MS20-O4 **The structure of nano-twinned rhombohedric $YCuO_{2.66}$ solved by electron crystallography.**

Holger Klein,[a] V. Ovidiu Garlea,[b] Céline Darie,[a] Pierre Bordet,[a]
[a] *Institut Néel, CNRS and Univ. J. Fourier, Grenoble, France,*
[b] *QCMD, Oak Ridge National Laboratory, USA*
E-mail: holger.klein@grenoble.cnrs.fr

$YCuO_{2+d}$ compounds can provide suitable models for studying frustrated spin-interactions and quantum fluctuations in low-dimensional triangular lattices. The introduction of extra oxygen atoms into the delafossite structure ($YCuO_2$) enhances the average formal valence of copper ((+2 for $\delta = 0.5$, and +2.33 for $\delta = 0.66$) and provides exchange paths between the resulting s = ″ spin moments [1]. In spite of being introduced almost a decade ago by Cava *et al.* [2], the crystal structure of $YCuO_{2.66}$ remained elusive. This was mostly due to the fact that only powder samples have been available.

Here, we report on the structure determination of the $YCuO_{2.66}$ phase by means of transmission electron microscopy (TEM).

To synthesize $YCuO_{2.66}$ an intermediate phase $YCuO_{2.5}$ was obtained by annealing of 2H-$YCuO_2$ at 460°C for 15h in flowing oxygen. $YCuO_{2.5}$ was then annealed at 400°C for 24h under oxygen pressure of 158 atm using an external heating autoclave [3]. Powder X-ray diffraction and powder neutron diffraction studies suggested a hexagonal cell (a = 6.22973 Å, c = 33.5065 Å) with space group $P6_3/mmm$. A Rietveld refinement even yielded a reasonable structure model. Careful examination of the sample by TEM revealed, however, the presence of twinning in the sample. All inspected powder particles (crushed in an agate mortar and deposited on an amorphous carbon membrane) showed twin domains with a width of about 10 to 15 nm. The individual domains were determined to be of rhombohedric symmetry (a = 6.23 Å, c = 33.5 Å in a hexagonal cell). The twin boundaries are parallel to the (0 0 1) planes and the direction of the c-axis is reversed from one domain to the next, thus mimicking a hexagonal symmetry. Precession electron diffraction (PED) patterns were recorded with a parallel beam and a selected area aperture from an area containing a large number of twin domains. Due to the rhombohedral extinction rules ($-h + k + l = 3n$) the reflection spots of both twin orientations superimpose in the systematic hkl rows with $-h + k = 3n$ while they are separated in the other rows. The comparison of the intensities of the separated spots yielded an estimate of the volume fraction of the two orientations and the intensities of the superimposed spots were partitioned according to the volume fraction. Diffracted intensities were extracted from PED patterns of three zone axes representing a data completeness of 44% up to 1.0 Å resolution. The structure was solved by direct methods in SIR2008 assuming a pseudo-kinematical approximation (I ~ F2) after applying a Lorentz-type correction for the precession geometry. The resulting structure can be described in terms of layers containing either Y or Cu. The cations form a triangular lattice in each layer. The present results will serve as a basis for further analysis of powder X-ray and neutron diffraction data that will enable precise determination of the oxygen positions.

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[2] Cava, R. J., Peck Jr, W. F., Krajewski, J. J., Cheong, S. W. & Hwang,H. (1994) *J. Mater. Res*., **9**, No. 2, 314
[3] Garlea V. O. et al . *Solid State Sciences* **8 (2006)**, 457

Keywords: precession electron diffraction; structure solution; twins

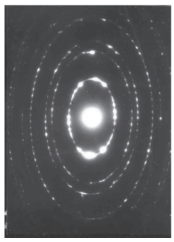
MS20-O5 **New electron diffraction rotation methods.**

M.G. Kyazumov, *Institute of Physics of the National Academy of Sciences of Azerbaijan, H.Javid av.33,Baku 370143.*
E-mail: elmira@physics.ab.az

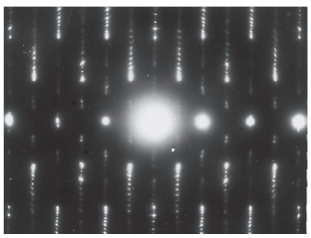
Last years the object of electron diffraction investigations become not only nano thicness samples, and also the samples having the nano- and mikro dimensional areas. Therefore the working out various new electron diffraction methods of the specific advantages is very actual.

Earlier we had been developed two methods[1(fig.1),2]: The following new electron diffraction methods has been developed:

- During registering of the diffraction process, monocrystal film rotates around one of the axes on the co-ordinate plane of reciprocal lattice which is located perpendicularly to the falling electron beam. This method concerns to the crystals with the hexagonal and higher symmetry (fig. 2);
- During registering, monocrystal film rotates around one of the co-ordinate axes of atomic or reciprocal lattice which is located perpendicularly to the falling electron beam. The rotation around each of these axes differs each other not only under the scheme of obtain of electron diffraction patterns (EDP) and also by the formulas of their decoding. Therefore each of them is represented as the separate method (fiq.3). Last methods concern to the crystals with low symmetry.



Fiq.1. PED pattern of GaSe, $\varphi = 55^o$.



Fiq.2. EDP of 2H polytype of $CdInGaS_4$ rotating around an axis h00.

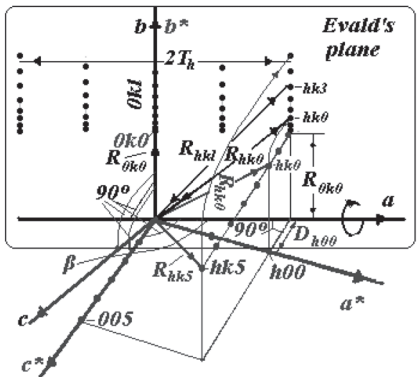


Figure 3. The scheme of obtaining of EDP from monoclinic crystal rotating around an axis *a* of atomic lattice.

[1] Zvyagin, B.B., Kyazumov, M.G., et.al. (1990). Kristallogr. (Sov.) v. 35, № 3, 602-.609.
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Keywords: 1 – electron diffraction method, 2 – thin films, 3 – layered compounds.