

16.1-2 KOSSEL-CAMERA STUDIES OF ISOTOPICALLY DISTINCT CRYSTALS BETWEEN 10K AND 370K  
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Most elements are solid solutions consisting of two or more stable isotopes. The volumes of these isotopes will be different at temperatures at which zero point energy is important, and for very hard materials such lattice parameter differences can be detected at RTP, but for most elements and compounds significant differences can only be expected near absolute zero.

A continuous-flow helium cold-finger (designed and made for us by Oxford Instruments Ltd) incorporates a large flange making a vacuum joint with an O-ring seal to the stage of a Stereoscan SEM. The cold finger can therefore be used in any other experiment on condition that a suitable vacuum jacket is provided, but it is used here with a Kossel camera (supplied by David Dingley of Bristol University) to obtain transmission divergent-beam patterns at various temperatures. The absorption conics on these patterns are sharp for focal spots  $\sim 1\mu$  diameter, and a radiation monitor attached to the camera is used as an exposure meter.

Cell parameters measured from Kossel patterns for which the magnification is not known must be obtained from ratios of measured lengths, not from the lengths themselves, but the ratios of lines joining selected intersections of adequate sensitivity can be combined to give very precise estimates of lattice parameters.

A particular advantage of using a SEM to obtain divergent-beam patterns is that a great variety of wavelengths can be employed by using suitable elements or compounds as the X-ray source, and computer programs can be used to select the optimum combination of crystal orientation and incident wavelength to give results in a specified temperature range.

Graphical simulation of the expected patterns, and graphs showing the behaviour of suitable ratios can be used to facilitate interpretation of the photographs, and accurate results even for organic crystals for which only a few low-angle reflections are strong enough to give good conics can be obtained if the crystals can stand the vacuum.

A particular advantage implicit in the use of the divergent-beam conics of radii (90- $\theta$ ) for isotope lattice parameter work is that their sensitivity increases as the d-spacing decreases, so that the precision of the measurements improves as the temperature is lowered towards the values at which the results are most significant.

As  $\theta$  approaches 90°, the resolution becomes infinite, and broadening of the conics due to the finite width of the spectral lines is easily observable. Indeed such films, which require exposure times of only a few minutes even on quite fine-grained film, offer a quick and simple method of profiling X-ray spectral lines if signal-to-noise problems can be overcome by suitable image analysis techniques. These problems arise because of the very small percentage reduction in intensity associated even with absorption conics of excellent visibility to the naked eye.

Results will be presented for members of various isotopic solid solution systems for which specimens are currently available to us as single crystals.

16.2-1 APPLICATION OF THE HIGH-ANGLE DOUBLE-CRYSTAL X-RAY DIFFRACTOMETRY (HADOX) TO PHASE-TRANSITION STUDIES. By A.Okazaki, N.Ohama, M.Sato and Y.Soejima, Department of Physics, Kyushu University, Fukuoka 812, Japan.

The method of HADOX (Okazaki and Ohama, J.Appl. Cryst. (1979) 12, 450) is advantageous in determining accurate peak profiles as a function of temperature. This therefore gives us, when we apply to phase-transition studies, not only the lattice constant vs temperature relation of high accuracy but also information on fine structures of the transition. The HADOX diffractometer (Ohama, Sakashita and Okazaki, J. Appl.Cryst. (1979) 12, 455) has been improved in several aspects; among them, the introduction of a microcomputer for automatic measurements and of a solid-state detector for separating observed peaks into components with different wavelengths are most important. Some results of the application of the new system are presented. For the structural phase transition in SrTiO<sub>3</sub> at 105 K, by using the in-situ topography in the HADOX arrangement, the precursor effect for the lattice constant is observed for a monodomain region. The effect can be discussed in terms of the soft R<sub>25</sub> mode as in the case of KMnF<sub>3</sub> (Sakashita and Ohama, Phase Transitions (1982) 2, 263). The peak profile of SrTiO<sub>3</sub> is anomalous below the transition temperature; it is examined as a function of the depth from the surface down to 55  $\mu$ m, by using white incident beams and a solid-state detector. The results are much more accurate than those of earlier experiments and reveal very detailed features of the transition.

16.2-2 A Wavelength Dispersion Diagram for a Monochromator/Diffractometer System with General Monochromator Orientation Angle,  $\phi_M$ .  
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A diffractometer for measurement of X-ray intensities from a small single crystal specimen, c, can have a crystal monochromator, M, located between the source and the specimen (ante-monochromator) with the monochromator crystal axis at a general angle,  $\phi_M$ , relative to the  $\omega$  axis of the specimen crystal (Mathieson, Rev. Sci. Instr., (1968), 39, 1834 - 37). The functional variation in  $\Delta\omega, \Delta 2\theta$  space of the  $\lambda$  dispersion of the M,c system is of concern for the measurement of Bragg reflections as  $\theta_c$  changes.

The locus of the  $\lambda$  dispersion of the M,c system in  $\Delta\omega, \Delta 2\theta$  space (Mathieson, Acta Cryst., (1982), A38, 378 - 387; Acta Cryst., (1984), A40, to be published) is deduced as follows

$$\frac{\Delta\omega}{\Delta 2\theta} = \frac{\tan\theta_c + \cos\phi_M \tan\theta_M}{2\tan\theta_c + \cos\phi_M \tan\theta_M} \quad (1)$$

From (1), the change in dispersion for the parallel and anti-parallel regions and for the crossed-crystal case (as well as the no-monochromator case) will be presented in a readily-appreciated diagrammatic form.