[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

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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

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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.

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

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