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XRF Microanalysis of Thick Objects

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Abstract. The conditions for investigation of specimens by the micro XRF method on the existing SR source VEPP-4M and the prospective SR source SKIF are compared.

INTRODUCTION

In a non-destructive study of samples by the method of x-ray fluorescence analysis, the absorption depth of the exciting radiation limits the thickness of the sample. When complex geological objects containing elements with high atomic numbers are under study, the excitation energy can be as high as several tens of keV and generate fluorescent photons from a depth of several hundred microns. However, the initiating radiation beam focused on the front plane of the sample gets defocused at such depths, and under the conditions discussed in the report its cross-section can become much larger than the focus size. This effect is especially significant at a small size of x-ray source, as in the case of the SKIF synchrotron radiation source. Based on the geometric model of the elemental resolution voxel the excitation energy and the focusing conditions can be proposed.

SIMULATION

Refractive x-ray lenses [1] are considered the most effective for focusing hard x-rays. Despite the low refractive index decrement, their aperture can be increased as compared with a classical lenses with the parabolic profile, through structural modification based on precision micro engineering [2]. At the same time, the low level of scattered background and constructive simplicity allow setting the lens in the vicinity to the object [3] to increase the photon flux onto the specimen.

The analysis of arrangement of the specimen and the lens was based on the fact that lenses designed for high photon energies can reproduce short focal lengths. Figure 1 presents a table of refractive lenses, which are located on one holder and can be fitted for photon energies of up to 200 keV.

TABLE 1. X-ray refractive lenses made of polymer on one substrate using deep x-ray lithography technique.

Lens number	Structure number	Aperture, μm	Curvature radius, μm	Structure length, μm
1	216	40.85	4.6	127
2	157	60	6.6	164
3	129	84.6	9.8	213
4	88	128.3	15.9	312
5	75	156.2	19.4	367
6	57	214.9	27.4	484
7	45	278.6	35.4	611
8	35	369	47.6	778

To keep the object intact for further research, we studied objects whose thickness was many times greater than the depth of fluorescence escape from the object. It is convenient to place such objects at 0 degrees to the axis of the initial x-ray beam, while the detector is located at 90 degrees to the axis, as shown in Figure 1.

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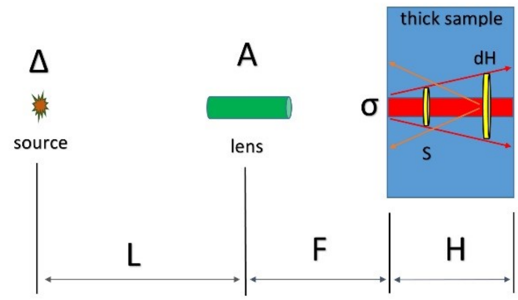
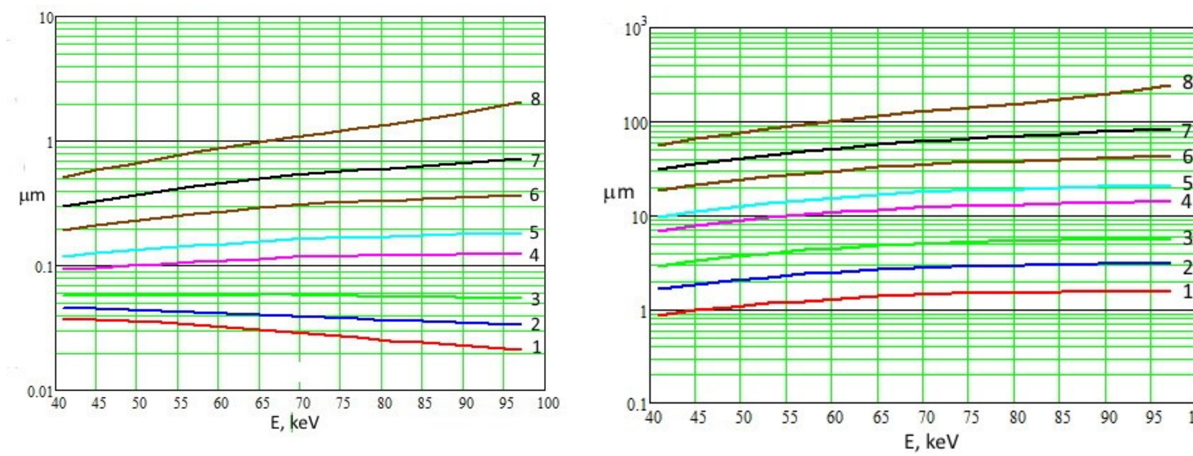


FIGURE 1. Beam geometry for simulation

For the geometry shown in Figure 1, a calculation of the flux of luminescent radiation to the detector was carried out for the above geometry of the detector and lens for two sources of synchrotron radiation: VEPP-4M and SKIF under development. In the comparison, the length of the synchrotron radiation out coupling beamlines was taken to be the same, 40 m.

A Cerium from the middle of the periodic table was chosen as the material of test object.

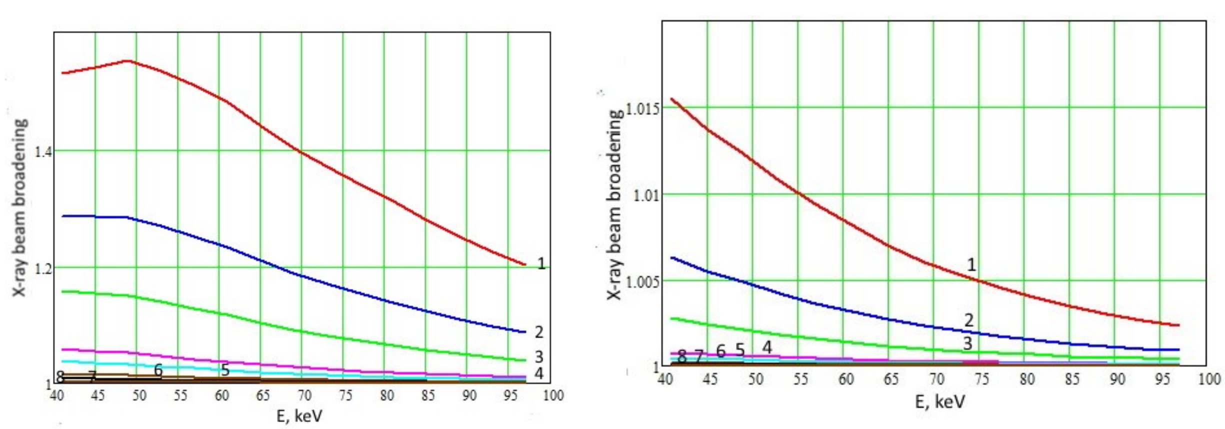
Figure 2 shows the calculation results. As seen from Figure 2., the focus size on SKIF can achieve approx. 20 nm (for the refractive profile of a lens made without aberrations) and is close to the diffraction limit for a long refractive lens [4], which is much better than the focus size for VEPP-4M, hardly reaching 1 μm.



(a) (b)

FIGURE 2. Calculated X-ray spot size for different x-ray refractive lenses for: (a) SKIF; (b) VEPP-4M

The expansion of x-ray beam passing through a thick specimen is represented in Figure 3, averaged over the beam path. From Figure 3 one can see that the average beam (relative) width changes significantly for lenses with a small aperture due to the expansion of the beam on the sample when the radiation source has a small transversal size (SKIF). For a source with a large transversal beam size, this effect is suppressed due to the beam size.



(a) (b)

FIGURE 3. Calculated broadening of X-ray beam for different x-ray refractive lenses for: (a) SKIF; (b) VEPP-4M

EXPERIMENTAL

Figure 4 shows the experimental setup for measuring the focus size of a refractive x-ray lens installed at beamline №8 of the storage ring of VEPP-4M [5]. The x-ray lens was mounted on the 6-axis precision stage. The x-ray knife was used to measure the focus. The Amptek silicon drift detector (XR-100SDD) was employed to measure the fluorescence from the knife-edge. The x-ray image sensor was installed behind the focus, at a distance of about 200 mm, and utilized for visualization of the x-ray image of the lens.

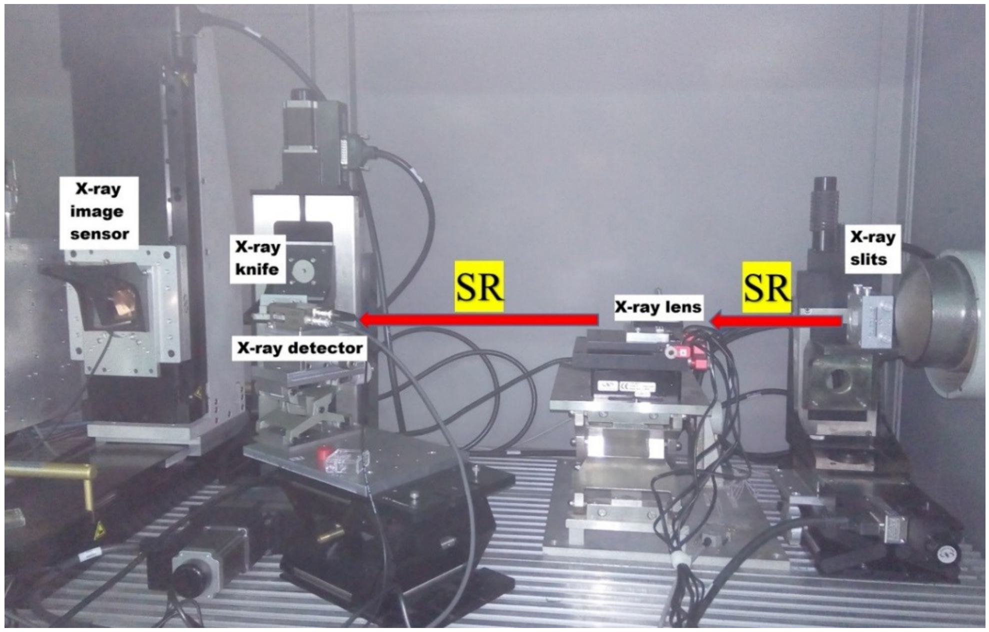


FIGURE 4. Experimental setup for measuring focus of refractive lens at the beamline №8 (VEPP- 4M)

The x-ray beam was generated in the 9-pole wiggler at a storage ring VEPP-4M current of 20 mA and electron energy of 4.5 GeV. The distance from the emission point to the lens was 32000 mm; the distance from the lens to the knife was 590 mm. The photon energy was 42 keV. Figure 5 shows the experimental dependence of the fluorescence intensity from the x-ray knife-edge in vertical direction (red line), derivative of the red line (blue line), and fitting with the Gaussian (black line). The width of the vertical cross section of the focused beam was 11.4 μm.

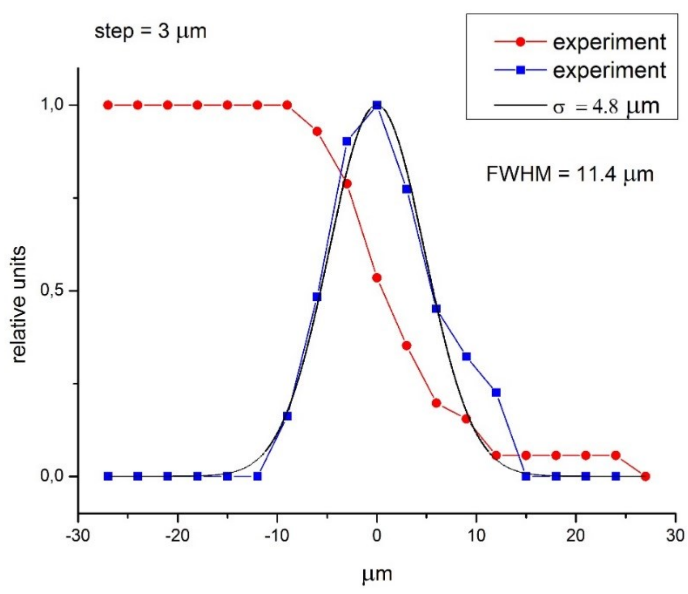


FIGURE 5. Red line: intensity of fluorescence caused by x-ray knife vs. knife-edge vertical position. Blue line: derivative of the fluorescence intensity from x-ray knife-edge (red line). Black line: fitting by Gaussian. FWHM of fitted curve is 11.4 μm

Figure 6 shows the x-ray fluorescence spectrum from a voxel behind a 11 µm x-ray focal spot. The vertical gain factor ≈ 10. This spectrum shows sharp suppression of the elastic and Compton scattering from the sample.

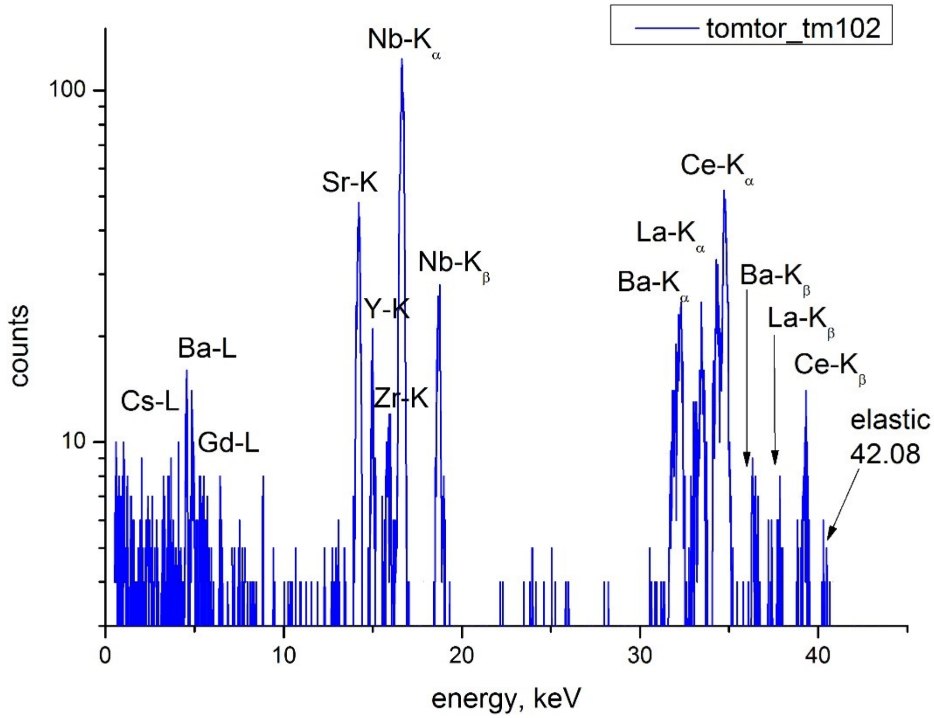


FIGURE 6. X-ray fluorescence spectrum from specimen voxel behind 11 µm x-ray focal spot

CONCLUSIONS

It is expected that the focal size for a SKIF is two or more orders of magnitude smaller than that for VEPP-4 synchrotron source in the high-energy range, but this advantage is reduced for a particularly small transverse size of the x-ray beam.

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