Chapter 2

Experimental section: ultra high vacuum (UHV) scattering chamber and the X1B-NSLS Beamline

In this chapter, we briefly describe the characteristics of X1B-NSLS beamline and the ultra high vacuum (UHV) scattering chamber.

2.1 History of X-Rays and Synchrotron radiation

Figure 2.1.1 (left) Wilhelm Conrad Roentgen (1845-1923) in 1896. (right) The famous radiograph made by Roentgen on 22 December 1895. This is traditionally known as "the first X-ray picture" and "the radiograph of Mrs. Roentgen's hand".
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The history of X-rays began in 1895 when Roentgen discovered “X-Rays” during his studies on light emission generated by discharging electrical current in highly-evacuated glass tubes. The electric discharge current between two plates inside a tube eventually became the basis of the conventional x-ray sources. By the time synchrotron radiation was observed almost a half-century later the scientific use of x-rays was well established. Some highlights include [1]

- 1909: Barkla and Sadler discover characteristic x-ray radiation (1917 Nobel Prize to Barkla).
- 1912: von Laue, Friedrich, and Knipping observe x-ray diffraction (1914 Nobel Prize to von Laue),
- 1913: Bragg, father and son, build an x-ray diffractometer (1915 Nobel Prize),
- 1913: Moseley develops quantitative x-ray spectroscopy and Moseley’s Law,
- 1916: Siegbahn and Stenstrom observe emission satellites (1924 Nobel Prize to Siegbahn),
- 1921: Wentzel observes two-electron excitations,
- 1922: Meitner discovers Auger electrons,
- 1924: Lindh and Lundquist resolve chemical shifts,
- 1927: Coster and Druyvesteyn observe valence-core multiplets, and
- 1931: Johann develops bent-crystal spectroscopy.

Synchrotron radiation generated by relativistic electrons in circular accelerators is only a half-century old and before that, at least, about another a half-century was spent developing the basics of modern x-ray physics. Thomson’s discovery of the electron was used as a theoretical basis for synchrotron radiation [2]. In 1897, Larmor derived an expression for the instantaneous total power radiated by an accelerated charged particle from classical electrodynamics. The following year, Liénard extended this result and showed the radiated power to be proportional to \((E/mc^2)^4/R^2\), where E is particle energy, m is the rest mass, and R is the radius of the trajectory [3]. In 1907, the discrete nature of atomic spectra was explained by Schott. By treating the motion of a relativistic electron in a homogeneous magnetic field, he obtained expressions for the angular distribution of the radiation as a function of the harmonic of the orbital frequency. At the beginning of 20th century, the basic modern theory of x-rays was established [3].

The first experimental observation of “synchrotron radiation” was made in 1940 on a 2.3-MeV betatron by Kerst at the University of Illinois. General Electric (GE) followed with a 20-MeV and then 100-MeV machine to produce high-energy x-rays for nuclear research. However these types of radiation were still considered a problem in electron accelerators because of the energy loss suffered by the electrons; it was not until mid 1960’s that synchrotron radiation started being used for research [4].
A further development of synchrotrons can be divided into several generations [1,4]. The first generation which is the next major advance was the development of parasitic electron storage rings, the basis for many of today's synchrotron sources. The Stanford Synchrotron Radiation Laboratory is one of the original examples.

The second generations were dedicated sources with adequate brightness to permit use with insertion devices such as undulators and wigglers [5]. Undulators which are an array of closely spaced vertically oriented dipole magnets of alternating polarity produce a high spectral brightness of the synchrotron-radiation source. Wigglers are similar to undulators but in general, have higher fields so can be thought of as a series of bending magnets whose spectra add incoherently to produce a continuous spectrum with high flux and short wavelength. The first wiggler was successfully installed in 1966 at the Cambridge Electron Accelerator (a 3-GeV storage ring) though it was used successfully only much later in 1987 by Schütz and coworkers at HASYLAB [6] in a study of magnetic circular dichroism in magnetic materials.

The third generation x-ray facilities, which operate in the present day, have been optimized for high brightness. Third-generation facilities specialize in either short-wavelength (high-energy or hard) x-rays or vacuum-ultraviolet and long-wavelength (low energy or soft) x-rays and the range in between (intermediate-energy x-rays). The European Synchrotron Radiation Facility (ESRF) in Grenoble, which operates at 6 GeV, was the first of the third-generation hard x-ray sources and started commissioning in 1994. It was followed by the Advanced Photon Source at Argonne National Laboratory (7 GeV) in late 1996, and SPring-8 (8 GeV) in Harima Science Garden City in Japan in late 1997. Of the long-wavelength sources, the Advanced Light Source at Berkeley (1.9 GeV) started in early 1994 and the Synchrotron Trieste (2.0 GeV) in Italy, the Synchrotron Radiation Research Center (1.3 GeV) in Hsinchu, Taiwan, and the Pohang Light Source (2.0 GeV) in Pohang, Korea. Recently (2005), the Canadian Light Source (2.3 GeV) also began commissioning.

Even this spectacular array of facilities is not enough to quell accelerator scientist’s ambitions. Even as the third-generation facilities enter their prime the development of fourth generation is already underway. Hard x-ray free electron laser (FEL) which based on a very long undulator is the best candidate for a fourth-generation source [1]. The peak brightness of such a device is expected to be many orders of magnitude higher than that of the third generation sources, and in addition supplies coherent pulses 100 fs or shorter. An example of a fourth generation source is TTF-FEL project at DESY which will culminate with a two-phase device operating at 6.4 Å. In the beginning it will have two end-stations, one of dedicated to VUV-FEL Raman spectroscopy. The first beam was delivered in the beginning of 2005.
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2.2 X1B beamline at National Synchrotron Light Source (NSLS)

RSXS experiments were carried out at the X1B linear undulator beamline of the NSLS [7]. The beamline is designed following the Dragon concept [8] having a spherical grating monochromator (SGM) with a movable exit-slit. The schematic of the beamline is shown in Fig. 2.2.1. The main optics consist of a horizontally focusing cylindrical mirror (HFM) and a vertical mirror focusing cylindrical mirror (VFM) to focus the beam on the entrance-slit of the SGM. The energy resolution and coherent flux depend on the slit openings, undulator gap, and the ring current. In our recent calibration [9], for a 10 μm setting of both slits, 36 mm gap of undulator and ~280 mA of the ring current, the energy resolution is of the order of 50 meV while the flux is about $10^8$ photon/s at 405 eV (N 1s absorption). The energy range extends from below 300 eV up to 1500 eV using four different gratings with groove densities (300, 600, 1200, 1600 lines/mm), which are optimized for different energies.

For more details of the undulator and of the X1 beam line up to the main mirror box, where the two branch lines separate (X1A and X1B), the reader is referred to Ref. 10. The HFM is a cylindrical focusing aluminum mirror coated with electroless nickel and gold, 2.5 degree angle of incidence, water-cooled, which focuses the undulator source horizontally onto the sample, which is located 14.21 meters from the source [11]. The VFM is a cylindrical focusing silicon mirror coated with gold, 2.5 degree angle of incidence, water cooled, 5:1 demagnification, which focuses the undulator source vertically onto the entrance slit of the monochromator, and is located 14.47 meters from the source [11]. The monochromator is an SGM with water cooled entrance slit (located 17.36 m from source) and moveable exit slit (located 23.5-24.4 m from source), four gold-coated silicon water-cooled spherical gratings (located 19.56 m from source). The total deflection angle is
6.6 degrees [11]. The last optical element is a refocusing mirror which is a Gold-coated silicon ellipsoidal mirror, ~5:1 demagnification, located 29.6 m from source. Focal spot (~100 μm (h) x ~30 μm (v)) is located 30.6 m from source [11].

Three endstations are currently available, for (1) soft x-ray scattering, located ~26 m from source, (2) soft x-ray emission and ARPES, located ~31 m from source, and (3) soft x-ray speckle (coherent) imaging, located ~31 m from source. Endstations (2) and (3) utilize, one at a time, the focused soft x-ray spot produced by the refocusing mirror.

2.3 Ultra high vacuum (UHV) scattering chamber

![Figure 2.3.1](image)

Figure 2.3.1 (left) Top view of the UHV Scattering chamber, (right) inside the Scattering chamber

As we discussed briefly in chapter 1 a new technique called resonant soft x-ray scattering is proposed to study charge and magnetic ordering in Cu-O systems. The idea of the required equipment is very simple; one must to do scattering or diffraction in the soft x-ray regime, i.e. an energy range of 200 – 2000 eV. There are two technical problems to overcome; one is the absorption of x-rays in air, and the second is the adsorption of Oxygen, Nitrogen etc. on the surface o samples at low temperatures. These two problems may be resolved by working under UHV conditions. Because one of the important edges we will want to work at is the O K edge we cannot tolerate more than about 1 monolayer of adsorbed gases.

An ultra high vacuum diffractometer for scattering soft x-rays was constructed during this study. The system is a five-circle diffractometer and mimics the functionality of a single-crystal diffractometer but operates in a vacuum of 2x10^{-10} mbar. The diffractometer consists of a six-degree-of-freedom sample stage, a two-axis detector arm, a channeltron detector stage with a multilayer for fluorescence rejection, a He flow cryostat, a 5 T magnet, a load lock or preparation chamber and a manipulator for transferring samples.
Actuation is done with vacuum stepper motors. The top view and inside of the diffractometer are shown in Fig. 2.3.1.

**Figure 2.3.2** A load lock and long-axis manipulator are mated to the main chamber of the diffractometer. The base pressure in the load lock is about $10^{-7}$ mBar before samples are transferred to the main chamber. A valve isolates the vacuum in the main chamber from the load lock.

The journey of the sample in vacuum starts from the load lock or a preparation chamber that is attached to the main chamber. A long axis manipulator is then used to transfer the sample to the main chamber. The diameter of the main chamber itself is about 1.2 m. To keep a good pressure in the main chamber a valve is installed in between the main chamber and the load lock. During transfer of the sample the pressure in the load lock was kept to better than $10^{-7}$ mBar. These are shown in **Fig. 2.3.2**.

In **Fig. 2.3.3** (top) a view inside the chamber is shown which consists of a sample, a sample holder, a long sample stage, Cu-braids, a multilayer for fluorescence rejection, two detector arms and one of the vacuum stepper motors, all of which are inside the UHV chamber. Sample cooling was done with a He flow cryostat connected to the sample via the Cu-braids, providing a base temperature of 18 K. At the bottom is shown a schematic diagram of 6 degree-of-freedom sample stage. The multilayer analyzer is also rotate-able independently from the detector.
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Figure 2.3.3. (Top) Inside of the main chamber. (Bottom) A schematic diagram of (i) 6 degree-of-freedom sample stage, (ii) a two-axis detector arm which moves in a horizontal geometry (P polarization), and (iii) a channeltron detector stage with multilayer analyzer for background rejection. All are actuated with vacuum-compatible stepper motors.
Figure 2.3.4 Zooming-in of the sample holder: (a) front view, (b) side view and (c) back view. It also shows the positions of three temperature sensors and a back-screw for tightening the sample holder to stage.

A zoom-in of the sample holder is shown in Fig. 2.3.4. Three temperature sensors were installed on the cold finger, at the end at the Cu-braids and in between the sample-holder holder and the sample holder. To have a better thermal contact, a screw was installed to allow a rigid clamping of the sample holder to sample stage. With this method we can reach sample temperatures down to 20 K. In principle, we could improve the temperature a bit more if radiation shields for the Cu-braids were installed.
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The last part we would like to point out is the channeltron-detector [see Fig. 2.3.5]. It consists of a slit, steel-grid, gold electrode which is covered with 1000 Å CsI and a channeltron electron multiplier. The slit which is located in front of the electrode is used to set the momentum resolution. The function of the steel grid, biased at -2000V is to reject electrons so only outgoing photons from the sample can travel to the electrode. To give high quantum efficiency we used the “unique” property of CsI [12]. Our earlier study showed that a thin film CsI exhibits a 100 times higher total electron yield than gold due to its wider band gap and lower work function. The gold, which is the substrate of the electrode, is important to avoid charging in the electrode since CsI is a very good insulator. A large number secondary electrons from the electrode are then caught by the front part of the channeltron. The channeltron itself is operated at 2500 Volts. We also apply a positive bias of 2000 V to the electrode to accelerate the secondary electrons into the detector. Instrument interfacing is done with LabVIEW and serial communication, which is controlled by an Epics-based VME (VERSAmodule Eurocard) system via a PC-based Channel Access Server. The front end is a Linux machine running Spec. The geometry of the experiment is explained in each chapter.
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References


