12.5-06   A NEW FORM OF NA-KURROL SALT STUDIED BY POWDER-PROFILE X-RAY DIFFRACTION. By A.Immirzi and W. Porzio, Istituto di Ingegneria dell'Università, Salerno and Istituto di Chimica delle Macromolecole del CNR, Milano, Italy.

Polymeric phosphates are being studied by means of powder profile X-ray diffraction (Rietveld method), using constrained refinement techniques (A.Immirzi, Acta Cryst. (1980) B36, 2378-2385; A.Immirzi, Gazz. Chim. Ital. (1980) 110, 381-387). Samples of Na-Kurrol salt type B (K.H.Jost, Acta Cryst. (1963) 16, 640-642), under prolonged grinding, are transformed into a more dense modification of higher crystallographic symmetry. The linear $(PO_3)_n$ anionic chains, which have approximate 4_1 helix conformation in type B phase gain exact 4_1 conformation and the crystallographic symmetry changes from $P2_1/n$ to $I4_1/a$. Crystal data are: a=b=13.167, c=5.925 Å, 16 $NaPO_3$ units per unit cell, D_c=2.64 g cm^{-3}. Structure determination was based only on X-ray powder diffraction data. The tetragonal symmetry was inferred from the 2Θ positions of a number of resolved peaks and the structure, deduced by trial, was refined by using the Rietveld method combined with constrained refinement according to two models with different degrees of freedom. In the first model fixed P-O bond lengths and free coordinates for Na^+ cations were used (11 variables), thus resulting in a final disagreement index R_2= $\Sigma|I_{obs}-I_{calcd}|/I_{obs}$=0.058. In the second model, the variables were reduced to 8 by imposing the local C_{2v} symmetry on each PO_2 unit and the R_2 obtained was 0.063 . The reduction of the number of structural variables appreciably increases their reliability. The resulting P-O-P and O-P-O bond angles as well as the O···O and Na^+···O packing distances are comparable with the values obtained in analogous compounds by means of single-crystal techniques.

12.5-07   POWDER NEUTRON DIFFRACTION STUDY OF THE NONSTOICHIOMETRIC SOLID SOLUTION OF LITHIUM TANTALATE $9LiTaO_3$:Ta_2O_5. By A. Santoro, R. S. Roth, and M. Austin, National Measurement Laboratory, National Bureau of Standards, Washington, DC 20234, USA.

The defective structure of nonstoichiometric $LiTaO_3$ has been investigated with the powder neutron diffraction technique and the profile fitting method of Rietveld (J. Appl. Cryst. (1969) 2, 65). The tantalum compound was selected instead of $LiNbO_3$ to avoid the complication that might arise with the possible reduction of Nb^{+5} to Nb^{+4}. The composition $9LiTaO_3$:Ta_2O_5 was chosen to introduce in the structure the largest possible number of defects allowed in the solid solubility range of Ta_2O_5 in $LiTaO_3$. The use of the powder method was adopted to avoid the need to grow large, homogeneous, and well-characterized, single crystals. The neutron diffraction data were collected with the five-detector diffractometer at the National Bureau of Standards reactor (Prince and Santoro, Natl. Bur. Stand. US Tech. Note 1117 (1980) pp. 11-12). Of the models proposed for explaining the deviations from the ideal composition of $LiTaO_3$, those of Lerner, Legras, and Dumas (J. of Crystal Growth (1968) 3, 231), of Nassau and Lines (J. Appl. Phys. (1970) 41, 533), and of Raeuber (Current Topics in Materials Science (1977) Vol. 1, ed. E. Kaldis, North Holland Publishing Co.) have been considered. Statistical tests on the results of least-squares fittings to the experimental data favor the model of Nassau and Lines (loc. cit.) over the other two.

12.6-01   DISORDER SCATTERING IN NEUTRON POWDER DIAGRAMS. By H. Boysen, Institut f. Kristallographie d. Universität, München, FRG.

Different kinds of disorder in crystals (0-, 1-,2-,3-dimensional, static or dynamic) give rise to diffuse intensity in reciprocal space in the form of peaks, streaks, planes and in the whole volume, resp. In a powder diagram this appears as a characteristic modulation of the background, which can be described by characteristic (corresponding to the dimensionality) functions containing the resolution of the diffractometer. From a refinement program valuable information on the parameters of the disorder can be drawn. In combination with the Rietveld program this also eases the treatment of the background. The reactor hall background has to be subtracted via a measurement with the empty can.

The case of glassy carbon serves as a good example, since it shows strong diffuse scattering of different origins. Glassy carbon consists of a network of folded fibrils constructed of graphitic carbon ribbons packed together in random orientation. This leads to diffuse peaks (hk0) and streaks (hkζ), an intense small angle scattering from small pores, incoherent scattering of carbon and 3-dim. disorder scattering from incorporated hydrogen. All these components have been included to refine the total pattern. An excellent agreement is found between the calculated and observed profiles. Parameters like particle sizes, strains, amount of H, interlayer distances, etc. can be determined.

12.7-01   THE FORMATION OF DEFORMATION TEXTURE IN STEELS UNDERGOING MARTENSITE TRANSFORMATIONS. By V.I. Yushkov, R.A. Adamesku, T.M. Gapeka, Ye.S. Machnev and P.V. Geld, Urals Polytechnical Institute, Sverdlovsk 620002, USSR.

An investigation by X-ray diffraction has been carried out on the peculiarities of texture formation in maraging-type steel having 15% Cr, 5% Ni and 2% Cu, and undergoing martensite transformation during heating and cooling, as well as during cold plastic deformation.

It has been found that hot rolling of the steel results in the formation of strained austenite texture in central layers of the sheet, this texture being characteristic of f.c.c. metals and alloys having high stacking fault energy. It is described by (135)[$2\bar{1}\bar{1}$], (110)[001], (110)[$\bar{1}12$] and (100)[001]-components. In contrast, the texture in the surface layers of the sheet is characterised by preferred orientation of (111)[$0\bar{1}\bar{1}$]-[$11\bar{2}$], (100)[011] and (211)[$01\bar{1}$]-types. The formation of the latter is due to friction.

The non-uniformity of austenite texture along the gauge of the sheet is inherited by martensite texture as well. In addition, the preferred orientations of martensite and the components of austenite texture have a Kurdumov-Sachs relationship.

The martensite texture in the central layers of the sheet is characterised by (111)[$11\bar{2}$], (211)[$01\bar{1}$], (100)[011] and (311)[$01\bar{1}$]-components, and that in the surface layers by (110)[$\bar{1}12$], (110)[$\bar{1}11$] and (211)[$\bar{1}11$]-components.

Strain martensite whose orientation coincides with that of quenching martensite is formed during the cold rolling of the investigated steel. Such strain-induced phase transformation makes a significant contribution to the mechanism of deformation.

The tendency of residual austenite to γ→ α transformation during the deformation is different in the surface and central layers of the sheet. This is accounted for by the orientation relationship of strain martensite formation. α-martensite is first of all formed in the sections of austenite having {100} <UVW> or {hkℓ} <011> -orientation.

Cold rolling with large amounts of reduction results in the formation of martensite texture typical for the deformation texture of b.c.c. metals and alloys: (100)[011], (211)[01$\bar{1}$], (111)[11$\bar{2}$] and (111)[01$\bar{1}$].

12.7-03   APPROXIMATION OF THE POLE DISTRIBUTION OF FIBRE TEXTURES BY SIMPLE FUNCTIONS. By <u>A. Apostolov</u> and I. Tomov, Institute of Physical Chemistry, Bulgarian Academy of Sciences, Sofia, Bulgaria.

The simple functions $1/[1+(\emptyset-\emptyset_0)^2/\alpha^2]^3$ and Gaussian are found to give a good approximation of intensity distribution in the range of the maxima of the pole figures. The values of the intensities, expressed as multiples of the random distribution by means of the above functions, are calculated and applied to the volume fractions of fibre (axially symmetrical) textures. The calculations are performed with a method developed previously (Tomov, I., H.J. Bunge, Texture of Cristalline Solids (1979), 3, 73).
The determination of the volume fractions of the texture components of two nickel samples (with initial matrix orientation <100> and <110>, respectively) was carried out, for three different pole figures, both with approximating functions and with numerical integration by Simpson's method. A good fit was obtained. The errors in the methods of computation are discussed.
Evaluating the volume fractions by approximating functions requires less experimental data and a shorter time for calculation. The sharper the texture, the more precise is the evaluation of the volume fractions.

12.7-02   DETERMINATION OF THE SHAPE OF CRYSTALLITES IN POLYCRYSTALLINE ZINC OXIDE. By <u>D. Louër</u>, R. Vargas, Laboratoire de Cristallochimie, Université de Rennes, 35042 Rennes Cédex, France and J.I. Langford, Department of Physics, University of Birmingham, Birmingham B15 2TT, England.

In size determination by means of diffraction broadening it is often of interest to know the shape of crystallites and their relations with the structure and the external form. For example, if there is evidence that the crystallites are prismatic, by X-ray line profile analysis the shape and size of the domains and their orientation with respect to the crystallographic axes can be deduced. However, data of high quality are required and suitable reflexions must be available.

As an example of this procedure, the case of polycrystalline zinc oxide produced by thermal decomposition of zinc hydroxynitrate was considered. Indeed, the differences in the broadening of the diffraction lines are explained with an anisotropic shape of the diffracting domains (Auffrédic, Ciosmak, Louër and Niepce, Proc. 9th I.S.R.S., Cracow, 1980, p. 590-593). In this case, microstrains are negligible and preliminary analysis of the breadths of several reflexions indicated that, on average, the crystallites could be regarded as cylinders of height 250 Å and diameter 90 Å, with the cylinder axis parallel to the c axis. It therefore seemed reasonable to assume that the crystallites are in fact hexagonal. There are, then, two likely orientations of the domans, with the base edge parallel to the [100] direction or to the [1 1 0] direction. Theoretical models for diffraction line profiles have been derived for materials with the crystallite shape of a cylinder or a hexagonal prism. The two models are compared to the zinc oxide powder pattern.