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Laboratory diffractometer-based XAFS spectrometer

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The device has been developed to allow XAFS spectra of good quality to be obtained using conventional powder diffractometer. The device is based on the mechanical system which synchronize the changes of curvature radius of bent crystal-monochromator with the Bragg angle. Due to high resolution (1-10 eV depending on the wavelength) and photon flux only several hours are needed to perform quality XAFS experiment. The device is easy to operate and can be successfully used both for XAFS investigations and for student's practice.

Keywords: XAFS spectrometer, XAFS laboratory experimental facilities

1.Introduction

XAFS experiments are performed mainly using synchrotron sources or dedicated laboratory facilities. Their high costs and limited availability for users prevents XAFS from becoming a routine structural tool. Although these facilities have a lot of advantages which are necessary for sophisticated experiments, most part of routine local structural investigations can be performed by means of much more simple, easy - to -operate and available laboratory devices. In this work we present the original laboratory EXAFS spectrometer, which can be easily performed on the basis of powder diffractometer. It has been made on the basis of principal design, described in (Shuvaev et al., 1984, Shuvaev et al., 1985) and since for many years was employed in research and student's practice.

The main difference of the present design concept from the other laboratory XAFS facilities (for example Knapp et al (1978), Oyanagi et al, (1980), Maeda et al (1982), Tohji et al (1983)) is performance of Iohan's focusing scheme via variation of the curvature radius of bent crystal monochromator synchronously with the Bragg angle by means of simple mechanical device which is the principal part of the EXAFS spectrometer. The advantages of this design concept are low cost, simplicity and convenience of operation along with good energy resolution and quite high photon flux in a wide energy range. Reliability of it's construction is proved via long-term exploitation in several research laboratories.

2. Design

The spectrometer is shown schematically at the Fig.1. The X-ray tube and scintillation detector lie on the goniometer circle. Thin plate-like flexible crystal-monochromator made of bulk SiO_2 (40×10×0.1 mm) is positioned at the center of the goniometer. The X-ray beam from the tube is Bragg diffracted by the crystal-monochromator onto the detector in conventional $\Theta/2\Theta$ geometry. The sample is posed in front of the detector slits. The monochromator is cylindrically bent so that its center, the tube focus and the detector slit lie on a horizontal Rowland's circle. In order to satisfy Johan's focusing geometry in each point of spectra, the curvature of the bent crystal-monochromator is changed automatically with Bragg angle by means of mechanical four-points crystal bender. The bending adjustment is performed using mechanical system which is the principal part of the laboratory EXAFS spectrometer (Fig.2). The system consists of a movable block which is linked to a cylindrical surface, which axis is displaced from the goniometer center. The focusing in all points of the spectra is achieved via proper adjustment of the cylindrical surface. The bending system has small dimensions (of about 15 cm in diameter and height), doesn't require any electronics for operation and ensures high precision of crystal bending. It is easy to install and easy to remove, so that powder diffractometer can be easily transformed into EXAFS spectrometer and vice versa.

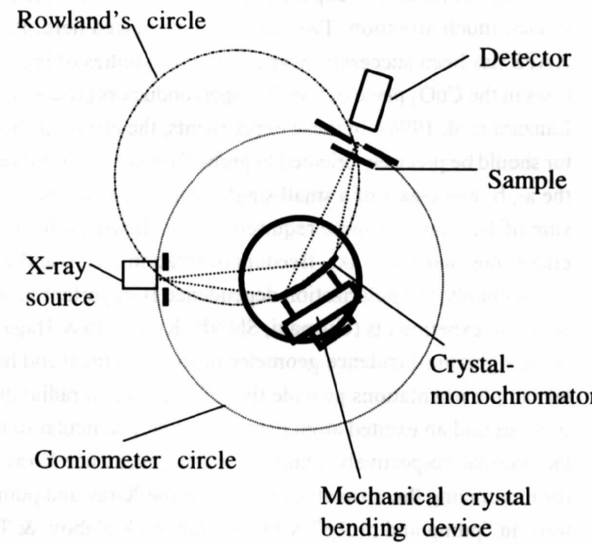

Figure1
Schematic view of the laboratory diffractometer- based XAFS spectrometer

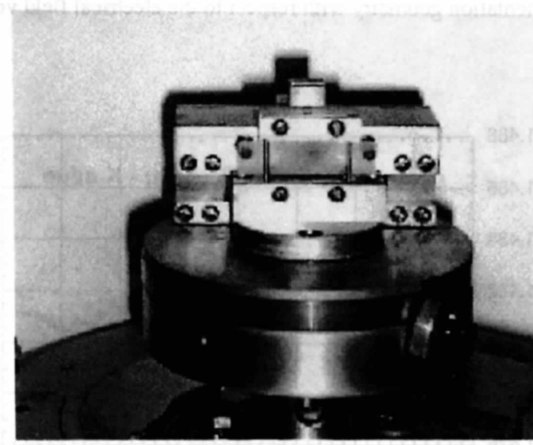

Figure 2
Crystal - monochromator bending system

3. Characteristics

The spectrometer has the following characteristics:

- photon flux up to 10^5 photon/sec at the tube voltage and current 30 kV and 25 mA respectively
- resolution from 1 eV (for Cr K-XAFS) to 10 eV (for Nb K-XAFS) with SiO_2 crystal-monochromator (better resolution in high energy region can be obtained using Ge crystal-monochromator)
- the energy range from 6 keV up to 20 keV

At the Figures 3 - 6 the spectra taken for only several hours by the laboratory XAFS spectrometer are presented. I_0 was measured by entire scan, however the device for automatic setting and removing of sample in each point of spectra is also available. The experimental details of the spectra measurements are presented in the Table1. Results of some other experiments, performed with the help of the EXAFS spectrometer, can be found for example in (Shuvaev et al., 1985, Shuvaev et al., 1989, Bugaev et al, 1995, Yagi et al, 1999).

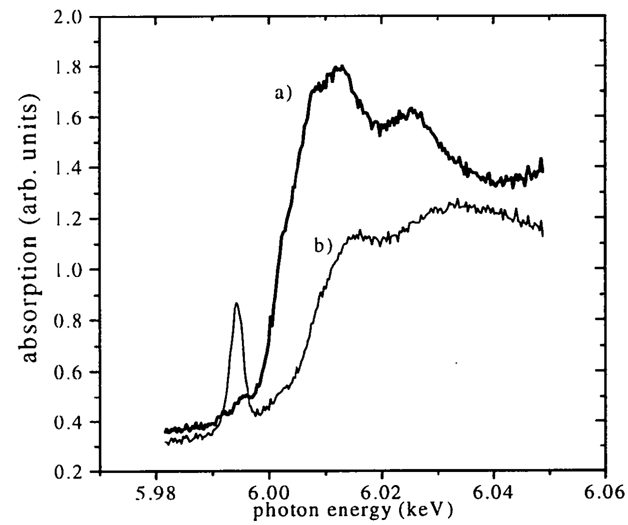

Figure 3
Cr K-edge XANES measured from powder samples of a)Cr_2O_3 and b) $(NH)_4Cr_2O_7$ using the laboratory XAFS spectrometer

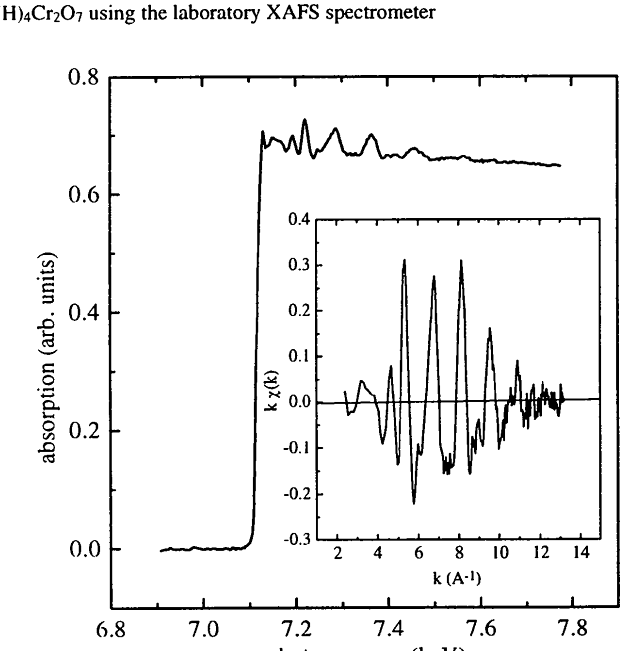

Figure 4
Fe K-edge EXAFS spectra obtained from powder sample of metal Fe using the laboratory XAFS spectrometer

Table 1
Experimental details.

	Cr_2O_3 and $(NH)_4Cr_2O_7$	metal Fe	metal Zn	$LiNbO_3$
Crystal-monochromator	SiO_2 (13-40) d=1.1801 A	SiO_2 (13-40) d=1.1801 A	SiO_2 (13-40) d=1.1801 A	SiO_2(24-60) d=0.80409 A
2Θ - range (°)	120.6-122.9	85-100	60-68	45-48.7
X-ray tube voltage and current	15 kV 25 mA	20 kV 25 mA	25 kV 30 mA	30 kV 25 mA
energy range (keV)	5.98-6.05	6.9-7.8	9.4-10.5	18.7-20.0
energy step	0.3 eV	3 eV	2.5 eV	3.5 eV
resolution	1.5 eV	2 eV	5 eV	10 eV
photon flux (photons/s)	10^4	3×10^4	6×10^4	8×10^4
time of measurements	4 hours	10 hours	6 hours	4 hours

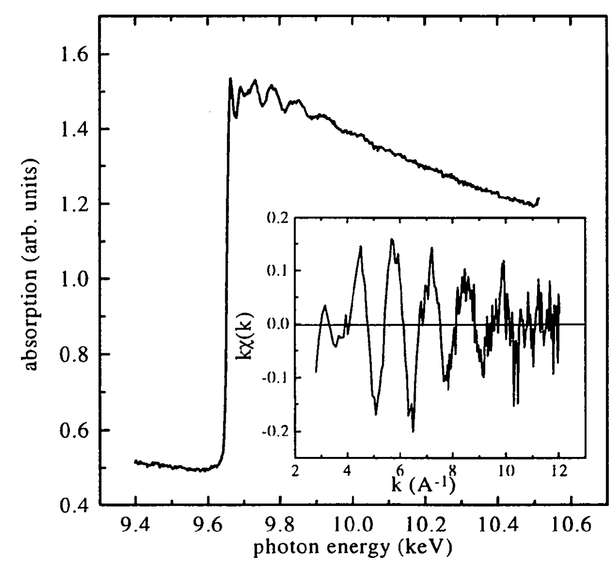

Figure 5
Zn K-edge EXAFS spectra of metal Zn thin film on the mica substrate measured using the laboratory XAFS spectrometer

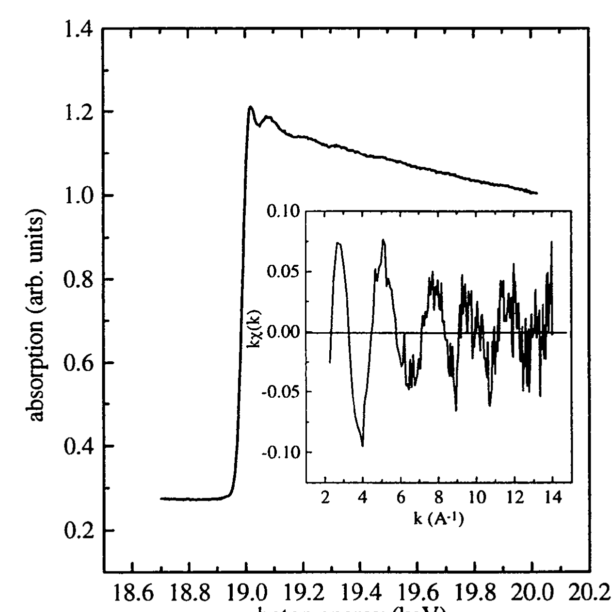

Figure 6
Nb K-edge EXAFS spectra , obtained from powder sample of $LiNbO_3$ using the laboratory XAFS spectrometer

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