

Poster Sessions

not to be of universal character when scaled against the correlation length and instead depends on the interaction of faulting. Furthermore, even in the case where it is clear the presence of large density of defects, and therefore independent faulting does not hold, direct methods still applies.

The developed procedure was used to analyse the layer disorder in $\text{RE}_2\text{Co}_{17}$ alloys (RE: rare earth) which undergo a reordering transition from rhombohedral to hexagonal arrangement when going from the light rare earth elements to the heavy rare earth. In all cases the structures show different degree of planar disorder. The X-ray diffraction experiments were conducted on the beam line XPD at the LNLS synchrotron facility in Campinas, Brazil. The increasing ordered character of the hexagonal phase was quantified as well as the evolution of the rhombohedral stacking through the value of the correlation length. A k parameter has been introduced in the decaying term of the probability correlation length, allowing to follow quantitatively the interaction of the faulting as well as the distance of the actual faulting to the random non-interacting model. The reconstructive phase transition in this system seems to follow a path where the new phase appears within the disordered phase and grows without homogeneous reordering of the disordered arrangement.

- [1] B.E. Warren, *X-Ray Diffraction*. Addison-Wesley **1969**. [2] D. Pandey, P. Krishna, *J. Phys. D: Appl. Phys.* **1977**, *10*, 2057–2068. [3] L. Velterop, R. Delhez, T.H. Keijser, E.J. Mittemeijer, D. Reefman, *J. Appl. Phys.* **2000**, *33*, 296–306. [4] D.P. Varn, G.S. Canright, J.P. Crutchfield, *Phys. Rev. B* **2002**, *66*, 40, 12086–12097. [5] E. Estevez-Rams, B. Aragon-Fernandez, H. Fuess, A. Penton-Madrigal, *Phys. Rev. B* **2003**, *68*, 064111–064123. [6] E. Estevez-Rams, U. Welzel, A. Penton-Madrigal, E.J. Mittemeijer, *Acta Cryst.* **2008**, *A64*, 537–548.

Keywords: defect, stacking, alloy

MS74.P21

Acta Cryst. (2011) **A67**, C678

MEM analysis of electron-density distribution in apatites

Oxana V. Magdysyuk,^a Robert E. Dinnebier,^a Pavel E. Kazin,^b Mikhail A. Zykin,^b Sander van Smaalen,^c Jeanette Netzel,^c ^a*X-Ray Diffraction Service Group, Max Planck Institute for Solid State Research, Stuttgart (Germany)*. ^b*Chemistry Department, Moscow State University, Moscow (Russia)*. ^c*Lab. of Crystallography, University of Bayreuth (Germany)*. E-mail: O.Magdysyuk@fkf.mpg.de

Recently, the possibility was demonstrated that 3d-metals ($M = \text{Cu}, \text{Ni}, \text{Zn}$) can be incorporated into strontium hydroxyapatite powders, where they partially occupy the channels and form linear O-M-O groups separated by hydroxide anions [1], [2]. The positions and isotropic displacement parameters of the atoms (isotropic ADPs) were refined by the Rietveld method. As unavoidable in Rietveld refinement, the strong correlations between the different crystallographic parameters do not allow the refinement of anisotropic ADPs in the neighborhood of Sr. In order to obtain information about the anisotropic ADPs of the in-channel atoms, the maximum entropy method (MEM) has been used as a complementary tool. In this work, the MEM was used to derive the most probable distribution of the electron density and to determine the ionic charge of atoms of hydroxyapatites with different content of metal atoms from powder diffraction data.

An almost completely *ab-initio* electron-density distribution can be obtained by the MEM using a combination of structure-factor amplitudes from a Le Bail fit of the diffraction data (disregarding any structural model) with phases from Rietveld refinement of the initial structural model [3]. The Le Bail fit provides amplitudes of structure factors as they would have been directly measured by single-crystal diffraction, except that for each group of overlapping reflections only

the sum of the intensities can be used. As an initial model for the determination of the phases of structure factors, the published atomic coordinates of the investigated compounds were used [1], [2] without metal and oxygen atoms in the channels of the apatite. Therefore, no prior structural information about locations of these atoms was used for the determination of the electron-density distribution. For the reconstruction of the electron density in the unit cell the computer program BayMEM [4] was used.

As examples, the two-dimensional maps of the electron-density distribution of the atoms of pure hydroxyapatite and hydroxyapatite with copper atoms in the channels $\text{Sr}_5(\text{PO}_4)_3\text{Cu}_{0.1}\text{O}_{0.2}(\text{OH})_{0.8}$ are shown in Fig. 1. The electron-density distribution of the Cu atoms is isotropic, suggesting the absence of positional and thermal displacement of the Cu atoms.

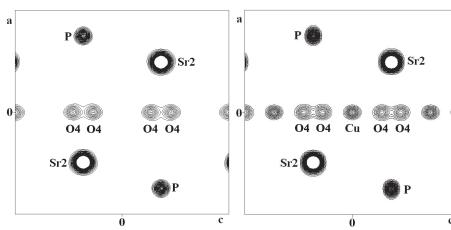


Fig. 1. Two-dimensional electron-density maps at $y=0$ of pure hydroxyapatite (left) and hydroxyapatite with copper atoms (right).

- [1] P.E. Kazin, A.S. Karpov, M. Jansen, J. Nuss, Yu. D. Tretyakov, *Z. Anorg. Allg. Chem.* **2003**, *629*, 344–352. [2] P.E. Kazin, O.R. Gazizova, A.S. Karpov, M. Jansen, Yu.D. Tretyakov, *Solid State Science* **2007**, *9*, 82–87. [3] A. Samy, R.E. Dinnebier, S. van Smaalen, M. Jansen, *Acta Cryst.* **2010**, *B66*, 184–195. [4] S. van Smaalen, L. Palatinus, M. Schneider, *Acta Cryst.* **2003**, *A59*, 459–469.

Keywords: MEM, apatite, powders.

MS74.P22

Acta Cryst. (2011) **A67**, C678–C679

Detour into two dimensions: a new method for powder structure solution

Dan Xie, Christian Baerlocher, Lynne B. McCusker, *Laboratory of Crystallography, ETH Zurich, (Switzerland)*. E-mail: dan.xie@mat.ethz.ch

A single-crystal charge-flipping algorithm [1] has been applied to 2-dimensional projections derived from X-ray powder diffraction data to retrieve structure factor phases. These phases proved to be as reliable as those obtained from high-resolution transmission electron microscopy (HRTEM) images or from precession electron diffraction (PED) data [2]. In particular, the stronger reflections tend to be correctly phased. The 2-dimensional electron density “images” obtained in this way show the same features as the corresponding HRTEM images, but with higher resolution. Application of the powder charge-flipping algorithm [3] to the full 3-dimensional powder diffraction data in conjunction with phases derived from several such (arbitrarily selected) projections was found to have a significant and beneficial effect on the structure solution.

The approach was first developed and tested using data collected on the complex zeolite TNU-9 [4], IM-5 [5] and SSZ-74 [6]. All three of these structures were originally solved by combining X-ray powder diffraction and electron microscopy data, because X-ray diffraction data alone were not sufficient. In all three cases, the phase information derived from 2-dimensional subsets of the X-ray powder diffraction data resulted in a significant improvement in the electron density maps generated by the powder charge-flipping algorithm. The inclusion of this phase information allowed all three structures to be determined