

01-Instrumentation and Experimental Techniques (X-rays,  
Neutrons, Electrons)

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dimensional intensity data were unable to be collected with an usual four-circle diffractometer. This indicates that if the data collection may be performed more quickly, a wide variety of reactions would be made clear on the basis of the structure analysis. We have designed and constructed a new type of diffractometer, which is composed of a  $\kappa$ -type goniometer, a no-screen type Weissenberg camera with two imaging plates as detectors and a reader for the imaging plate with a laser. The crystal is mounted on a  $\kappa$ -type goniometer and is aligned automatically by two still photographs. Then the three-dimensional intensity data were collected within an hour with the Weissenberg camera using two imaging plates alternately for recording and reading the data. All the operations are computer-controlled. Some examples showing the dynamical structural changes have been obtained using the new diffractometer.

## MS-01.01.06 SUB-NANOSECOND TIME RESOLUTION IN LAUE DIFFRACTION USING A THIRD GENERATION SYNCHROTRON

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The high brilliance of third generation synchrotrons will make it possible to shorten the exposure time in diffraction measurements by a factor  $\sim 10^2$ - $10^4$  compared to existing SR sources. Of particular interest for time resolved crystallography is the Laue method which makes optimal use of the polychromatic synchrotron spectrum and permits the recording of near complete data sets in a single shot for high symmetry crystals.

We present here the design of the white beam station BL3 at the ESRF, which will use a soft wiggler with 48 poles and a maximum field of 0.76 T at 20 mm gap giving a critical energy of 16 keV. The deflection parameter K can be varied between 2 and 5. The polychromatic radiation is focused by a toroidal mirror and ray-tracing calculations show that the 10 mA single bunch mode will produce  $4 \times 10^{10}$  photons onto a small  $0.2 \times 0.2$  mm<sup>2</sup> sample from one electron bunch. The duration of the single bunch is 50 psec and simulations of the diffraction pattern from myoglobin predicts 500-750 usable reflections.

Single bunch exposures may therefore open up the possibility of capturing short lived excited states in a reaction scheme, but it has also the advantage that the diffraction pattern may be free of radiation damage since the time scale associated with the migration of free radicals is supposed to happen on a much longer time scale. The beamline will be installed in September 1993 and first beam is expected in the 4th quarter of 1993.

## PS-01.01.07 DECONVOLUTING LAUE MULTIPLE DIFFRACTION SPOTS BY THE DENSITY MODIFICATION METHOD.

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In a Laue diffraction pattern 10-20% of the spots result from the exact superposition of two or more reflections which are 'harmon-

ics', e.g.  $hkl$ ,  $2h, 2k, 2l$  ..... For the solution of large or difficult structures the intensities of the remaining 80-90% of the reflections, measurable as singles, may not be sufficient, and evaluation of the intensities of the components of the multiple spots is therefore important. A new procedure for this deconvolution using real-space density modifications on the Patterson map is given. This is a further development based on a procedure in reciprocal-space related to Direct Method (Hao, Campbell, Harding and Helliwell, *Acta Cryst.*, 1993, A49, in the press). It has been tested with Laue diffraction data from 4-Zn insulin and Cytochrome C Peroxidase (CCP). 304 and 1134 reflection intensities were evaluated from multiple spots of insulin and CCP respectively; the R-factors showing the agreement with the high quality monochromatic data are 0.24 and 0.21.

## PS-01.01.08 DEVELOPMENT OF X-RAY FOCUSING OPTICS FOR MICRODIFFRACTION.

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X-ray microfocusing optics capable of focusing hard (5-30 keV) x-rays to submicron focal spot size with high focusing efficiency are of great importance for spatially resolved microdiffraction as well as many other applications such as microscopy, microspectroscopy, and microanalysis. The availability of such optics opens up new opportunities to extend the capabilities of many conventional x-ray techniques with high spatial resolution. For example, identification of crystallographic phase, orientation, local structure, and strain can be studied using the spatially resolved microdiffraction technique. For this purpose, we have developed two types of microfocusing optics, transmission phase zone plates and reflecting focusing optics using an ellipsoid mirror. A 0.6-micron spatial resolution and a 33% focusing efficiency were measured from a phase zone plate using synchrotron x-rays. In this paper, we will present the recent developments in x-ray focusing optics for x-ray microdiffraction and other applications. The parameters that are relevant to microdiffraction experiments will be discussed.

## PS-01.01.09 TIME-RESOLVED ENERGY-DISPERSIVE POWDER DIFFRACTION STUDY OF THE FORMATION OF CORDIERITE FROM A CHROMIUM DOPED CORDIERITE GLASS.

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The crystallisation of cordierite and spinel from a non-stoichiometric chromium doped cordierite glass has been studied at a number of temperatures under isothermal conditions using energy-dispersive powder diffraction. A quartz like intermediate was found to briefly exist before the onset of crystallisation. A complete kinetic analysis will be presented together with a detailed discussion of the chemistry of formation of cordierite.