from different complex materials with scanning microbeam SAXS/WAXS. Results from the imaging of nanostructural parameters such as shape, size and orientation of nanoscale inhomogeneities in bone and other hierarchical biocomposites are presented. Moreover, a unique combination of *in-situ* bending deformation with X-ray nanobeam scanning is demonstrated for single carbon fibres.

Keywords: bone, carbon materials, small-angle scattering

MS48.27.2

Acta Cryst. (2005). A**61**, C64

Engineered Bone from Bone Marrow Stromal Cells: a Structural Study by an Advanced X-ray Microdiffraction Technique

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One of the most recent therapeutic strategies for the reconstruction of damaged large bony segments includes the tissue engineering approach. It takes advantage of the patient's own cells, which are isolated, expanded in vitro, loaded onto a bioceramic scaffold and reimplanted into the lesion site. Bone marrow stromal cells (BMSC) are the most commonly used cell type.

A structural characterization of the engineered bone is largely desirable. An important point is to evaluate if the BMSC extracellular matrix deposition on a bioceramic scaffold recapitulates the ontogeny of the natural bone development. Moreover the investigation of the interaction between the newly deposited bone and the scaffold results particularly interesting. Indeed the chemistry and the geometry of the scaffold used to deliver BMSC in the lesion site determine spatial organization of the new bone and the bone-biomaterial integration.

We investigated for the first time the local interaction between the newly formed mineral crystals in the engineered bone and the biomaterial by means of microdiffraction, using a set-up based on an X-ray waveguide. We demonstrated that the newly formed bone is well organized inside the scaffold pore, following the growth model of natural bone, and that there is a good adhesion with the scaffold. Combining Wide Angle (WAXS) and Small Angle (SAXS) X-ray Scattering with high spatial resolution, we were able to determine the orientation of the crystallographic c-axis inside the bone grains, and the orientation of the mineral crystals and collagen micro-fibrils with respect to the scaffold. Moreover from a quantitative analysis of both the SAXS and WAXS patterns the grain size appears to be compatible with the model for early stage mineralization.

Keywords: SAXS, WAXS, bone mineralization

MS48.27.3

Acta Cryst. (2005). A**61**, C64

Polychromatic Microdiffracion Measurements of Mesoscale Structure and Dynamics

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Polychromatic x-ray microdiffraction combined with differential aperture microscopy is a powerful new method for studying the local crystallographic structure of materials. This approach extends single-crystal methods to virtually all materials including materials characterized by heterogeneity at the atomic *and* mesoscopic length scales. Defects such as grain boundaries, surfaces, precipitates, second phases, strain, dislocations, vacancies, interstitials, site substitutions and other disruptions of perfect periodicity all have signatures best studied using single crystal methods. Here we describe emerging x-ray microbeam techniques that exploit "single-crystal like" x-ray diffraction measurements on subgrains in typical polycrystalline materials. We show how polychromatic and tunable monochromatic measurements on small sample volumes can bring single-crystal techniques to real materials and reveal their atomic and mesoscopic defect structures. This emerging revolution in materials science is certain to address long-standing issues of materials behavior.

Keywords: phase, texture, strain

MS48.27.4

Acta Cryst. (2005). A**61**, C64

SR X-ray Microdiffraction Systems at SPring-8: Present Status and Applications

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In Hyogo beamline of SPring-8, we are developing research programs using an x-ray microbeam for applications to the wide range of science. Two types of the x-ray microbeam system have been developed; one is for a high spatial resolution and the other is for a high angular resolution. The former uses a Fresnel zone plate (FZP) for the x-ray focusing element, and the sub-micrometer beam is available [1]. By adopting a narrow slit in front of the FZP, a microbeam with a relatively small angular divergence can be also available [2]. The system is used, for example, for measurements of strain distribution in laser diodes with a è-2è diffractometer and structural analysis of polymers with a diffractometer using imaging plate detectors. The smallest beam size ever achieved is 70 nm in FWHM at 10 keV. The latter system uses a total reflection focusing mirror with a bent-cylindrical shape and the microbeam possesses very a small angular divergence of several arcsec with the beam size of a few micrometers [3]. The system is used for the estimation of local crystallinity of various semiconductor crystals for electronic devices. The both systems are opened for many kinds of users.

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Keywords: synchrotron radiation instrumentation, synchrotron X-ray diffraction, X-ray microanalysis

MS48.27.5

Acta Cryst. (2005). A**61**, C64-C65

Hard X-ray Nanoprobe with Refractive X-ray Lenses

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A hard x-ray nanoprobe based on refractive x-ray lenses allows one to apply hard x-ray analytical techniques, such as diffraction or fluorescence analysis, with high spatial resolution. This is particularly useful to investigate heterogeneous samples in materials, environmental and life science.

Using nanofocusing lenses (NFLs) [1-2], a nanobeam with a lateral extension down to 50 x 50 nm^2 is currently feasible at third generation synchrotron radiation sources in the hard x-ray range. The beam divergence of about 1 mrad is sufficient for many diffraction experiments. The beam size can be expected to be reduced to below 10 nm in the future [3].

We report on nanodiffraction experiments in materials science performed at beamline ID-13 of the ESRF. The coherence properties of the nanobeam are discussed in view of diffraction from small objects with coherent radiation.

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Lett., 2005, **94**, 054802.
Keywords: X-ray nanobeam, nanodiffraction, refractive X-ray lenses

MS49 Charge Spin and Momentum Densities in Material Science

Chairpersons: John Charles Spence, Brummerstedt Iversen

MS49.27.1

Acta Cryst. (2005). A**61**, C65

High-Energy Synchrotron Radiation for Charge Density and Materials Science Experiments

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Recently it has been shown that high-energy synchrotron radiation is an excellent tool for the measurement of charge densities, because there is no significant affection of the data by absorption and extinction in most practical cases [1,2]. Thus, the enhancement of the data quality compared to 'low-energy' data sets now allows detailed comparisons between experimental and theoretical charge densities, even in the case of 'new materials' like high-Tc superconductors [3].

On the other hand high-energy synchrotron radiation is also very useful for 'classical' materials science experiments, e.g. texture or stress and strain analyses, because of the large intrusion depth, i.e. the possibility of studying not only academic but also 'realistic' samples (size). GKSS is currently building up two high-energy materials science beamlines at DESY, Hamburg, Germany. The concepts of the beamlines will be presented here. Both will be equipped with materials science diffractometers, which can also be used for charge density studies.

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Keywords: synchrotron radiation experimental, charge density, materials science

MS49.27.2

Acta Cryst. (2005). A**61**, C65

Charge Density Studies of Ultra High Resolution Protein Structures

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The recent advances in synchrotron radiation and crystallogenesis methods have bought bio-crystallography in a context favorable to subatomic resolution protein structures. At this resolution, electron density reveals fine details related to the deformation of the valence electron density due to chemical bonding and intermolecular interactions. A spherical atom model of electron density does not allow to take into account these features in the refinement. However, in small molecules charge density studies, the Hansen & Coppens [1] multipolar model is commonly used, and allows the asphericity of the atomic electron density to be parameterized and quantified against experimental data.

Here we will show how charge density studies principles can be applied with the software MoPro [2] on protein structures obtained at subatomic and atomic resolution, using specific methods like the multipolar parameter transferability from our experimental database [3]. We will also present derived electrostatic properties based on the multipolar formalism and computed on high resolution Human Aldose Reductase – inhibitors complexes [4] of pharmacological interest.

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Keywords: charge density, protein structure, very high resolution

MS49.27.3

Acta Cryst. (2005). A**61**, C65

Hypervalency – Experimental Charge Density Uncovers a False Concept

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Recently we synthesised and experimentally determined the charge density in molecular species containing so-called hypervalent central atoms. In those compounds formally the amount of valence electrons at the central atom exceeds the number of eight. Typical text book examples are SiF_6^{2-}, PF_6 or SO_3. Historically 3d orbitals are employed to explain the valence expansion and the generate sp^3d or sp^3d^2-hybrid-orbitals. However, the promotion of a phosphorus 3p electron to the d-orbital 16 eV are required but only 1 to 5 eV received by each covalent bond. Theoretical chemistry uncovered hypervalency as a false concept long time ago.[1] We investigated the phenomenon in terms of experimental charge density and topological analysis[2] of the hexacoordinated silicon complex $[F_2Si\{O(Me_2NN)\ CPh\}_2]$, the lithiumiminophosphoranate $[(Et_2O)Li\{Ph_2P(CHPy)(NSiMe_3)\}]$, and the sulfur triimide $S(N^tBu)_3$.[3]

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Keywords: hypervalency, topological analysis, sulfur

MS49.27.4

Acta Cryst. (2005). A**61**, C65

Measurements of Electron Densities in Solids

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This talk reports the recent progress in the measurement of electron densities in inorganic crystals and its significance for our understanding of bonding and electronic structure [1]. The talk is organized in two parts. The first part first emphasizes the importance of accuracy in experimental structure factors for electron density mapping and the challenge of studying inorganic crystals, which is then followed by an introduction of the convergent beam electron diffraction technique for accurate structure factor measurement. The second part of the talk reports the study of electron density in several inorganic crystals of materials interest with focus on transition metals and ions. Comparison between experiment and theory will be made to highlight the significance of experimental electron density and the need for further study. The talk will be concluded by looking into future challenges and opportunities in materials science for crystallography.

* The talk is based on work performed in collaboration with B. Jiang, M. Kim, M. O'Keeffe and J.C.H. Spence. The work was supported by NSF and the author thanks DOE BES for supporting his current research.
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Keywords: electron density, electron diffraction, crystal electronic structure

MS49.27.5

Acta Cryst. (2005). A**61**, C65-C66

Orbital-wise Decomposition of Magnetic Compton Profiles and Spin Moments in UGe_2

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The uranium ferromagnet UGe_2 has drawn much attention because of possible coexistence of superconductivity and ferromagnetism [1].