Acta Cryst. (2002). A**58** (Supplement), C182

A NEW COMPOSITE STRUCTURAL DESCRIPTION OF Ca_xMO_2 COMPOUNDS (M = Cu-Co)

S. Lambert D. Grebille
Crismat Ismra Boulevard Du Marechal Juin CAEN 14050 FRANCE

In recent years, a constant attention has been focused onto copper in low dimensional structural environments showing an aperiodic character and related to interesting physical properties (high Tc superconductors, spin ladder compounds). Recently, a new composite structure was isolated in the Ca-Co-Cu-O system. The structure of the $Ca_{0.82}(Cu_{0.65}Co_{0.35}O_2)$ compound, determined by single crystal X-ray diffraction using the 4-D formalism [1,2] corresponds to two interpenetrating orthorhombic F sublattices, exhibiting incommensurate periods along the [100] direction. The two subsystems are respectively constituted of 1-D infinite $[MO_2]$ chains (M = Cu or Co) and of Ca layers. This structure is closely related to oxide structures already depicted [3], but the 4-D composite description gives a new interpretation of the Ca/(Cu+Co) ratio in the above formula. The actual symmetry of this structure class is now proved to be rigorously described in the superspace formalism. The modulation functions have been refined and allow a spectacular distance accommodation between the two sublattices. Moreover, the structure refinement shows that a substitution of Co for Cu in this type of compound is possible, which could be compatible with a charge localization of the type Cu^{2+}, Co^{3+}.

References
[1] T. Janssen et al, International tables for crystallography, vol C, 797 (1992)
[2] refinement program JANA, V. Petricek et al, Acad. of Sc. of the Czech Republik, Phaha (1998)
[3] T.G.N. Babu et al, Mat. Res. Bull. 26, 499-506 (1991); T. Siegrist et al, Chem. Mat. 2, 192-194 (1990)

Keywords: APERIODIC STRUCTURE COPPER OXIDE 4D REFINEMENT

Acta Cryst. (2002). A**58** (Supplement), C182

STRUCTURAL CHARACTERIZATION OF YV_4O_8

M. Onoda A.C. Dhaussy Y. Kanke
National Institute for Material Science Advanced Materials Laboratory Namiki 1-1 TSUKUBA IBARAKI 305-0044 JAPAN

The structure of a multiple-domain crystal of YV_4O_8 was refined on the assumption that α-form, β-form and their respective twin forms intergrowth mutually. The model was expressed as a commensurate composite crystal with two kinds of subsystems, one is V_4O_8 part and the other is Y part, and refinement was performed using high-dimensional formalism and all reflections from all domains simultaneously. Next, diffuse streaks observed in the X-ray and electron diffraction patterns were simulated using the matrix method which has been used for one-directional disorder such as stacking faults.

Keywords: **COMPOSITE CRYSTAL, DIFFUSE SCATTERING, PHASE TRANSITION**

Acta Cryst. (2002). A**58** (Supplement), C182

INCOMMENSURABILITY AND DISORDER IN 4'4-DIETHOXYAZOBENZENE STRUCTURE

CB Pinheiro[1] M Gardon[2] G Chapuis[1]
[1]Universite De Lausanne Institut De Cristallographie BSP - Dorigny LAUSANNE 1015 SWITZERLAND [2]Institut furExperimentalphysik, Universita Wien - Strudlhofgasse 4, A-1090 Wien - Austria

The 4,4'-Diethoxyazobenzene ($C_{16}H_{18}N_2O_3$) presents two distinct solid state phases by cooling from the melting point around 403 K down to 100 K. Phase I, stable above 356 K, is modulated [1]. Its average structure was initially described in the monoclinic space groups *Cc* and *C*2 with four independent molecules per unit cell [1]. The central oxygen (also named azoxy group) was considered to be randomly distributed between two sites. An irreversible phase transition takes place at 356 K leading to a triclinic structure, space group *P*1, with 2 independent molecules per unit cell [1].

Recent experiments carried on X-ray diffractometers equipped with Image Plate and CCD area detectors have shown that the 4,4'-Diethoxyazobenzene modulated phase is also disordered as indicated by the presence of organized and strong diffuse scattering rods as well as many first and second order satellite reflections in the reciprocal space. A new refinement of the average structure of the modulated phase indicates that the entire 4,4'-Diethoxyazobenzene molecules should be split in order to properly describe all the atomic displacement parameters. In this new refinement the position of the azoxy group defined the conformation of the entire molecule and four distinct orientations could be observed in the crystal structure. The refinement of the incommensurate phase is taking into account discontinuous modulation between the four possible orientations observed in the average structure refinement.

References
[1] Sciau, P., Lapasset, J. and Moret, J. (1988). Acta Cryst. C44, 1089-1092.

Keywords: **ORGANIC COMPOUNDS, MODULATION, DISORDER**

Acta Cryst. (2002). A58 (Supplement), C182

FREE PARAMETERS IN SYMMETRY ADAPTED MODELS FOR MODULATED STRUCTURE REFINEMENT

L. Pytlik W. Sikora F. Bialas
University of Mining and Metallurgy Faculty of Physics and Nuclear Techniques Al. MIckiewicza 30 KRAKOW 30-601 POLAND

Models used for refinement of modulated structures are usually presented as sets of Fourier coefficients describing the occupation probabilities or displacement vectors of particular atoms(molecules). The use of symmetry is restricted to imposing extra constraints for symmetry equivalent atoms. However the same model can be described using smaller number of parameters. The essential fact is that structural phase transitions usually take place according to one or more irreducible representations of the high symmetry space group. Such a property allows calculation of the symmetry reduction model and the respective atomic translations or occupation probabilities. Practical implementation of the above procedure can be found in 'MODY' program, written by W.Sikora and P.Czapnik, calculating the basis functions for a given representation. The calculated basis functions exhibit some interesting features: as a rule they are orthogonal and they contain only a limited number of parameters which describe the structure on all atomic sites. As the basis functions are usually complex an additional restriction is imposed: the coefficients of the linear combination of basis functions should be selected in such a way that the resulting structure model (displacements or probabilities) has to be real. The respective system of equations can be reduced to a linearly independent set and then solved for the unknown coefficients. After such operation the final structure model contains clearly defined minimum number of free parameters, which can be easily transformed to the equivalent set of Fourier coefficients. Examples will be given for some typical cases.

Keywords: **STRUCTURE REFINEMENT SYMMETRY**