o.m4.p3 **An Investigation of Polar Twinning in $KTiOPO_4$ and $KTiOAsO_4$.** P.A.Thomas, T. Lyford. Department of Physics, University of Warwick, Coventry CV4 7AL, UK *P.Rejmankova-Pernot, F. Lorut, J. Baruchel European Synchrotron Radiation Facility, BP 220, F-38043 Grenoble Cedex, France.* S.P.Collins *CLRC, Daresbury Laboratory, Warrington, UK.*
Keywords: twinning.

$KTiOPO_4$ (KTP) and $KTiOAsO_4$ (KTA) are well-known nonlinear optical materials which used for frequency-doubling and parametric oscillation applications. They are isostructural polar crystals described by orthorhombic space group $Pna2_1$. In principle, they are ferroelectric crystals, although they do not conform strictly to this definition at room temperature as the large ionic conductivity, brought about by mobile potassium ions and sub-stoichiometry on the potassium sites, does not allow a switching field to be sustained. However, they can be switched at low temperatures and this allows the writing of periodic gratings of inversion domains into the crystals for the purpose of creating so-called "quasi-phase-matched" optical devices.

We have been investigating gratings of induced inversion domains using advanced x-ray techniques[e.g.,1-4]. Two techniques have principally been used: (1) high-resolution diffraction at Station 16.3 of Daresbury Laboratory, which reveals structural information about arrays of domains through rocking curves with well-developed satellite reflections; (2) Bragg-Fresnel imaging[3,4] at ID19 of the ESRF. The detailed structural information accessible with these two techniques is presented, compared and contrasted. It is shown that it is possible to locate the pivot atom on a domain wall through which the twinned structures are matched, using the pseudo-symmetry approach of Thomas & Glazer, (1991[5]). It is further shown that it is possible to locate unambiguously the position of the inversion centre invoked in the twinning, even though this is not constrained to lie on an atomic position. Structural models describing the domain matching are developed for both KTP and KTA for induced walls in the {100} and {010} orientations and are shown to be consistent with the experiment for domain walls in the {100} orientation. The implications for natural twinning in crystals of this family are also discussed.

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o.m4.p4 **XRD/NMR structure determination of twinned β-Cu_7PSe_6 and α-Cu_7PSe_6 polymorphs.** E. Gaudin[1,2], F. Taulelle[1], M. Evain[2], F. Boucher[2], V. Petricek[3], *[1]RMN et Chimie du Solide, UMR 7510 CNRS, Université Louis Pasteur, 4 rue Blaise Pascal, F-67070 Strasbourg Cedex. [2]Laboratoire de Chimie des Solides, I.M.N., UMR C6502 CNRS, Université de Nantes, 2 rue de la Houssinière, BP 32229, F-44322 Nantes Cedex 3. [3]Institute of Physics Academy of Sciences of the Czech Republic Na Slovance 2, 180 40 Praha 8, Czech Republic.*
Keywords: phase transition, twinning, space group determination.

Cu_7PSe_6 belongs to the argyrodite family. It undergoes two phase transitions at 320 K and 250 K. In the γ-Cu_7PSe_6 high temperature form the Cu^+ ions are mobile within the framework [PSe_6] and their diffusion paths are evidenced by XRD by means of a combination of a non-harmonic Gram-Charlier expansion of the Debye-Waller factors and of a split model[1]. For the ambient temperature and low temperature forms of this compound the structure determination on single crystal was obscured by twinning. A method for searching crystallographic information from NMR has been established. This strategy is based on the concepts developed by Brown[2]. The sites symmetry observed by NMR can be related to compatible space groups[3]. XRD and NMR used together for structure search and resolution allowed us to find both polymorphs structures. By taking into account a merohedric twinning, the refinement of the β-Cu_7PSe_6 room temperature form structure leads to the residual factors R = 0.0297 (Rw = 0.0245)[2]. The pseudo-merohedric twinning of the α-Cu_7PSe_6 low temperature form has been described with six twin laws (R = 0.0466 / Rw = 0.0486)[4].

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