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Examples of Order-Disorder in Macromolecular Crystals. Andrey Lebedev. *Structural Biology Laboratory, University of York, UK.*
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Several examples of macromolecular crystal twins and partially disordered crystals are discussed. Most of them possess one-dimensional disorder and belong to a class called order-disorder (OD) structures. The OD-structures are composed of molecular layers, which are stacked in such a manner that all pairs of adjacent layers are equivalent, whereas twinning or disorder is caused by the presence of non-equivalent triplets. The OD-twins can belong to various geometrical categories of twins including twins by reticular merohedry and allotwins composed of individuals with different crystallographic symmetries. The standard macromolecular crystallographic software does not handle these two categories and detwinning may be required before structure solution or refinement. However, detwinning for OD-twins can be replaced by a more robust procedure of demodulation. A similar procedure applies to partially disordered OD-structures, however there may be an additional problem in this case, the choice of the reference structure that represents predominating ordered domains. This choice is not always obvious or unique. The same problem occurs in several apparently trigonal or hexagonal examples with two-dimensional disorder and there is an example with three-dimensional disorder. A proper treatment of such disordered cases requires a refinement procedure that is similar to twin refinement but also accounts for interference terms between ordered domains.

Keywords: macromolecular X-ray crystallography, order-disorder phenomena, twinning

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Automatic detection of twinning and missed symmetry. P. Zwart

FA1-MS02-T03

High-resolution X-ray Diffraction Study of Single Crystals with Twins: Application to Perovskite Ferroelectrics. Semen Gorfman[a], David Walker, Kaustuv Datta, Dean Keeble, Pam Thomas.
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In the current work we present the details of a powerful technique to investigate precisely the splitting of individual Bragg reflections due to the twinning by utilizing two different types of diffractometer: (1) Oxford Diffraction Gemini R CCD for exploring large areas of reciprocal space and (2) Panalytical X'Pert Pro MRD for high resolution studies of selected areas of reciprocal space. In doing so and supposing that twinning originates from the phase transformation from the higher to the lower symmetry phase, we aim to establish the lower symmetry of a single twin domain in the twinned crystals unambiguously.

The developed technique has been applied to a few important ferroelectric systems, such as potential lead-free piezoelectric $Na_{0.5}Bi_{0.5}TiO$ and (1-x)BiScO–xPbTiO [1] to understand their symmetry more accurately in comparison to the conventional powder diffraction experiment. In particular we managed to show that the true symmetry of the $Na_{0.5}Bi_{0.5}TiO_3$ is monoclinic in contrary to the commonly accepted rhombohedral identified by means of the powder diffraction.

[1] Datta, K., Gorfman, S., Thomas, P. A. (2009) *Appl. Phys. Lett* **95,** 251901

Keywords: Perovskite structures, Twins, High-resolution X-ray diffraction.

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"Hexagonal" single crystals. Yurii Prots, Horst Borrmann. *Max-Planck-Institut für Chemische Physik fester Stoffe, Dresden, Germany*
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Developments in X-ray single crystal diffraction are vastly driven by the general availability of a broad range of detectors with fascinating properties such as a large active area, high resolution and extended dynamic range. This allows to obtain reliable data of high accuracy both for weak and strong reflections, but even at high redundancy within a relatively short amount of time. Application of modern data reduction programs which usually include very useful graphical tools allows for a very detailed analysis of the data obtained. As a consequence, structure solution and refinement become a "routine business" in many cases. However, certain samples are calling for troubles, which may be even enhanced when automatic procedures are applied.
In this contribution we present few examples of single crystal diffraction experiments for which the initial unit cell is always a hexagonal one. According to the particular problem data were deconvoluted before or in course of structure solution and refinement. In any case models were cross-checked against powder diffraction data both from laboratory and synchrotron X-ray sources. As an independent method metallography was extensively used. Discussed intermetallic compounds include the "simple" structure of YbMgGa (ZrNiAl type, *P*62*m*, a = 7.7417 Å, c = 4.1178 Å) [1], the orthohexagonal structure of $Yb_2Pt_6Al_{15}$ (own type, *Cmcm*, a = 12.7966 Å, b = 7.38813 Å, c = 16.3604 Å) [2] as well as different types of twinning: quite trivial reverse-obverse twinning observed in K_4ZnAs_2 (Na_4HgP_2 type, *R*3*m*, a = 5.7529 Å, c = 26.866 Å) [3], but also rather complicated cases of ScAuGa (TiNiSi type, *Pnma*, a = 6.6010 Å, b = 4.3628 Å, c = 7.5631 Å) and HfPdGe (own type, $P2_1/m$, a = 6.6573 Å, b = 3.9359 Å, c = 11.4411 Å, β = 90.102°), where multiple twinning with three or six domains, respectively, is observed [4,5]. Peculiarities of the observed diffraction patterns as well as a strategy for structure solution and refinement are discussed.

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