

### Easy crystal centring in a modified Gandolfi camera

X-ray powder patterns can be simulated for a single crystal provided that it is rotated about its centre of gravity in arbitrary directions while it is exposed to the X-rays. A camera which allows such a movement to be made has been described by Gandolfi (1967) and is commercially available.\* It represents essentially a Debye-Scherrer camera in which the sample support may be turned about two different axes, one being coaxial with the axis of the film cylinder (axis A, see Fig. 1) and the other (axis B) forming an angle of about  $45^\circ$  with it. These axes intersect in the X-ray beam and rotate with different velocities. In order to obtain powder patterns of good quality (*i.e.* showing sharp and continuous diffraction lines) and to reduce the exposure time, it is essential that the crystal is situated exactly at the point of intersection of these axes. This requires a careful and often time-consuming centring procedure under a telescope, which may be cumbersome in practice because it involves a simultaneous alignment of the crystal with respect to two axes. The purpose of this note is to describe a slight modification to this camera which allows one to align the crystal separately for each axis and thus to centre it more rapidly in the camera.

In the modified version the alignment is actually done in two steps. As can be seen from Fig. 1, the sample support can be fixed in two positions, an axial position (position 1) in which it turns about axis A, as in a Debye-Scherrer camera, and a peripheral position (position 2) in which it turns about axis B corresponding to the Gandolfi geometry. The crystal to be investigated is first mounted in position 1 and aligned with respect to the cylinder axis (A). This can be done in a straightforward manner by displacing the brass pin supporting the glass fibre and crystal within the sample holder by means of, for instance, adjustable screws as illustrated in Fig. 1. Once the crystal is aligned, the sample support is removed and mounted in position 2. Centring with respect to the second axis (B) is then not necessary and one only has to find the point of intersection of axes A and B in order to align the crystal in the X-ray beam. This can be achieved very simply by rotating the crys-

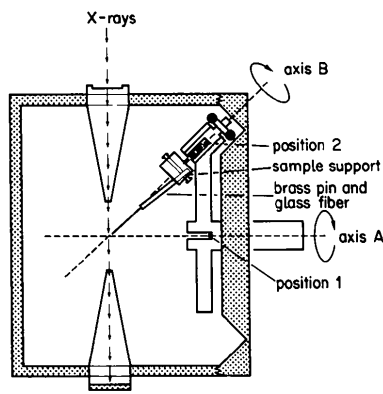


Fig. 1. A schematic drawing of the modified Gandolfi camera.

tal support about axis A (and axis B) and by translating it parallel to axis B until the amplitude of the precession-like movement of the crystal as observed under the microscope has become minimal. This two-step procedure is more convenient and less time-consuming than the conventional procedure and has been tested on a newly constructed camera. However it could also be implemented on the original camera because the modifications required are only minor. These consist essentially of drilling a hole coaxial with the cylinder axis corresponding to the usual Debye-Scherrer geometry to provide a position for the sample support.

The camera was constructed by members of the Atelier de la Section de Physique, to whom we extend our sincere thanks.

A. GRÜTTNER  
K. YVON

Laboratoire de Cristallographie aux  
Rayons X

M. DELALOYE

Département de Minéralogie  
University of Geneva  
Geneva  
Switzerland

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## Meeting Report

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### Crystallography in materials science (Joint Spring Conference: The Institute of Physics Crystallography Group and Materials and Testing Group), University of Bath, 10-12 April 1978

The attendance at this conference was relatively small (about 47 delegates) and there were more delegates who were crystallographers than materials scientists.

The Conference had four themes: (a) phase transitions; (b) disordered and amorphous materials; (c) texture and preferred orientation; (d) general X-ray applications including medical materials.

In the first session Professor G. A. Saunders (Bath) gave an introductory lecture on the investigation of soft phonon modes, mainly by ultrasonics. Starting with definitions of order parameters and of types of phase transitions the lecturer showed how a displacive phase transition, for example in barium titanate, removes symmetry and can be used as a measure of order. At a second-order transition the vibrational (phonon) frequency approaches zero. This is at the Brillouin zone centre in ferroelectrics such as barium titanate, but at the zone boundary in antiferroelectrics (strontium titanate).

In elastic phase transitions (acoustic rather than optical phonon mode softening) shear may cause a change of lattice from for example cubic to tetragonal. Such martensitic transformations are found in  $\text{Ni}_3\text{Sn}$ , which has been studied acoustically over a range of temperatures. Indium-thallium is another example, in which experimental observations of mode softening at 22% Tl are confirmed by calculation from pseudo-potentials. It was agreed in answer to questions that study of the martensitic behaviour of iron would be valuable, but the experimental problems of operating at high temperatures were formidable.

Of the shorter contributions the phase changes in diamantane and triamantane were discussed by A. R. Bates, T. E. Jenkins and D. J. Somerford and by R. Cernik, E. H. M. Evans, R. Hine and J. P. G. Richards respectively, both groups being from University College, Cardiff. In the first study the phase transitions at 35 and  $127^\circ\text{C}$  were studied by ultrasonics, Raman scattering and dilatometry and the lower-temperature transition shown to be as-

\* Officina Elettrotecnica di Tenno, 38060 Tenno (Trento), Italy.

sociated with a soft librational mode at the centre of the Brillouin zone. In the second study a disorder parameter was defined, for the transition starting at 0°C and becoming complete at about 30°C, based on measured and calculated structure factors.

In the paper on order-disorder phenomena in complex oxides  $\text{Li}_2\text{TiO}_3\text{-MgO}$  (A. R. West and M. Castellanas, Aberdeen) a different order parameter was derived and shown to be exponentially related to the deviation of sample temperature from the critical value (1220°C). This study was by X-ray diffraction. M. Pickering and R. W. H. Small (Lancaster) reported on the structures and transitions of disordered phases of 2,3-dibromo-2,3-dimethylbutane. Three phase transitions were detected by DSC and studied by X-ray diffraction. In the two highest-temperature phases the crystal structures have been found, but the lowest-temperature phase has not yet been characterized. The change of entropy between the two higher-temperature phases agrees well with the calculated value.

The first session of the second day opened with an invited lecture by Professor A. Leadbetter (Exeter) on scattering studies of molecular and liquid crystals. Professor Leadbetter gave a clear description of scattering techniques as applied to several systems intermediate between crystal and liquid, *viz.* liquid crystals and crystals with rotational disorder. X-ray diffraction gives a time averaged picture of the structure, intense diffuse scatter indicating the existence but not the time scale of disorder. Quasielastic incoherent neutron scattering complements this information by giving time scales and, in addition, directions and amplitudes of movements. A particularly intriguing finding was that molecules in liquid crystals migrate from one layer to another.

The first short contribution in the second session was by D. M. Glover (CERL) on 'melting' transitions in crystalline potassium soaps. X-ray diffraction data on potassium caprate, in which a phase transition occurs at 76°C, was presented and showed that the 'melting' involved the six carbon atoms farthest from the ionic end group. R. Waring and A. H. Windle (Cambridge) presented an analysis of radial distribution functions in polymer science with special reference to polymethyl methacrylate. The main point of the paper was that termination errors in the Fourier transform method using X-ray, electron or neutron scattering data lead to a ripple which confuses the radial distribution function. Modification of the

normalization procedure was discussed. D. M. Nicholas, J. Stranger, P. M. Crankshaw and D. N. Davis (Thames Polytechnic) discussed both amorphous and crystalline nickel-phosphorus alloys as studied by X-ray diffraction and SEM. The alloys were prepared by both electrolytic and electroless deposition on a mild steel substrate.

The third session opened with a lecture by Professor I. M. Ward (Leeds) who surveyed the subject of texture and preferred orientation in polymers. He described the methods of computation of elastic and optical properties using orientation distribution functions and gave examples from semicrystalline (polyethylene) and amorphous (PMMA) materials. He also outlined the methods at present available for finding the orientation distribution function.

The lecture was followed by the contribution of D.M. Sadler and A. Keller (Bristol) on neutron scattering studies in polyethylene, in which it was shown that the method of deuteration of part of the polymer allows calculation of the form of the molecular trajectory in crystals. Solution-grown crystals appear to have molecules regularly folded in rows, which themselves fold about the *c* axis to form a 'superfold'. In marked contrast to this is the behaviour of melt-grown crystals, in which the molecules follow the pattern of random re-entry, but with some memory of the melt configuration. The second short contribution in the third session was also from Bristol, by J. Stejny, D. Dlugosz and A. Keller. It dealt with the preparation, electron microscopy and electron diffraction analysis of single crystals of poly(sulphur nitride). This is an inorganic polymer of interest in the field of solid-state physics in that it is superconducting. The chain orientation is uniform over the whole macroscopic crystal and the crystal is in consequence highly anisotropic.

R. Lovell and A. H. Windle (Cambridge) gave a short contribution on the structure of oriented polymer glasses, showing that in PMMA oriented by mechanical means the X-ray diffraction pattern can be used to narrow down the range of possible molecular conformations present. Calculation of possible sequences of bond rotation angles showed that the best agreement with the X-ray and conformational energy evidence was to be obtained with three *trans* followed by one *gauche* bond (*tttg*).

In session 4 Dr T. C. Tranter (University Hospital of Wales, Cardiff) presented an invited paper on the use of X-rays in the study of natural dental materials. He described the application of X-ray diffraction

techniques in the study of the structure of tooth enamel. Significant differences in crystallite size were found between samples of healthy tooth enamel and samples exhibiting dental caries. Samples of the latter type were found to have a larger average crystallite size than samples of the former, indicating that preferential dissolution of smaller crystallites by food acids had occurred. The talk was followed by a wide ranging discussion, which touched on the effect of diet on tooth decay and the possible effectiveness of fluoridization on enhancing crystal growth in tooth enamel.

L. S. Dent Glasser (Aberdeen) gave a short contribution on X-ray diffraction studies of cement and paint, contrasting direct powder diffractometry with the use of Guinier films and a newly developed microdensitometer. The aim was to establish the composition of an amorphous product mechanically inseparable from the reactants in both materials studied.

P. Stothart (N. I. Research in Dairying, Reading) gave an account of SAXS and WAXS from reverse-osmosis membranes of cellulose acetate (600–3000 Å thick). A model consisting of 230 Å particles packed into weakly diffracting paracrystallites was proposed.

The third paper, on cathodoluminescence, birefringence and X-ray topography as techniques for the study of diamond, was contributed by Mai Chen-Hung and A. R. Lang (Bristol). Dr Mai (Peking, China) read the paper, which was concerned with the differences between the three techniques and their application to radiation-damaged diamond.

In the final contribution to the conference T.F.J. Quinn and C.J.L. Tye (Aston, Birmingham) referred to the effects of heat treatment on the lattice parameters of PCTFE. They showed that the two tetragonal forms of the polymer unit cell were differently represented in annealed and in quenched polymer, the larger unit cell being more common in annealed material.

During the conference delegates and their guests were entertained at a Civic Reception in the Assembly Rooms in Bath and enjoyed a guided tour of the Museum of Costume.

R. G. C. ARRIDGE

*The H. H. Wills Physics Laboratory  
University of Bristol  
Bristol BS8 1TL  
England*

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