All of the well-loved program systems have their roots in ideas formed about 30 years ago, and have evolved slowly under the care and attention of individuals or small groups. These programs ***express*** the knowledge held by these people, but they do not document it.

The equation $A^T.A.\delta x = A^T.\Delta F$ sums up what happens in least-squares, but it requires a lot of code to convert this into even a simple useable program, and a massive amount of understanding of the problem and environment to turn it into a user-friendly program.

The principal writers and care-takers of the most popular programs are now in the final phases of their careers. When they shuffle off their mortal coils, devotees may be able to keep some of the programs running for a short while, as a kind of working museum. Every thing in not broken yet, so there is nothing to fix. However, if the community is to avoid re-inventing very may wheels in the future, there is urgent need to properly document current knowledge, and use it to create better wheels.

**Keywords: computing, least-squares, mortal coils**

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**MS21 BASIC TO INDUSTRIAL APPLICATIONS OF STRESS AND STRAIN ANALYSES WITH SYNCHROTRON AND NEUTRON RADIATIONS**
***Chairpersons:*** Alain Lodini, Lyndon Edwards

**MS21.25.1**
*Acta Cryst*. (2005). A**61**, C32
**Insights into Deformation Mechanisms from *in-situ* Diffraction Experiments**
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The usage of neutron and synchrotron x-ray diffraction as a tool to measure internal stresses has increased significantly in recent years. While a great deal can be learnt about the influence of processing and fabrication routes on materials by studying samples after processing, it is often beneficial to carry out controlled loading experiments. Loading samples *in situ* in the diffracting beam provides a direct insight into the micromechanical deformation mechanisms contributing to the macroscopic response of the sample as a whole, under user imposed environmental conditions. Combined with micromechanical modelling a great deal can be learnt regarding the way that the various mechanisms operate and interact, for example different slip modes and/or phase transformations. While both neutrons and synchrotron x-rays probe bulk rather than surface properties, the two techniques provide different opportunities and different challenges for such experiments. The techniques and capabilities will be explored via examples of studies of the deformation of metal and ceramic polycrystals.

**Keywords: deformation, internal stress, plasticity**

**MS21.25.2**
*Acta Cryst*. (2005). A**61**, C32
**Strain Mapping Methods and Instruments: Recent Advances and Future Implications**
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Neutron diffraction is now a relatively mature technique for strain mapping in engineering materials and components. Synchrotron X-ray diffraction methods are developing rapidly, and for some applications offer more efficient data collection. The fundamental principles of diffraction methods for strain determination, and subsequent calculation of stress, are well-understood. Much of the improvement in the applicability of neutron and synchrotron X-ray methods in the last 10 years has been achieved by improvements in instrumentation and the development of dedicated diffractometers for strain measurement and mapping. This has allowed for better sample positioning and accommodation of bulkier and weightier samples. At the same time, there have been improvements in neutron optics and our understanding of beam attenuation effects.

This talk will review some of these developments, in the context of the new engineering-oriented diffractometers that have been developed in the last five years. Results will be presented from applications that could not have been achieved ten years ago. It will also look forward to the possibilities of future developments which will further expand the scope and applicability of engineering diffraction measurements of strain.

**Keywords: residual stress analysis, strain mapping, engineering materials**

**MS21.25.3**
*Acta Cryst*. (2005). A**61**, C32
**Contribution of Numerical Simulation to Stress Evaluation by Neutron or Synchrotron Diffraction**
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As demonstrated by various round robin tests, stress evaluation by neutron diffraction or synchrotron radiation is reliable when the probe volume is completely immersed in the studied material. However, near surface measurements or acquisitions carried out close to interfaces are much more difficult to analyze, due to parasitic shifts of the diffraction peaks which are obtained in such condition.

This study shows the contribution of numerical simulations to solve this problem. It demonstrates that a complete modeling of diffractometers by a Monte Carlo method allows defining precisely the size and shape of the probe used. It permits then predicting the evolution of the diffracted intensity versus the position of this volume in the matter. This approach allows also determining and correcting all systematic shifts of the diffraction peaks which appear when measurements are carried out near the surface or close to an interface. The calculations finally let to define the real analyzed depth, accounting for the local conditions of diffraction and absorption in the material. The experimental procedures implemented thanks to the numerical simulations strongly improve the space resolution of the neutron and synchrotron stress evaluation methods and reduce the uncertainties of the results. To this last end a new method for a global analysis of stress fields was developed which greatly improves the precision of measurements.

**Keywords: stress, synchrotron radiation neutron, simulation**

**MS21.25.4**
*Acta Cryst*. (2005). A**61**, C32-C33
**Study of Elasto-plastic Deformation in Mg Alloy Using Synchrotron Radiation**
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Strain scanning using monochromatic and white beam X-rays is becoming increasingly popular for measuring the residual and live stresses within engineering samples and test pieces.

This work presents the results of a study of elasto-plastic deformation in bent bars of magnesium alloy using 68.5 keV monochromatic synchrotron X-rays and white radiation. We have developed a fast monochromatic method where an aperture is scanned across an image plate exposing a fresh part of the plate at each step, and the sample is simultaneously scanned through the X-ray beam. A complete set of 'diffraction segments' are recorded on the image plate showing peak positions, texture and peak broadenings as a function of position in the sample. The measurements made with the energy dispersive, white beam technique are consistent with the new monochromatic method. We demonstrate that information about plastic deformation can be successfully extracted not only from peak shape variation, but also from the relative peak positions (difference strains) between different reflections. The difference arises as a consequence of elastic and plastic anisotropy of grains in response to loading, and sheds light on the micromechanics of polycrystalline

systems. The plastic strain distribution obtained in this way provides a general correlation between plastic deformation history and the properties of the observed diffraction peaks.

**Keywords: strain scanning, synchrotron radiation, magnesium alloy**

**MS21.25.5**

*Acta Cryst*. (2005). A**61**, C33

**Depth-resolved Strain Measurements by Energy-variable X-ray Diffraction**

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Characterization of the microstructure of materials with spatial resolution is one of key issues in materials related fields from nano-technology to non-destructive testing of manufactured articles. Depth resolved strain/stress measurements by diffraction methods are of particular interest. In order to improve depth resolution of x-ray diffraction, we are developing novel technique for synchrotron beam lines – energy-variable diffraction (EVD) [1]. The method is based on our ability to precisely change energy of synchrotron radiation and, in a result, to accurately control the x-ray penetration depth. Comprehensive analysis of x-ray trajectories, taking into account the instrument misalignment, change of the height of an incident x-ray beam with energy, and variable penetration of x-rays into the sample depth, allowed us to receive analytic expression for the diffraction profile measured by EVD and to show that the maximum diffraction intensity registered in the detector is coming from certain depth, which is energy-dependent [2]. This finding opens a way for measuring residual strains with high depth resolution by changing the x-ray energy in small enough steps.

Experimental examples taken with differently scaled metal/metal and metal/ceramic multilayers as well as structures from nature (seashells) demonstrate the capabilities of the method.

[1] Zolotoyabko E., Quintana J. P., *Rev. Sci. Instr.*, 2002, **73**, 1663. [2] Zolotoyabko E., Quintana J. P., *J. Appl. Cryst*.,2002, **35**, 594. [3] Zolotoyabko E., Quintana J. P., *Nucl. Instr. & Meth. B*, 2003, **200**, 382. [4] Zolotoyabko E., Pokroy B., Quintana J. P., *J. Synchr. Rad*.,2004, **11**, 309.

**Keywords: residual strains, X-ray diffraction, multilayers**

## MS22 SINGLE PARTICLE X-RAY DIFFRACTION IMAGING

***Chairpersons:*** Janos Hajdu, Henry Chapman

**MS22.25.1**

*Acta Cryst*. (2005). A**61**, C33

**Diffraction Imaging of the Yeast Cell: First Results**

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We have developed an apparatus for soft x-ray diffraction microscopy (XDM) of dry or frozen hydrated biological specimens. The microscope, stationed at beamline 9.0.1 of the Advanced Light Source, can collect nearly complete three-dimensional diffraction data to 10 nm resolution. Diffraction patterns, from eight angular orientations of a whole and unstained freeze-dried yeast cell, were recorded with the microscope and phased using the difference map algorithm. The resulting images portray the natural complex refractive contrast of the cell to 30 nm resolution and their agreement provides confidence in the accuracy of the imaging technique. New techniques for handling noisy and incomplete diffraction data were developed and improved the convergence of the algorithm. The effects of large doses on the structure of the cell were also investigated and it is determined that dry specimens suffer from shrinkage while frozen hydrated cells are stable with doses as large as $5\times10^9$ Gray.

**Keywords: X-ray diffraction, X-ray imaging, X-ray microscope**

**MS22.25.2**

*Acta Cryst*. (2005). A**61**, C33

**Imaging Magnetic Nanostructures by X-ray Holography**

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While holography has evolved to a powerful technique in the visible spectral range, it is difficult to apply at shorter wavelength as no intrinsically coherent (soft) x-ray laser is yet available as a light source. The progression from visible light towards shorter wavelength is motivated by the increase in spatial resolution that can be achieved. Of equal importance is the possibility to exploit special contrast mechanisms provided by scattering in resonance with transitions between electronic core and valence levels.

We demonstrate imaging of non-periodic objects by x-ray spectro-holography at 50 nm spatial resolution. Magnetic domain patterns forming in thin film Co-Pt multilayers with perpendicular anisotropy are imaged using x-ray magnetic circular dichroism contrast at 778 eV photon energy. The images are obtained by direct Fourier inversion of the coherent scattering pattern, without the need of phase retrieval or an iterative computing process. Holography at this wavelength was made possible by combining the sample with a nanostructured mask. [1] This approach is particularly valuable for future single shot and/or single molecule imaging experiments at free electron x-ray lasers. At such sources, the coherent x-ray flux will be sufficient to record a coherent x-ray diffraction snapshot using a single x-ray pulse with a duration of a few femtoseconds.

[1] Eisebitt S., Lüning J., Schlotter W. F., Lörgen M., Hellwig O., Eberhardt W., Stöhr J., *Nature*, 2004, **432**, 885.

**Keywords: holography, coherent scattering, electronic structure and magnetism**

**MS22.25.3**

*Acta Cryst*. (2005). A**61**, C33

**Prospects for X-ray Diffraction Imaging of single Biological Molecules**

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Short x-ray pulses from x-ray free electron lasers (XFELs) may enable diffraction imaging of single biological molecules. This would allow the determination of the structure of many molecules that have, to date, resisted crystallization. Since the appropriate sources will not be available for a few years, experimental design currently has to be done through simulations and modeling. Various aspects of the models are tested through experiments on currently available light sources.

In this presentation we will discuss numerous issues of the injection, irradiation, and imaging process. We will present our plans to model all aspects of the diffraction imaging endeavor, and the progress that we have made to date. Specifically, we will present an analysis of the pulse length and photon energy requirements by combining results from a continuum damage model [1] with a fluence requirement model [2]. We will further discuss several means to alleviate the pulse requirements, and compare the requirements with parameters of two planned x-ray lasers. Finally, we will present results from recent 3D imaging experiments at a resolution down to 10nm.

[1] Hau-Riege S.P., London R.A., Szoke A., *Phys. Rev. E*, 2004, **69**, 051906. [2] Huldt G., Szoke A., Hajdu J., *J. Struct. Biol.*, 2003, **144**, 219.

**Keywords: diffraction imaging of non-crystalline specimens, biological molecules, radiation damage studies**

**MS22.25.4**

*Acta Cryst*. (2005). A**61**, C33-C34

**Imaging of Atom Clusters by hard X-ray free Electron Laser Pulses**

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We study the possibility of imaging a small cluster of atoms by