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Fluorescence EXAFS spectrometer for Kurchatov synchrotron radiation center. First experiments

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Abstract

The paper presents fluorescence EXAFS spectrometer developed at Kurchatov Synchrotron Radiation Center. The spectrometer allows one to study elements from Cr ($Z = 24$) up to Bi ($Z = 83$). The important peculiarity of the spectrometer is the possibility to study thick undisturbed samples, retaining their history—annealing, seasoning, hardening and so on. The spectrometer consists of beam position monitor, channel cut monochromator, intensity monitor, sample holder and detector. The detectors consist of photomultipliers with scintillators. The EXAFS spectra of Ni–Cr alloys are measured and processed as the examples.

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Keywords: Synchrotron radiation; EXAFS spectrometer

1. Introduction

Now, Kurchatov Synchrotron Radiation Source [1] is equipped with several experimental stations. The spectrometer presented here uses widely expanded EXAFS method, which can determine the distance from absorbing the atom to some of its coordination spheres. The amplitude of thermal vibration, the sort of adjacent atoms and coordination numbers can be determined. The method can be applied to crystals, amorphous solids and even to liquids. Diluted samples can be investigated either [2,3].

2. The spectrometer

The spectrometer is mounted on the bending magnet beamline of big storage ring of Kurchatov

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Synchrotron Radiation Center [1]. The schemes of the spectrometer and its electronics are shown in Figs. 1 and 2, respectively. Additionally, Fig. 2 shows the system developed for stabilization of SR vertical position. Without such a system drift of the SR beam can achieve 1 mm, and reasonable result cannot be attained. Fluorescent screen placed at an angle of 45° to the SR axis registers non-used part of white SR beam. TV camera registers this SR beam optic image. The image is put into computer 2 supplied with the developed software [4]. The part of corresponding interface is shown in Fig. 3. Computer 2 is connected by a feedback loop with the storage ring control system. The system described here assured the stability of SR beam vertical position with the accuracy better then 10 μm.

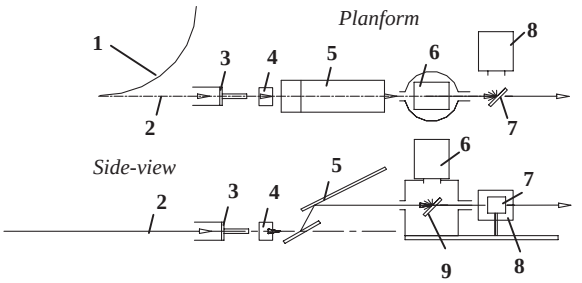

Fig. 1. EXAFS spectrometer scheme. (1) storage ring, (2) white SR beam, (3) SR beamline Be foils, (4) beam position monitor, (5) monochromator, (6) beam intensity monitor, (7) a sample to be investigated, (8) fluorescence detector, (9) monitor scatterer.

Channel cut monochromator Si(2 2 0) supplies photons with the energies from 6 up to 20 keV. The distance from the emitting point to the monochromator is 18.5 m. The resolution of the spectrometer with vertical slit 0.3 mm is 0.5 eV, which is near to optimal resolution for EXAFS measurement. On the left-hand side of Fig. 2 the system of monochromator movement is shown [5]. It consists of piezoelectric drive and angle encoder. Minimal angle step of the drive is 1 s of arc. The accuracy of total angle positioning is 5 s of arc. The system for stabilization of vertical SR position and the monochromator are used for refractive lens test too.

Monitor detector registers radiation, which is scattered by a thin organic film placed right after the monochromator. This monitor gives us the possibility to consider a slow decrease of the intensity conditioned by storage ring current decrease as well as narrow minimums in the intensity conditioned by multi-wave reflection processes in the monochromator crystal. Detector for measuring fluorescence of a sample is placed in storage ring orbit plane at an angle of 90° to input beam. Such a position provides the minimum noise level, which is conditioned by elastic scattering on

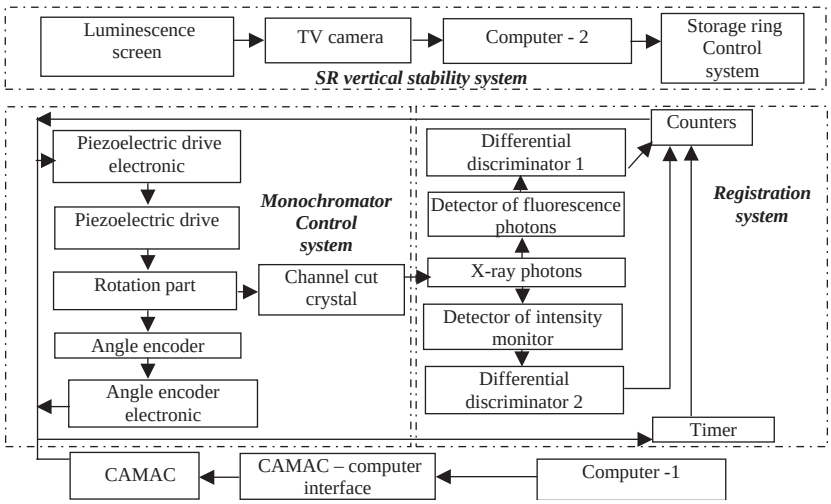

Fig. 2. EXAFS spectrometer electronics.

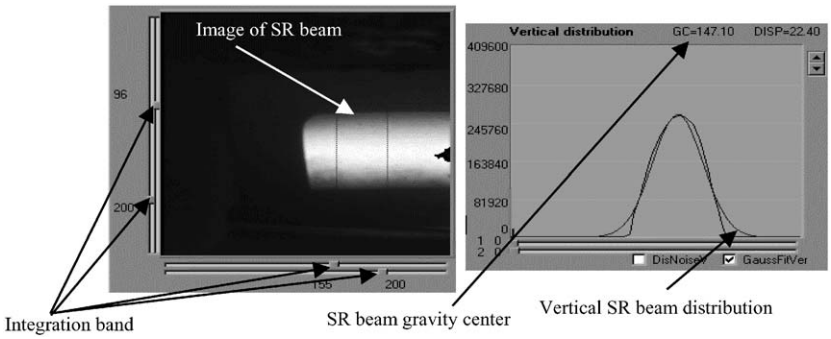

Fig. 3. Part of interface of the code for SR vertical position beam monitor.

the sample. The monitor and fluorescence detectors consists of photomultipliers with NaI(Tl) scintillators in counting mode. Electronics blocks are developed in CAMAC standard. A software complex was developed to control the spectrometer. The typical time for measuring one experimental spectrum is about 3 h.

3. Experiment and preliminary data processing

To verify the spectrometer we compared measured fluorescence spectrum of Cu foil with corresponding absorption spectrum of the same sample measured at ESRF. Processing of the both spectra gives practically the same Fourier transformations. Ten samples of Ni-based alloys with different Cr contents were investigated. These alloys are very interesting for atomic energy industry because some of them have high radiation and corrosion resistibility. EXAFS spectra on K-edges of Ni and Cr were measured.

Primary spectra have about 5000 experimental points. To improve statistical uncertainty we made averaging of five adjoining points. Now we make preliminary processing of the spectra. The processing is not trivial and applying different methods of processing we can get different results. We give one of the possible methods of preliminary processing here. Effective absorption coefficient was obtained as the ratio of fluorescence count to monitor count. Delicate effects connected with scattering geometry were not considered.

Experimental results were preliminarily processed with the help of VIPER code developed by Klementiev [6]. To find one atom (mean) absorption coefficient $\mu 0$, we used spline interpolation mode. Fig. 4 shows one of the experimental spectrum and corresponding preliminary processing. In the upper left part of Fig. 4 one can see effective absorption coefficient. The upper right part of Fig. 4 shows oscillation of absorption coefficient that is XAFS function. In the left bottom part of Fig. 4 one can see Fourier transformation of XAFS function. They usually call such a Fourier transformation as pseudo-radial distribution of electron density around investigated atom. In the right bottom part of Fig. 4 we placed filtered XAFS function made by back Fourier transformation after cutting some highest harmonics. This function can be used on the next processing stage with structure model of investigated sample and function of electron cross-section scattering on sample elements.

In the first approximation we can presume that a peak situated near 2 Å region of Fourier transformation corresponds to the first coordination sphere. In the experiment, oscillation amplitude is approximately constant and almost do not depend on Ni concentration in a sample. This leads to a notable changing of Fourier maximum with only slight changing of its position. Structure of radial function near 3–5 Å region differs from

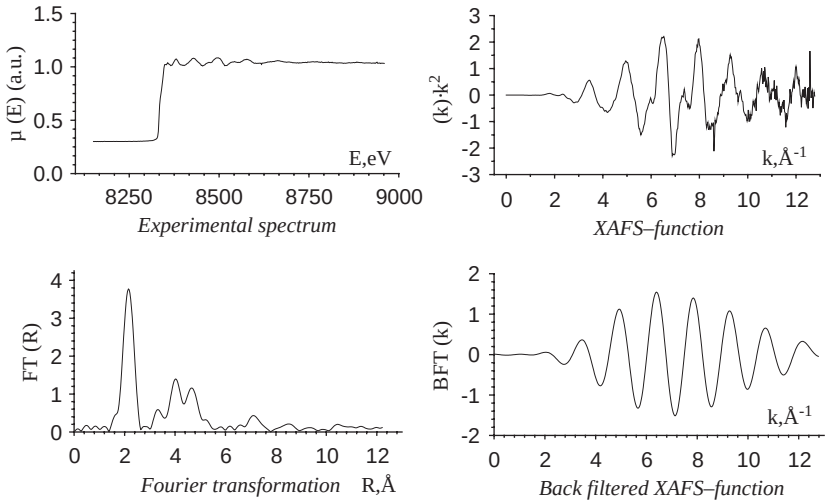

Fig. 4. EXAFS spectrum of alloy Cr—32.11%, Mo—1.28% Ni—base, and its preliminary processing.

spectra to spectra. Maybe these parts correspond to the second coordination sphere.

4. Conclusion

The fluorescence EXAFS spectrometer is on-stream at Kurchatov Synchrotron Radiation Center. Taking into account the energy band of the spectrometer one can see that a number of elements, which are interesting for atomic energy industry, can be investigated beginning from Cr ($Z = 24$) up to Bi ($Z = 83$). Lighter elements can be investigated by measuring absorption K-edges, and heavier ones by measuring L-edges. The important peculiarity of the spectrometer is the possibility to investigate thick undisturbed samples, which conserve their history—annealing, seasoning, hardening and so on. A number of EXAFS spectra of some Ni–Cr alloys were measured and preliminarily processed.

Acknowledgments

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