MS20 ADVANCES IN COMPUTATIONAL METHODS FOR SMALL MOLECULE CRYSTALLOGRAPHY
Chairpersons: Simon Parsons, Giovanni Luca Cascarano

MS20.25.1
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Phasing via full Pattern Powder Decomposition by Monte Carlo and Patterson Methods
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In a recent paper [1] a new full pattern decomposition technique has been suggested using linear ternary codes resulting from modifications of the Hamming codes. The resultant decomposition procedure consists of only 27 decomposition trials: to each of them 20 direct methods trials are applied, for a total of 540 tangent processes among which the correct solution may be found.

The above method has been combined with a technique [2] which modifies the Patterson map to obtain, by inversion, better estimates of the structure factor moduli.

The resultant procedure has been introduced in EXPO2005, and proved quite useful to solve crystal structures from powder diffraction data.

[1] Altomare A., Caliandro R., Cuocci C., da Silva I., Giacovazzo C., Moliterni A.G.G., Rizzi R., *J.Appl. Cryst.*, 2004, **37**, 204-209. [2] Altomare A., Foadi J., Giacovazzo C., Moliterni A.G.G., Burla M.C., Polidori G., *J.Appl. Cryst.*, 1998, **31**, 74-77.
Keywords: direct phasing, powder diffraction, ab-initio structure determination

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Ab-initio Structure Solution without the Use of Atomicity
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Charge flipping [1] is an amazingly simple ab-initio structure solution method that is based on the existence of extended zero regions in the electron density, but not directly on atomicity. The algorithm is iterative: the real-space modification simply changes the sign of electron density below a threshold, while in reciprocal space observed moduli are prescribed without any weighting. It was tested using synthetic data for a wide range of structures [1], and was shown to work on real data of both normal [2] and modulated [3] crystals. Recently, we have found an efficient modification of the charge flipping algorithm [4] that complements the phase exploration in reciprocal space. In the modified algorithm weak reflections are treated separately, their calculated moduli are let to change freely and their calculated phases are shifted by $\pi/2$. Paradoxically, it is better not to use observed moduli of weak reflections, in the search for a solution they create only unwanted constraints. The improvement is drastic, in some cases the success rate is increased by a factor of ten, in other cases a previously unsolvable structure becomes solvable by the modified algorithm.

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[1] Oszlányi G., Sütő A., *Acta Cryst. A*, 2004, **60**, 134. [2] Wu J.S., Spence J.C.H., *et al.*, *Acta Cryst. A*, 2004, **60**, 326. [3] Palatinus L., *Acta Cryst. A*, 2004, **60**, 604. [4] Oszlányi G., Sütő A., *Acta Cryst. A*, 2005, **61**, 147.
Keywords: ab-initio structure determination, algorithms, single-crystal diffraction

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A DS5 (Direct-Searcher Automatic System Version 5) Program for small Molecules Running on Windows PCs
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New developed ***DS5*** (Direct-Searcher automatic system ver. 5) for the crystal structure analysis of organic compounds running on PCs is one program that is integrated into one from more than 20 main programs and subroutine/graphic libraries of the *DS*SYSTEM* series [1,2]. Three features of the ***DS5*** are: input instruction data have compatibilities with *Shelx* series [3], calculation sequences are controlled by subprogram names that are prepared by user, and graphical outputs are displayed on PC with Postscript/HP-GL files. All functions of the ***DS5*** are inherited from of the *DS*SYSTEM4*.

1) Data reduction (6): *ABSORP, AFCR, CONVERT, LQPARM, RDEDIT, SPACEG*
2) Calculation (8): COMPARE, *DISTAGL, LSBF, PARST, ROTEN, SFFR, Shake (ShakePSD), THMA*
3) Display and Plot (4): *DISTAGL, ORTEP3, PLUTO, ROTENP*
4) Publication (3): *DISTAGL, PARSTC, TABLES*
5) Document (2): *Manual, Help File*

[1] Okada S., Okada K., *Z. Kristallogr*., 2000, **215**, 131. [2] Okada K., Okada S., *J. Chem. Inf. Comput. Sci*., 1997, **37(3)**, 522. [3] Sheldrick G. M., SHELXL97: Program for the Refinement of Crystal Structures. University of Göttingen, 1997.
Keywords: computer programs, organic compounds, structural analysis software

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Maximising the Information Obtainable from Diffraction Experiments
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In this presentation I will describe some of the methods we have been developing in recent years for the analysis of both single crystal and powder diffraction data. In particular I will address how using modern and flexible software, such as the Topas-Academic package written by Alan Coelho, one can perform innovative analysis without needing access to source code. Complex analytical approaches can then be rapidly developed to address specific problems by those without high levels of computational expertise.

To illustrate the importance of software flexibility I will describe how simulated annealing techniques widely use for structure solution from powder diffraction data can be used to solve complex oxide superstructures from single crystal data. I will also describe methods we've developed for "surface fitting" diffraction data. By treating a set of variable temperature/time/pressure powder or single crystal data as an ensemble rather than as unconnected individual measurements, one can dramatically increase the useful information extractable from experimental data. Whilst specific examples will be used to highlight possibilities, the methods to be described have wide ranging applicability.
Keywords: methodology of diffraction analysis, powder diffraction, single-crystal X-ray methods

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Small Molecule Crystallographic Computing – What is the future?
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A well known proverb amongst programmers is 'If it's not broken, don't mend it'. This is good advice, but what should be done if it is clear that very soon something is going to stop working?

In recent years small molecule crystallographic programs have disappeared one by one. This usually happens when there is no one left who really understands the program, and who can support it in a changing environment. For a short while after the programs become unavailable, old crystallographers bore young crystallographers by repeating 'I remember when it was easy to do …. with program X'. Then people forget that it was ever possible, and then some one re-discovers the process. The wheel is re-invented.

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

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

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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

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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

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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

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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