P.22.01.1

Acta Cryst. (2005). A**61**, C475

Structure of $LaSrCuO_{3.5}$

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The oxygen deficient $La_{1.07}Sr_{0.93}Cu_{0.92}O_{3.52}$ has been synthesized and the structure has been determined using a combination of transmission electron microscopy, X-ray diffraction and neutron diffraction.

X-ray diffraction showed a basic K_2NiF_4 type structure of a=3.7583(3) Å and c=13.005(3) Å in space group *I4/mmm*. However some reflections remained unexplained. Therefore electron diffraction was performed which showed the presence of an incommensurate modulation and allowed the detemination of the superspacegroup and modulation vectors as *I4/mmm*($\alpha\alpha$0, α-α)00*mg*, q_1=0.22a* + 0.22b* and q_2=0.22a*-0.22b*. Following these observations neutron diffraction was used to determine the origin of these modulations.

The five-dimensional model obtained using the super space formalism shows oxygen-vacancy ordering, implying three different environments present for the copper in the structure: octahedral, pyramidal and square plane coordination. Significant displacements in the (a,b) plane occur for all atoms, no displacement is observed along the c-axis.

High resolution electron microscopy images support the model.

Keywords: incommensurate structures, copper oxides, structural determination

P.22.01.2

Acta Cryst. (2005). A**61**, C475

HMT-Resorcinol: An Example of Modulated Structure in Substituted HMT Adducts

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Hexamethylenetetramine (HMT) has a tendency to create modulated structures when forming adducts with hydroxyl moieties. Many examples have been reported recently [1], [2], [3], [4], [5]. We report here a model of the incommensurately modulated structure of HMT-Resorcinol solved at room temperature, in the superspacegroup Xmcm(0b0)s0s (where the X centering corresponds h+k+m=2n). The model consists mainly in an occupation modulation of the Resorcinol, which can adopt two different positions related by a mirror plane. This compound undergoes a lock-in phase transition of second order at about 278K yielding a commensurate structure. X-ray diffraction data have been collected at 120K, with LT cell parameters related to the room temperature ones by ($\mathbf{a}_{LT}$=$\mathbf{a}_{RT}$, $\mathbf{b}_{LT}$=2$\mathbf{b}_{RT}$, $\mathbf{c}_{LT}$=4$\mathbf{c}_{RT}$). A comparison of the supercell model and a commensurate model derived from the superspace formalism will be presented. .

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Keywords: superspace refinement, incommensurate structures, modulated structure

P.22.01.3

Acta Cryst. (2005). A**61**, C475

The Way of Crystal of [cpMe_4SiMe_2N(t-Bu)]$TiCl_2$ from Disoder thru Modulation to Twinning

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Constrained geometry catalysts based on *ansa*-{(*tert*-butylamido-κ*N*)dimethyl(η^5-2,3,4,5-tetramethylcyclopentadienyl)silane} dichlorotitanium (IV) (**1**) allowed for a large scale production of new polymers and copolymers of specific properties [1] The structure of compound **1** was investigated by X-ray diffraction a [2,3], however the geometrical parameters were not published in full.

From our reinvestigation [4] follows that the *tert*-butyl moiety is disordered in two positions over mirror operation of Pnma space group at room temperature. The new measurement of **1** at low temperatures revealed two phase transitions by reordering t-Bu moiety. The results of first one can be described in supercel 4**a**,**b**,**c** (**1**b), and space group $Pna2_1$, the second one (**1**c) as 3**a**,**b**,**c** and $P2_1/n11$ pseudomeroedricaly twinned into the lattice symmetry mmm. Whole pathway can be unified by applying the modulation concept, superspace group Pnma(a00)0s0 with two q-vectors (1/4,0,0) and (1/3,0,0) for **1**b and **1**c, respectively, affording unique opportunity to test various models (commensurate versus incommensurate etc.).

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Keywords: modulated crystal structures, phase transitions, catalyst structure

P.22.01.4

Acta Cryst. (2005). A**61**, C475

Structure of the Pseudodecagonal Al-Co-Ni Approximant PD4

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The similarity of crystalline approximants to the quasicrystals in terms of their compositions, densities and diffraction patterns, makes them very important for determining the structures of quasicrystals. Several crystal structures of these approximants were determined [1–2].

Several periodic pseudodecagonal (PD) structures were found in the Al-Ni-Co system. They are named PD1, PD2 etc. and exhibit diffraction patterns with almost perfect tenfold symmetry and quasiperiodic reflection arrangements [3].

X-ray diffraction data (0.85 Å resolution) from a single crystal of PD4-phase was collected using Oxford XcaliburTM 3 diffractometer. The unit cell parameters for this structure are a = 101.302(9) Å, b = 32.102(2) Å, c = 4.1803(4) Å.

The phase problem for the approximant PD4 was solved by direct methods in the non-centrosymmetric space group *Bbm*2. The refinement of the structure using the deduced model is in progress.

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Keywords: quasicrystal crystallography, direct methods, structure simulation

P.22.02.1

Acta Cryst. (2005). A**61**, C475-C476

Diffuse Scattering from Composite Crystals Containing Stacking Faults

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The compound $(Ca_2CoO_3)_{0.62}CoO_2$, a potential candidate for a thermoelectric material, has been revealed to be a misfit-layered compound, which consists of two interpenetrating monoclinic subsystems, CoO_2-part of CdI_2-type sandwiches and Ca_2CoO_3-part of ordered three-atom-thick NaCl-type blocks [1]. Sometimes reflections with specific indices showed rather large line-widths in powder diffraction patterns of the compound. They suggested occurrence of stacking disorder in one subsystem or in two subsystems. In some powder patterns of misfit-layered sulfides such as $(PbS)_{1.12}VS_2$, composed of VS_2 sandwiches and two-atom-thick NaCl-type PbS