

[3] K. A. Müller, W. Berlinger, M. Capizzi, H. Gränicher, *Solid State Commun.* 8, 549 (1970). [4] J. Leist, J. Sidoruk, H. Gibhardt, K. Hradil, M. Meven, G. Eckold, accepted in *diffusion fundamentals*.

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Monoclinic superstructure of mullite-type KAl_9O_{14} .
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The chemical composition of the Al, Si–mullites is given by the general formula $Al_2(Al_{2+2x}Si_{2-2x})O_{10-x}$. The amount of Si^{4+} substituted by Al^{3+} is correlated with the sum of oxygen vacancies necessary to retain charge balance. In mullite-type aluminates $X^{2-2x}Al_2(Al_4)O_{10-x}$ negative charge is compensated by incorporation of equivalent number (2-2x) positively charged X cations [1].

Single crystals of KAl_9O_{14} (or $K_{0.67}Al_6O_{9.33}$) were grown using a flux method. Purity of the sample was confirmed by SEM-EDX analyses. Colourless needle-like prisms of KAl_9O_{14} , some as long as 1 cm, show with cross-polarised light twin planes perpendicular to the needle axis. The structure was determined from single-crystal data and refined to a residual $R|F| = 0.031$. The compound crystallizes in the monoclinic space group $P2_1/n$ ($a = 8.1937(6)$, $b = 7.6734(6)$, $c = 8.7930(6)$ Å, $\beta = 110.747(6)^\circ$, $V = 516.99(7)$ Å³, $Z = 2$). A detailed analysis based on oriented sections of reciprocal space revealed that the crystal is also subject to twinning by partial merohedry which was accounted for during the refinement. Furthermore, distinct one-dimensional diffuse scattering parallel to $c^*-(a^*/3)$ could be observed.

Crystals of KAl_9O_{14} have a typical mullite-type structure with linear edge sharing AlO_6 octahedral chains connected with inner-chain groups comprising two AlO_4 tetrahedra and one AlO_5 trigonal bipyramid. The 4+1 coordinated aluminium has four shorter (1.75-1.86 Å) and one longer bond (2.16 Å) to oxygen atoms. K-atoms are located in the centre of cavities originating from oxygen vacancies. Differences between the calculated powder diffraction pattern of this structure and known powder diagrams of $K_{0.67}Al_6O_{9.33}$ [2] clearly confirmed that this structure is a new polymorph.

The monoclinic superstructure of KAl_9O_{14} can be transformed to a $3 \times c$ orthorhombic supercell ($a = 7.69$ b = 7.68 c = 8.82 Å). A similar ($3 \times a$) superstructure was postulated for the 4:1 mullite earlier [3].

[1] Schneider H., Komarneni S., *Mullite*, WILEY-VCH, Weinheim, 2005 [2] Fischer, R.X.; Schmücker, M.; Angerer, P.; Schneider, H.; *Am. Mineral.*, 2001, 86, 1513. [3] Fischer, R.X.; *Am. Mineral.*, 1994, 79, 983.

Keywords: KAl_9O_{14} , mullite, superstructure

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Structural characterization of a new Pb/Sr based ferri-manganite presenting incommensurate shear structure by combining different techniques of transmission electron microscopy.

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Perovskite related iron and manganese based Crystallographic Shear (CS) structures have first been discovered in the Pb rich part of the Pb-Sr-Fe(Mn)-O diagram. Recent studies on the CS perovskite $PbFeO_{2.5}$ [1], [2], [3] evidenced the role of the lone pair cation in the shear process and a large family of compounds $Pb_{4m+3n}Fe_{4(m+n)}O_{10+9n}$ with different shear $1/2[110]_p(h0l)_p$ (p denotes perovskite) mechanisms has been defined. The $1/2[110]_p(\bar{3}05)_p$ structure of $Pb_{18}Fe_{20}O_{48}$ ($Pb_{0.9}Fe_{0.24}$) [1] will be used as a comparative example to describe the structure of our Pb-site deficient perovskite-like compound.

The latter is a ferri-manganite synthesized for the composition $Sr_{0.61}Pb_{0.18}(Fe_{0.75}Mn_{0.25})O_{2.29}$ and its new modulated CS structure, related to perovskite, was characterized by transmission electron microscopy. The Selected Area Electron Diffraction study allowed to describe the structure in a C centered monoclinic supercell with the parameters : $a_m = 27.595(2)$ Å, $b_m = 3.8786(2)$ Å, $c_m = 13.3453(9)$ Å and $\beta = 100.125(5)^\circ$, refined from powder X-ray diffraction data. The structure determined from the High Resolution Transmission Electron Microscopy (HRTEM) and High Angle Annular Dark Field Scanning Transmission Electron Microscopy (HAADF STEM) studies corresponds to a $1/2[110]_p(203)_p$ CS structure, following the concept of a phasoid with two coexisting variants (“1” and “2”) with the same monoclinic unit cell. The difference takes place at the translational interface, creating two local phases coexisting in the same particle with either double or single tunnels, where Pb atoms are likely located [4]. The incommensurability phenomena are associated with the formation of defective boundaries through structural unit groups, which are common part of the normal sequences encountered in the two variants, leading to a slight misalignments of the “1” and “2” monoclinic supercells along \bar{a}_m [5].

The structural resolution carried out from the precession electron diffraction data allowed to identify the 2 monoclinic phases “1” and “2” with the good positions of the different cations.

This compound exhibits an insulating behavior coupled with a strong antiferromagnetism with the presence of a small coercive field hysteresis [5].

[1] Abakumov A.M., Hadermann J., Bals S., Nikolaev I.V., Antipov E.V., van Tendeloo G., *Angew. Chem., Int. Ed.*, 2006, 45, 6697. [2] Hadermann J., Abakumov A.M., Nikolaev I.V., Antipov E.V., van Tendeloo G., *Solid State Sci.*, 2008, 10, 382. [3] Abakumov A.M., Hadermann J., van Tendeloo G., Antipov E.V., *J. Am. Ceram. Soc.*, 2008, 10, 1807. [4] Lepoittevin C., Hadermann J., Malo S., Pérez O., van Tendeloo G., Hervieu M., *Inorg. Chem.*, 2009, 48, 8257. [5] Malo S., Lepoittevin C., Pérez O., van Tendeloo G., Hervieu M., *Chem. Mater.*, 2010, 22, 1788.

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