effect of particle statistics can be evaluated from the ω-scan as well as the φ-scan data in the synchrotron parallel-beam geometry.

It has been proved that the effective number of diffracting crystallites is increased by a factor of about 100, by applying continuous φ-rotation of a flat specimen during the measurement, as predicted by the theory [3]. Errors caused by particle statistics, which should be incorporated in any analysis of powder diffraction data, will also be discussed.

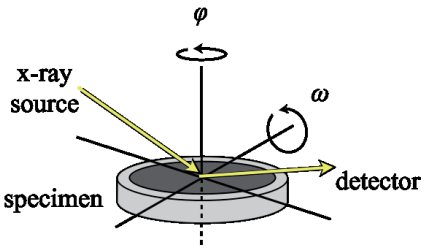

Fig. 1 Definitions of φ & ω

[1] L. Alexander, H.P. Klug, E. Kummer, *J. Appl. Phys.* **1948**, *19*, 742-753. [2] T. Ida, T. Goto, H. Hibino, *J. Appl. Cryst.* **2009,** *42*, 597-606. [3] P.M. de Wolff, *Appl. Sci. Res.* **1958**, *7*, 102-112.

Keywords: powder, particle, statistics

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A new mechanism of negative thermal expansion in an interpenetrated framework material

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Negative thermal expansion (NTE) is an unusual property that has only been recorded in a small number of materials. However, we have recently found that metal-organic framework (MOF) materials have a number of structural characteristics that promote NTE.[1]

Conventionally, porous materials tend to support transverse vibrations, as motion into the empty pores is unhindered. Hence, in frameworks in which the previously empty space is occupied either by guest molecules or an interpenetrating framework, NTE is diminished. Here, however, we show that framework interpenetration can increase the magnitude of NTE over a similar framework when the normal thermal expansion of the intermolecular contacts between the two frameworks results in a structural distortion – a phenomenon unreported in other materials.

The studied material is MOF-14, a highly porous copper-carboxylate framework which forms a singly interpenetrated network.[2] We present single-crystal and powder diffraction data demonstrating the structural change of the material over a wide temperature range, clearly showing the nature of this new mechanism for NTE.

[1] Y. Wu, A. Kobayashi, G.J. Halder, V.K. Peterson, K.W. Chapman, N. Lock, P.D. Southon, C.J. Kepert, *Angew. Chem. Int. Ed.* **2008**, *47*, 8929-8932. [2] B. Chen, M. Eddaoudi, S.T. Hyde, M. O'Keeffe, O.M. Yaghi, *Science* **2001**, *291*, 1021-1023.

Keywords: negative thermal expansion, metal-organic framework, interpenetration

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Advances in guinier-type powder diffraction

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X-ray powder diffraction may be considered the main 'workhorse' in almost any field regarding solid state and materials sciences. Advances with instrumentation in recent years provided for major improvements with established methods but also induced the development of new and more specialized applications. The application of shorter wavelength (higher energy) X-rays while looking for higher resolution in the diffraction experiment is a principal contradiction calling for combinations of 'smart' solutions.

The Huber G670 Guinier system is very versatile and reliable, but at the same time simple and flexible for modifications towards more dedicated experiments. The imaging plate based detector system is well suited for any kind of typical lab-based X-ray sources ranging from Cr-Kα (5.4116 keV) up to Ag-Kα (22.1054 keV). However, much higher energies or synchrotron radiation may be used without any problems.

We have started to evaluate improvements and dedicated applications of the instrument on a broad basis. Concerning conditioning of the primary X-ray beam several options will be presented including 1-D and 2-D multilayer optics along with Johansson-type crystal monochromators. Such devices become very demanding in many respects when high-energy X-rays are targeted. Spot shaped beams are not commonly used with Guinier-type setups, however, a small but brilliant spot provides an excellent basis for studying small samples in general, but in particular with diamond-anvil cells or for typical samples in the fields of art and cultural heritage. Any setup discussed may be easily adapted to a broad range of X-ray sources. New ideas and developments towards improvement and modifications of the imaging plate itself but also for the entire detector system will be carefully considered.

Finally, improvements of the unit with respect to resolution but also concerning signal-to-noise ratio will be discussed with respect to the camera setup and the detector. Diffraction data of well defined samples will be presented for any particular setup evaluated. In numerous experiments we got increasingly convinced that Yttriumoxide is in many respects superior to most generally used standard materials.

Keywords: powder, guinier, optics

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Real-time behaviour of crystallizing cocoa butter. Time-resolved SAXS-WAXS study

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Almost 80% of cocoa butter (CB) in chocolate is crystalline. This is due to four different mono-unsaturated triacylglyceroles (SOS, POP, POS and SOA) which are structurally closely related and crystallise together. Their crystal structures are determined from XRPD together with the structures of the two most stable polymorphs of CB itself [1], [2]. In chocolate generally the β-V polymorph is present rather than the somewhat more stable form β-VI.

We studied the crystallisation process of pure CB time-resolved