Journal of
Synchrotron Radiation

ISSN 0909-0495

Received 2 May 2011
Accepted 19 October 2011

Synchrotron-based spectroscopy of X-ray channeling through hollow capillary microchannels inside glass plates

M. I. Mazuritskiy

Department of Physics, Southern Federal University, Rostov-on-Don 344090, Russian Federation.
E-mail: mazurmik@gmail.com

Here, soft X-ray synchrotron radiation transmitted through microchannel plates is studied experimentally. Fine structures of reflection and XANES Si L-edge spectra detected on the exit of silicon glass microcapillary structures under conditions of total X-ray reflection are presented and analyzed. The phenomenon of the interaction of channeling radiation with unoccupied electronic states and propagation of X-ray fluorescence excited in the microchannels is revealed. Investigations of the interaction of monochromatic radiation with the inner-shell capillary surface and propagation of fluorescence radiation through hollow glass capillary waveguides contribute to the development of novel X-ray focusing devices in the future.

Keywords: X-ray spectroscopy; X-ray scattering; XANES; X-ray fluorescence channeling; X-ray capillary waveguides.

1. Introduction

Capillary optics is one of the fastest growing X-ray optical technologies (Pfeiffer *et al.*, 2002; Sun *et al.*, 2009) because of its superior capacity of generating high-flux-density beams in the micrometre and submicrometre range. The properties and peculiarities of X-ray beams formed by polycapillary optics have been intensively studied over the past 20 years (Kumakhov & Komarov, 1990; Bilderback *et al.*, 1994; Dabagov *et al.*, 1995; Dabagov & Uberall, 2007). Investigations of X-ray waveguides and microcapillary optics based on the phenomenon of multiple total external reflection (Dabagov, 2003) are devoted to ultrafocusing properties and partially coherent beams. Application of the wave optics methods allows the processes of radiation transmission by the guides to be described in detail. In microchannels the propagation features are defined by the interaction of radiation with a curved surface and characterized by the waves that propagate close to the capillary walls. A basic point of X-ray beam transportation in a capillary channel (as an X-ray waveguide) is the mode regime (Spiller & Segmuller, 1974; Dabagov, 2003; Bukreeva *et al.*, 2010) associated in particular with the surface channeling of X-ray radiation, first determined by Dabagov (see Bellucci & Dabagov, 2003; Dabagov & Uberall, 2007).

It is known that the pioneering papers (Dabagov & Okotrub, 2004; Okotrub *et al.*, 2005) presented experimental results on X-ray fluorescence channeling in carbon nanostructures. Microchannel plate (MCP) systems can be applied to focus and collimate X-rays. The optics can provide a highly effective low-weight device and may be ideally suited for use in many applications. One of the goals of this paper is to present experimental data on the transmission of X-ray fluorescence radiation emitted by the walls of the silicon glass microchannels through microcapillary structures. First experimental results on the spectroscopy of X-ray fluorescence transportation in the MCP were published earlier (Mazuritskiy, 2006).

Grazing-incidence X-ray methods based on the analysis of the secondary radiation (fluorescence, reflection) from a solid surface as a result of interaction with primary radiation have a great scientific and applied interest. If the grazing angle is less than the Fresnel angle, and incident photons are capable of exciting the atomic levels, then both X-ray fluorescence and elastic scattering are observed. Inelastic scattering and the propagation of fluorescence radiation have been studied in the anomalous dispersion region of the energy range near the Si L-absorption edge.

2. Experimental set-up

An MCP sample (thickness $\sim$0.5 mm) consisting mainly of SiO_2 glass substrate (without special treatment) had a regular structure (hexagonal type in the transverse cross section), holey cylindrical channels (pore) 8 µm in diameter and centre-to-centre spacing (pitch) size 10 µm. Monochromatic beam (divergence <10 mrad) penetrated the microchannels of the polycapillary structure (see Fig. 1). The primary radiation was directed almost perpendicularly to the plate surface of the MCP, corresponding to grazing incidence onto the microchannel walls. Samples were electrically insulated from the metal (Fe) surface of the holder. Radiation passing through the microchannels was absorbed by the holder and detected as an electrical photocurrent.

The grazing angles between the monochromatic incident radiation and the microchannel walls were adjusted by the rotation of the sample (around the X, Y axes; see Fig. 1). In the first step the polycapillary structure was aligned to the 'zero' position where capillary channels were parallel to the primary monochromatic beam. Fine structure was not observed in the spectra at the zero position. The angle between the primary beam and the microchannel walls was fixed for each spectrum. The fine structure was measured at the exit

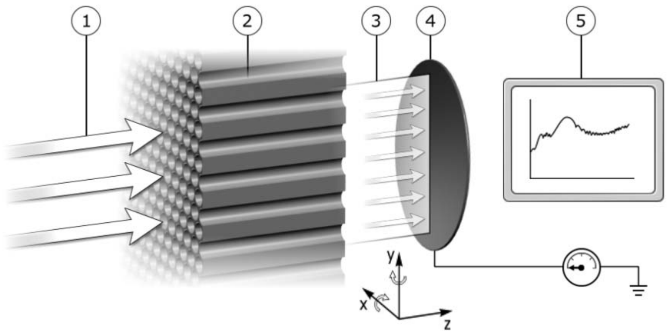

Figure 1
Layout of the experiment: 1, primary monochromatic radiation; 2, microchannel plate; 3, X-ray radiation at the exit of microchannels; 4, metal holder of the sample (collector) and detector of radiation; 5, system for recording.

of the MCP for various grazing-incidence angles of the primary beam onto the channel walls.

The X-ray absorption near-edge fine structure (XANES) and reflection spectra were excited by monochromatic synchrotron radiation with photon energies varying in the vicinity of the Si L-absorption edges. The fine structure of the spectra has been studied at different grazing angles including the X-ray total external reflection regime (less than the critical angle). The critical angle (about 10°) corresponds to the experimental data (Tripathi *et al.*, 2002) for the glass in the vicinity of the Si $L_{2,3}$-absorption edge (100–140 eV). The Si L_1-edge of absorption corresponds to a photon energy of $\sim$160 eV. The spectra were measured with a resolution of 0.1 eV on the spectrometer MUSTANG RGBL-PGM (Fedoseenko *et al.*, 2001) at the BESSY synchrotron centre. The intensities of the spectra presented in the figures have been normalized to the intensity of the incoming beam.

3. Results and discussion

Fig. 2 shows the fine structure of the Si $L_{2,3}$-XANES obtained in this work for the same sample under different experimental conditions. Spectrum 1 corresponds to radiation propagating through microchannels and absorbed directly by the holder. The angle of the primary radiation to the walls of the MCP channels was 3.5°. Spectrum 2 corresponds to the classical total electron yield spectrum of the same MCP measured in the mode of absorption of primary monochromatic radiation by the sample. An electrical photocurrent in the sample passing through the holder was detected. In this case the sample was in electrical contact with the holder and the angle between the primary radiation and the surface of the channel walls was large ($\sim$30–45°) and fixed. The presented spectra were mathematically transformed by background subtraction for correct comparison of the fine structures. A spectral background estimation was carried out by a polynomial fitting to the smooth regions of the spectrum (apart from the peaks) below and above the absorption edge.

It should be underlined that the fine structures of the spectra 1 and 2 in Fig. 2 are similar and correspond to the fine structure of Si L-absorption spectra published earlier (Wu *et al.*, 1998; Gilbert *et al.*, 2003). It is known that fluorescence yield spectra refer to ionization of the atomic inner-shell in a sample at the first stage. When the outer-shell electron fills the inner-shell vacancy in an ionized atom a characteristic photon will be emitted. The absorption peaks A, B, C and D of the XANES spectra are determined by multiple-scattering resonances of the photoelectrons excited at the atomic absorption sites and scattered by neighbour atoms (Wu *et al.*, 1998). Spectrum 1 is the XANES spectrum detected as X-ray fluorescence radiation at the exit of microchannels. So, the spectroscopic features define the possibility of the transportation of excited Si L-fluorescence in media through the microcapillaries.

The interaction between soft X-rays and matter is classified into absorption and elastic scattering. In the ideal case it is possible to consider the incident monochromatic beam after interaction with a smooth surface as being split into two beams: mirror reflected and refracted. To discriminate between XANES and reflection spectra it is necessary to analyze their fine structures. One of the properties of XANES spectra in comparison with X-ray reflection spectra is a relatively high difference in intensity for the energies under and above the Si $L_{2,3}$-absorption edge. A sharp peak in the fine structure in the immediate vicinity of the absorption edge (above the energy) is an additional peculiarity of the XANES spectrum. These features make it possible to specify whether a spectrum is a reflection or an XANES spectrum. It is especially important for grazing-incident experimental geometry and photon energies in the vicinity of an absorption edge. Below we will analyze the fine structure of the spectra presented in this work and also compare them with spectra published earlier.

Spectra 2 and 3 in Fig. 3 are reflection spectra because their fine structure corresponds to that presented by Filatova *et al.* (1995) for a

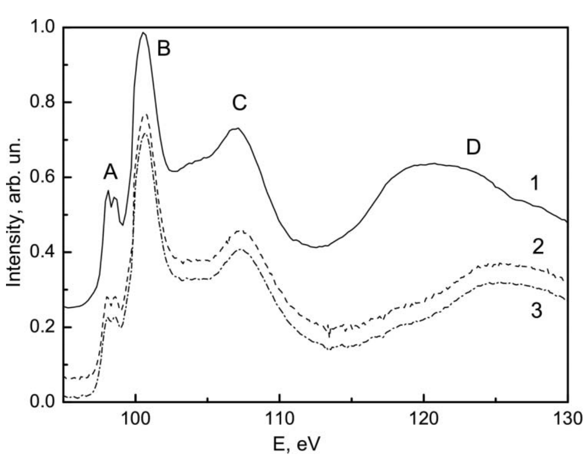

Figure 2
XANES Si $L_{2,3}$ spectra of MCP: 1, radiation passing through the microchannels; 2, radiation absorbed by sample; 3, smoothed curve of spectrum 2 using a three-point spline interpolation.

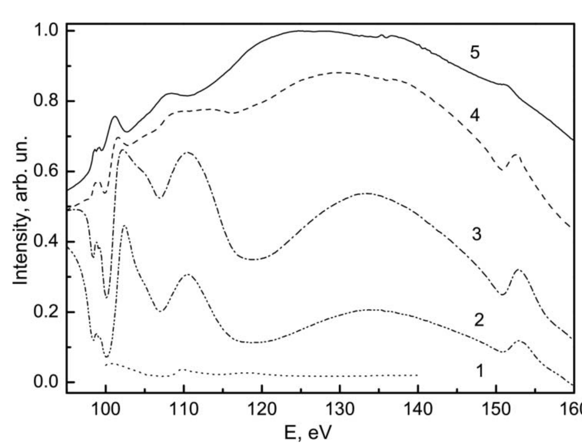

Figure 3
XANES Si $L_{2,3}$ spectra on the exit of the MCP for various angles of incident radiation onto the channel walls: 1, 9°; 2, 6.5°; 3, 5.5°; 4, 4.5°; 5, 3.5°.

flat SiO_2 surface. However, for the MCP in our case the primary radiation enters long and narrow channels, so that the reflected rays cannot be directly emitted. The propagation of X-ray radiation through capillaries at grazing angles less than the critical angle of total external reflection is realised by multiple reflections on the capillary inner surfaces and can be described within the general formalism of X-ray channeling (Dabagov, 2003). It should be underlined that the fine structure of spectra measured at the output of the MCP is similar to that of simple reflection spectra.

For angles equal or less than 3.5° (see Fig. 3, curve 5) the fine structure unambiguously determines the XANES spectra for SiO_2 published earlier by Wu *et al.* (1998) and Gilbert *et al.* (2003). It is known that the spatial distribution of fluorescence is almost isotropic. Therefore, a low intensity of X-ray fluorescence at the output of the MCP should be detected. On the contrary, the (arbitrary) intensity of spectrum 5 in Fig. 3 is increased in comparison with the reflection spectra. So, X-ray fluorescence excited in the microchannels can propagate mainly through the capillaries at grazing angles less than the critical angle. In other words, analysis of the fine structure of XANES spectra makes it possible to conclude that X-ray fluorescence is selectively transported along the hollow MCP channels.

The fine structure of spectrum 4 in Fig. 3 can be presented as a linear combination of curves 3 and 5 (transformation from reflection to XANES spectrum). In this case we assume the propagation of both reflected radiation and X-ray fluorescence together through the microchannels of the MCP.

The propagation of radiation in capillary systems depends on its interaction with the inner walls of the channels and is determined by the permittivity. Resonance phenomena in X-ray spectroscopy are observed near the atomic absorption edges if the photon energy of the incident radiation is close to the energy of the electronic transition from an inner electron shell to unoccupied free states. Correct theoretical calculations of the propagation of X-ray fluorescence in the capillary systems require detailed information on the real and imaginary parts of the permittivity in the vicinity of an absorption edge which can be obtained from theoretical calculations and experimental reflection or XANES spectra. The modes of radiation propagation in a glass capillary waveguide and interference between the incident and reflected (fluorescence) waves can be studied. On the basis of the experimental and theoretical data, important results can be reached in the applications of soft X-ray optics particularly regarding the extreme focusing of radiation by the polycapillary systems.

The author would like to thank the Russian–German laboratory at the BESSY synchrotron centre for technical support (project No. 2008_1_70987).

References

Bellucci, S. & Dabagov, S. B. (2003). *J. Phys. Condens. Matter*, **15**, 3171–3178.
Bilderback, D. H., Hoffman, S. A. & Thiel, D. J. (1994). *Science*, **263**, 201–203.
Bukreeva, I., Pelliccia, D., Cedola, A., Scarinci, F., Ilie, M., Giannini, C., De Caro, L. & Lagomarsino, S. (2010). *J. Synchrotron Rad.* **17**, 61–68.
Dabagov, S. B. (2003). *Phys. Usp.* **46**, 1053–1075.
Dabagov, S. B., Kumakhov, M. A., Nikitina, S. V., Murashova, V. A., Fedorchuk, R. V. & Yakimenko, M. N. (1995). *J. Synchrotron Rad.* **2**, 132–135.
Dabagov, S. B. & Okotrub, A. V. (2004). *Spectrochim. Acta B*, **59**, 1575–1580.
Dabagov, S. B. & Uberall, H. (2007). *Nucl. Instrum. Methods Phys. Res. A*, **580**, 756–763.
Fedoseenko, S. I., Iossifov, I. E., Gorovikov, S. A., Schmidt, J.-S., Follath, R., Molodtsov, S. L., Adamchuk, V. K. & Kaindl, G. (2001). *Nucl. Instrum. Methods Phys. Res. A*, **470**, 84–88.
Filatova, E., Stepanov, A., Blessing, C., Friedrich, J., Barchewitz, R., Andre, J.-M., Le Guern, F., Bac, S. & Troussel, P. (1995). *J. Phys. Condens. Matter*, **7**, 2731–2744.
Gilbert, B., Frazer, B. H., Naab, F., Fournelle, J., Valley, J. W. & de Stasio, G. (2003). *Am. Mineralog.* **88**, 763–769.
Kumakhov, M. A. & Komarov, F. F. (1990). *Phys. Rep.* **191**, 289–350.
Mazuritskiy, M. I. (2006). *JETP Lett.* **84**, 381–383.
Okotrub, A. V., Dabagov, S. B., Kudashov, A. G. *et al.* (2005). *JETP Lett.* **81**, 34–39.
Pfeiffer, F., David, C., Burghammer, M., Riekel, C. & Salditt, T. (2002). *Science*, **297**, 230–234.
Spiller, E. & Segmuller, A. (1974). *Appl. Phys. Lett.* **24**, 60–64.
Sun, T., Zhang, M., Liu, Z., Zhang, Z., Li, G., Ma, Y., Du, X., Jia, Q., Chen, Y., Yuan, Q., Huang, W., Zhu, P. & Ding, X. (2009). *J. Synchrotron Rad.* **16**, 116–118.
Tripathi, P., Lodha, G. S., Modi, M. H., Sinha, A. K., Sawhney, K. J. S. & Nandedkar, R. V. (2002). *Opt. Commun.* **211**, 215–223.
Wu, Z. Y., Jollet, F. & Seifert, F. (1998). *J. Phys. Condens. Matter*, **10**, 8083–8092.