Determining microfocused x-ray beam size by x-ray fluorescence correlation spectroscopy

Jin Wang, Parlapalli V. Satyam, Oliver H. Seeck, Zhonghou Cai, and Sunil K. Sinha
Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439 USA

Introduction

The advent of third-generation synchrotron x-ray radiation sources promises x-ray beams possessing high brilliance, low emittance, and more importantly, larger coherent volumes in the beam. In turn, these properties allow the use of Fresnel zone plates to focus x-rays to a beam spot well below 1 μm in the hard x-ray region [1]. Recently, it has been demonstrated that x-ray fluorescence correlation spectroscopy (XFCS), which utilized microfocused x-ray beams, is a powerful method to study particle dynamics in colloidal suspensions [2]. To optimally use the method, one of the requirements is that the x-ray illuminating volume in the samples is small. This requirement has been satisfied by using an x-ray Fresnel zone plate to focus the incident x-ray ray beam to submicron size. For determining the size of a microfocused x-ray beam at the sample position, the most commonly used method is performing positional scans with a sharp knife edge while monitoring either the transmission or fluorescence intensity. However, the results from such measurement are the convolution of the beam size and vibration in the experimental setup. Therefore, so-determined beam size at the focal point has usually been significantly larger than the designed values, especially for zone plates with a submicron theoretical focused beam size. In this report, we outline a novel method utilizing XFCS for precisely determining the characteristics of the vibration, as well as the actual x-ray beam size by making use of the dynamic nature of the XFCS technique.

X-ray fluorescence is simply proportional to the product of the number of fluorescent atoms in the x-ray beam and the x-ray exciting field intensity. While a knife edge intercepts an x-ray beam, relative motion (vibration) causes fluctuation in the x-ray fluorescence intensity. The fluorescence autocorrelation function, $g_f(t) = \langle I_f(0)I_f(t)\rangle / \langle I_f(0)\rangle^2$, yields direct information about the vibration, where, for small and simple harmonic oscillation, the time-dependent fluorescence intensity can be expressed by $I_f(t) = I_0[1 + f\cos(\omega t)]$, and, where ω is the angular frequency of the vibration, I_0 is the amplitude of the static or averaged fluorescence intensity, and f (<1) is the normalized vibration amplitude by the beam size. Generally, the vibration frequency has a small distribution, σ, around ω. If $\sigma << \omega$ is satisfied, the correlation function can be expressed as:

$$g_f(t) = 1 + \frac{f^2}{2}\cos(\omega t)\exp(-\sigma^2 t^2/2). \qquad (1)$$

Therefore, the intercept of the correlation function, $g_f(0)$, should not exceed 1.5. For practical reasons, we always use the reduced correlation function, $g_f(t) - 1$ in the following calculation, presentation, and discussion.

Methods and Materials

The zone plate used in the XFCS study was designed for focusing a coherent x-ray beam (8 keV photon energy) to about 0.25 μm in the vertical direction with a focal length of 50 mm. To determine the focused beam size, a knife edge consisting of a broken metal thin film, namely 200 Å-thick Cr deposited on a polished silicon wafer, was scanned through the beam, while the Cr Kα fluorescence was monitored. Such a metal film has an edge with sharpness better than 100 Å over a length of a few micrometers examined by electron microscope. Therefore, the accuracy of the measured beam size would be on the same order. However, the so-determined beam size at the focal point has usually been significantly larger than the designed values due to mechanical vibration.

The experiment was carried out at the 2-ID-D undulator beamline of the Advance Photon Source (APS). The schematic of the setup is shown in Figure 1. Monochromatic beam at 7.5 keV was focused by a zone plate with a diameter of 50 mm, a focal length of 47 mm, and an efficiency of 30% at this photon energy. The beam size was first measured with Cr knife edge scans through the beam. The scans were done at different positions close to and away from the focal point of the zone plate. Then, XFCS measurements were performed at each position on the zone plate axis where the fluorescence fluctuation caused by vibration is the greatest; in turn, the XFCS data possess the highest sensitivity to the vibration-induced fluorescence fluctuation.

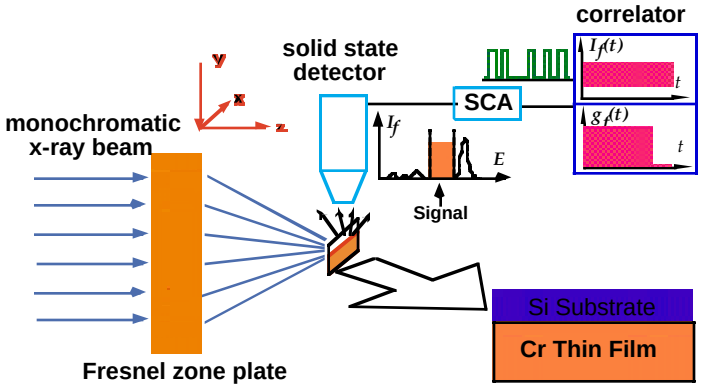

Figure 1: Schematic of the experimental setup. A chromium knife edge (shown in the magnified view) was used to scan the microfocused beam while Cr Kα fluorescence was monitored by an energy-dispersive detector and fed to a correlator. The coordinate system is also defined in the figure.

Results

To prepare the correlation measurement, the initial scans were performed in a conventional fashion. The apparent beam size at different locations on the zone plate axis near the focal point (from both sides) was measured by vertical scans using the Cr knife edge. Scans at 52 and 48 mm from the zone plate are shown in Figure 2. The data were fit with models assuming that the beam cross section has a Gaussian intensity distribution or a uniformly illuminated circular disk. The fit with the latter model yields a much lower χ^2 and is shown in the figure. The effect of the relative vibration between the sample stage and zone plate smeared the definition of the beam spot. This can be observed more obviously at the position 1 mm within the focal point (data taken at z = 48 mm). With this model, the apparent beam size was then characterized by the radius of the disk, $r(z)$, while z is the distance from the zone plate. Furthermore, the measured beam spot radius as a function of the measurement location is presented as an inset in Figure 2B.

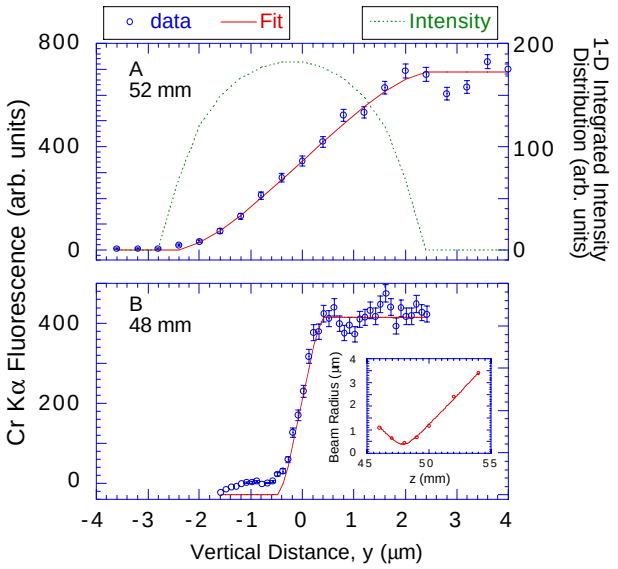

Figure 2: The experiment (circles) and fit (line) Cr Kα fluorescence yield during the knife edge vertical scans at z = 52 mm (A) and 48 mm (B). In A, the 1-D integrated intensity of the beam spot is shown (broken line). Also shown as an inset in B is the apparent beam radius at the different locations (circles) and a simple fit (line) described in the text.

The data can be quantitatively fit by the convolution of a fixed focused spot size and a linearly diverging spot size as $r(z) = \sqrt{r_0^2 + \left[\alpha(z - z_0)\right]^2}$, where r_0 is the apparent focused beam radius at z_0, and α is the divergence if the beam. To determine the true beam size, the autocorrelation of Cr fluorescence was measured while the knife edge was set on the zone plate axis (the half-height point in the scans in Figure 2).

The correlation functions and the fits to data with Eq. 1 are shown in Figure 3. The data were fit by a vibration model with a frequency, $\omega = 122\ s^{-1}$, and $\sigma = 6.6\ s^{-1}$. The correlation functions can be measured accurately to $[g_f(t) - 1] < 10^{-3}$. The fit using a fixed vibration amplitude matches the experimental measurement extremely well. The intercept of the correlation functions increases as the measurement was taken closer to the focal point of the zone plate. It is noted that, at z = 48 mm, the intercept approaches 0.5. This indicates that the vibration amplitude had exceeded the true beam spot size. Therefore, at this point, the fit would not reliably yield the vibration amplitude. Measurements farther from the focal point would be necessary. In turn, the determination of the intrinsic focal beam size requires XFCS spectra at a series of axial locations. Here, the vibration amplitude was determined to be 0.59 μm, and by deconvoluting the vibration from the apparent beam size shown in the inset to Figure 2, the intrinsic beam size at the focal point is found to be 0.32 μm (radius).

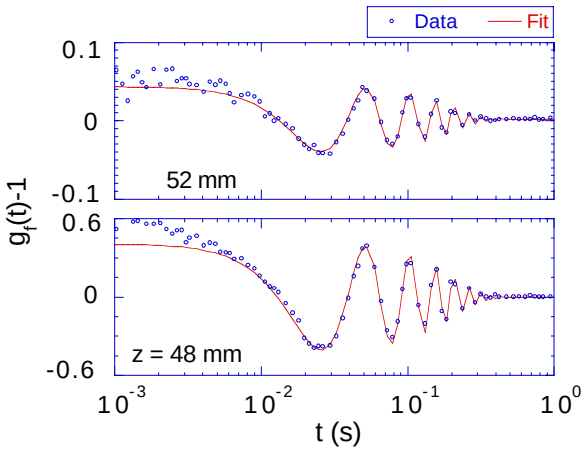

Figure 3: The experimental (circles) and theoretical (line) fluorescence autocorrelation functions (reduced), $g_f(t) - 1$, at different distances to the zone plate. Note the different y scales.

Discussion

What distinguishes this beam size determination technique from convensional ones is the capability of separating the dynamic and static contributions to the measured size. Mechanical vibrations with submicron amplitude, which are associated with optical setups, have not been able to critically affect the performance of either visible or x-ray optics due to the fact that the focused beam size has well exceeded the microsize. However, with the development of hard x-ray focusing devices and the perfection of x-ray sources, the focused beam size has recently been reduced to submicron size. Therefore, reducing the vibrations on nanometer scale starts to play an important role in the process of improving the performance of the x-ray microfocusing elements. On the other hand, the availability of such devices and the XFCS method provide a means to characterize the fast, nanosized vibrations for the first time. With the sensitivity afforded by the method, vibrations with an amplitude of 3% of the beam size, which will result in a

correlation function interception of 0.005, can be detected readily. Correspondingly, with the smallest x-ray beam size available (about 20 nm in the soft x-ray region), the detectable vibrations can be extended to a few nanometers. The frequency of the vibrations can be as high as MHz after the beam intensity fluctuation due to synchrotron source structure is accurately normalized.

Acknowledgments

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