## X-RAY DIFFRACTION FROM α-QUARTZ CRYSTALS UNDER ELECTRIC FIELDS

<u>R. Guillot<sup>1</sup> P. Alle<sup>1</sup> P. Fertey<sup>1</sup> N.K. Hansen<sup>1</sup> E. Elkaim<sup>2</sup> <sup>1</sup>laboratoire De Cristallographie Et Modelisation De Materiaux Mineraux Et Biologiques, Universite Henri Poincare Faculte Des Sciences - B.P. 239 Vandoeuvre-Les-Nancy 54506 France <sup>2</sup>lure, Centre Universitaire Paris Sud, B.P. 34 - 91898 Orsay Cedex, France</u>

The aim of our work is to analyze by diffraction techniques the correlations between structural and physical properties of crystals onto which an electric field is applied. In the laboratory in Nancy we have, based on the ideas of previous work [1], build a device using a field switching technique. It consists of a high voltage supply, the electronics for switching the field, and synchronous counting on four chains combined with a control for stepscanning the diffraction profiles [2]. By using this stroboscopic technique, it is possible to measure very small changes in the Bragg angles due to the strain resulting from the converse piezoelectric effect, and also to measure minute changes in the Bragg intensities due to polarisations of atomic structure and electron density. A first measure was carried out at LURE with the 4-circle diffractometer WDIF-4C on α-quartz, since perfect crystal samples can easily be obtained. Furthermore, quartz is a well-characterised piezo-electric material. Electrodes were vapour deposited onto the (1 1 0) extended faces of a crystal plate of dimensions 5x5x0.5 mm. Our measurements reproduce the known piezo-electric coefficients of a-quartz. We have measured changes in Bragg intensities for 27 reflections, 21 unique. Different atomic models have been investigated in search for an explanation of the origin of piezoelectric effect. Our conclusions are compared with a previous study with a similar technique [3].

[1]A. Paturle, H. Graafsma, H. -S. Sheu, P. Coppens & P. Becker (1991) Phys. Rev. B43, 14683-14691.

[2] R. Guillot, P. Allé, P. Fertey, N. K. Hansen & E. Elkaïm (2002) J. Appl. Cryst. in print.

[3] U. Pietch, J. Stahn, J. Davaasambuu & A. Pucher (2001) J. of Physics and Chemistry of Solids 62, 2129-2133.

# Keywords: ELECTRIC FIELD PIEZO-ELECTRIC EFFECT $\boldsymbol{\alpha}$ QUARTZ

### Acta Cryst. (2002). A58 (Supplement), C327

### INFLUENCE OF RARE EARTH ELEMENTS ON THE STRUCTURE AND PROPERTIES OF THE PEROVSKITE-LIKE PHASES IN Ru-Sr-Ln(Ce)-Cu-O SYSTEM

<u>G.M. Kuzmicheva</u><sup>1</sup>I.E. Kostyleva<sup>2</sup> E.P. Khlybov<sup>3</sup> A.S. Andreenko<sup>1</sup> Academy of Fine Chemical Technology 86 Vernadsky PR MOSCOW 11757 RUSSIA

<sup>1</sup>M.V.Lomonosov State Academy of Fine Chemical Technology, Moscow, Russia <sup>2</sup>L.F.Vereschagin Institute for Physics High Pressure, Troitsk, Russia <sup>3</sup>International Laboratory of High Magnetic Fields and Low Temperatures, Wroclaw, Poland

RuSr2LnCu2O8-d Perovskite-like cuprates with (Ru-1212) and RuSr<sub>2</sub>(Ln,Ce)<sub>2</sub>Cu<sub>2</sub>O<sub>10-d</sub> (Ru-1222)composition are a new class of compounds in which a magnetic order and superconductivity coexist. We report the preparation, X-ray study, electrical and magnetic properties of nonsuperconducting and superconducting phases with 1212 (space group P4/mmm) and 1222 (space group I4/mmm) types structure based on Ru. Polycrystalline samples of nominal compositions RuSr<sub>2</sub>LnCu<sub>2</sub>O<sub>8-d</sub> and RuSr<sub>2</sub>(Ln,Ce)<sub>2</sub>Cu<sub>2</sub>O<sub>10-d</sub> with Ln=La,Pr,Nd,Sm,Eu,Gd,Tb,(Gd,Y); (Ru,Cu)Sr<sub>2</sub>YCu<sub>2</sub>O<sub>8-d</sub> and  $(Ru,Cu)Sr_2(Y,Ce)_2Cu_2O_{10\text{-}d}$  were prepared by the solid-state reaction of stoichiometric quantities of high-purity oxides via a three-step procedure with several intermediate regrindings. As a rule, the superconducting properties appear in the presence of flowing oxygen either at atmospheric pressure or at high pressure. A characteristic feature of Ru-Sr-Ln(Ce)-Cu-O system is the formation of (Ln,Sr)(Ru,Cu)O<sub>3-d</sub> phase with cubic perovskite structure (sp. gr. Pm3m) as an impurity. The La, Pr, Nd and Tb elements have the effect of increasing the cubic perovskite concentration in a sample. The Ru-1212 and Ru-1222 structures contain a rotation and tilt of the RuO<sub>6</sub> octahedra. The formal charge of Cu (FC Cu) calculated from the electroneutrality condition of refined phase composition achieves an optimal value (FC Cu=2.15) for the superconducting Ru-1212 (Tc=39 for Gd) and Ru-1222 (Tc=40K for Ln=Nd, Sm, Eu, Gd and Tc=10 K for Ln=(Gd,Y)) phases. Our study shown that the starting composition and different treatment conditions of samples are due to changes in structure, superconducting properties and madnetic ordering.

## Acta Cryst. (2002). A58 (Supplement), C327

## RARE-EARTH ELEMENTS IN MOLECULAR CONDUCTORS, CRYSTAL AND ELECTRONIC STRUCTURES

<u>O.N. Kazheva</u><sup>1</sup> E. Canadell<sup>2</sup> N.D. Kushch<sup>1</sup> O.A. Dyachenko<sup>1</sup> <sup>1</sup>Institute of Problems of Chemical Physics of RAS Institutskii Prosp., 18 CHERNOGOLOVKA MOSCOW REGION 142432 RUSSIA <sup>2</sup>Institut de Ciencia de Materials de Barcelona, Spain

Using rare-earth elements in electroconducting salts - a novel approach to the creation of molecular conductors. In the course of study of the influence of rare-earth elements on the structure and properties of conducting compounds synthesized new radical cation salts on the basis we of bis(ethylenedithio)tetrathiafulvalene (ET) and tetramethyltetraselenafulvalene (TMTSF) containing a complex anions of rare-earth elements. The new conducting  $(ET)_5[M(NCS)_6NO_3]C_2H_5OH$  (M = Dy, Y, Ho) and (TMTSF)<sub>3</sub>[Y(NO<sub>3</sub>)<sub>5</sub>]<sub>2</sub>C<sub>6</sub>H<sub>5</sub>Cl salts were synthesized by electrocrystallization, studied by X-ray crystallography at 110 and 295 K, and their electronic band structures were examined. In the (ET)<sub>5</sub>[M(NCS)<sub>6</sub>NO<sub>3</sub>]C<sub>2</sub>H<sub>5</sub>OH (M = Dy, Y, Ho) salts pentagonal bipyramid [M(NCS)<sub>6</sub>NO<sub>3</sub>]<sup>4-</sup> anion structure seems to be the reason for forming an earlier unknown packing type of the radical cation layers - $\Omega$ -type, which determines semiconducting properties of the compounds. In the new (TMTSF)<sub>3</sub>[Y(NO<sub>3</sub>)<sub>5</sub>]<sub>2</sub>C<sub>6</sub>H<sub>5</sub>Cl semiconductor directions of the TMTSF stacks in the neighboring organic layers are practically perpendicular. Such a disposition of conducting stacks allows to regard the  $(TMTSF)_3[Y(NO_3)_5]_2C_6H_5Cl$  salt as a precursor of 2D conductors formed by 1D conducting layers characterized mutually perpendicular directions of conductivity. In general the creation of a crystal with 2D conductivity using 1D blocks is a novel promising approach in crystal engineering of molecular conductors.

This work was supported by the RFBR (project No. 02-03-32794).

Keywords: MOLECULAR CONDUCTORS; CRYSTAL STRUCTURE; ELECTRONIC STRUCTURE

Acta Cryst. (2002). A58 (Supplement), C327

#### CRYSTALLOGRAPHIC ASPECT OF SPINAL OXIDE M<sub>3</sub>O<sub>4</sub> FORMED DURING ATOMISATION OF STAINLESS STEEL TYPE OF 310 P. Liu

R & D Cetre, AB Sandvik Steel Physical Metallurgy R & D Centre, AB Sandvik Steel, SANDVIKEN 811 81 SWEDEN

Oxides may form during atomization of stainless steels. The effects of formation of oxides on the properties depend upon their shape, size and distribution. The studied stainless steel was type of 310 with a nominal composition of  $24_{26}$  Cr<sub>19</sub>22 Ni<sub>61</sub>603; 2 Mn<sub>61</sub>603; 1.5 Si<sub>61</sub>603; 0.25 C in wt %. The oxides formed during atomization of stainless steel was extracted from the matrix and studied using Jeol 2000 FX transmission electron microscope operated at 200 kv.

Particles of spinal oxide of  $M_3O_4$  with a size ~300 nm were observed. EDX analysis indicated that it corresponds to  $MnCr_2O_4$  (*Fd-3m*, a = 8.437 Å). With a tilting of a crystal of the spinal oxide it shows a spherical shape at the zone axis <111>; rhombus at the <110> and cubic at <100>. The different shape reflects the crystallographic symmetry at the particular zone axis. This implies that the crystal had a high interfacial energy with the matrix and likely formed before the matrix was solidified.

Reference

1. E. Whipple and A. wold, J. Inorg. Nucl. Chem., 24, 1962, 23-27.

# Keywords: OXIDE, STAINLESS STEEL, AND ELECTRON MICROSCOPY