

KN3 Phase Homology and Structural Prediction in Extended Series of Inorganic Bi-based Compounds. Mentré Olivier,^a Diana Endara,^a Almaz Aliev,^a Marie Colmont,^a Marielle Huvé,^a UCCS, CNRS UMR 8181 Université Lille Nord de France, USTL, F-59655 Villeneuve d'Ascq, France, E-mail: olivier.mentre@ensc-lille.fr

we are interested in the organization of bismuth-based 1D sizeable building-units (BUs) into new XO_4 containing frameworks ($X=P, As, V$). In the $Bi_2O_3-X_2O_5-MOx$ (M =various cations) ternary diagrams, structural relationships between the phases in competition have been generalized with respect to systematically found sizeable BUs. For the first time we present here an unified model which allows a generalization of most of the reported compounds of these chemical systems and an easy distinction of pertinent BUs and comprehension of their assembly into the final edifice. The concerned crystal-types mainly follow from the X/M for Bi cationic substitution in the $-Bi_2O_3$ fluorine-like structure. In its ideal form, the $-Bi_2O_3$ is better described from a regular lattice of edge-sharing $(O)Bi_4$ "anti-tetrahedra" [1,2]. It involves that all cations align along a "square-grid". Experimentally, we observe that XO_4 tetrahedra substitute the Bi-sites with important constraints, such that the "cationic grid" persists but strongly distorted. The degree of distortion is proportional to the ratio of XO_4 . Strikingly, sizeable 1D(2D)-ribbons(planes) of $O(Bi)_4$ tetrahedra persist (=BUs), surrounded by XO_4 groups. On the basis of such an extended rational model, the structural prediction, formulation and elaboration of novel archetypes have been successfully achieved for several terms [3,4]. Currently, the characterization of variously sized BUs from $n=1$ to emphasizes the thermo-dynamical stability of various structural types, despite their closed chemical compositions. We will pay special attention to several aspects such as HREM/XRD complementarity, disorder-order duality and recent tailormade compounds including polar materials.

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KN4 Towards Routine Structure Solution using Precession Electron Diffraction. Paul A. Midgley, Alexander S. Eggeman, *Department of Materials Science and Metallurgy, University of Cambridge, Pembroke Street, Cambridge, CB2 3QZ, UK.* E-mail: pam33@cam.ac.uk

Although x-ray and neutron diffraction will always be the techniques of choice to solve the vast majority of crystal structures, there are many examples where the small beam available in an electron microscope is invaluable in helping to first visualise and then solve the structures of small precipitates, interfacial phases etc. Precession electron diffraction [1] is the electron analogue of the x-ray technique but where the beam, rather than the sample, is precessed about the optic axis of the electron microscope to form a diffraction pattern whose intensities are integrated through the Bragg condition. In practice the beam is scanned in a hollow cone above the specimen and then de-scanned below the specimen to bring the beam back onto the optic axis. The geometry of the precessed electron diffraction pattern is then identical to a conventional pattern but with a far larger number of reflections visible due to the rocking nature of the beam. It has been found over the past decade or so that precessed intensities can be used to solve structures treating the intensities in a pseudo-kinematic fashion. Of course, in general, the strong interaction of the electron beam with the crystal leads to multiple scattering and so although precessed intensities can be used successfully for solving structures, there remains the question of why they can be used in this way. In this paper we will explore the reasons why precession diffraction works for structure determination, focussing on the geometry of the technique, the variation of intensities with precession angle and specimen thickness and how these 'mimic' the behaviour of kinematic intensities. New structure solution algorithms, based on charge flipping methods [2], will be discussed that are suited to electron intensities. Finally, we will discuss the issue of structure refinement using precession data. Refined structures using precession data often have very high R-values, primarily because we are comparing kinematic and non-kinematic intensities. Recently we have proposed an alternative refinement method [3] that considers the rank, or order, of the reflection, rather than the intensity and refines the structure with respect to that rank. Initial work shows the structure refinements are indeed improved over those using conventional refinement procedures.

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