

MS30.P13

Acta Cryst. (2011) A67, C419

Diffuse scattering study of γ -pigment red 170

Rangana Warshamanage,^a Anthony Linden,^a Hans Beat Burgi,^{a,b} Dmitry Chernyshov,^c ^aInstitute of Organic Chemistry, University of Zürich (Switzerland). ^bDepartment of Chemistry and Biochemistry, University of Bern (Switzerland). ^cSwiss-Norwegian beamlines at European Synchrotron Radiation Facility, Grenoble (France). E-mail: rangana.warshamanage@access.uzh.ch

Pigment Red 170 is an economically important automotive coating in the paint industry, but subject to fading. Structurally it has four polymorphic forms namely the α , β , γ and δ -polymorph. Among them the γ -polymorph shows superior pigment properties. [1] It has been found that modifications of some of the molecule's substituents can affect the durability, but a rational approach to such modifications requires an understanding of the crystal structure of the pigment. Despite the importance of Pigment Red 170 in many industrial applications only a few structural studies have been carried out. [1,2] Only recently, Schmidt *et al.*, have determined the structures of the α - and γ -phases from a combination of X-ray powder diffraction and crystal structure prediction algorithms. [1,2] According to the description given, both phases have layer-like structures.

Single crystal data of the γ -phase have been collected by Schmidt on a sealed tube diffractometer. They show diffuse scattering. We have collected Synchrotron data from the same crystal and found that the observed rods of diffuse scattering are typical of faulted layer stacking. There is currently some uncertainty concerning the correct unit cell that should be used to describe the average structure. The Synchrotron data suggest a larger unit cell than that derived by Schmidt. We will describe our results from the attempts to derive the correct unit cell and solve and refine the average structure.

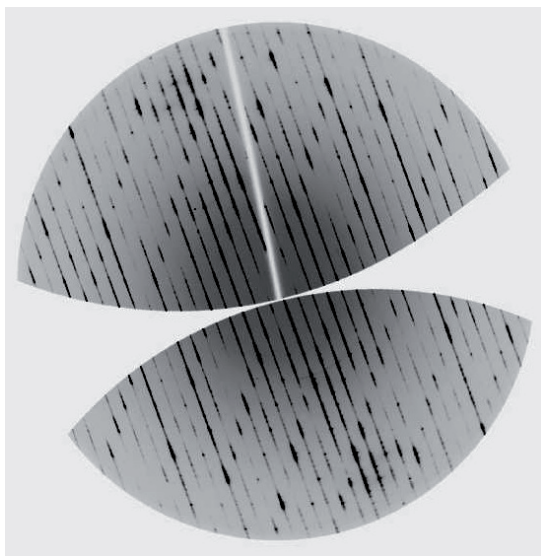


Fig. 1. The $h2l$ reciprocal lattice plane from the γ -phase of Pigment Red 170 collected on the BM01A beamline at SNBL (ESRF).

[1] M.U. Schmidt, D.W.M. Hofmann, C. Buchsbaum, H.J. Metz, *Angew. Chem. Int. Ed.* **2006**, *45*, 1313-1317. [2] W.D.S. Motherwell, H.L. Ammom, J.D. Dunitz, A. Dzyabchenko, P. Erk, A. Gavezzotti, D.W.M. Hofmann, F.J. J. Leusen, J.P.M. Lommerse, W.T.M. Mooij, S.L. Price, H. Scheraga, B. Schweizer, M.U. Schmidt, B.P. van Eijck, P. Verwer, D. E. Williams, *Acta Cryst.* **2002**, *B58*, 647-661.

Keywords: disorder, diffuse_scattering, pigment

MS30.P14

Acta Cryst. (2011) A67, C419

Real structure of $\text{Sr}_{1-x}\text{Ba}_x\text{Si}_2\text{O}_2\text{N}_2:\text{Eu}^{2+}$ ($0.5 < x < 0.8$) phosphors by TEM and XRD

Oliver Oeckler, Tobias Rosenthal, Markus Seibald, Wolfgang Schnick, Department of Chemistry, LMU Munich (Germany). E-mail: oliver.ockler@gmx.de

Oxonitridosilicates (sions) such as $\text{Sr}_{1-x}\text{Ba}_x\text{Si}_2\text{O}_2\text{N}_2:\text{Eu}^{2+}$ are known as efficient phosphors for pc-LED applications. The silicate layers consist of highly condensed SiON_3 tetrahedra interconnected by N^{3-} atoms while metal atoms are located between those layers. The emission wavelength can be tuned by changing the atomic ratio Ba: Sr, ranging from blue to yellow-green [1]. The corresponding crystal structures have been discussed controversially in the literature. The compounds exhibit a wide variety of real-structure effects like twinning, intergrowth and stacking disorder [2,3]. In order to determine complex structural phenomena in mixed Sr/Ba sions, a combination of electron microscopy and diffraction as well as X-ray diffraction methods has been employed successfully. For $\text{Sr}_{0.5}\text{Ba}_{0.5}\text{Si}_2\text{O}_2\text{N}_2:\text{Eu}^{2+}$, diffuse intensities observed in X-ray powder and electron diffraction patterns are fully explained by simulations based on a model including stacking disorder as well as twin and anti-phase boundaries. The unexpected red-shifted luminescence can be understood based on the detailed knowledge of structural features.

For $\text{Sr}_{1-x}\text{Ba}_x\text{Si}_2\text{O}_2\text{N}_2:\text{Eu}^{2+}$ ($x \approx 0.25$), single-crystal X-ray data show that the crystal structure is a variant of that of $\text{BaSi}_2\text{O}_2\text{N}_2$; however, the metal atom layers are corrugated in a similar fashion as in $\text{SrSi}_2\text{O}_2\text{N}_2$ which otherwise exhibits a different structure. The Ba rich compounds exhibit disordered silicate layers and show pronounced diffuse intensities in diffraction patterns. Additional intergrowth of (pseudo)orthorhombic and triclinic domains in $\text{Sr}_{1-x}\text{Ba}_x\text{Si}_2\text{O}_2\text{N}_2:\text{Eu}^{2+}$ ($x \approx 0.25$) is evident in HRTEM images, SAED patterns and reciprocal lattice sections calculated from X-ray data. The structures of the individual domains are corroborated by HRTEM simulations based on structure models developed from single-crystal data combined with information from electron diffraction. Experimental luminescence spectra show two maxima which correspond to the two different structure types building up the intergrown domains.

[1] V. Bachmann, C. Ronda, O. Oeckler, W. Schnick, A. Meijerink, *Chem. Mater.* **2009**, *21*, 316-325. [2] O. Oeckler, F. Stadler, T. Rosenthal, W. Schnick, *Solid State Sci.* **2007**, *9*, 205-212. [3] J. A. Kechele, O. Oeckler, F. Stadler, W. Schnick, *Solid State Sci.* **2009**, *11*, 537-543.

Keywords: HRTEM, diffuse scattering, oxonitridosilicate

MS30.P15

Acta Cryst. (2011) A67, C419-C420

Water-mediated hierarchal structure in room temperature ionic liquids

Hiroshi Abe,^a Masami Aono,^a Yusuke Imai,^a Takahiro Takekiyo,^b Yukihiro Yoshimura,^b ^aDepartment of Materials Science and Engineering, ^bDepartment of Applied Chemistry, National Defense Academy (Japan). E-mail: ab@nda.ac.jp

Room temperature ionic liquids (RTILs) have been big subjects of environmental friendly "green chemistry". Almost zero vapor pressure in RTILs is one of outstanding features. We have already reported anomalous behaviors of *N,N*-diethyl-*N*-methyl-*N*-2-methoxyethyl ammonium tetrafluoroborate ([DEME][BF₄])-H₂O mixtures by simultaneous X-ray diffraction and DSC measurements.[1-5] In particular, at 1 mol% H₂O, two kinds of superstructures, volume