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Large-distance refocusing of a submicrometre beam from an X-ray waveguide

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Among the several available X-ray optics for synchrotron radiation producing micrometre and submicrometre beams with high intensity, the X-ray waveguide (WG) can provide the smallest hard X-ray beam in one direction. A drawback of this optics is that, owing to the divergence at the exit, a nanometre-sized spot on the sample can only be obtained if this is within a few micrometres of the WG exit. Another limitation is that in planar WGs the beam is compressed in only one direction. Here, using a dynamically bent elliptical Si/Pt mirror, the guided X-ray beam has been refocused at ~ 1 m from the waveguide exit. The large working distance between the device and the submicrometre focus leaves some space for sample environment (vacuum chamber, furnace, cryostat, magnets, high-pressure device *etc.*) and allows cross-coupled geometries with two WGs for efficient compression in two directions.

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1. Introduction

X-ray optical elements able to focus synchrotron radiation beams down to submicrometre size are gaining more and more importance owing to the widespread potential applications in microdiffraction, microimaging, microfluorescence etc. Among these, waveguides (WG) for hard X-rays can achieve the smallest beam size [a few tens of nanometres full width at half-maximum (FWHM)] with good efficiency. The first attempt to fabricate and study waveguides for hard X-rays was made in 1974 by Spiller & Segmüller (1974), but only at the beginning of 1995 was it experimentally demonstrated that an X-ray beam could exit from the end face of a waveguide with submicrometre size in one direction (Feng et al., 1995; Lagomarsino et al., 1996; Jark et al., 1996). Since then, great improvements in efficiency have been achieved (Jark et al., 2001) and applications in different fields have been demonstrated (Di Fonzo et al., 2000; Zwanenburg et al., 1999; Lagomarsino et al., 1997; Cedola et al., 2003; Müller et al., 2000). A drawback of the WG is that the minimum size of the beam is achieved at the exit of the waveguide. The divergence of the beam (~1 mrad) causes a broadening of about 100 nm every 100 μ m of free-space propagation after the end of the WG. The fact that the beam is compressed only in one direction is another characteristic of the WG, which in some applications (e.g. microimaging) is a serious drawback. In principle, a cross-coupled geometry similar to that of the Kirkpatrick-Baez reflecting mirrors (i.e. two waveguides at 90° to each other) could be adopted. However, the physical dimensions of the waveguides and the broadening effect in free-space propagation described above make this solution unpractical. A twodimensional WG has been fabricated and tested (Pfeiffer et al., 2002), based on the microfabrication principle, but its efficiency is quite low. If, however, the beam provided by a WG could be efficiently refocused at some distance from the WG exit, both drawbacks mentioned above could be overcome. The scope of this paper is the experimental demonstration that refocusing of the beam generated by an X-ray WG in a submicrometre spot is in fact feasible.

2. Experiment

Keywords: X-ray waveguides; submicrometre beam; elliptical mirror.

We used for this purpose an elliptical mirror provided by the optics group of the ESRF. The experiment was carried out at the optics beamline BM5 of the ESRF. Fig. 1 shows a schematic view of the experimental arrangement. The beam from the bending-magnet source was monochromated by a double-Bragg reflection Si(111) monochromator. The WG consisted of a \sim 130 nm-thick C film cladded in between two Cr layers, one (20 nm-thick) on the substrate side and the other (3 nm-thick) on the free-space side. The WG, with its surface lying in the vertical plane, was put on the first tower of the



Schematic view of the experimental set-up.

BM5 main diffractometer and adjusted to its first resonance mode TE₀ (Jark et al., 1996). A pair of slits at about 110 mm from the WG allowed passage of the guided beam but cut the direct and the reflected beams (Jark et al., 1996). The mirror was put on the second tower with its center at a distance $L_{\rm M}$ of ~450 mm from the WG exit. The mirror consisted of a Pt-coated Si substrate, reflecting the beam from the WG by total external reflection with a nominal grazing angle of 4 mrad. An elliptical shape was given to the substrate in order to focus the exit beam from the WG in a 1:1 geometry. Under these conditions the WG-mirror distance $L_{\rm M}$ is equal to the mirror-focus distance $L_{\rm F}$, and a theoretical magnification of 1 is achieved. The length of the mirror was 170 mm, so that it could accept the whole diverging beam exiting from the WG. The shape was given to the mirror before the experiment with a dynamical bender (Ziegler et al., 2001; Hignette et al., 2001). The deviation from the stigmatic ellipse in terms of slope errors, measured with a long trace profiler, was of the order of 2 µrad. A scintillator detector was then put on the detector arm of the second tower in order to measure the refocused beam intensity. In front of the detector, a knife-edge controlled by a piezo actuator was used to measure the spot size. The knife-edge was a Ta foil with an edge carefully polished to about 100 nm roughness. The minimum step used in the experiment for the knife-edge translation was 125 nm. The mirror-knife-edge distance $L_{\rm KE}$ could be varied over a large range. For perfect 1:1 focusing, L_F should be equal to L_M . However, small misalignments can vary the focal distance $L_{\rm F}$, and at the same time can deform or broaden the focused spot. Therefore, keeping the mirror profile fixed, we attempted to optimize the mirror alignment acting on two parameters: the incidence angle θ_{I} and the mirror position y_M transverse to the incident beam direction (see Fig. 1). In order to find the focus position (i.e. the minimum spot size), for any couple of values θ_{I} and y_{M} we carried out knife-edge scans for a number of different $L_{\rm KE}$ values.

3. Results

Fig. 2 shows five beam profiles (closed points) for the θ_I and y_M couple which gave the best results in terms of spot size. The profiles were obtained by differentiating knife-edge scans after proper smoothing. The best scan in Fig. 2 has a FWHM of 0.85 µm. Although this is already quite a good result, we tried to understand the reasons why the refocused beam has a dimension significantly higher than the beam at the exit of the WG (about 65 nm FWHM). To this purpose a



Figure 2

Beam intensity profiles as obtained by derivation of knife-edge scans for five different values of the mirror-knife-edge distance L_{KE} (closed points). The variation ΔL_{KE} with respect to a reference value coincident with the focal distance L_F is marked on the different profiles. For each profile the abscissa indicates the knife-edge translation. The full line represents calculations (see text).

ray-tracing code was written which allows simulation of the elliptical mirror properties and that can take into account possible misalignments, slope errors and experimental conditions. The ray-tracing code considers the beam exiting from the WG exit as a Gaussian beam; the mirror is initially taken as an ideal elliptical surface with reflectivity $R(\theta)$, where θ is the angle of incidence. The mirror slope error is taken into account as a parabolic deviation of the real surface from the ideal one. The knife-edge is schematized as a perfect screen, with zero transmission for positions intercepting the beam, and a step size corresponding to that of the experiment. In Fig. 2 the results of the simulations for the five scans are reported, assuming a perfect alignment but a small slope error of 2 µrad. (*i.e.* the value found by the long trace profiler). Note the double peak present both in the theoretical and experimental scans for smaller distances L_{KE} . It is also worth noting that, by considering only the mirror misalignment without slope error, we were not able to simulate the behaviour of the experimental curves as a function of the $L_{\rm KE}$ distance.

4. Discussion

In the case considered here the refocused distance was quite high, of the order of 1 m. Under these conditions any small slope error introduces a significant broadening of the refocused spot size. The reason we adopted this distance was purely due to the experimental set-up and not for intrinsic reasons. Much smaller distances (and also much shorter mirrors) can be used in a more compact set-up. Simple calculations show for example that at a WG–mirror distance of 100 mm the entire beam from the WG could be intercepted by a mirror as short as 18 mm. A shorter mirror length makes the mirror preparation easier, and a shorter mirror-focus distance causes less spatial broadening for a given slope error.

It is worthwhile comparing the proposed WG-mirror combination with the up-to-date Kirkpatrick-Baez (KB) mirrors. In fact, deep submicrometre spot sizes have been obtained with KB mirrors (Hignette et al., 2001; Mori et al., 2002) and therefore one could ask about the advantages of refocusing the WG exit beam. The main point is that with KB mirrors the spot size depends on the source demagnification factor, and therefore on the ratio between the source-optics distance and the optics-focus distance. This can impose serious limits on the minimum spot size achievable. For example, with a standard synchrotron radiation beamline of length 40 m and with a source dimension of 80 µm, in order to achieve a 0.05 µm spot size the demagnification ratio must be 6.25×10^{-4} , and the mirror-edge-focus distance should be as small as \sim 7 mm. This short mirror-focus distance would give no space for another focusing element (as in KB geometry) in the orthogonal direction. Obviously with exceptionally long beamlines the mirror-focus distance could be significantly improved and this geometrical limit could be overcome. Another concern is the limited angular acceptance of the WG. In fact the WG we used for this experiment had an angular acceptance of the order of 3 µrad (Jark & Di Fonzo, 2004). However, in the case considered above, a mirror would have a comparable small angular acceptance. In fact, as we have demonstrated recently (Bukreeva et al., 2004), an elliptical mirror efficiently accepts a maximal angular range Φ given approximately by $\Phi \simeq (5/6)\theta_c M$, where θ_c is the critical angle of the mirror and M is the magnification factor which reduces the source to the desired spot dimension. In the example mentioned earlier, *i.e.* a Pt-coated Si mirror and a magnification ratio of 6.25×10^{-4} , the mirror acceptance Φ would be about 3 $\mu rad,$ a value comparable with the WG angular acceptance. Comparison between the WG-mirror combination and just mirrors can also be made in terms of the gain, *i.e.* the ratio between the exit flux density and the input flux density. For a WG with resonant beam coupling the best measured gain value is of the order of 100, for conditions similar to those described in the present experiment; for an ideal WG the gain can be about three times higher (Jark et al., 2001). The corresponding mirror with optimum length would instead have a gain of the order of 1000 (Bukreeva et al., 2004). The advantage of the WG-mirror combination is that with the WG the beam dimension is defined only by the thickness of the guiding layer and not by the source dimension and the demagnification ratio. Therefore spot sizes in the 100 nm range or below could be reached virtually at any beamline, without the need of a large source-mirror distance, and even with table-top laboratory X-ray sources, as recently demonstrated (Pelliccia et al., 2005). This would be absolutely impossible with just mirrors. Concerning the minimum spot size achievable, the limits are given by geometrical aberrations and by diffraction. The former can now be kept very low, especially in short mirrors, with advanced fabrication methods (Mori et al., 2002). Diffraction limits can also be kept in the range 10-20 nm with the proper choice of experimental conditions. Therefore, spot sizes below 100 nm with the refocusing scheme described here become realistic.

This could open the way for new kinds of applications with WGs in many fields: the large working distance between device and focus would leave some space for sample environment (vacuum chamber, furnace, cryostat, magnets, high-pressure devices *etc.*). In microfluorescence the signal-to-noise ratio should be dramatically improved by placing the sample far away from the WG end. Moreover, cross-coupled geometries with two WGs could be adopted for efficient compression in two directions. Another opportunity offered by WGs is coherence: the beam from the WG is fully coherent, and if the mirror is perfect enough the coherence is preserved in the refocused beam, allowing phase-contrast microscopy with very high spatial resolution. Finally, the sensitivity of the refocused spot profile to tiny slope errors could even suggest the use of a WG beam as a fine *in situ* mirror profiler. We gratefully acknowledge Olivier Hignette and Amparo Rommeveaux (Metrology Laboratory, Optics Group) for providing the mirror and bending it to the correct shape before the experiment. We acknowledge partial support from the CNR project 'Luce di Sincrotrone'.

References

- Bukreeva, I., Dabagov, S. & Lagomarsino, S. (2004). Appl. Opt. 43, 6270-6277.
- Cedola, A., Stanic, V., Burghammer, M., Lagomarsino, S., Rustichelli, F., Giardino, R., Nicoli Aldini, N. & Di Fonzo, S. (2003). *Phys. Med. Biol.* 48, N37–48.
- Di Fonzo, S., Jark, W., Lagomarsino, S., Giannini, C., De Caro, L., Cedola, A. & Müller, M. (2000). *Nature (London)*, **403**, 638–640.
- Feng, Y. P., Sinha, S. K., Fullerton, E. E., Grübel, G., Abernathy, D., Siddons, D. P. & Hastings, J. B. (1995). Appl. Phys. Lett. 67, 3647–3649.
- Hignette, O., Rostaing, G., Cloetens, P., Rommeveaux, A., Ludwig, W. & Freund, A. (2001). *Proc. SPIE*, **4499**, 105–116.
- Jark, W., Cedola, A., Di Fonzo, S., Fiordelisi, M., Lagomarsino, S., Kovalenko, N. V. & Chernov, V. A. (2001). *Appl. Phys. Lett.* 78, 1192–1194.
- Jark, W. & Di Fonzo, S. (2004). J. Synchrotron Rad. 11, 386-392.
- Jark, W., Di Fonzo, S., Lagomarsino, S., Cedola, A., Di Fabrizio, E., Brahm, A. & Riekel, C. (1996). J. Appl. Phys. 80, 4831–4836.
- Lagomarsino, S., Cedola, A., Cloetens, P., Di Fonzo, S., Jark, W., Soullié, G. & Riekel, C. (1997). Appl. Phys. Lett. 71, 2557–2559.
- Lagomarsino, S., Jark, W., Di Fonzo, S., Cedola, A., Müller, B. R., Riekel, C. & Engstrom, P. (1996). J. Appl. Phys. **79**, 4471–4473.
- Mori, Y., Yamauchi, K., Yamamura, K., Mimura, H., Sano, Y., Saito, A., Ueno, K., Endo, K. Souvorov, A. Yabashi, M., Tamasaku, K. & Ishikawa, (2002). *Proc. SPIE*, **4782**, 58–64.
- Müller, M., Burghammer, M., Flot, D., Riekel, C., Morawe, C., Murphy, B. & Cedola A. (2000). J. Appl. Cryst. 33, 1231–1240.
- Pelliccia, D., Cedola, A., Scarinci, F. & Lagomarsino S. (2005). J. Phys. D, 38, A213–A217.
- Pfeiffer, F., David, C., Burghammer, M., Riekel, C. & Salditt, T. (2002). *Science*, **12**, 230–234.
- Spiller, E. & Segmüller, A. (1974). Appl. Phys. Lett. 24, 60-61.
- Ziegler, E., Hignette, O. & Morawe, C. (2001). Nucl. Instrum. Methods, A467, 954–957.
- Zwanenburg, M. J., Peters, J. F., Bongaerts, J. H. H., De Vries, S. A., Abernathy, D. L. & van der Veen, J. F. (1999). *Phys. Rev. Lett.* 82, 1696–1699.