

This will be demonstrated on a number of molecular structures solved from laboratory data and validated later with the use of synchrotron and/or neutron data.

Keywords: crystal structure determination X-ray powder data, synchrotron powder diffraction, neutron powder diffraction

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Close Contact Penalty Functions in Direct Space Methods and Energetic Considerations in Structure Refinement

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When solving crystal structures from powder diffraction data using direct space methods, all available information such as molecular structures and space group symmetry is used to limit the number of degrees of freedom. When limited quantity of information is available from a powder diffraction pattern (e.g., broad peaks, preferred orientation, positions of weak scatters) and/or the number of degrees of freedom is large, it may be necessary to add extra chemical information in order to obtain a solution. This chemical information can be that the generated structures should be energetically stable, where the potential energy contributes to a combined figure of merit alongside the powder pattern similarity, R_{wp} .

Considering the fact that viable solutions should not contain overlapping atoms, adding a simple close contact penalty that prevents solutions with non-viable intermolecular interactions from being generated is adequate for the global optimization process, which aims at locating a rough, refinable solution.

During Rietveld refinement an accurate description of the potential energy should be used in combination with the R_{wp} in a weighted optimization process. The a priori determination of the weighting factor might not be intuitive; in such cases a Pareto optimization (a posteriori preference articulation) can be used to obtain an appropriate value so as to ensure that structures that are both chemically viable and in close agreement with the experimental powder pattern.

Keywords: ab-initio structure determination, Rietveld structure refinement, potential energy

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Differential Thermal Expansion and Laboratory Capillary X-ray Powder Diffraction: Progress, Practicalities and Performance

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The challenge for experimental XRPD applied to crystal structure determination, is to obtain the best possible estimates of diffracted intensities. The chance of achieving this is enhanced by using (i) low- T data collections to decrease thermal vibration and therefore boost the intensities seen at higher diffraction angles and (ii) new PSD detector technologies yielding improved signal-to-noise and count-rates. Importantly, significant improvements to the extracted intensity information available from overlapping peaks may be gained by using multiple low- T data collections, thereby exploiting differential thermal expansion (DTE) [1,2]. Here we present the exploitation of DTE to retrieve an immense amount of accurate intensity data from a series of laboratory capillary X-ray powder diffraction data sets collected at several temperatures. We describe a validated method for intensity extraction and include details on the impact of improved PSD detectors and high intensity laboratory X-ray sources on this approach.

[1] David W. I. F., Shankland K., McCusker L. B., Baerlocher Ch. in *Structure Determination from Powder Diffraction Data*, Ch. 1, David et al. (Eds), Oxford University Press, Oxford, 2002. [2] Zachariasen W. H., Ellinger F. H., *Acta Crystallogr.*, 1963, **16**, 369.

Keywords: X-ray powder diffraction, differential thermal

expansion, crystal structure determination

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Rapid Structure Solution using Global Optimisation and Distributed Computing

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The use of global optimisation methods has helped to increase the size and complexity of molecular organic structures that can be solved directly from powder diffraction data. These methods require many repeat runs to be performed in order to confirm the location of the global minimum in parameter space; this is particularly true for very complex structures, where success rates in locating the minimum may fall to only a few percent. Fortunately, these multiple runs can be performed independently of each other and as such, they are ideally suited to the notion of grid-type computing. We have recently adapted the DASH [1] program to run under the GridMP [2] distributed computing system; the current setup allows up to eighty DASH simulated annealing runs to be executed in parallel on *existing* desktop resources. Initial results show not only impressive performance gains but also indicate that new computational routes that were previously closed to us (due to their compute-intensive requirements) are now open. For example, full characterisation of a hybrid Monte Carlo (HMC) method of structure determination from powder diffraction data required many months of CPU time; the work was performed in a matter of days using a grid-adapted version of the HMC code [3].

[1] David W.I.F., Shankland K., Shankland N., *Chem. Commun.*, 1998, 931-932. [2] <http://www.ud.com> [3] Markvardsen A.J., Shankland K., David W.I.F., Didlick G., *J. Appl. Cryst.*, 2005, **38**, 107-111.

Keywords: powder diffraction, structure determination, parallel computing

MS26 RECENT ADVANCES IN QUASICRYSTAL RESEARCH

Chairpersons: Koh Saitoh, Ron Lifshitz

MS26.25.1

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Fabricating Novel Symmetry Nanoscale Systems using Quasicrystal Surfaces

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Quasicrystals are metallic alloys which have unusual structural properties: they are aperiodic and may display symmetries not observed in periodic materials. The surfaces of these materials offer opportunities for the fabrication of nanostructures and thin films which themselves have unusual symmetries and structures. In turn, measurements of such nanostructured systems may offer insights into the larger question of the relationship between physical properties and aperiodicity. Several such systems have been fabricated and are under investigation in our laboratory, and some examples will be shown.

One spectacular case is that of an ultrathin film grown by the deposition of copper atoms on the five-fold surface of the icosahedral $Al_{70}Pd_{21}Mn_9$ quasicrystal [1]. STM images show that the in-plane structure comprises rows having separations of $S=4.5\pm 0.2$ Å and $L=7.3\pm 0.3$ Å, whose ratio is the Golden mean $\tau=1.618...$ within experimental error. The sequences of such row separations form segments of terms of the Fibonacci sequence, indicative of the formation of a pseudomorphic Cu film. We have recently demonstrated that such films can also be grown using magnetic elements such as Co, Fe and Ni. Characterisation of their magnetic properties has also been undertaken and will be discussed.

[1] Ledieu J., Hoefft J.T., Reid D.E., Smerdon J.A., Diehl R.D., Lograsso T.A., Ross A.R., McGrath R., *Phys. Rev. Lett.*, 2004, **92**, 135507.

Keywords: quasicrystals, nanostructures, scanning tunnelling microscopy

MS26.25.2*Acta Cryst.* (2005). A61, C38**Maximum or Minimum Density Principle Determines the Bulk Terminations of Quasicrystals?**Zorka Papadopolos^a, Gerald Kasner^b, ^a*Institut für Theoretische Physik, Universität Tübingen, Germany.* ^b*Institut für Theoretische Physik, Universität Magdeburg, Germany.* E-mail: zorka.papadopolos@uni-tuebingen.de

In [1] we announced the “thick layer” concept of the bulk terminations in quasicrystals. We consider a bundle of dense atomic planes in the bulk, not necessarily extremely close together, as a candidate for a termination. We show that only “thick” layers can terminate the bulk of decagonal Al-Cu-Co (d-AlCuCo) in the 2fold directions. Also, the secondary electron images indicate that certain “thick” 5fold and 2fold layers are favoured as terminations of icosahedral Al-Pd-Mn (i-AlPdMn). In particular, the 2fold terrace-like surfaces of i-AlPdMn containing pits are perfectly explained in this framework.

Following a suggestion of Sharma et al. [2], that a gap in the bulk might define a termination, we introduce a minimum density rule on low density “thick” atomic layers as well and show that, in the framework of the model of i-AlPdMn, such a rule does not match the observed step heights on either the 2fold or 5fold surfaces.

[1] Papadopolos Z. et al., *Phys. Rev. B*, 2004, **69**, 224201. [2] Sharma H.R. et al., *Phys. Rev. Lett.*, 2004, **93**, 165502.**Keywords:** quasicrystals, surface structure, scanning tunneling microscopy**MS26.25.3***Acta Cryst.* (2005). A61, C38**Diffuse Scattering and Phasons in the i-Zn-Mg-Sc Phase and its 1/1 Approximant**Marc de Boissieu^a, T. Ishimasa^b, S. Francoual^{a,c}, ^a*LTPCM, UMR CNRS 5614, St Martin d Hères, France,* ^b*Hokkaido University, Sapporo, Japan,* ^c*ILL, Grenoble, France.* E-mail: boissieu@ltpcm.inpg.fr

Phasons modes are excitations characteristic of the quasicrystalline state. In the same way than phonons modes lead to thermal diffuse scattering, phason modes lead to diffuse scattering in the diffraction pattern. This diffuse scattering can be calculated in the framework of the hydrodynamic theory of icosahedral phases.

Diffuse scattering has been extensively studied in the i-AlPdMn phase. It has been shown that most of the observed diffuse scattering is due to phason modes, which are interpreted as pre-transitional fluctuations.

We present an extensive room temperature study of the diffuse scattering in the i-ZnMgSc quasicrystal and its 1/1 periodic approximant. For this purpose, absolute scale measurements of the X-ray diffuse scattering have been carried out on the D2AM beam line (ESRF). Whereas the diffuse scattering measured in the 1/1 approximant can be accounted for by the thermal vibrations (TDS), a supplementary contribution to the signal is observed in the quasicrystal. This extra contribution is larger for reflections having a larger Q_{per} component, indicating that it is due to phason modes. This demonstrates that although both phases share the same clusters, phason modes are a characteristic of the quasiperiodic long range order. Using the absolute scale measurement, we find that the amount of diffuse scattering is about for time smaller in the i-ZnMgSc than in the i-AlPdMn phase. This demonstrates the high structural quality of the i-ZnMgSc phase, which is also evidenced by the large number of high Q_{per} reflections observed in the diffraction pattern.

Keywords: quasicrystal, diffuse scattering, phasons**MS26.25.4***Acta Cryst.* (2005). A61, C38**A High Pressure High Temperature Study of Co-rich d-Al-Co-Ni**
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A basic key in the understanding of the stabilization mechanisms of quasicrystals is the knowledge of their stability ranges within the p-T field. In the last decade, the Al-Co-Ni system has become the model system for the study of decagonal quasicrystals [1]. The decagonal phase in this system shows a quite large compositional stability range, going along with changes of the structural disorder and the formation of superstructures as a function of temperature.

Ni-rich decagonal quasicrystals were found to be stable within the applied experimental frameworks (Al₇₂Co₈Ni₂₀ up to 70 GPa from powder [2], Al₇₀Co₁₂Ni₁₈ up to 10 GPa from single crystal [3], both at ambient temperature). The Co-rich decagonal phase was not yet studied at non-ambient conditions. This phase is neighbored by the recently discovered W-phase [4], which is the highest approximant phase in the Al-Co-Ni system, and therefore the structural behavior of this decagonal phase at non-ambient conditions is of special interest.

The results of an in-situ high pressure high temperature study on Al₇₃Co₂₁Ni₆ up to about 16 GPa and 973 K will be discussed.

[1] Steurer W., *Z. Kristallogr.*, 2004, **219**, 391. [2] Hasegawa M., Tsai A.P., Yagi T., *Phil. Mag. Lett.*, 1999, **79**, 691. [3] Krauss G., Miletich R., Steurer W., *Phil. Mag. Lett.*, 2003, **83**, 525. [4] Sugiyama K., Nishimura S., Hiraga K., *J. Alloys Comp.*, 2002, **342**, 65.**Keywords:** high pressure, high temperature, quasicrystals**MS26.25.5***Acta Cryst.* (2005). A61, C38**Mesoscopic Archimedean Tilings in Polymeric Stars**Yushu Matsushita^a, Wataru Kawashima^a, Atsushi Noro^a, Atsushi Takano^a, Nobuo Tanaka^b, Tomonari Dotera^c, ^a*Department of Applied Chemistry, Japan.* ^b*Ecotopia Science Institute, Nagoya University, Nagoya, Japan.* ^c*Department of Polymer Chemistry, Kyoto University, Kyoto, Japan.* E-mail: yushu@apchem.nagoya-u.ac.jp

Four mesoscopic Archimedean tiling patterns have been observed for the molten ABC star-branched terpolymers composed of polystyrene(S), polyisoprene(I), and poly(2-vinylpyridine)(P) of the type S_{1.0}I_{1.0}P_x. The copolymers exhibit (6³), (4.8²) and (4.6.12) Archimedean tiling[1] when x are 0.7, 1.2 and 1.9[2], respectively, while the molecule of the type S_{1.0}I_{1.0}P_{1.3} shows more complex (3².4.3.4) tiling pattern with mesoscopic length-scale. Namely the side length of the polygons are about 80nm. In this structure the circumstance of a molecule splits into multiple sites and consequently two microdomains with different sizes and shapes are formed for one component. Moreover the experimental results were well explained with the predicted results based on free energy theorem using Monte Carlo method. This pattern has been observed for the other materials on much shorter length-scale, therefore, the experimental fact observed in the present study is demonstrating that the complexity is universal over different hierarchy.

[1] Grunbaum B., Shephard G. C., *Tilings and Patterns*, Freeman, New York, 1986.[2] Takano A., Matsushita Y. et al., *Macromolecules*, 2004, **37**, 9941.**Keywords:** archimedean tiling, ABC star-branched terpolymer, hierarchy**MS27 DETECTORS: DEVELOPMENTS AND REQUIREMENTS FOR X-RAY, SYNCHROTRON AND NEUTRON SOURCES****Chairpersons:** Naoto Yagi, Christian Broennimann**MS27.25.1***Acta Cryst.* (2005). A61, C38-C39**Development of Very-high Rate and Resolution Neutron Detectors in DETNI**Burckhard Gebauer^a, Svyatoslaw S. Alimov^a, Andrea Brogna^b, Siro Buzzetti^c, Wladyslaw Dabrowski^d, Thomasz Fiutowski^d, Martin Klein^b, Caterina Petrillo^e, Francesco Sacchetti^e, Christian J. Schmidt^b, Hans Kristian Soltveit^b, Christian Schulz^a, Robert Szczygiel^f, Ulrich Trunk^b, Clara Turconi^a, Dr. Thomas Wilpert^a, ^a*Hahn-Meitner-Institut Berlin, Glienicke Str. 100, D-14109 Germany.* ^b*Physikalisches Institut der Universität Heidelberg, Philosophenweg 12, D-69120 Heidelberg, Germany.* ^c*INFN & Dipartimento di Elettronica e Informazione, Politecnico di Milano, Piazza L. da Vinci 32, I-20133Milano, Italy.* ^d*Faculty of Physics and Nucl. Techniques, AGH*