

MS78.P12

Acta Cryst. (2011) A67, C692**Structural and functional study of ComE, a key actor of *S. pneumoniae* competence**

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S. pneumoniae is the leading cause of community-acquired infections worldwide. Its global success might in part be explained by its genetic transformation, which consists of the internalization and the incorporation in the bacterial chromosome of exogenous DNA. Transformation is turned on in cells which are in a physiological state called competence. The induction of the competent state depends on the two-component regulatory system ComD-ComE. The response regulator (RR) ComE is phosphorylated by the histidine-kinase ComD, and acts as transcriptional activator of a large set of competence-specific operons. These genes encode in particular proteins involved in binding, uptake, processing, and recombination of exogenous DNA, but they are also involved in the virulence of *S. pneumoniae*¹. The prototypical RR contains a conserved regulatory domain, linked to a variable effector domain. The majority of RRs (63%) contain DNA binding effector domains. Among them, 5% interact with DNA through the unusual and recently characterized LytTR domain². They form the AgrA/LytR RR family and regulate production of many important virulence factors. Despite their interest for drug development, no full-length RR structure has been determined yet within this family. ComE belongs to the AgrA/LytR family. We initiated a structural study of the ComE non-phosphorylatable D58A mutant, and solved its X-ray 3D structure at 3.4 Å resolution. As expected, ComE D58A contains a conserved regulatory domain and a LytTR domain. The two domains are linked by a long non-structured linker. Interestingly, ComE D58A forms a dimer in the crystal. The regulatory domains are linked by a two-fold symmetry axis within the dimer, whereas the LytTR domains are related by both a translation and rotation (head-to-tail). This asymmetric dimer configuration is facilitated by the flexibility of the linker. The sequence of the ComE promoter is known and consists of two direct repeats. Although ComE D58A was crystallized without DNA, the dimer conformation seems to be consistent with promoter binding, which incited us to set out for a functional mechanistic study.

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Keywords: ComE, competence, Response Receptor

MS79.P01

Acta Cryst. (2011) A67, C692**Full structure solution of aluminides using precession electron diffraction data**

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It is well-known that if single crystals of sufficient size are available, single crystal X-ray diffractometry is used for structure solution. Else, structure of the new phase should be solved by other methods. In the era of nano-science, standard powder X-ray diffraction is not always applicable due to the peak broadening effect, thus electron

crystallography (EC) emerges as important and sometimes the only possible tool for structure determination of nano-sized crystals. One of the essential problems causing a limited use of EC for this purpose is the dynamical nature of electron scattering. In the past decade, researchers have shown that influence of dynamical effects on the intensities of diffracted beams can be substantially reduced if, instead of conventional illumination of the specimen, beam precession technique (PED) is used [1], [2]. Following this finding, several structures of zeolites, complex oxides and minerals were solved using PED technique. Until today, structure of aluminides, which belong to the wide class of intermetallic compounds, was never solved fully (including atomic positions) using solely EC methods. It must be noted that strategies for structure solution of intermetallics should be different from those of zeolites and complex oxides, since for intermetallics no strict constraints on coordination polyhedra, interatomic distances and angles can be applied.

Current research focuses on development of strategies for full structure determination of aluminides. The work was done in two steps: a) full structure solution of a case study, for this purpose structure of well-known Mg₁₇Al₁₂ (β) phase was investigated; and 2) structure determination of novel aluminides found in ternary Al-T₁-T₂ systems (where T₁ and T₂ are transition metals).

The main steps in structure solution methodology used in this work were: 1) determination of geometry of the unit cell using electron diffraction (ED); 2) space group estimation evaluating the symmetry of the PED patterns using the methodology described in [3]; and 3) development of atomic model applying direct methods (utilized in SIR program [4]) on PED datasets. The extraction of the intensities from the experimental PED and conventional selected-area ED (SAED) patterns was performed using ELD program [5]. Symmetry considerations were applied on electron diffraction data and several datasets were prepared merging data extracted from different zone axis patterns. It should be noted that due to uncertainty, the intensities were treated as kinematical ($I \propto |F_{hkl}|^2$) and as dynamical ($I \propto |F_{hkl}|$). It was found that data should be treated as kinematical only.

Present research proves beyond any doubt that PED intensities could be used as an input for direct methods and as a result full atomic model of aluminides can be derived, while SAED data was proved to be dynamical and unsuited for this purpose. Different approaches and influence of method's variables on structure solution were checked and proper strategies for structure determination of aluminides were suggested.

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Keywords: solution, TEM, intermetallic

MS79.P02

Acta Cryst. (2011) A67, C692-C693**The anion deficient perovskite-based homologous series A_nB_nO_{3n-2}**

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We have discovered a new A_nB_nO_{3n-2} homologous series of anion deficient perovskites, where the anion deficiency occurs due to

periodically spaced crystallographic shear (CS) planes. The CS planes delimit quasi two-dimensional blocks of the perovskite structure with a thickness of $(n-2)$ BO_6 octahedra, n being the number of the homologue in the series.

Using the appropriate chemical composition and thermal treatment conditions we have isolated in a single phase form different members of the homologous series with $n = 4$ ($\text{Pb}_{4-2x}\text{A}_{2x}\text{Fe}_4\text{O}_{10}$ ($\text{A} = \text{Sr}, \text{Ba}$)), $n = 5$ ($\text{Pb}_{2.9}\text{Ba}_{2.1}\text{Fe}_4\text{TiO}_{13}$, $\text{Pb}_2\text{Ba}_2\text{BiFe}_5\text{O}_{13}$), $n = 6$ ($\text{Pb}_{3.8}\text{Bi}_{0.2}\text{Ba}_2\text{Fe}_4\text{Ti}_{1.8}\text{O}_{16}$, $\text{PbBaBiFe}_3\text{O}_8$) and $n = 7$ ($\text{Pb}_{4.5}\text{Bi}_{0.2}\text{Ba}_{2.4}\text{Fe}_{4.9}\text{Ti}_{2.1}\text{O}_{19}$) [1], [2].

The crystal structures of the obtained compounds have been determined using a combination of diffraction and electron microscopy techniques: X-ray (XRD) and neutron powder diffraction (NPD), electron diffraction (ED), and high angle annular dark field scanning transmission electron microscopy (HAADF-STEM). The compounds crystallize in an orthorhombic crystal system with the lattice parameters $a \approx a_p\sqrt{2}$, $b \approx b_p$, $c \approx 9.7\text{\AA} + (n-2)a_p\sqrt{2}$, where $a_p \approx 4.06\text{\AA}$. The space groups are $Pnma$, $Ammm$ and $Imma$ for the 4th, 5th and 6th members, respectively. The $\text{A}_n\text{B}_n\text{O}_{3n-2}$ structures can be represented as a sequence of atomic layers $-\text{ABO}-(\text{O}_2-\text{ABO})_{n-2}-\text{O}_2-\text{ABO}-\text{ABO}-(\text{O}_2-\text{ABO})_{n-2}-\text{O}_2-\text{ABO}-$. HAADF-STEM reveals a long range ordered sequence of the perovskite modules with a uniform thickness for members with $n = 4-6$.

In this type of structures, the cations are partially ordered. At the CS planes, tunnels are created in which only the lead and bismuth cations are situated. The formation of tunnels allows the spatial accommodation of the lone electron pair of these cations. The coordination number of the B cations at the CS planes is 5 and these BO_5 tetragonal pyramids are occupied exclusively by Fe^{3+} cations. The remaining Fe^{3+} cations as well as Ti^{4+} cations are located in the BO_6 octahedra of the perovskite modules. The CS planes introduce edge sharing connections of the transition metal-oxygen polyhedra at the interface between the perovskite blocks. This results in intrinsically frustrated magnetic couplings between the perovskite blocks due to a competition of the exchange interactions between the edge- and the corner-sharing metal-oxygen polyhedra.

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Keywords: perovskite, homologous series, lead

MS79.P03

Acta Cryst. (2011) **A67**, C693

Electron crystallography based on inverse dynamic scattering

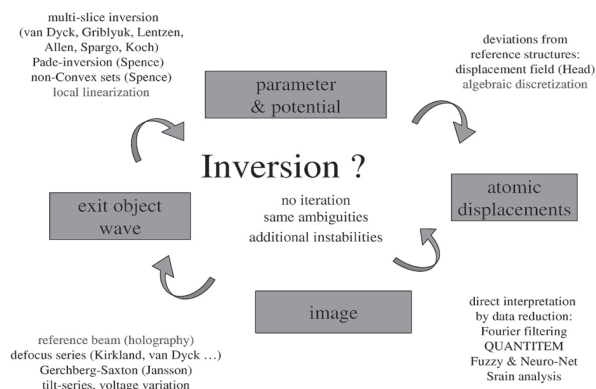
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Electron crystallography and electron microscope tomography may enhance structure investigations via local object analysis. However, this has in contrast to X-ray techniques the disadvantage, that an ill-posed inverse problem for the highly nonlinear dynamical theory has to be solved. Fig. 1 schematically shows all necessary steps of the object analysis: anticlockwise the trial-and-error technique, clockwise the direct solution of the inverse problem. Authors which proposed partial solutions are listed, too. Details are to be found in [1] with respective references. In [1], [2] also a special solution of the inverse problem for the local parameter analysis is given. Step 1 of the inversion is solved, by replacing the image by a hologram or a defocus series, which makes the problem linear, and finding the exit object wave by inverse Fourier transform. Step 2 yielding the object structure directly from the exit

wave, is the real challenge of inversion. Step 3, the analysis of lattice defects, has no explicit solution yet.

The solution proposed here for step 2 can be characterized in short as follows [1], [2]: The first step of inversion yields the moduli and phases for all reflections of the experimental exit plane waves Φ^{exp} as function of lateral (pixel) positions (i,j) . Theoretical waves Φ^{th} are then calculated using the dynamical scattering matrix \mathbf{M} for an a priori model characterized by the number of beams and the scattering potential \mathbf{V} . With a suitable experimentally predetermined a priori beam orientation \mathbf{K}_0 and sample thickness t_0 as a free parameter, a perturbation approximation yields both Φ^{th} and \mathbf{M} as linear functions of the parameter to be retrieved. Its analytic form enables the inverse solution of $\|\Phi^{\text{exp}} - \Phi^{\text{th}}\| = \text{Min}$ yielding directly for each image pixel the local thickness $t(i,j)$, the local beam orientation $\mathbf{K}(i,j)$, and further data of the parameter space.

The linearization transforms the ill-posed problem to a well posed one and enables the analytic inverse. However, the solution is ill-conditioned. With the generalized and regularized inverse in [2] an enhancement of the solution is given. By applying second order perturbation and using a mixed type potential $V(i,j) = q_k(i,j) V^k$ in [1] with the parameters $q_k(i,j)$ also local potential variations can be retrieved [3]. Smoothing, coupling of different parameters via additional a priori information, including the iteration of the start values, if the retrieved parameter goes beyond the confidence region, finally help to avoid modeling errors.



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Keywords: inverse problems, electron holography, analytic approximation

MS79.P04

Acta Cryst. (2011) **A67**, C693-C694

Automatic diffraction tomography (ADT) with precession on 6H-SiC and NiTe

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Automated electron Diffraction Tomography (ADT) in combination with precession electron diffraction (PED) has been recently used in the investigation of nano-crystalline materials. By the use of this method