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A new approach for in-laboratory XAFS equipment

Takeyoshi TAGUCHI,[a]* Qi-Fan XIAO[b] and Jimpei HARADA[a]

[a]X-ray research laboratory, Rigaku Corporation, 3-9-12 Akishima, Tokyo 196-8666, Japan, [b]X-ray Optical Systems Inc., 30 Corporate Circle, Albany, NY 12203, U.S.A.. Email:takey@rigaku.co.jp

Abstract. A so-called linear spectrometer with bent-crystal has been usually used for an in-laboratory EXAFS apparatus, because of its high efficiency of utilizing X-rays from an ordinary X-ray tube and its better energy resolution. In a linear spectrometer, the monochromator crystals have to be aligned very accurately along a Rowland circle, so the goniometer movement becomes very complicated. The mechanism of the monochromator can be very much simplified when parallel X-rays are used. We propose to use polycapillary optics to obtain a quasi-parallel X-ray beam from a conventional X-ray source and measure EXAFS spectra by a simple system utilizing an ordinary powder diffractometer.

Keywords: in-laboratory XAFS, polycapillary

1. INTRODUCTION

Almost two decades have already passed since the first workshop on laboratory EXAFS facilities was held at the University of Washington in 1980 (Stern, E. A. Ed., 1980). Since then, a lot of technological improvements have been made on laboratory equipment, such as the use of new material filaments, e.g. LaB6, to eliminate undesirable characteristic lines, high current X-ray generators (Sakurai & Osaka 1997), fast detectors, etc (Tohji, K., Mizushima, T. & Udagawa, Y. 1990). As the result, performance of laboratory based EXAFS machines are now at a remarkably higher level as compared with that at the time of the last workshop. Most commercial in-laboratory system use yet a bent crystal to monochromatize and to focus X-rays, but this makes the goniometer large and its movement complicated. An ordinary powder diffractometer can be used to measure EXAFS spectra by slit collimation, but the X-ray intensity is lower than the linear spectrometer. (Kuroda, H., Sato, T. and Asakura, K. 1985) The recent development of X-ray optical elements, i.e. mirror and capillaries, are remarkable and allow us to consider more simple optical system for X-rays. A multilayer mirror can collect X-rays with a fairly large capture angle, but can select a narrow energy range. This is not suitable for XAFS applications because we need to collect X-rays in wide energy range. Therefore, we examined capillary optics for this purpose. In this paper we present the comparison of the examined system with existing systems; specifically a linear spectrometer (named since monochromatized wavelength is "linear" to the distance between an X-ray source to a monochromator crystal. Udagawa, Y. 1993) and those used in synchrotron radiation facilities.

2. EXPERIMENTAL

A schematic experimental setup is shown in Fig.1. A rotating anode X-ray generator (RIGAKU RU-200, 60kV-200mA) was used as an X-ray source. In order to produce a quasi-parallel (divergence < 0.25 degrees) X-ray beam, a polycapillary collimating optic (XOS Inc. X-8-L10; designed to collect 4.21 degree x 4.21 degree Cu radiation from a standard line source with dimensions of 0.5 x 10 mm^2 in point focus geometry) was mounted on a three axis stage and was placed at 100mm away from the source point. The transmission efficiency of the optic, which depends on the X-ray source size, was 33% for a 0.25mm diameter source. The position of the collimating optic was adjusted so that the maximum flux could be obtained. A flat crystal monochromator was set at the sample position on an ordinary wide angle goniometer (RIGAKU RINT-2000) and monochromatic X-rays were measured in front of and after a sample (a transmission geometry) with a proportional counter and a scintillation counter.

3. RESULTS AND DISCUSSION

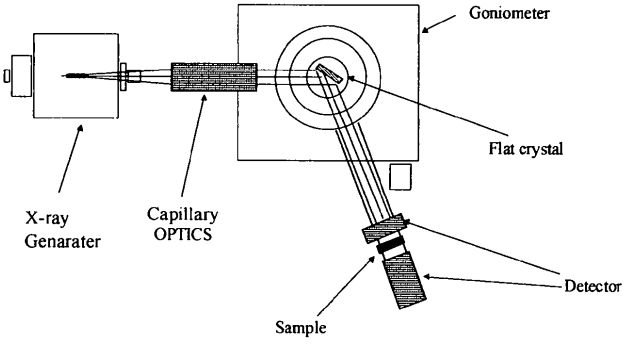
The energy resolution of this instrument was estimated by the measurement of the energy width of the W $L\alpha_1$ line (@8397.6eV, natural width is 6.50eV FWHM), using a Ge(220) flat crystal. The observed width of W $L\alpha_1$ was wider than 80eV. This is in agreement with the calculated value from the equation $\Delta E = \frac{E}{\tan\theta}\Delta\theta$, where E is the measured energy, θ is the Bragg angle at energy E and $\Delta\theta$ is the divergence angle of output beam, which is the same as the critical angle θ_c in which total reflection occurs in the capillary channel. Therefore the divergence of output beam should be made as narrow as possible so as not to lose too much intensity and to improve the energy resolution. When a soller slit was placed in front of the counter and using the 2nd order reflection, better energy resolution was obtained, but with a loss of X-ray intensity. We then compared the X-ray flux at the sample position (Fig.1) with and without the capillary collimating optic. When we measured the flux without the collimating optic, we put a 0.5mm wide slit at the position of the collimating optic and also in front of the detector. Figure 2 shows the X-ray intensity with and without the collimating optic, and the ratio of them. From the geometrical setting and transmission efficiency, we expected an intensity ratio of 43 at 8keV, but the actual ratio measured was about half of this value. This reduction is due to the X-ray source size. The collimating optic can collect X-rays from the area of 0.5 x 0.5 mm^2, but the source used was 0.5 x 1 mm^2. Thus about half X-rays were not collected by the optic. Applying a soller slit to restrict the divergence angle, or to improve the energy resolution, we expected an intensity loss of 57% (60% open area, angular restriction 0.17degree). Therefore the ideal ratio goes down to about 12 at 7keV. The measured ratio with a soller slit was 6.7 (Table 1). Misalignment of system could be a reason for this reduction. Yet, we get about 7 times more photons than restricting the divergence by slit. More than 20,000 photons/sec were counted with the collimating optic (20kV-200mA, Ge(440), @7keV). In order to accumulate 1,000,000 photons at a sampling point, one needs 50 sec. With 200 sampling point, total time to measure one EXAFS spectrum is less than 3 hours. Figure 3 shows a comparison of the iron EXAFS spectra measured with the capillary collimating optic and a spectrum obtained with a linear spectrometer (R-EXAFS Super). The X-ray flux at the sample point is more than 100,000 photons/sec (14kV-300mA,

Ge(220), DS 2 degree, RS 0.2mm, @7keV) and the total acquisition time for iron EXAFS was just over 1 hour (390 sampling points, 10 sec/point). The iron EXAFS spectrum measured at the Photon Factory, KEK, Tsukuba, is shown in Fig.3 as a reference. The spectra from linear spectrometer and the synchrotron are identical. The spectrum of capillary optic system is inferior to the other two in energy resolution. Detail of the oscillations, or high frequencies could not be reproduced, but rather good agreement is seen in the power spectra of the FOURIER transform.

4. SUMMARY AND CONCLUSION

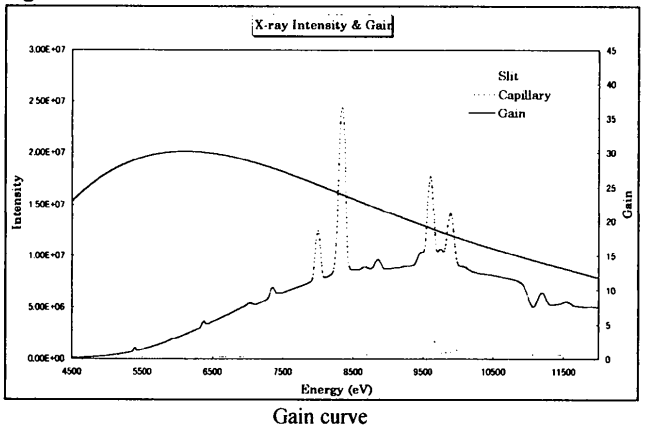
The linear spectrometer, which is used for most in-laboratory EXAFS system, is superior to the system examined here in energy resolution and X-ray intensity. Nevertheless the system where a polycapillary is used as an X-ray collimating optic with a wide angle goniometer occupies only 1/3 the area to be compared with the existing in-laboratory EXAFS system and the X-ray intensity is higher than when only slits are used. Therefore, we conclude that the system examined here can provide a modest way to get EXAFS spectra using an inexpensive flat crystal with a small modification to an existing powder diffractometer.

Figure 1



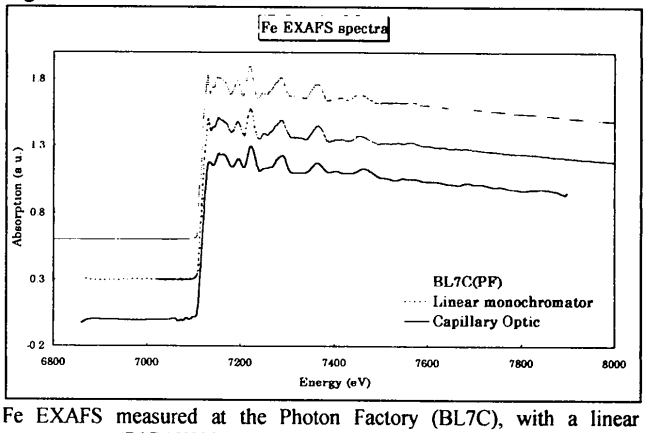
Experimental setup

Figure 2



Gain curve

Figure 3



Fe EXAFS measured at the Photon Factory (BL7C), with a linear spectrometer (RIGAKU R-EXAFS Super) and a system described here

Table 1

	FWHM (measured)	ΔE (calculated)	Gain
Capillary + Ge(220)	83.5	84.9	
Capillary + Ge(440)	27.9	30.8	
Capillary + Ge(220) + SS	40.5	59.1	
Capillary + Ge(440) + SS	19.4	21.4	6.7
0.5mm slit x2 + Ge(440)	17.6	19.2	1
Ge(220) R-EXAFS	11.8	6.2	(35)

Measured and calculated energy resolution and gain (relative intensity normalized by the slit collimation)

The iron EXAFS data measured at the Photon Factory is courtesy of Prof. Kuroda, H. (Science University of Tokyo).

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