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REPORT OF THE CRYSTAL OPTICS PANEL FOR INSTRUMENTATION ON THE SRS

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Science Research Council

Daresbury Laboratory

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1. INTRODUCTION

The Crystal Optics Panel is one of four such Panels set up by the Science Board of the SRC to provide recommendations and advice on the type and range of instrumentation desirable for the new Synchrotron Radiation Source (SRS).

The areas covered by each Panel of users were Crystal Optics (Chairman Dr. W. Hayes); Grating Optics (Chairman Dr. G.V. Marr); Beam Lines (Chairman Dr. I.H. Munro); Computing (Chairman Dr. H. Sherman). The Panel reports were submitted to the Physics Committee of the SRC and referred to the Synchrotron Radiation Research Committee.

The general terms of reference for the Panels were:

To consider the type and range of instrumentation for the experimental programme on the new Synchrotron Radiation Source and, in particular, to make recommendations under the following headings:

- (i) the requirements for the research proposals in the SRRC Panel Report;
- (ii) the adaptation of the existing and proposed equipment on the NINA SRF;
- (iii) the need for instrumental design and development;
- (iv) the arrangement and instrumentation of beam lines;
- (v) the systems for the control of apparatus and the collection and analysis of data.

The Panel concluded that demand for access to the SRS in the x-ray region is likely to vary greatly from one field of research to another. Areas where the demand is expected to be large are:

- (i) Extended x-ray absorption edge fine structure (EXAFS);
- (ii) x-ray photo-electron spectroscopy (XPS);
- (iii) Topography;
- (iv) Small angle scattering (SAS);

- (v) Lattice structures and interferometry;
- (vi) x-ray absorption microscopy and lithography.

These are treated as "high-priority" areas. The number of research groups working in the following areas is expected to be comparatively small: Non-linear optics, inelastic scattering (Compton, Raman), diffuse scattering, holography, atomic spectroscopy, x-ray fluorescence, Fourier transform spectroscopy, and radiometry. These are therefore designated "second-priority" areas.

The Panel has adopted the position that it should specify the instrumentation requirements of the high-priority areas, where it is anticipated that the demand will be both great and immediate. Other fields, where demand is expected to be small, could be catered for by granting access to equipment in the high-priority areas.

Before proceeding to the detailed recommendations, the Panel wishes to emphasise some matters which it considers of the utmost importance to the successful exploitation of the SRS in the x-ray region.

- (i) Firstly, the Panel is emphatic on the need for a wiggler magnet to produce from the outset increased intensity at wavelengths of about 1.5 Å and below. Without it the SRS will be of limited use to SAS workers. Likewise, the wiggler is considered to be of great importance in the fields of x-ray topography and absorption microscopy. Furthermore, for workers in such potentially interesting areas as inelastic scattering, non-linear optics and other processes requiring high intensity at short wavelengths, a wiggler will be an essential part of the facility.
- (ii) The Panel recommends that Daresbury Laboratory take immediate initiative in establishing a community of x-ray users holding regular meetings at Daresbury.
- (iii) To extract the maximum amount of useful x-radiation from the SRS, it has been proposed that optical components be placed close to the ring, even within the main ring vacuum vessel. Although at the time of preparation of this report, the feasibility of such

an arrangement had not been established, the Panel recommends that the possibilities of "close-coupling" should be thoroughly investigated, in view of the benefits to be had from the improved radiation-collection efficiency.

(iv) The Panel is conscious of the rapid development of x-ray techniques, and the growth of research in the crystal optics regime. In view of this, the Panel has quite often refrained from making firm recommendations on details of instrumentation that will be required in 1979/80. The Panel therefore feels that a further survey should begin not more than two years before the SRS is commissioned and that this should be an on-going process.

(v) There is comparatively little x-ray equipment at the present SRF (see Table 1). It follows that there will have to be substantial acquisition of new instruments to equip the SRS facility to the level envisaged in this report. The manner in which this should be done requires urgent consideration (see section 9).

The outline of the report is as follows:

<u>Section 2</u> deals with monochromators, detectors and other instruments common to different research fields.

<u>Section 3</u> contains the Panel's recommendations on meeting the needs of the high-priority areas; these are summarised in Table 1.

<u>Section 4</u> outlines the proposed development programme, and discusses the provision of a test facility for this purpose; it also includes the topic of radiometry.

<u>Section 5</u> discusses computing requirements.

<u>Section 6</u> contains recommendations on ancillary equipment, workshop facilities, etc.

<u>Section 7</u> sets out beam characteristics of importance to x-ray users.

<u>Section 8</u> outlines the Panel's tentative suggestions on beam-line layout.

<u>Section 9</u> contains some general recommendations for future action.

The membership of the Crystal Optics Panel as originally appointed was:-

Dr. W. Hayes (Chairman)	(University of Oxford)
Dr. J.H. Beaumont	(University of Oxford)
Dr. G.A. Brooker	(University of Oxford)
Dr. M.J. Cooper	(University of Warwick)
Dr. M. Hart	(University of Bristol)
Dr. H.E. Huxley	(University of Cambridge)
Professor D.C. Phillips	(University of Oxford)
Dr. K.R. Lea	(Daresbury Laboratory)

During the course of the Panel's work Professor Phillips resigned and Dr. D.K. Bowen (University of Warwick) and Dr. H.C. Watson (University of Bristol) were co-opted on to the Panel.

2. COMMON INSTRUMENTATION REQUIREMENTS

2.1 Monochromators

High-resolution monochromators giving wavelength coverage from about 0.1 Å to about 20 Å will be required. Below about 6 Å, Ge and Si crystals are most suitable for monochromators. At longer wavelengths the situation is less clear-cut, although gypsum and β-alumina crystals may prove satisfactory. Below 4 Å, a helium gas environment for the monochromators will suffice, but for longer wavelengths, an evacuated chamber will be required for the monochromator. This does not necessarily entail a common vacuum with the storage-ring beam line, as a separately pumped chamber isolated by a very thin beryllium window whould serve up to about 10 Å.

The simplest monochromator design employs a plane crystal mounted on an axis. Monochromatisation occurs when incident "white" synchrotron radiation undergoes Bragg scattering from the plane surface. Although this is an easy device to set up, it should be pointed out that the exit beam direction rotates through twice the angle of rotation of the crystal as the wavelength is varied, and this necessitates repositioning the experimental equipment.

This single-axis design can be improved by replacing the plane crystal with a channel-cut crystal. The radiation now undergoes two Bragg reflections successively from the inward-facing surfaces. Within the bandpass for Bragg reflection in high-quality silicon or germanium, a comparatively small intensity loss of order 10% is suffered in the double reflection. The particular attraction of the channel-cut crystal is that it produces a monochromatic exit beam that is fixed in direction, parallel to the incident beam. However, a monochromator based on a single channel-cut crystal, preceded by a fore-slit to control the resolution, possesses the following additional characteristics, which could be inconvenient in practice: (i) the exit beam undergoes a lateral displacement as the wavelength is varied; (ii) the ultimate resolution involves a convolution of the source size, the distance from the tangent point on the electron orbit, the x-ray collimation and the crystal reflectivity.

A still more sophisticated design combines the foregoing with its mirror image, i.e. two axes, each carrying a channel-cut crystal. Such a double channel-cut crystal monochromator gives an exit beam fixed in both direction and position, a property of particular value when working with small non-uniform samples. Also, this design can eliminate spurious reflections, and allow large beam sizes to be used because the angular resolution does not depend on beam size. The wavelength calibration is determined internally by the relative positions of the two crystals; whereas the calibration of the single crystal monochromator depends on its orientation with respect to the electron beam direction at the tangent point.

The energy resolution achievable with such single-axis and double-axis monochromators is about 6 parts in 10^5 for the case of the 220 reflection in silicon, or about 9 parts in 10^6 for the 440 reflection. For applications requiring resolutions of this latter order the crystal or crystals must be stepped through angular increments of order 0.2 arc s. A major difficulty with the two-axis system appears to be that of construction to the requisite tolerances.

The output flux from a double-axis, channel-cut crystal monochromator has been calculated to be 10^9 photons/s. This figure was based on measurements made at the NINA synchrotron, and the following representative characteristics:

Storage ring operating at 2 GeV and 1 A; crystal acceptance angles of $\pm 10^{-5}$ rad vertically by 2×10^{-4} rad horizontally; double crystal monochromator tuned to 1 Å, with a resolution $\delta\lambda/\lambda = 5 \times 10^{-5}$.

The presence of higher orders, e.g. $\lambda/2$ from Si 440 reflection, $\lambda/3$ from 660 reflection, etc., in the monochromator output when nominally set for the fundamental wavelength λ from the 220 reflection, is a potential source of serious error in some experiments. Unless the detector is energy-sensitive so that order-sorting can be carried out at the detection stage, some other method will have to be tried to suppress higher orders. Suggested methods are (i) selection of Bragg reflections with missing higher orders; (ii) use of dissimilar Bragg reflections in double-crystal monochromators; (iii) use of filters (in limited regions); (iv) adjusting the energy of the SRS to reduce the higher order intensities; (v) using adjustable grazing incidence mirrors for selective reflection of only the first-order wavelength; and (vi) use of differential absorption of x-ray wavelengths in double ionisation chambers.

We mention in passing that in some experimental applications, the high resolution that crystal monochromators can provide is not required, and a wider spectral bandwidth, with greater photon flux, would be preferable. A fluorescent source, stimulated by the primary synchrotron radiation beam, might offer a viable alternative to a monochromator in such cases, for, despite the poorer directional properties of the fluorescent radiation, the intensity may be large as a result of integrating the primary beam intensity from short wavelengths up to the K-edge of the fluorescent element. Such a device would provide a fixed wavelength output, with a set of interchangeable fluorescent materials to provide a series of desired wavelengths. Unlike a conventional x-ray tube source, the fluorescence lines have little background. A fluorescence source would be easy to set up, since it does not require careful alignment, and is well suited to experiments needing a divergent beam.

2.2 Detectors

There are a considerable variety of x-ray detectors available. These include (i) photographic plates; (ii) scintillation counters, using e.g. NaI(Tℓ) crystals; (iii) ionisation chambers and proportional counters; (iv) cooled solid-state detectors, e.g. Si(Li); (v) multiwire chambers, one- or two-dimensional; and (vi) TV-linked image intensifier systems.

The photographic method is inexpensive and straightforward to use although processing times can be very long (up to several hours). Permanent two-dimensional records are obtained with, in the case of nuclear plates, a limiting resolution of ¼ μm determined by grain size. The resolution is further limited in the case of x-rays to 1 μm or worse by photo-electron tracking and by statistical noise (the latter cannot always be eliminated by long exposure because nuclear plates, though slow by ordinary standards, are excessively fast to hard x-rays and limiting densities are rapidly reached). Lead iodide plates may offer limited improvement in resolution.

In a scintillation counter, a quantum counting efficiency approaching 100% can be achieved between about 0.2 and 2.5 Å. Counting rates are limited by the lifetime of the fluorescing state, which for thallium-activated sodium iodide is about 0.3 μs. The pulse size varies inversely with the wavelength, offering some energy resolution, but not as much as a proportional counter. Over the same wavelength range, this latter detector offers a high detection efficiency, energy resolution of order 10% (a one standard deviation figure) and a resolving time of order 0.2 μs (one standard deviation). Maximum counting rates of 10^5 s^{-1} apply in both cases.

The cooled solid state detectors, based on lithium-drifted silicon crystals, for example, provide virtually 100% detection efficiency at wavelengths of order 1 Å, and excellent energy resolution (∿ 200 eV). For wavelengths shorter than 0.5 Å, Ge(Li) detectors are more efficient. Their counting rates are severely limited to a maximum of about 10^5 s^{-1}, a limitation of the electronics, and not the basic detector deadtime. Development of faster electronics would be extremely valuable.

Multiwire proportional chambers are position-sensitive detectors which can collect data simultaneously at many different points in an x-ray diagram. Both the linear-position-sensitive and the area-sensitive detectors are the subject of development work in several laboratories, but some present-day performance figures may be listed for guidance:

detection efficiency up to 80%; energy resolution 25%; spatial resolution ∿ 0.1 mm using a 256 x 256 wire grid; readout capability of 10^6 to 10^7 s^{-1}.

X-ray sensitive TV cameras are presently available with resolutions of about 10 to 20 μm. Much interest attaches to achieving higher resolution (∿ 1 μm) with this detecting medium.

All of the detectors mentioned above are likely to be required in the x-ray research programmes at the SRS.

3. REQUIREMENTS OF HIGH-PRIORITY RESEARCH FIELDS

3.1 EXAFS (Extended X-ray Absorption Edge Fine Structure)

A typical EXAFS apparatus comprises:

(i) a scanning x-ray monochromator

(ii) an absorption sample, perhaps mounted in a liquid nitrogen or liquid helium cryostat

(iii) one or more x-ray detectors to measure the absorption in the sample.

Monochromators will be required to give wavelength coverage from about 0.2 Å to about 9.5 Å and energy resolution of about 5 eV. For studying the extremely sharp threshold structure, however, a resolution of better than 1 eV is desirable. In the region of 1 Å, the low-order Bragg reflections from Si or Ge achieve this.

The EXAFS appears as an oscillatory structure superimposed on the continuum absorption; the intensity variation amounts to only a few per cent of the total absorption due to the x-ray edge. To obtain an accuracy of 1% on the EXAFS structure necessitates therefore an overall measurement precision of order 1 in 10^4. A 100% efficient detector, capable of counting 10^8 incident photons/s would achieve a measurement with statistical uncertainty of 1 in 10^4 in 1 second. The limited counting rate of present-day solid-state detectors implies a counting time of up to 1000 s per point on an EXAFS spectrum, which is uncomfortably long. Clearly, development of higher counting rate solid-state detectors is very desirable. In the circumstances, it appears preferable to use ion chambers, coupled to electrometers, to cope with the high flux levels available from the SRS. However, solid-state detectors will be valuable for making corrections to the data for the higher-order content of the radiation.

The Panel recommends that three scanning monochromators be provided for EXAFS work: one to be a single-axis instrument, the other two to be double-axis instruments. All three should be non-vacuum instruments.

3.2 XPS (X-ray Photoelectron Spectroscopy)

The basic equipment required for XPS work consists of an x-ray monochromator, an evacuated sample chamber, and an electron spectrometer to analyse the energy of photo-electrons.

Since the main research interest appears to lie in the vacuum region of the x-ray spectrum, specifically 4 - 20 Å, a vacuum monochromator is necessary. Because the XPS spectrum is sensitive to the location of the excitation beam on the specimen, a double-axis channel-cut crystal monochromator design is indicated, as this provides an exit beam fixed in position. However, a single-axis monochromator would serve adequately for fixed wavelength applications, and for the less critical scanned-wavelength investigations.

The resolution of the monochromator should match, or even exceed that of the electron spectrometer. Thus a photon bandwidth of ∿ 0.1 eV would be required at present. For some XPS investigations, it will be important that the synchrotron radiation should not be depolarised by the monochromator.

To maximise the output intensity, the vacuum monochromator should be situated close to the ring, although the actual position will be influenced by the need to find space close by for the analyser.

The electron spectrometer deals with photoelectrons whose energies extend from essentially zero up to a maximum equal to the photon energy. Since the latter can be 12.4 keV ($\lambda \approx 1$ Å), high voltage insulation is required of the electron energy analyser to scan the full range of the photoelectron spectrum. Insulation of this order is provided in at least one commercial instrument, already acquired by Daresbury Laboratory for use at the SRF.

The energy resolution of electron energy analysers has reached the level of about 0.1 eV, but even so, some energy distribution curves show signs of still finer, unresolved structure. Thus it appears unwise to specify any particular figure for energy resolution, other than the best that technology can provide. In this regard, new techniques merit careful examination, such as the electron time-of-flight method of energy analysis in use at the Stanford Synchrotron Radiation Project.

In addition to a spatially fixed analyser, there is growing interest in examining angular dependence in XPS. Thus the angle between the electric vector in the incident x-ray beam and the normal to the sample surface, and the emission direction of the photoelectrons, are parameters likely to be studied in future XPS work. An analyser with a suitable angular degree of freedom seems to be called for.

The Panel recommends that a scanning vacuum monochromator, covering the 4 - 20 Å range, be provided for XPS. This at the outset should be a single-axis instrument† with provision to add a second axis at a later date. The Panel also recommends that access to a monochromator out of the vacuum should be provided for XPS research at wavelengths shorter than 4 Å.

The Panel recommends that two electron energy analysers be provided for XPS. Both analysers would include a sample chamber and sample preparation unit. One instrument would incorporate sample and analyser rotational facilities.

3.3 X-ray Topography

This is a technique for observing lattice imperfections directly in an image formed by means of diffraction contrast. Either continuous or monochromatic x-radiation can be used, with a resolution of about 1 μm on the resulting topographs. Compared with conventional x-ray tube sources, the synchrotron provides a major breakthrough by reducing topographic exposure times 1000-fold. X-ray topographs are as convenient to take as electron micrographs; it is anticipated that the demand to undertake x-ray topography at the SRS will be very great and may possibly approach the present-day use of transmission electron microscopy.

Ideally, the sample is placed at a sufficient distance from the tangent point to be completely illuminated by the direct x-ray beam. A photographic plate or TV detector is placed behind the crystal (for transmission) at a distance great enough to avoid overlapping between the direct beam and the many diffracted images that are produced.

† It should be emphasised that this is the only instrument recommended in the report to cover the long-wavelength end of the x-ray range.

The resolution, d, in this type of topographic method is given by

$$d \sim \frac{hb}{a}$$

where h is the larger source dimension, a the source-specimen distance and b the specimen-film distance. Since the SRS electron beam is designed to be 3 mm x 0.1 mm (one standard deviation) the resolution will be limited by the horizontal (3 mm) dimension. It is better to consider the source size as 12 mm x 0.4 mm, (taking $\pm 2\sigma$ in both directions) which will contain approximately 90% of the electron beam. For 1 μm resolution it follows that b/a must be $\lesssim 10^{-4}$. If b = 10 mm, then a = 120 m, and it is clear that the longest available beam line ($\sim$ 80 m) must be used for x-ray topography. A beam with 20 mrad horizontal and 0.3 mrad vertical divergence measures 24 mm x 1600 mm at 80 m. Most users will find 24 mm an adequate beam height but provision should be made for one camera to incorporate vertical scanning so that large crystals $\sim$ 100 mm diameter (currently in production in the semiconductor industry) can be examined.

The apparatus required consists of simple but stable two-axis goniometers, designed for adjustment by remote control, together with photographic or TV means of recording the image. The remote control can be relatively simple since setting accuracies need only be of the order of 1 mm and 1° (though stability must be of the order of 1 μm and 1 arc s). However, it would be useful if the throughput could be increased by adding a pre-programming facility so that the goniometer could be switched rapidly between different reflections.

The Panel foresees the x-ray topographic facilities at the SRS being very extensively used for (i) routine (but rapid) conventional work and (ii) dynamic experiments on such phenomena as the deformation and mechanical properties of materials, magnetic and ferroelectric properties, and phase transformations. Because of the anticipated high level of demand, and consequent high throughput at the SRS, the accent must be on ease, convenience and speed of use, with capability to set up and take exposures at a rate of about ten per minute. For the dynamic studies, there is a great need for a rapidly-changeable high-resolution recording medium. A multiple-plate holder for nuclear plates capable of stepping and changing the plates rapidly be remote or automatic control

will certainly be required. Moreover, the development of a television camera, directly sensitive to x-rays, with a resolution of 1 μm (as compared with 20 μm presently available) would be of great value.

The Panel recommends the provision of four continuous-radiation topographic cameras, as specified below:

(a) Three cameras with horizontal aperture 25 mm, without any scanning facility.

(b) One camera with aperture 120 mm with vertical scan facility.

Two of the cameras specified in (a) plus the large-specimen camera (b) should be mounted near the end of a long beam line (without wiggler) occupying, say, the space between 75 m and 80 m. The x-ray topography beam line should be sighted on a tangent point where the horizontal dimension of the source can be made as small as possible, so as to achieve the best resolution. A rough vacuum line with beryllium windows is adequate. Source slits may be required.

The remaining camera should be mounted at the end of a beam line that is fitted with a wiggler. This is essential for the study of heavier elements (e.g. tungsten) by topography since these elements absorb x-rays of the normal SRS spectrum very strongly. Again, the beam line should be as long as practicable, but since it is unlikely that the highest resolution will be achieved or needed with the shortest wavelengths, a beam line of 25 - 50 m can be accepted.

There may be a more specialised need for a topographic apparatus using monochromatic radiation. However, the Panel does not recommend the construction of a tunable monochromator for this purpose unless demand greatly increases. For occasional work, access to one of the EXAFS monochromators should be granted.

3.4 SAS (Small Angle Scattering)

The diffraction or scattering of x-rays by crystalline matter occurs at smaller angles the larger the lattice spacings. For 1 Å radiation, and lattice spacings of the order of 100 Å, the scattering angle is about 0.5°. Measurement of the spatial distribution of the scattered radiation determines the lattice spacings in the crystal. Biological specimens are probably of the greatest interest, although their inherent weakness of diffraction, generally small size, and large magnitude of their repeat distances, make SAS measurements difficult to perform. Extension of these studies to transient phenomena, such as the contraction of a muscle, is dependent on having available sources of exceptionally high intensity, since the time available to record the diffraction pattern is short. The all-important advantage of the SRS to these latter experiments is the much higher x-ray intensity available, compared with conventional x-ray tube sources. Even so, the available flux is by no means in excess of requirements, and all possible steps will have to be taken to maximise the x-ray intensity arriving at, and transmitted through, the SAS camera. In this connection the Panel emphasises that the prospects for SAS research are very dependent on the provision of a wiggler magnet.

An existing camera design embodies double focusing of the x-radiation, first by a cylindrically-curved grazing-incidence mirror (focusing in the vertical direction) followed by a bent single crystal of quartz or germanium, which focuses in the horizontal direction. The crystal also monochromatises the radiation, typically at $\lambda = 1.5$ Å. The biological sample is supported in this converging, monochromatic beam, and the diffraction pattern it produces is recorded by a detector placed in the focal plane, typically 1 to 2 m away. There are some adjustable apertures designed to collimate the beam and minimise background scattering. At the SRS, such a camera might have a horizontal aperture of 20 mm, a vertical aperture of about 3 mm and to capture the maximum flux, it should be placed as close to the tangent point as possible, e.g. 5 metres or less. If necessary, the crystal might precede the mirror and be positioned inside the shield wall (see section 8).

There are alternative camera designs, e.g. a non-focusing plane-crystal SAS camera, and another employing undispersed incident radiation and energy-resolved detection of diffracted beams. These have been tested, and their benefits vis-à-vis the double-focusing camera have been debated. It will be important to keep abreast of developments and refinements of new and old camera designs, so that instruments built for the SRS may achieve the best possible performance.

Position-sensitive detector systems, such as the multiwire proportional

chamber and x-ray sensitive television camera, will likely be employed in SAS cameras whilst the cooled solid-state detector appears to be essential in the version of SAS camera which uses energy-resolved detection. Active development work on various detector systems is being pursued in a number of places, e.g. Hamburg, Paris, Cambridge and Stanford. In view of this, the Panel does not wish to specify the preferred detector for SAS work, as any decision taken now could well be subject to change before the SRS becomes operational.

The Panel recommends that three SAS cameras be provided to meet the anticipated great demand. Two of these should be of the double-focusing variety. A decision on the design of the third camera should be deferred until closer to the time for starting its construction.

The Panel recommends that the electron beam height at the tangent point be maintained constant to better than ± 10 μm (if possible over periods of days or weeks) as a pre-requisite to obtaining the highest resolution from the double-focusing camera design.

3.5 Lattice Structures and Interferometry

Conventional x-ray sources are adequate for all but the most demanding lattice structure problems. It follows, therefore, that the SRS facility will be used by only those crystallographers who have problems where particular advantage can be gained from a free choice of wavelength, with high intensity, low angular divergence, or high degree of polarisation of the primary beam. The monochromated beam will also reduce specimen damage. Apart from a few interested protein crystallographers, potential users are hard to identify. We can assume, however, that future applications will include work with small or unstable crystals, and with specimens from which either accurate lattice parameter or structure amplitude measurements are required. Part of the programme will be concerned with the interferometric measurement of x-ray optical constants which are required for structure analysis.

Users of the SRS facility are likely to fall into one of two main categories:

(a) those who require rapid collection of vast amounts of structure amplitude data;

(b) those who make relatively few measurements but require precise control of the experimental conditions.

The experimental needs of the first group are relatively straightforward and typically satisfy the requirements of the protein crystallographer. Experiments performed by the second group are likely to be less routine in nature, and instrumentation specified for these users must be sufficiently flexible to accommodate unidentified demands. Taking into consideration these requirements and the expected use of the SRS facility, it is anticipated that the needs of lattice structure users will be satisfied by two beam line locations and the following equipment:

(i) A low-resolution scanning monochromator covering the range 0.5 to 4 Å and producing a uniform beam of diameter 0.05 to 0.20 mm. The sample should be mounted on a crystal oscillation system similar in concept to that marketed by Enraf-Nonius, but with a "large" area detector and associated computer readout.

(ii) A high-resolution scanning monochromator covering the widest possible wavelength range, and producing a uniform beam of approximately 1 mm in diameter. The crystal sample should be mounted on a computer-controlled orienting system equipped with beam shaping facilities, a "small" area detector device and associated computer readout.

The Panel recommends that two instruments be provided for work on lattice structures at the SRS, one of each type discussed above.

3.6 X-ray Absorption Microscopy and Lithography

The instrumentation for absorption microscopy (micro-radiography) comprises:

(i) a tunable monochromator;

(ii) a rigid specimen mounting table to take special stages, with a scanning option;

(iii) a high-resolution recording medium.

Demand for such a facility is anticipated not only from biologists,

but from materials scientists, in fields such as phase transformations, study of defects in fabrication processes, and studies of polymers. The ability to tune through a specific absorption edge means that element-sensitive discrimination can be obtained. In addition to "static" absorption microscopy, dynamic studies are likely to become very important, for which a rapid recording medium, such as a television camera, would replace the photographic plates implicit in (iii) above.

The monochromator needed for absorption microscopy should operate in air, or rough vacuum, covering a wavelength range from 0.1 to 4.0 Å, with a beam width of 120 mm. The constraints on resolution are the same as for topography, and a beam line length of order 80 m will be required. The reflecting crystal surfaces should be carefully etched and polished so that they yield a structureless surface topograph. This is essential for uniform x-ray illumination over the sample. Longer x-ray wavelengths are more suited to measurements on thin tissue specimens, in order to obtain higher contrast. Ultimately, a vacuum x-ray monochromator scanning out to 10 Å will be required, but this does not warrant a high priority. The wiggler will be essential for many technological applications of absorption microscopy.

The Panel recommends that one single-axis scanning monochromator, covering the 0.1 to 4.0 Å region, be provided for x-ray microscopy and lithography.

4. DEVELOPMENT PROGRAMME (INCLUDING RADIOMETRY)

In assessing instrumentation requirements, the Panel has encountered several areas where improvements to existing x-ray technology are badly needed, and some areas where performance data are unavailable. This has led the Panel to recommend a development programme on selected problems within applied x-ray optics, the better to exploit the short-wavelength output of the SRS. Such a development project should proceed in three phases: first, some urgent survey work at the NINA synchrotron radiation facility before the closure of the synchrotron; second, some work covering the time span without synchrotron radiation in the U.K.; thirdly, development work utilising the intense beams available when the SRS is commissioned for which a test facility will be needed at Daresbury Laboratory. Some of the work might well be contracted to specialists outside Daresbury Laboratory

The Panel recommends a development programme under the following headings:

(i) Incorporation of high-accuracy rotary-drive systems in a double-axis, two-crystal monochromator.

(ii) Examination of various techniques for monochromator order sorting, such as adjustable grazing-incidence mirrors for selective reflection of first-order wavelengths, use of dissimilar crystals in double-crystal monochromators, and the use of filters.

(iii) Development of existing detectors to achieve improved performance in respect of count rates, energy and spatial resolution, and fast readout capabilities.

(iv) Investigation of problems caused by radiation damage to such x-ray optical components as mirrors, windows and crystals, as a result of the high fluxes that will be encountered at the SRS.

(v) Design and development of topographic cameras, goniometers and cassettes.

(vi) Provision of a radiometry test facility at the SRS to enable such activities as the following to be pursued:

testing of x-ray optical component performance

experimental verification of grating theory

spectral intensity comparisons and fluorescence conversion efficiencies

development of a calibrated x-ray attenuator.

Item (vi) would require a test chamber, occupying a permanent beam line position, and equipped with a wide-range vacuum monochromator and various detectors. The chamber should be a multi-purpose vessel, able to serve as a precision reflectometer for mirror evaluations.

5. COMPUTING REQUIREMENTS

Adequate computing facilities will form a vital link in the successful exploitation of the SRS. From the point of view of users, this means that all scanning monochromators should be under computer control, and data acquisition should likewise be automated. There will be need for remote positioning facilities for experimental stages, for controlled variation of parameters of interest, and general remote handling operations.

It is important that computer control facilities be simple to operate by the experimentalist, and offer a degree of flexibility to allow new requirements to be incorporated.

Experiments involving position-sensitive detectors such as multiwire proportional chambers, TV image intensifier systems, etc. will produce data at very high rates; these experiments will require a dedicated computer with a sizeable main store, which must have access to the bulk store of the central computer. For some purposes, video-tape equipment will be required.

Users will also want to analyse their spectra on-line wherever possible, but in some instances, e.g. crystallographic work, the large-scale computing facilities that are required would probably have to be provided off-line.

Existing computing facilities at the Daresbury Laboratory Synchrotron Radiation Facility provide an example of the application of computing control equipment which meets the diverse needs of experimental research projects. Recommendations for the extension and improvement of these computing facilities to meet the increased demands of SRS users are the prime responsibility of the Computing Panel, to whose report further reference is directed.

6. ANCILLARY EQUIPMENT AND SERVICES

In compiling this report, the Panel has become increasingly conscious of the need for additional laboratory equipment and facilities, e.g. for the preparation of samples, without which users will find themselves severely handicapped. The efficient utilisation of synchrotron radiation will be impossible if these essential services are not readily available on site.

The Panel recommends that the following provisions be made to meet the needs of research workers in the x-ray region:

(a) Facilities and services

A versatile mechanical technician for small jobs and modifications.

A research workshop, equipped with basic machine tools, where users themselves can undertake simple jobs.

General chemical, biochemical and biological laboratory facilities.

Two or three photographic dark rooms, with automatic film-processing facilities.

X-ray preparation room (including x-ray generator and general camera facilities for pre-alignment work).

Adequate storage space for users' equipment when not on-line.

(b) Items of Equipment

X-ray detectors: photographic, scintillation and proportional counters, solid-state detectors, and multiwire proportional chambers. It is envisaged that two x-ray sensitive TV cameras will also be needed. (See section 2.2).

Anti-vibration mounts for sensitive equipment, e.g. interferometers. Such mounts should be installed during the construction of the SRS.

Laser alignment facilities.

Liquid nitrogen and liquid helium cryostats for mounting solid EXAFS specimens.

Sample preparation facilities integral to the electron spectrometer for XPS.

"Universal" mounting system for x-ray cameras and diffractometer assemblies.

"Automatic" nuclear-plate cassettes.

X-ray beam shutters equipped with timers for controlling exposure times.

Goniometer designs that are rugged, and designs that are disposable.

(c) Assorted items

Gas cells with various windows, e.g. mylar, Be or Al, for gas and vapour samples.

Conventional evaporator.

High temperature sputtering evaporator.

Conventional polishing equipment.

Ion beam polishing equipment.

Optical microscope (x 1000 magnification).

Stereo-zoom microscope (for sample manipulation).

Micro-manipulator.

7. IMPORTANT BEAM CHARACTERISTICS

The size and positional stability of the electron beam at the tangent point have considerable effect on the performance capability of x-ray instruments, particularly spectroscopes, double-axis instruments, and those in which focusing of the beam is attempted, e.g. in the double-focusing SAS camera.

The Panel wishes to stress that the beam size should not exceed the figures put forward in the Design Study Report, viz. $\pm 1\ \sigma$ dimensions of $\pm$ 0.1 mm (vertically) by $\pm$ 3 mm (horizontally) averaged over one day. The beam height should be maintained constant to $\pm$ 10 μm, and the angular direction kept constant to $\pm$ 0.1 mrad (i.e. to $\pm$ 1 mm at 10 m). Continuous interaction between the users and the machine construction committee should be maintained to achieve this specification.

The improvement in flux output at short wavelengths to be obtained with a wiggler magnet makes a signficant difference to the prospects of research in SAS, topography, absorption microscopy and inelastic scattering. The Panel urges that the wiggler be available from the start of the SRS, for otherwise the SRS in 1979 will offer only one-tenth the output in the 1 Å region of at least one competing machine built previously, and less flux than NINA for wavelengths less than about 0.6 Å. The Panel concludes that 50 mrad of wiggler aperture will be required, made up of 30 mrad for SAS cameras, 5 mrad for topography and 15 mrad for absorption microscopy and inelastic scattering. This Panel stresses the importance of urgent consideration of wiggler aperture by the Beam Lines Panel.

8. BEAM LINES

The x-ray beam lines are best thought of as serving a dual purpose. They provide a low absorbance path and shield the users from the main beam. For the latter purpose, where possible, they are best made from sufficiently thick metal to provide complete radiation protection. (This Panel has urged on the Beam Lines Panel the importance of an early resolution of the radiation protection problem).

The question which then arises is whether to evacuate the lines and terminate them with beryllium or polymer windows or to fill them with He or H_2. With a gas filling, window absorption could be reduced since thinner windows would be sufficient to maintain a slight over-pressure. Windows could also be dispensed with, apart from the Be window on the SRS, by directly coupling the beam line to the SRS and the experiment. The following table gives the % transmission of H_2 and He at 1 atmos pressure for absorption paths appropriate to a standard beam line and the long topography line. For comparison, a 0.008" pure Be window transmits 55% at 3.95 Å. The transmission is sensitive to the presence of high-Z impurities, because of the Z^4 dependence of the absorption coefficient.

% Transmission of H_2 and He Columns at 1 atmos. for four wavelengths

gas and path length	λ(Å) 3.95	3.05	2.54	1.93
20 m H_2	81	88	90	92
20 m He	31	55	70	82
60 m H_2	53	67	74	77
60 m He	2.9	17	34	56

On the longer lines, He would attenuate the x-rays quite considerably. H_2, however, would probably be acceptable were it not for the danger of explosion when flushing initially. Even on the short beam lines He produces an attenuation of 70% at the longest wavelength. For these reasons and because the beam line walls must be thick enough to provide radiation protection, it would probably be best to opt for evacuated beam lines. One advantage of an evacuated system would be the

ease of ensuring it was working properly simply by means of vacuum gauges.

The Panel has drawn up tentative assignments to beam lines of the instruments recommended in the report. These are:

Line 1 (Wiggler line): Aperture 50 mrad, maximum length 25 m, Be window

(a) 3 SAS cameras as close as possible to the tangent point.

(b) Be window at 10-20 m for high energy EXAFS or Raman work with an extension to 25 m for topography.

(c) Absorption microscopy (12 cm aperture).

Line 2: Aperture 20 mrad, length ∿ 80 m, Be window (see figs. 1 & 2)

†(a) EXAFS at 15 m.

†(b) EXAFS at 30 m.

†(c) 3 topography ports at 70 m.

†(d) 1 interferometry/absorption microscopy port at 70 m.

Line 3: Aperture 20 mrad, length ∿ 25 m, Be window (see fig. 3)

(a) Raman/fluorescence/spare at 5 m.

(b) Lattice structure at 8 m.

(c) Terminal EXAFS for large experiments at 11 m.

Line 4: Aperture 20 mrad, length ∿ 8 m, differential pumping vacuum line

†(a) XPS/radiometry vacuum monochromator

†Experiment requires anti-vibration mount built into the floor.

Schematic layouts for lines 2 and 3 are shown in figs. 1, 2 and 3. One of the major difficulties in deciding on a layout for a beam line system is the inevitable lack of space between an in-line experiment and the adjacent continuing line. For example, in fig. 1, the beam centre in EXAFS 1 is only 6 cm from the nearer edge of the beam continuing to EXAFS 2. The lack of space is also clearly shown in fig. 2, where all the separate experimental lines emerging from a possible branching link (see fig. 1) are very close together. The branching link is as close as possible to the tangent point to keep the pipework down to manageable dimensions.

There will be a main shutter for each port, which is best placed immediately after the first Be window either in He or in a vacuum. On a vacuum x-ray line this shutter would be placed immediately after the differential pumping system. In addition to this, shutters on individual experiments will be necessary. These could either be in line or it may be more convenient to have them immediately in front of an experiment. It will be essential to have the shutters for individual experiments under control from the associated experimental position. If either shutter is in a He enclosure, the pneumatic operating system must not leak air into the enclosure. At Stanford He is used to operate the pneumatic system for this reason.

It is essential to allow free access past the end of an individual shuttered line. Early guidance from the Beam Lines Panel on this point is important in view of its relevance to situations typified by the topography lines in fig. 1.

It will be necessary to have apertures in the lines to prevent grazing incidence reflections off large areas of beam pipe. It would also be useful when setting-up to have fast-acting lead shutters in the lines for photographic exposures with timers down to 0.1 s. The alternative of inserting such a shutter between an experiment and the end of a beam line may not always be convenient.

Laser alignment should be provided for all the x-ray beam lines. To minimise interference with the machine vacuum on "beryllium-windowed lines", it is recommended that these should be mounted downstream of the main beam shutter. Subsequent Be windows would have to be mounted on retractable gate valves with substitute glass-windowed valves. This should present no problems with conventional vacuum beam lines.

To assist in deciding on layout details near the tangent point it will be necessary to obtain drawings of the following items from the Beam Lines

Panel:

1. A conventional bending magnet port with no differential pumping and a Be window mounted as close as possible to the tangent point.

2. A conventional bending magnet port differentially pumped for vacuum x-ray experiments.

3. A wiggler magnet port with no differential pumping and a Be window as close as possible to the tangent point. Since wiggler magnets will be in straight sections the SR beam will not diverge from the electron beam until it enters the next bending magnet. This adds 2.5-3 m to the closest approach of the Be window to the tangent point.

The detailed layout for the wiggler line and line 4 will be very dependent on the particular vacuum and shielding systems near the tangent point. These should become clearer when the drawings mentioned above become available. There is a possibility of close-coupling a Bragg reflector to the synchrotron radiation by putting it in the ring itself. For ease of manipulation and adjustment it could, e.g., be slid through a gate valve. Considerable gains in intensity would be achieved if the crystal can survive the irradiation, and beam outside the 20 mrad cone could be used which would otherwise be wasted.

9. RECOMMENDATIONS FOR FUTURE ACTION

The Crystal Optics Panel is aware that even if its recommendations are accepted in principle, there may be no mechanism available to put them into practice at the right time.

Some of the development programmes mentioned in the report are best pursued by the interested research groups in their own laboratories, or other places where the appropriate technical expertise already exists, rather than by trying to set up a development group at Daresbury. This approach can, however, only work if the SRC is willing to fund such work as part of its SRS programme. It is most important that the SRC announces its policy, in this respect, at an early stage so that potential SRS users may know how to proceed.

Furthermore, the acquisition of conventional pieces of equipment, and the establishment of a group of x-ray technicians serving the x-ray area should not be left until the SRS exists. A senior technician familiar with running an x-ray laboratory should be engaged at least two years before the machine is available, and he should be closely involved in the planning of the ancillary x-ray facilities, purchase of equipment, and the hiring of other technical staff for that area.

Finally, there needs to be some 'watch-dog' mechanism which will raise the alarm if the design, as it develops, changes in some important way (e.g., beam position stability) from the figures used for this present study. Such a mechanism should be established immediately.

TABLE 1: Summary of Panel's Recommendations in High-priority Areas

Report Section	Research Field	Instruments Recommended	Available at SRF
3.1	EXAFS	3 scanning monochromators (in air) - one single-axis design - two double-axis designs 1 eV resolution; range 0.3 to 4 Å. Also access to XPS monochromator for work at longer wavelengths.	A double-axis scanning monochromator under construction at SRF, March 1976
3.2	XPS	1 vacuum scanning monochromator (single-axis initially) 2 photo-electron spectrometers. Also access to EXAFS monochromator for $\lambda < 4$ Å.	AEI ES 200B electron spectrometer, 0.3 eV resolution
3.3	Topography	4 cameras - all to have goniometers with a "universal" mount.	Parts to make one camera
3.4	SAS	3 cameras - 2 of double-focusing design; one of design to be specified later.	Two different models of SAS camera at SRF.
3.5	Lattice Structures and Interferometry	1 scanning monochromator with sample oscillation system and area detector. 1 scanning monochromator and computer-controlled goniometer.	
3.6	Absorption Microscopy and Lithography	1 scanning monochromator (in air) single axis, channel-cut crystal design. 0.5 - 4.0 Å.	
4 (vi)	Development Programme and Radiometry	1 wide-range vacuum monochromator, and test chamber	

FIGURE CAPTIONS

Fig. 1 Line 2: Schematic representation of beam division on the 70 m, 20 mrad beam line.

Fig. 2 Line 2: Branching link for 70 m line.

Fig. 3 Line 3: Schematic representation of beam division among experiments on a 20 mrad line.

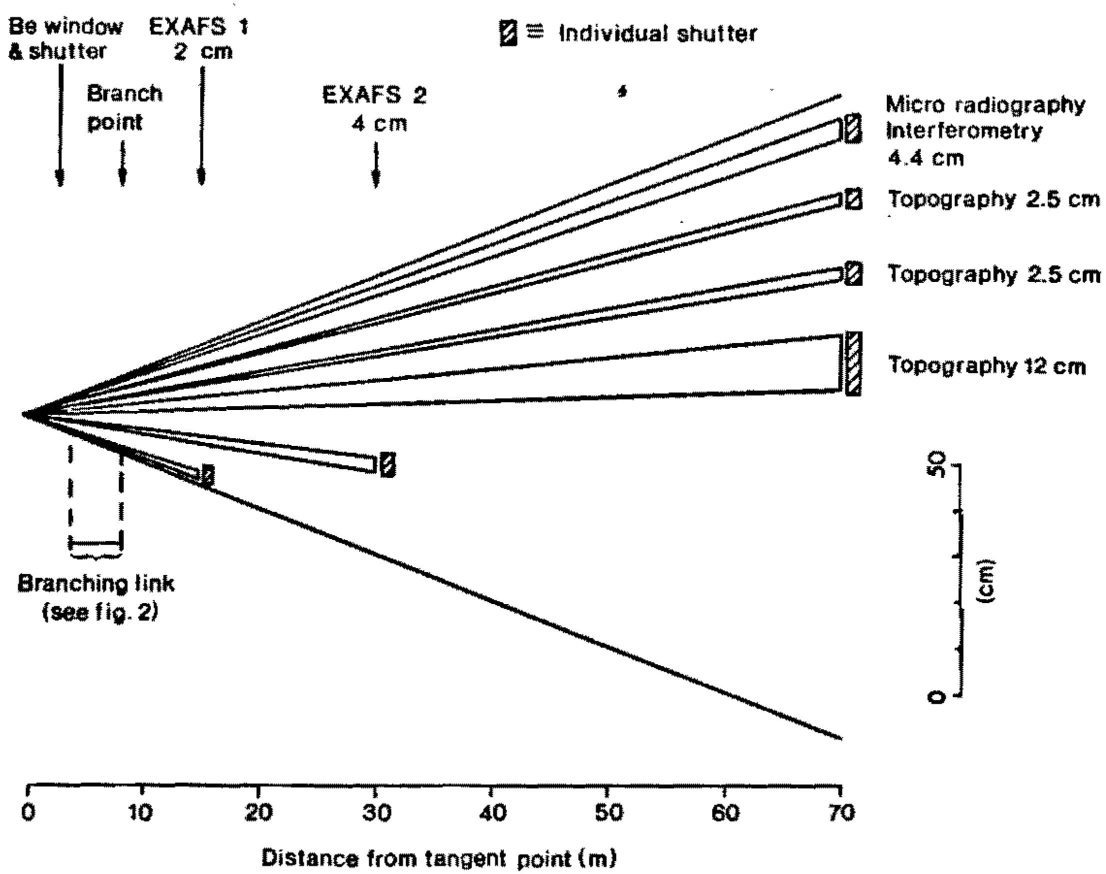
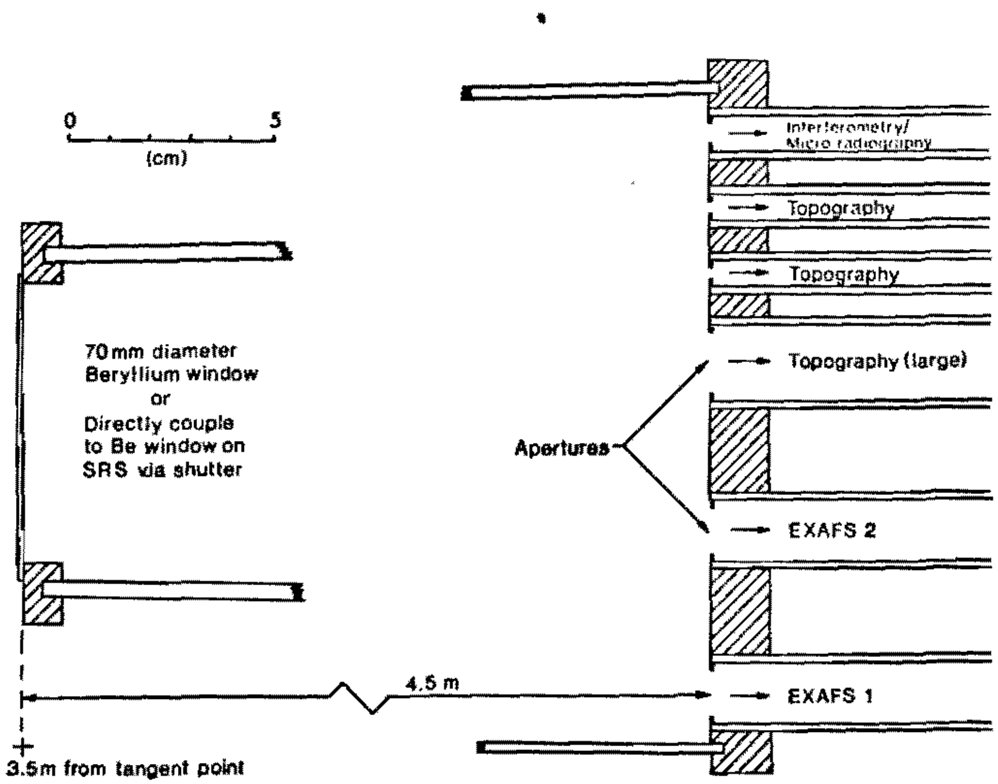
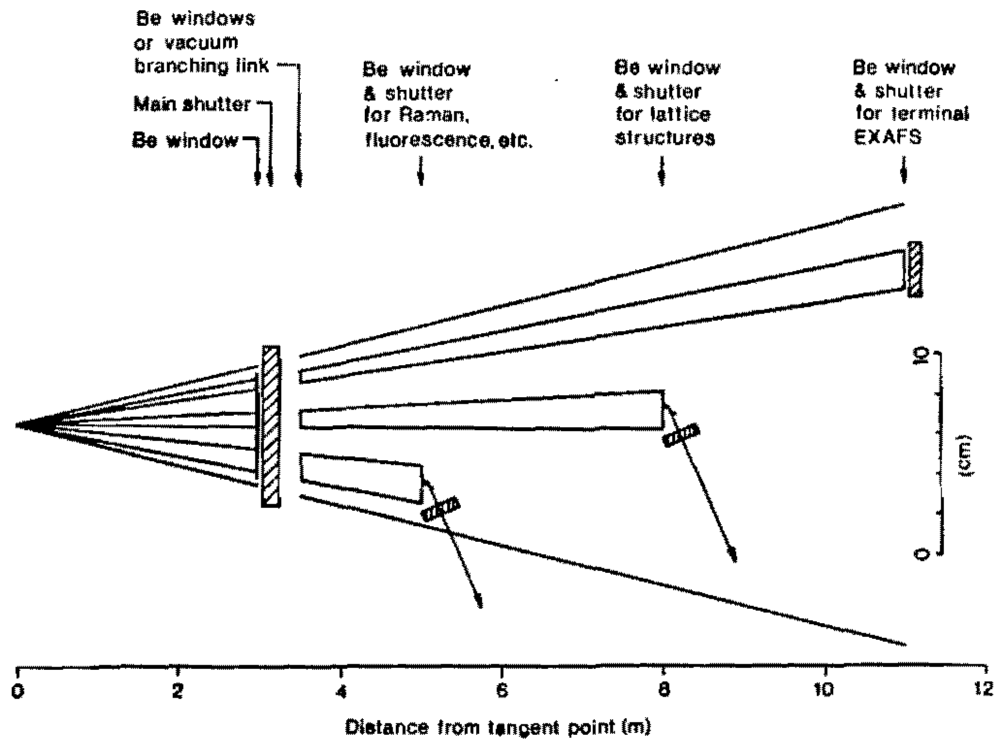


Fig.1



0
5
(cm)
Interferometry/
Micro radiography
Topography
Topography
Topography (large)
70mm diameter
Beryllium window
or
Directly couple
to Be window on
SRS via shutter
Apertures
EXAFS 2
4.5 m
EXAFS 1
3.5m from tangent point

Fig. 2



Be windows
or vacuum
branching link
Main shutter
Be window
Be window
& shutter
for Raman,
fluorescence, etc.
Be window
& shutter
for lattice
structures
Be window
& shutter
for terminal
EXAFS
10
(cm)
0
0
2
4
6
8
10
12
Distance from tangent point (m)

Fig. 3