refinements were carried out for the five specimens using GSAS software (R_{wp}<10%). The peak profiles were fitted with pseudo-Voigt functions and the background was described by the Chebyshev function of the first kind.

The lattice parameters were refined and the best peak shape was found to be a Lorentzian with slight asymmetry. In enamel the hexagonal lattice parameters were found a=9.4463(39)Å and c=6.8848(51)Å. Atom positions and bond lenghts also were refined. The average crystallite size measured by the diffracting planes was calculated using the Debye-Scherer equation. For the enamel, the crystallite size was 28 nm. For dentin and circumpulpal dentin, the values found were 21nm and 16nm, respectively. These data suggest that average crystallite size increases from circumpulpal dentin to enamel.

It is well-known that the shape of the profiles of diffraction depends on the spectral contribution of X-ray source, geometric parameters of the experimental setup and the characteristics of the material microstructure (crystallite size and microstrain effects). The influence of the first two factors in the broadening of diffraction peaks could be minimized through the use of synchrotron radiation and the adoption of high-precision diffractometer. Therefore, in this work, the instrumental effects were minimized and the broadening of the peaks is predominantly due to microstructural characteristics of the dental tissues.

[1] P. Anderson, J.C. Elliott, U. Boze, S.J. Jones *Archives of Oral Biology* **1996**, *41*, 281-290. [2] R.M. Wilson, J.C. Elliott and S.E.P. Dowker *American Mineralogist* **1999**, *84*, 1406-1414. [3] M. Al-Jawada, A. Steuwer, S.H. Kilcoyne, R.C. Shore, R. Cywinski, D.J. Wood *Biomaterials,* **2007**, *28*, 2908-2914.

Keywords: tooth, diffraction, rietveld

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Mayaro virus non structural protein 3 macro domain, via powder diffraction on a single urchin like crystal

Yves Watier[a], Nicolas Papageorgiou,[b] Coutard Bruno,[b] Lantez Violaine,[b] Gould Ernest A.,[b] Fitch Andrew N.,[a] Wright Jonathan P.,[b] ; Canard Bruno,[b] Margiolaki Irene,[c] [a]*European Synchrotron Radiation Facility, ESRF, BP-220, 38043 Grenoble, FRANCE,* [b]*Architecture et Fonction des Macromolécules Biologiques, CNRS and Universités*