A4 - Structure and Properties of Bio Materials

A4 - O1

IN-SITU SYNCHROTRON STUDIES OF TEXTURE IN WOOD DURING MECHANICAL TESTS

J. Keckes[1], I. Burgert [2], M. Müller [3], K. Kölln [3], S. V. Roth [4], S. E. Stanzl-Tschegg [5], P. Fratzl[2]

[1]Erich Schmid Institute for Materials Science, Austrian Academy of Sciences and Institute of Metal Physics, University of Leoben, Austria
[2]Max-Planck-Institute of Colloids and Interfaces, Department of Biomaterials, Potsdam, Germany
[3]Institute for Experimental and Applied Physics, University Kiel, Germany
[4]European Synchrotron Radiation Facility, Grenoble, France
[5]Institute of Meteorology and Physics, University of Agricultural Sciences, Vienna, Austria

The exceptional mechanical properties of biological materials reside in their complex architecture at all hierarchical levels supported by specific molecular mechanistic phenomena. In order to understand the structure-property relationship in wood, in-situ synchrotron diffraction studies on individual wood cells and on wood foils combined with tensile tests were performed at ID1 and ID13 beam-lines of ESRF, respectively.

The wood foils of the dimensions 5 x 50 x 0.2 mm and the individual wood cells of about 20 μm in diameter and 500 μm in length were strained in tensile stages under various strain rates monitoring strain-stress development and collecting x-ray diffraction patterns using a 2D CCD detectors. For the experiment on individual cells, a very precise piezoelectric tensile stage with simultaneous stress/strain control was used while the samples were examined with beam of 2 m [1].

The diffraction data obtained from both wood foils as well as from wood cells demonstrated significant changes in texture due to straining. By relating the mechanical data with the texture information from cells and from foils, it was possible to separate deformation mechanisms inside the cell-wall from those mediated by cell-cell interactions. The data indicate the presence of a dominant recovery mechanism in the cell-wall which reforms the amorphous matrix between helical cellulose microfibrils, recovering its mechanical function. This mechanism dominates to the tensile behaviour of different wood tissues. Moreover, the comparison of the mechanical and the microstructural results from various wood types allowed to draw relevant general conclusions regarding the role of different microstructural features of wood on its mechanical behaviour as well as to deduce wood architecture units progressively optimised during evolution.

[1] J. Keckes, I. Burgert, K. Frühmann, M. Müller, K. Kölln, M. Hamilton, M. Burghammer, S.V. Roth, S. Stanzl-Tschegg & P. Fratzl (2003), Cell-wall recovery after irreversible deformation of wood, *Nature Materials* **2**, 811-814.

A4 - O2

DEFORMATION MECHANISMS AT THE FIBRILLAR LEVEL IN BONE

***H. S. Gupta[1], W. Wagermaier[1], P. Roschger[2], Sergio S. Funari[1] and P. Fratzl[1]**

[1]Biomaterials Department, Max-Planck-Institute of Colloids and Interfaces, MPIKG-14424, Potsdam, Germany
[2]Ludwig Boltzmann Institute of Osteology, Hanusch Hospital and Hospital for Traumatology, Meidling, Vienna, Austria

Introduction: The deformation mechanisms leading to fracture in bone involve structural events at different levels of the hierarchy simultaneously, including but not limited to microcracking between fibrils, lamellar pullout, and sub-micron level deformation mechanisms. At the level of 10 – 100 nanometers, bone material consists of Type I collagen fibrils impregnated with poorly crystalline mineral (hydroxyapatite) particles. The combination of the ductile organic and the brittle inorganic material results in both high stiffness and high toughness, through as yet incompletely understood fracture and energy absorption mechanisms. The measurement of the strains in the building blocks – like collagen fibrils and molecules, mineral particles, and assemblies of the above – are essential in understanding the local mechanical environment in the extracellular matrix, which serves to signal for anabolic or catabolic changes (modeling and remodeling) of bone.

Purpose: To determine how external stresses and strains are coupled to submicroscopic deformation mechanisms at the fibrillar level in parallel fibered bone and partially mineralized turkey leg tendon (MTLT).

Approaches: Time-resolved small angle X-ray diffraction combined with *in-situ* tensile testing on parallel-fibred bone (from the periosteum of bovine femur), with 8 keV synchrotron radiation at the A2 (Soft Condensed Matter Research) beamline (HASYLAB, Hamburg).

Results: Parallel fibred bovine bone deforms elastically up to 1 – 1.5 %, but subsequently (above the elastic yield point) shows reduced stiffness and increasing damage. While the fibril extension is proportional to the extension of the total tissue during the whole deformation, the strain in the fibril remains less (1/3 to 1/2) of the tissue strain. Therefore, some of the deformation must occur by slippage between fibrils, presumably within a proteoglycan-rich extra-fibrillar matrix. While the much lower mineralized MTLT shows a similar behavior for small strains, it behaves differently above the yield point, with a fraction of the fibrils relaxing back to the unstressed state while others show much more extensibility before fracture. This bimodal behavior is probably related to the inhomogeneous mineralization of the fibrils inside MTLT.

Conclusions: Deformation mechanisms of the bone material involve an elastic deformation of fibrils as well as a component of extra-fibrillar deformation in the surrounding matrix, which can be quantified by *in-situ* tensile testing combined with synchrotron small angle X-ray scattering.

Supported by Grant FWF P143.

A4 - O3

STRUCTURE AND INTERACTIONS OF WEAKLY ORDERED LIPID MODEL MEMBRANES

Georg Pabst, Michael Rappolt, Heinz Amenitsch, and Peter Laggner

Institute of Biophysics and X-ray Structure Research, Austrian Academy of Sciences, Schmiedlstraße 6, A-8042 Graz, Austria

We have developed a global x-ray data analysis technique, which accounts for the quasi-long range order and diffuse scattering displayed by phospholipid membranes [1, 2]. Such systems are frequently taken as mimics for biological cell membranes. With our method we are able to determine structural parameters, such as the membrane thickness, and fluctuations that depend on the bilayer bending rigidity. We demonstrate the applicability of the method on various lipid/water systems including phase transition phenomena and give a comparison and outlook to surface diffraction on solid supported lipid films.

[1] G. Pabst, M. Rappolt, H. Amenitsch, and P. Laggner, Phys. Rev. E 62, 4000 (2000)-

[2] G. Pabst, R. Koschuch, B. Pozo-Navas, M. Rappolt, K. Lohner, and P. Laggner, J. Appl. Cryst. 36, 1378 (2003).

A4 - O4

PROTEIN POLYCRYSTALLOGRAPHY

R. B. Von Dreele

IPNS/APS, Argonne National Laboratory, Argonne, IL 60549 USA
e-mail: vondreele@anl.gov

The application of powder diffraction to polycrystalline proteins has seen a number of significant advances. Faster data collection techniques have reduced the sample size requirement to less than 1mg and allowed acquisition of high quality data in < 30sec permitting *in situ* exploration of protein crystallization, reactions and radiation damage effects. Recent developments in data analysis include the use of multiple data sets in combined stereochemically restrained Rietveld refinements and Pawley refinements to enhance the information content of the powder data and possibly provide a route to protein structure solution.

A4 - O5

RECENT ADVANCES IN PROTEIN POWDER DIFFRACTION STUDIES AT ESRF

Irene Margiolaki, Jonathan P. Wright, Andrew N. Fitch and Robert B. Von Dreele

European Synchrotron Radiation Facility (ESRF), BP220, F38043 Grenoble Cedex 9, France and Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439

Obtaining the atomic structure of large macromolecules like proteins depends on the availability of good quality single crystals. Following the recent reports of crystal structure refinement (Von Dreele, 1999) and solution (Von Dreele, 2000) of some small protein structures from powder diffraction data, the requirement for a single crystal might be relaxed. Since many materials of interest do not readily form single crystals, the availability of the powder technique widens the spectrum of samples which might be characterised crystallographically. Unfortunately, the collapse of three-dimensional reciprocal space into a one-dimensional powder diffraction pattern leads to a catastrophic loss of information. There is not only the usual phase problem, owing to significant peak overlap it is frequently not even possible to determine the intensity of individual Bragg reflections, but only their sum. Nevertheless, powder diffraction data gives a range of complementary information which can be more difficult to obtain from a single crystal. The peak shapes depend on the microstructure of the material, accurate unit cell parameters can easily be determined and the sample generally survives under nastier conditions.

The powder diffraction technique has developed dramatically in the last 20 years, however, the application to macromolecular crystallography remains in its infancy. Initially, we identified turkey egg-white lysozyme (TEWL) as a good candidate material for a trial powder diffraction study for several reasons. Owing to the similarity to hen egg-white lysozyme (HEWL), a great deal is known about the system already, although TEWL will hopefully be more representative of a typical microcrystalline sample in comparison to HEWL, which readily forms large crystals of excellent quality. We would like to know if it is possible to exploit molecular replacement techniques with powder data: the TEWL structure was originally solved in this way. Furthermore, we aim to illustrate the kind of complementary information which can be derived simply from powder data when a sample goes through a catastrophic phase transition. We report refinements of the room temperature crystal structure from powder data, the variation of the unit cell parameters with pH of the precipitation medium and an investigation into the processes occurring when the sample is frozen. Finally, recent results from a series of X-ray powder diffraction experiments in different systems such as insulin, trypsin and conclavin-A will be presented.

Von Dreele, R. B. (1999) J. Appl. Cryst. **32**, 1084-1089.

Von Dreele, R. B. (2000) Acta Cryst. D**56**, 1549-1553.

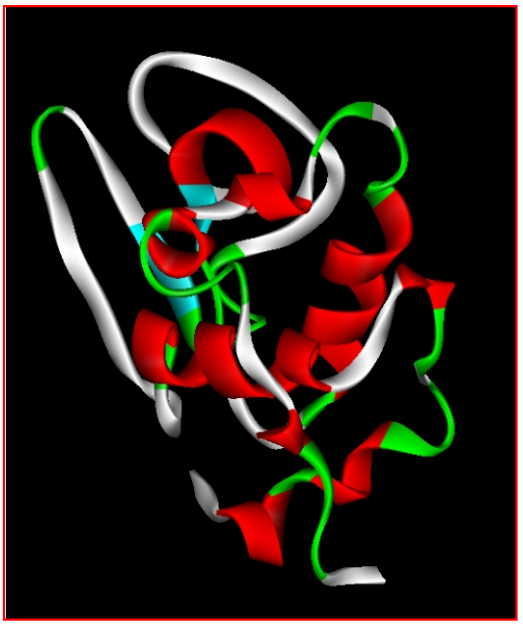

Figure 1. Refined conformation of TEWL at 295 K (pH 6.0) illustrated as ribbon.