VESPERS: A Beamline for Combined XRF and XRD Measurements

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Abstract. VESPERS (VEry Sensitive Elemental and Structural Probe Employing Radiation from a Synchrotron) is a bending magnet beamline that is just beginning construction at the Canadian Light Source. The beamline has several novel design elements that are intended to increase its operating flexibility and availability to users. First, there is a requirement to deliver a microscopic beam with a variable bandwidth, thus enabling the generation of Laue diffraction patterns and high yield X-ray fluorescence spectra from the same region preferably simultaneously. Thus, the bandpass of the VESPERS monochromator can be readily changed to focus radiation into the same 2-4 micron diameter area that is either polychromatic or having a bandwidth of 10%, 1.6% or 0.01%. This allows the user to change the diffraction pattern to suit the complexity of the crystal and the spectral signal to noise ratio to suit the detection sensitivity required. Second, the beamline is designed to have two branches capable of operating simultaneously and virtually independently using the same primary optics. These design features are accomplished using four separate beams originating at four pinholes at the entry to the Primary Optical Enclosure. The compound focus design uses spherical mirrors to focus both polychromatic and pre-monochromatic beams onto the intermediate slits. A pair of bendable K-B mirrors in the experimental hutch is used to demagnify the beam further down to micron size. The photon energy of this beamline is 6-30 keV.

Keywords: X-ray diffraction, X-ray fluorescence, Laue diffraction, X-ray absorption spectroscopy, microscopic strain, mineral trace analysis, synchrotron, broadband radiation, pink radiation.
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INTRODUCTION

VESPERS (VEry Sensitive Elemental and Structural Probe Employing Radiation from a Synchrotron) is a beamline that is just beginning physical construction at the Canadian Light Source, after two years of conceptual and detailed planning. The objective of VESPERS is to deliver a micro-focussed hard X-ray beam to solid materials in such a manner that a microscopic volume could be analysed by X-ray diffraction (XRD) and X-Ray fluorescence (XRF) either simultaneously or sequentially. In addition, it should also be possible to obtain XANES measurements on the same sample volume. Therefore, this is a microprobe capable of providing a high level of complementary structural and analytical information.

Laue diffraction will be the principal structural technique. Laue diffraction uses polychromatic X-rays to diffract from many crystallographic planes in a material without the necessity of rotating the sample in the X-ray beam. However, to be able to index reflections from all crystal planes there is an additional requirement to be able to switch the bandwidth of the incident X-ray from pink to near monochromatic in a routine manner. Therefore, the VESPERS monochromator is equipped to produce X-rays with several widely differing bandwidths. X-ray fluorescence is used to detect the presence of different chemical elements within the analytical volume. The sensitivity and speed with which a particular element is detected depends on many factors: the X-ray flux, the excitation energy of the fluorescence line used, the background from X-rays scattered from other elements and so

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on. Since the VESPERS monochromator can produce a range of X-ray energies *and* bandwidths, it is possible to alter the excitation conditions to suit the analytical requirement: for example, for highest analytical counting speed and multielement detection, a band pass of 10% or even pink radiation might be selected, while for higher sensitivity (but lower counting speed), a band pass of 1% or even 0.01% could be chosen. The availability of a very narrow bandpass setting also allows the absorption edge of a particular element to be scanned, thereby permitting the chemical state of a particular element to be sensed.

In anticipation of a large and expanding number of users, VESPERS is configured as a two-branch line, both halves of which can function simultaneously and with a significant degree of independence.

THE BROAD CONCEPT

The broad bandwidth required for Laue diffraction is provided by radiation produced from a bending magnet filtered by mirrors. The resultant X-ray beam can be used in a completely polychromatic form, or as highly monochromatic radiation, or an intermediate case where the wavelength is broadened by some fraction of the beam energy. There is, in addition, a necessity to achieve co-focussing of an X-ray beam that is, at times, mono-energetic, broadened to a defined bandpass, or completely polychromatic radiation.

Achievement of co-focussed radiation pathways into a microscopic spot on a sample requires a correction in the paths of the monochromatic and polychromatic beams. MacDowell and co-workers [1] achieved this at Beamline 7.3.3. at the Advanced Light Source (ALS) in Berkeley, California using a four crystal monochromator; however, the radiation losses involved are believed significant compared to a solution developed by Ice *et al.* [2] at MHATT-CAT then UNI-CAT (34-ID) at the Advanced Photon Source (APS) in Argonne, Illinois that involved the use of different slits in the monochromator. We have adopted the latter concept for VESPERS.

In VESPERS, the primary optical components of the primary stage are located in the Primary Optical Enclosure (POE) hutch (see Figure 1, please be noted that in order to keep the figure clarified only one of two branches is shown in the figure). Four beams are defined by a fixed mask with four entrance apertures, two vertically offset apertures for each branch. For one of the branches (see Figure 1), two vertical focussing mirrors produce parallel beam pathways, one of which is pre-monochromatic (which will be monochromatized by the monochromator downstream) and the other will remain polychromatic. Their paths are made co-incident by an offset within the monochromator.

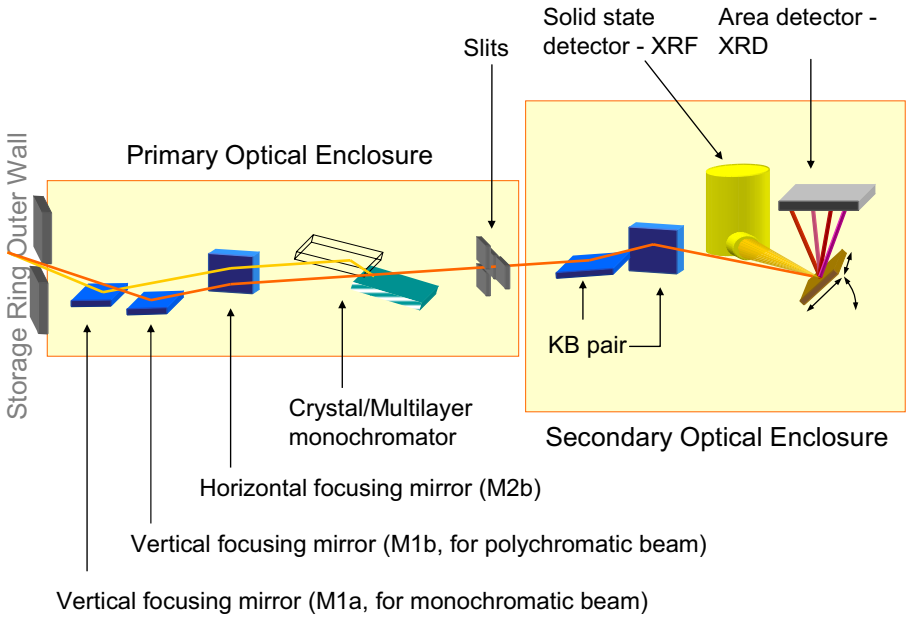

FIGURE 1. Schematic diagram of the main components of VESPERS beamline. To keep the figure clarified, only one of two branches is shown here.

The double crystal/multilayer monochromator in the POE is engineered to produce highly monochromatic or broader bandpass radiation. This is achieved using double Si (111) crystals onto which are deposited two multilayer stripes - one to produce a bandwidth of 10% and the other 1.6%. Each multilayer stack is graded differently and is designed to achieve high reflectivity. An uncovered section of each crystal surface is used to produce highly monochromatic radiation with a bandpass of 0.01%. The bandpass is changed by a physical sideways movement of the monochromator across the path of the incident X-ray beam. If polychromatic radiation is desired, the monochromator moves out of the optical pathways of the incident beams and the pre-monochromatic beam is blocked. Therefore, only polychromatic beam can pass through.

The convergence point of the primary focussing stage is at the location of ~1:1 image of the source near the end of the POE hutch. Intermediate slits are placed here and thus act as the source for the secondary focussing stage. The slits are used to define beam illumination size on the sample if a spot diameter less than 4 μm (FWHM) is desired. The minimum practical spot size presently achievable with this configuration is probably no smaller than 1 μm (FWHM).

In the Secondary Optical Enclosure (SOE, i.e., experimental hutch), a sample stage is shown surrounded by detectors for Laue diffraction patterns (a CCD camera), and XRF (an EDX detector). Within this enclosure the beam pathway passes through a beryllium window that terminates the vacuum, followed by ion chambers to measure beam flux. A bendable K-B mirror pair enclosed in a helium atmosphere comprises the secondary focussing elements.

The sample focus point is 7.5 cm downstream from the end of the second K-B mirror. The sample stage is normally angled at 45 degrees (φ and θ) to the incoming beam, as well as to the two major detectors. The sample is positioned on a stage whose motions in x, y, and z are motor-controlled with a reproducibility of 0.5 μm. Above the sample stage is a thin (50 μm) platinum wire that can be step-scanned across the sample surface and will allow the measurement of the three-dimensional distribution of phases within a sample. This process, called differential aperture X-ray microscopy (DAXM), was developed by Larson *et. al.* at Oak Ridge National Laboratory (ORNL), Oak Ridge, Tennessee [3].

The mean X-Ray energy to be used in most VESPERS experiments is ~15 keV. X-rays with energies ranging from 6-30 keV will be sufficiently intense for use in specialised experiments. At 15 keV, a flux of $\sim 2\times10^{11}$ photons/s is calculated for a 10% bandpass beam into a 4 μm diameter on the sample.

The beamline software and hardware are being configured so that user control can be directed from a remote station, for example, at the University of Western Ontario, using the CANARIE broadband network.

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