

unit cells in the sample. If a crystal structure shows disorder, i.e. if different cells look differently, some of the scattered intensity is lost from the Bragg peaks and distributed throughout reciprocal space as diffuse scattering.

The interpretation of such scattering is far from routine. Sometimes it can be done with qualitative arguments and simple simulations; sometimes it requires computationally demanding modeling including Monte-Carlo (MC) simulations combined with optimization of the parameters of the MC simulation [1,2]. Fortunately this computationally most demanding approach involving global optimization using differential evolution or other population based methods is embarrassingly parallel and therefore well suited for taking advantage of the rapidly growing accessibility of parallel computers [3].

We develop software (ZODS: Zürich – Oak Ridge Disorder Simulations) for analyzing diffuse scattering from disordered single crystals whose average structure is (at least approximately) known. ZODS aims to take advantage of parallel processing in various computational environments ranging from personal computers to supercomputers.

The refinement of the diffuse scattering model is parallelized on two levels. (1) A global optimization method calculates an objective function at many points in parameter space simultaneously. (2) Calculation of the objective function at one point involves simultaneous simulation of multiple disordered crystals with the same set of parameters, merging of the corresponding intensities and calculation of the objective function at that point.

The software is tested for correctness and scalability with experimental data for β -NaLaF₄ [4] and artificial test data.

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The disordered and modulated crystal structure of the zeolite SSZ-57

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SSZ-57 has the most complex zeolite structure found to date, with both modulations and significant disorder. High quality X-ray diffraction data could be measured on a ca. 2 x 2 x 8 μ m microcrystal using a Pilatus 6M pixel detector installed on the beamline X06SA at the Swiss Light Source. The high performance of the experimental setup allowed a full data set (1800 frames, oscillation angle: 0.1° / frame) to be collected in just 15 minutes. The Bragg structure can be described in the three-dimensional space group $P4m2$ ($a = b = 20.091$ Å, $c = 110.0$ Å). A better representation of the symmetry, of the sparse density of Bragg reflections and of systematic extinctions in the diffraction pattern, however, was obtained in the four-dimensional super-space group $I4_1/amd(00\gamma)s00s$ ($\gamma = 0.125$), which allowed a refinement with only 648 parameters instead of the 5433 parameters required with the three-dimensional approach. The framework of SSZ-57 is related to that of ZSM-11 (MEL, $I\bar{4}m2$, $a = b = 20.12$ Å, $c = 13.44$ Å). The MEL units are stacked along **c**, but the sequences are interrupted by 12-rings having their channel direction perpendicular to the **c**-axis.

MEL units on either side of the 12-rings are rotated with respect to one another by 90° around **c**. Subsequent 12-rings are rotated in the same manner, so there are two mutually perpendicular 12-ring channels per three-dimensional unit cell. The presence of significant disorder did not allow the structure to be understood in detail without considering information from diffuse scattering. In particular, the distribution of the 12-rings, which is of crucial importance to the understanding of the catalytic properties of this zeolite, could not be determined. Monte Carlo modeling of the real structure resulted in an excellent agreement between the experimental and calculated diffuse scattering with the application of just a few rules. When looking along their channel directions, the 12-rings can be stacked directly on top of each other, or, alternatively and with higher probability, they can be shifted to lie above one of the neighboring 10-rings along the positive or negative **c**-directions. Along perpendicular directions in the **a,b** planes, the 12-rings form (almost) long-range ordered chains without any off-sets along **c**. The positions of the 12-rings along **c** are uncorrelated within the constraints given by the average structure. Though simple from a conceptual point of view, the complexity of the structure required a Monte Carlo computer model describing more than 10⁹ atoms. The handling of such a huge model only became feasible with the application of some simplifications, but these did not seriously hamper the modeling of the experimental observations.

Keywords: zeolites, disorder, modulated structures

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X-ray scattering as a tool for the study of finite size effects in polymers

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The current trends in Polymer Science evolve towards the development of nano-scaled materials. Much effort has been carried out in the last decade to understand the influence of confined media in fundamental phenomena [1]. There is a general agreement that the polymer morphology and nanostructure, which in turn control the physical properties of the material, changes under spatial confinement. The present work contributes to the understanding of finite size effects in polymer materials by means of X-ray scattering methods. Nanolayered polymer systems are employed for this purpose.

Multilayered films with thousands of alternating layers of two immiscible polymers were prepared using a layer multiplying coextrusion process [2]. Films of poly(ethylene terephthalate) (PET) and polycarbonate (PC) with individual layer thicknesses of 65 nm are here investigated. Both materials are initially in the glassy state. Information on the appearance and evolution of PET lamellae (typically several nanometers in one dimension) was obtained by means of isothermal experiments using small-angle X-ray scattering (SAXS) in a synchrotron radiation source. Room temperature wide angle X-ray diffraction studies were also used to provide complementary information of chain orientation.

Real time SAXS studies during the crystallization of nanolayered PET at 150 °C reveal that lamellae oriented with the basal surfaces parallel to the layer stacking (flat-on lamellae) appear. The distribution of directions normal to the lamellar surfaces broadens as crystallization proceeds. This could be explained as arising from the insertion of new lamellae with slightly inclined surfaces between the first developed crystals. Analysis of the SAXS profiles using the intensity profile method allows determining the long period *L* (periodicity along the

lamellar stacks), the thickness of the crystalline lamellae and that of the amorphous layers, l_c and l_a respectively ($L = l_c + l_a$) [3]. Results reveal that PET lamellae in the ultra-thin layers exhibit larger L values than those within the PET control material due to the occurrence of larger amorphous regions between lamellar crystals. In addition, finite-size effects are found to delay the crystallization process. However, the final crystal lamellar structure is similar for both, nanolayered and bulk material.

Room temperature WAXS measurements of the crystallized multilayered materials reveal uniaxial symmetry and indicate that edge-on lamellae are also generated upon crystallization. The simulation of uniaxially oriented patterns with the help of the CLEARER2 software package [4] reveals that the c -axis of the edge-on lamellae lies parallel to the layer surfaces. It is proposed that two lamellar populations develop: edge-on lamellae appear close to the interphases while flat-on lamellae are preferentially located in the layer core.

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Modeling of X-ray diffuse scattering in $Rb_2Li_4(SeO_4)_3 \cdot 2H_2O$
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A title rubidium and lithium selenate hydrate has monoclinic symmetry ($P2_1/c$ space group) and room temperature lattice parameters: $a = 5.256 \text{ \AA}$, $b = 5.178 \text{ \AA}$, $c = 26.739 \text{ \AA}$, $\beta = 93.11^\circ$. Its structure consists of four planar packages perpendicular to c -axis. Selenium tetrahedra are present in each of the four packages but in the two of them Se positions are occupied partially (SOF = 0.5) together with accompanying oxygens forming two alternative tetrahedra [1]. As a result, beside the Bragg reflections coming from the average structure unit cell (Fig. 1a), additional diffraction effects in the form of long, strongly structuralized streaks, parallel to c^* and crossing a^*b^* reciprocal plane at $h \pm \frac{1}{2}$, $k \pm \frac{1}{2}$, are observed (Fig. 1b).

It appeared that the origin of observed diffuse streaks is the different arrangement of selenium atoms in the partially occupied packages, locally fulfilling conditions for C -centering in the $2 \times 2 \times 1$ superstructure cell. Characteristic structuralizations of the diffuse streaks is caused by a switching of SeO_4 tetrahedra forming oxygens into two alternative positions.

Preliminary model hunting and followed Monte Carlo simulations

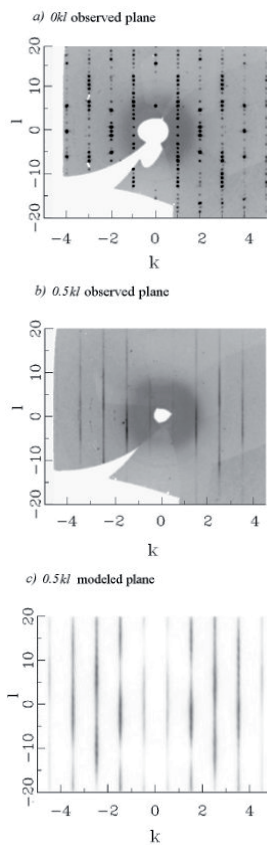


Fig. 1

allowed us to find the best agreement between experimental and calculated diffuse scattering effects for generated sequences of Se-substituting packages (Fig. 1c). Different types of selenium planar domains and ways of multi-plane packaging were also tested and verified.

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Mesomorphic phase in oriented poly(pentamethylene 2,6-naphthalate)

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The thermotropic liquid crystalline (LC) behavior of polymers with rigid mesogenic units interconnected through flexible spacers has been extensively over the past two decades. In the main-chain LC polymer flexible spacers are constrained by the mesogenic units to which they are linked, thus to have some orientational order. Among these polymers poly(alkylene 4,4'-biphenylene)s whose mesophase existence and transition behavior have been intensively studied. These BB- n polyesters invariably form smectic mesophase when n varies from 3 to 9. Polyester based on 2,6-naphthalene dicarboxylic acid, poly(m -methylene 2,6-naphthalate) is another example of LC polymer that possibly show mesophase. In this family, as was noted in BB- n family, the macroscopic thermal and mechanical properties exhibit odd-even fluctuations as the number of methylene group in PmN increases. However, the existence of mesophase in these polymers is relatively rare and has been reported only in PEN and PBN. In this report, we present the mesophase structure in poly(pentamethylene 2,6-naphthalate) (PPN).

Keywords: WAXS, polymer, orientation

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Crystal structures of high-entropy alloys of high melting temperature

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In the recent years importance is being given to high-entropy alloys (HEA) where the alloy consists of multiple elements in high proportion (5–35 atomic %) for their many interesting properties [1]. Higher number of constituent elements increases the configurational entropy of the system, which influences the short-range ordering and coloring-problem. A lot of studies have been done in the alloys with Al and other transition metal elements. The most common of the base system has been Al-Co-Cr-Fe-Ni with other few metals. In this study we have focused on system containing high melting temperature refractory metals like W, Mo, Nb, Ta and V. The refractory HEA has potential application in the aerospace industry where there is an ever increasing need for a functional material with sufficient strength at high