

U7C beamline and XAFS station of national synchrotron radiation laboratory

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The XAFS station on beamline U7C of National Synchrotron Radiation Laboratory (NSRL) was completely constructed in December 1998. The source for XAFS station is from a 3-pole superconducting wiggler with magnetic field of 6 T inserted in the straight section of the storage ring. Using a Si(111) double crystal monochromator with a fixed slit, the X-ray intensity at the sample position is about 3×10^9 photons/second at the energy of 8980 eV of Cu K-edge. The Keithley 6517 electrometers are used to record the electron charges that are produced in the ionization chambers. A high ratio of signal to noise has been obtained for the XAFS spectra of Cu, Ni and Fe foils. Furthermore, the XAFS spectrum of Cu foil in NSRL is in good agreement with that obtained in BSRF and KEK.

Keywords: U7C beamline of NSRL, XAFS station, measurement of electron charge.

1. Introduction

XAFS has become one of the widely used techniques in the applications of synchrotron radiation because of its capability to get local structure information on condensed matter in the field of physics, chemistry, materials and life science (Koningsberger, Lytle et al, 1999).

The National Synchrotron Radiation Laboratory (NSRL) in Hefei, P. R. China possesses a 200 MeV linac for second generation synchrotron radiation. The energy of the electrons in the storage ring is 0.8 GeV and the characteristic wavelength from bending magnet is 24 Å (516.6 eV). In order to meet the requirement for XAFS measurements, a superconducting wiggler has been inserted in the straight section of the storage ring. According to the theoretical calculation (Yan et al., 1996), the photon flux from a 6 T superconducting wiggler can reach 1.3×10^{10} photons/s/mrad/(0.1%B.W.) at the Cu K-edge. The photon energy of the characteristic wavelength is 2550 eV and the available energy can be extended to 15500 eV. It is about 5 times as high as that coming from bending magnet. In order to achieve the performance in the designed energy range, the beamline optics should be adjusted to the optimal position for each XAFS experiment. The design of the U7C XAFS beamline and station was started from 1994, the installation and construction were completed in December 1998, and it was open to users from November 1999. This report is an introduction to the instrumentation and characterization of the XAFS beamline and station.

2. Beamline and Monochromator

The insertion device for XAFS measurements is a single period, 3-pole superconducting wiggler with operating magnetic

field of 6 T. A Si(111) double crystal monochromator is used for the scan of energy which is described in detail elsewhere (Xu et al, 1998). A HUBER 420 goniometer is employed to drive the two separate crystals with (+n, -n) arrangement in the monochromator for Bragg angle rotation. The goniometer is controlled by using an accurate HUBER 9011 stepping motor controller. The schematic structure and performance parameters of the XAFS beamline are shown in Figure 1 and summarized in Table 1. As can be seen, the beamline is composed of three sections, e.g. the front end, the differential segment and the monochromatic system.

Table 1

The parameters of the monochromator and XAFS beamline

Acceptance angle of the monochromator (H×V)	1×0.1 mrad ²
Bragg angle range	10°-60°
Photons at sample position (Cu K-edge)	3×10^9 photons/second
Photon energy range	4.1-12.5 keV
Energy resolution ($\Delta E/E$)	3×10^{-4}
Spot size at the sample (H×V)	10×1 mm ²

3. XAFS station

3.1 Instrumentation

As we see in Table 1, the intensity of the flux to the station is lower, hence the detecting system is the key factor for the measurement of XAFS signals. In order to increase the ratio of signal to noise, we employ the Keithley 6517 electrometers to detect directly the electron charges (instead of the current) in the ionization chambers that are filled with N₂/Ar mixture gases. Keithley 6517 is an instrument with very high accuracy, sensitivity and large dynamic range. It can resolve charges as low as 10^{-14} coulomb and measure as high as 2.1 μC. The accuracy of it can be kept at the level of 0.4%, which is at least one power better than the traditional instrument used in the current measurement. According to Table 1, the measured photon flux at the sample position for XAFS station is 3×10^9 photons/s/mrad/(0.1%B.W.). If the measured charges are in the range of 10^{-7} to 10^{-10} coulomb, then the resolution of the instrument is in the range of 1 to 10 pC according to the specification of the instrument. Based on the above, the ratio of noise to signal is about 10^{-4} .

3.2 Method

In the coulomb function of the Keithley 6517, an accurately known capacitor of value C is connected in the feedback loop of the amplifier so that the voltage V_s in equation (1) can be scaled and displayed as charge. Equation (1) is the relation of the parameters in the charge measurement XAFS.

$$V_s = \frac{1}{C} \int_0^t i dt = \frac{Q_s}{C} \quad (1)$$

where V_s is the voltage imported to the inner capacitor, i is the current produced in the ionization chamber, Q_s is the scaled charge by the inner capacitor of Keithley 6517. Therefore, the

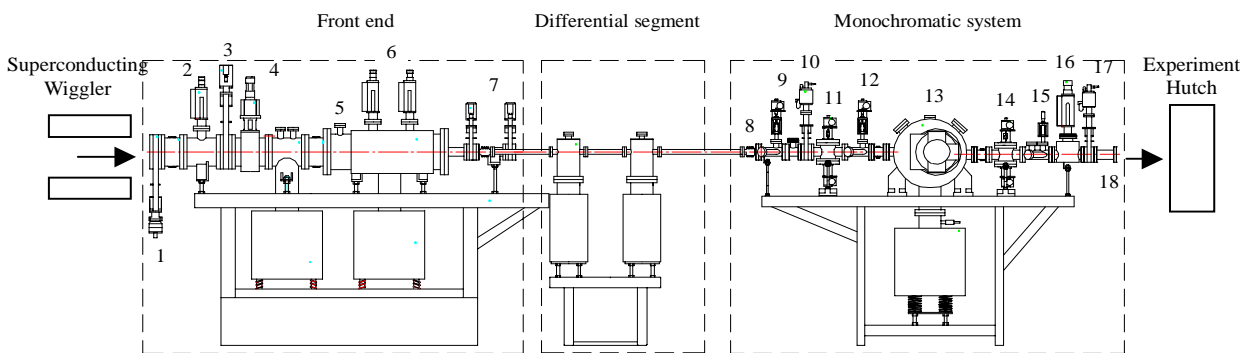


Figure 1. Side view of XAFS beamline at NSRL

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|-----------------|-----------------------|----------------------------------|-----------------------|-------------------------|
| 1. Handle valve | 2. Water cooling mask | 3. Pressure valve | 4. Fast control valve | 5. Separating diaphragm |
| 6. Beam stop | 7. Pressure valve | 8. Absorption Be window | 9. Diaphragm | 10. Pressure valve |
| 11. Entry slit | 12. Flux monitor | 13. Double crystal monochromator | 14. Exit slit | 15. Fluorescent screen |
| 16. Beam stop | | | | |

absorption coefficient μ , which represents the intensity of XAFS spectra, can be calculated according to:

$$\mu = A \ln \frac{Q_{s1}}{Q_{s2}} \quad (2)$$

where A is a constant, Q_{s1} and Q_{s2} are the charges displayed on the front and back Keithley 6517 electrometers respectively.

Figure 2 illustrates the schematic diagram of the detecting system for charge measurement XAFS. As indicated, the process of this kind of measurement is much simpler than current measurement method used in the traditional XAFS technique, however, the measuring precision is much higher.

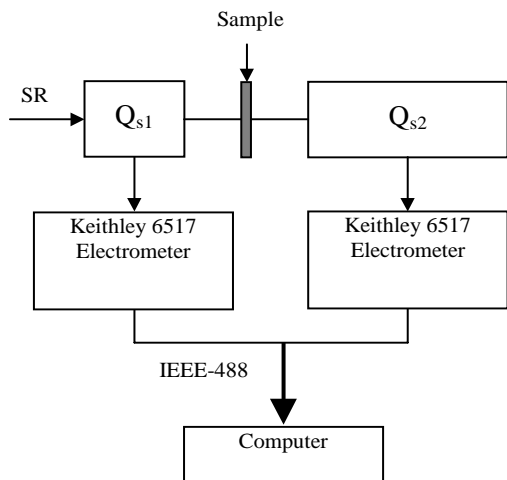


Figure 2 Schematic diagram of the detect system for charge measurement XAFS.

Figure 3 shows the curve of photon flux versus photon energy, which is drawn based on the electron charge (Q) accumulated in the front ionization chamber. The relation between Q and the number of photons (N_0) is approximately as the following equation:

$$Q = \frac{E}{30} N_0 (1 - e^{-\mu_x d}) \quad (3)$$

where E is the photon energy, μ_x is the absorption coefficient of

the gas in the ionization chamber (Here we used the 1:1 mixture gases of Ar and N_2), d is the length of the front ionization chamber. Obviously, the intensity of the incident X-ray after 5000 eV is stable enough to do XAFS measurement, but it decreases before 5000 eV that is mainly attributed to the stronger absorption of air to the softer X-rays.

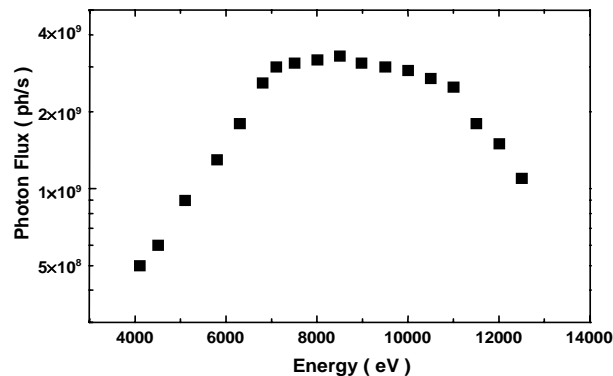


Figure 3 Photon Flux of XAFS station at sample position determined by charge measurement.

4. Test Results and Comparison

Recently, XAFS measurement in transmission mode has been made on XAFS station of NSRL. In the conditions of 6 T wiggler operation mode, the storage ring operating at 0.8 GeV, and beam current 50-150 mA, the EXAFS spectra of Fe, Ni and Cu foils at K-edge were measured. As shown in Figure 4, the K-edges of Fe, Ni and Cu located at 7111, 8331 and 8980 eV are set to the same energy for comparison, respectively. From the curves, we can clearly observe that the backgrounds preceding the absorption edges are very smooth and the oscillations afterwards are very reasonable, indicating that high ratios of signal to noise have been obtained on the XAFS station of NSRL.

Figure 5 is the comparison of EXAFS spectra for Cu foil measured at NSRL, BSRF and KEK respectively. From the magnitude and frequency of the oscillations, it can be seen that the EXAFS spectrum of Cu foil obtained at NSRL is in good agreement with those measured at BSRF (Wei et al, 1997) and KEK (Nomura et al., 1993). Considering the low flux at NSRL

for XAFS measurement, the three results are comparable.

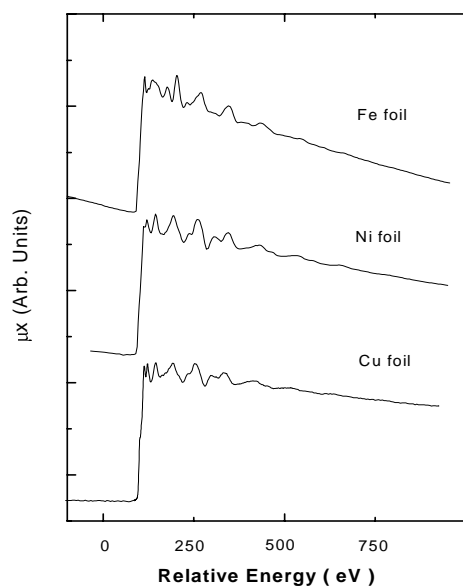


Figure 4
EXAFS oscillations for Cu, Ni and Fe foils determined at NSRL.

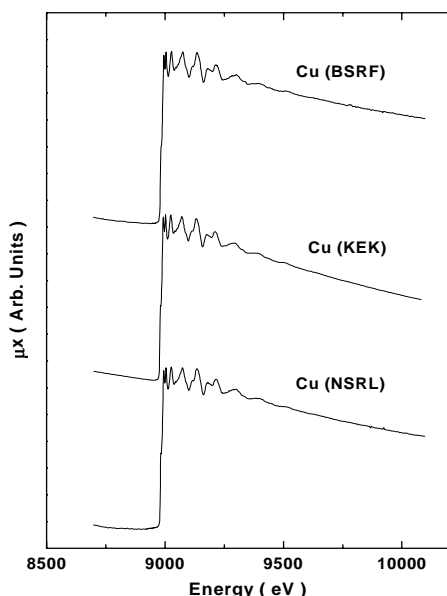


Figure 5
Comparison of the Cu K-edge EXAFS obtained at NSRL, KEK and BSRF.

Figure 6 demonstrates the radial distribution functions (RDF) for Cu foil after data processing and Fourier transform. This result displays clearly that the first neighbor coordination is 0.226 nm far from the central Cu atom and two characteristic features of fcc Cu structure appear at 0.416 and 0.480 nm which correspond to the third and fourth coordination respectively. The magnitude difference of oscillations among the three curves is less than 2%. Therefore, although the intensity of the light from NSRL is much lower than that of the other two, these results indicate that the XAFS station at NSRL has been in well working status.

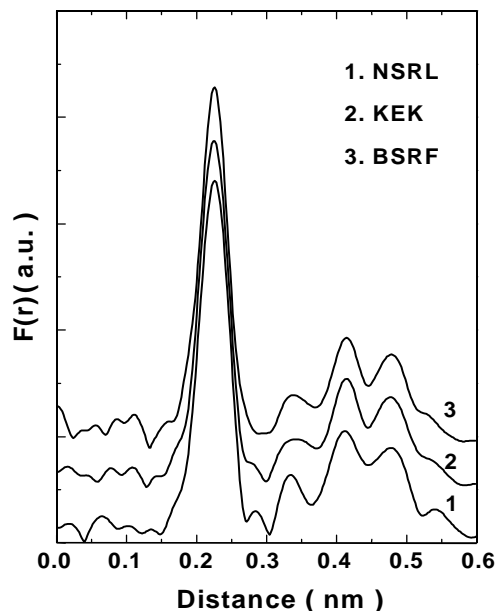


Figure 6
Comparison of radial distribution functions for Cu foils measured at BSRF, KEK and NSRL

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