High-reflectivity Cr/Sc multilayer condenser for compact soft x-ray microscopy

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The condenser is a critical component in compact water-window x-ray microscopes as it influences the exposure time via its efficiency and the resolution via its numerical aperture. Normal-incidence multilayer mirrors can reach large geometrical collection efficiencies and match the numerical aperture of the zone plate but require advanced processing for high total reflectivity. In the present article we demonstrate large-diameter normal-incidence spherical Cr/Sc multilayer condensers with high and uniform reflectivity. Dc-magnetron sputtering was used to deposit 300 bilayers of Cr/Sc with a predetermined d-spacing matching the $\lambda=3.374$ nm operating wavelength on spherical substrates. The mirrors show a uniform reflectivity of ~3% over the full 58 mm diameter condenser area. With these mirrors an improvement in exposure time by a factor of 10 was achieved, thereby improving the performance of the compact x-ray microscope significantly. © *2006 American Institute of Physics.* [DOI: 10.1063/1.2400665]

I. INTRODUCTION

Soft x-ray microscopy in the water-window region ($\lambda=2.3-4.4$ nm; approximately 0.3–0.5 keV) is an attractive technique for natural-contrast, high-resolution imaging of thick organic material.[1] We have developed the first compact water-window microscope with subvisible resolution.[2] The condenser is a critical component in such microscopes since it strongly influences both exposure time and resolution. In the present article we describe a normal-incidence spherical Cr/Sc multilayer condenser which allows ten times improvement in exposure time compared to previous systems.

Most of the present operational x-ray microscopes in the water window are based on synchrotron radiation sources.[1] Due to the high brightness of this source the requirements on the efficiency of the optics can be slightly relaxed while still obtaining acceptable exposure times. Condenser concepts include large-diameter zone plates[3] and rotating condensers for undulator beams.[4] Compact microscopes show promise for use in the small-to-medium-scale application laboratory, thereby increasing the impact of x-ray microscopy. Intrinsically such microscopes rely on much weaker and primarily incoherent sources, resulting in different and increased requirements on the x-ray optics. Thus, the condenser is a critical component. For the water-window elliptical mirrors have been combined with discharge sources at $\lambda=2.48$ nm.[5] Normal-incidence multilayer mirrors[2–6] and Wolter-type mirrors[7] have been employed with laser-plasma sources at $\lambda=3.37$ nm. Diffractive optics have been attempted with both types of sources.[8,9] The normal-incidence multilayer condenser offers several advantages, including high collection efficiency, well-defined spectral selectivity, high numerical aperture (NA), and ease of alignment, that are described in more detail in Sec. II. The challenges include uniformity, low roughness and intermixing, and short and accurate d-spacing.

Multilayer deposition technology for extreme ultraviolet (EUV) mirrors (Mo/Si) is currently being pushed to unprecedented accuracy, now reaching 70% reflectivity at 13.5 nm.[10] During the past five years short-d-spacing normal-incidence water-window mirrors have shown significant progress, increasing the normal-incidence reflectivity from a few percent to tens of percent.[11] W/B_4C mirrors have demonstrated a local reflectivity of at least 1.9% at 3.4 nm.[6] Cr/Sc mirrors were pioneered by Salashchenko and Shamov[12] Kuhlmann *et al.* showed that the normal-incidence reflectivity of dc-magnetron-sputtered Cr/Sc multilayer mirrors can be considerably improved by the application of a suitable bias voltage during the growth process,[13] resulting in a reflectivity of 14.8% close to the Sc edge.[14] Simultaneously Eriksson *et al.* used dc-magnetron sputtering with tailored ion assistance to achieve 5.5% at the $\lambda=3.37$ nm line that is of importance for microscopy and 14.5% at the Sc edge at $\lambda=3.11$ nm.[15] For even shorter wavelengths the requirements on roughness and intermixing become extremely demanding. Recently, Gullikson *et al.* included barrier layers and demonstrated near-normal-incidence reflectivities of 17% at $\lambda=2.73$ nm (Cr/Ti) and 9% at $\lambda=2.42$ nm (Cr/V), as well as 32% at $\lambda=3.11$ nm (Cr/Sc).[16] Although impressive, the above numbers were reached on small substrates. Useful normal-incidence condensers require uniform deposi-

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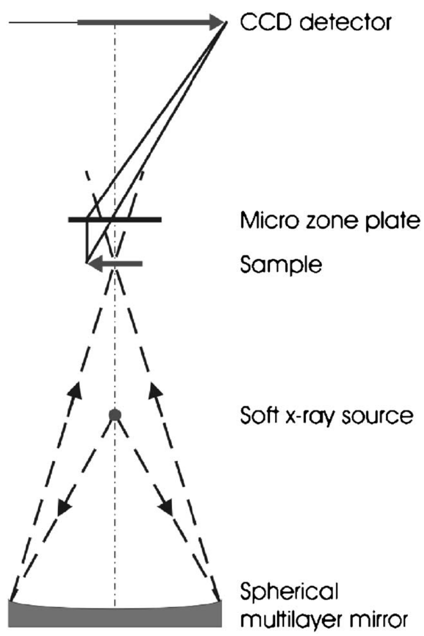

FIG. 1. Experimental arrangement of the compact x-ray microscope with a normal-incidence multilayer condenser mirror.

tion over large areas with a predetermined, accurate, and reproducible d-spacing in order to achieve high collection efficiency. This is a difficult fabrication problem.

In the present article we describe the fabrication of large-diameter normal-incidence Cr/Sc spherical multilayer condenser mirrors with high and uniform average reflectivity (3%) for a selected spectral emission line ($\lambda = 3.374$ nm) of a laser-plasma source. The condenser properties are demonstrated with a liquid-methanol-jet laser-plasma target in our compact x-ray microscope, showing the improvement in exposure time by an order of magnitude.

II. MULTILAYER CONDENSERS FOR COMPACT MICROSCOPY

The requirements on the multilayer condenser are determined by the experimental arrangement of the compact x-ray microscope. We therefore start by giving a brief introduction to this system.[2–7] Figure 1 shows the compact full-field x-ray microscope. The high-resolution imaging is performed with an $f = 0.5$ mm nickel zone plate with an outermost zone width of 30 nm.[18] The source is a 100 Hz, negligible-debris, high-brightness methanol liquid-jet laser plasma.[19] We operate at the strong $\lambda = 3.374$ nm carbon line ($1s-2p$ in CVI) which has a narrow linewidth ($\lambda/\Delta\lambda \approx 500$).[20] The use of a single narrow-linewidth line is necessary in order to avoid chromatic aberrations in the zone-plate imaging. The source is combined with a 58 mm diameter, 343 mm radius of curvature W/B_4C spherical normal-incidence multilayer condenser. The mirror has an average reflectivity of 0.3%–0.5% and a spectral bandwidth of $\lambda/\Delta\lambda \approx 80$.[6] A back-illuminated 13×13 μm^2 charge coupled device (CCD) camera detects the image at $1000\times$ magnification.

The condenser is a critical component in the microscope. Here normal-incidence spherical multilayer mirrors offer significant advantages compared to diffractive optics. The NA can easily be matched to the NA of the zone-plate optics, which is important for resolution. For comparison we note that with the typical 1:1 imaging of diffractive-optics condensers this is very difficult, since the outermost zone width of the large-diameter condenser in this case has to be fabricated with an outermost zone width equal to half of the micro-zone-plate's. The large NA of the multilayer condenser also results in a high geometrical collection efficiency, resulting in shorter exposure times. In addition, the limited bandwidth of multilayer mirrors offers spectral selectivity of the source spectrum, thereby avoiding blurring by other source emission lines in the subsequent highly chromatic micro-zone-plate imaging. Finally, the alignment of the microscope is easy since the spherical mirror reflects also at visible wavelengths. Compared to elliptical-mirror condensers used at grazing incidence, which may also offer large collection efficiencies and NA, the multilayer condenser has a significant advantage related to its spectral selectivity and the much simpler alignment. The alignment of elliptical mirrors with small high-brightness sources is notoriously difficult due to the significant aberrations of such mirrors. However, the demands on the multilayer fabrication are severe. Interdiffusion and roughness must be minimized for high reflectivity at these short d-spacings. The uniformity of the d-spacing must be very high over the full area of the mirror. In addition, these microscopes are operated with narrow-linewidth laser-plasma sources, where the emission wavelength is determined by an atomic transition. Thus, the d-spacing must not only be uniform but also match the predetermined emission line over the full mirror area. In the next section we describe the fabrication of large-diameter uniform spherical multilayer mirrors with high normal-incidence reflectivity.

III. MULTILAYER CONDENSER FABRICATION

The normal-incidence multilayer condensers were fabricated on fused silica substrates having a diameter of 58 mm and a radius of curvature of 350 mm. In the microscope the mirror images the $\lambda = 3.374$ nm laser-plasma source onto the sample plane (Fig. 1) with a 282 mm source-condenser distance and 462 mm condenser-sample distance. Thus, the NA is 0.063, which matches the NA of a 27 nm outer zone width zone plate. The main challenge of the multilayer deposition is to simultaneously achieve a high and uniform reflectivity at the correct spectral position (the $\lambda = 3.374$ nm line with $\lambda/\Delta\lambda \approx 500$) over the full working aperture.

From the Introduction it is clear that the Cr/Sc material combination has suitable optical and technological properties for high-performance mirrors in the water window.[14,15] Our model calculations[21] show that at $\lambda = 3.374$ nm, Cr/Sc has a calculated maximum reflectivity of 42.8%, compared to 9.5% of W/B_4C multilayer systems used in the present x-ray microscope arrangement. Thus, a Cr/Sc coating was chosen for the $\lambda = 3.374$ nm condenser mirrors.

The number of periods N necessary to obtain the highest possible reflectivity of Cr/Sc multilayer mirrors depends on the wavelength and the angle of incidence. In general, the reflectivity increases with increasing number of periods N, but on the other hand, the reflectivity may decrease for very

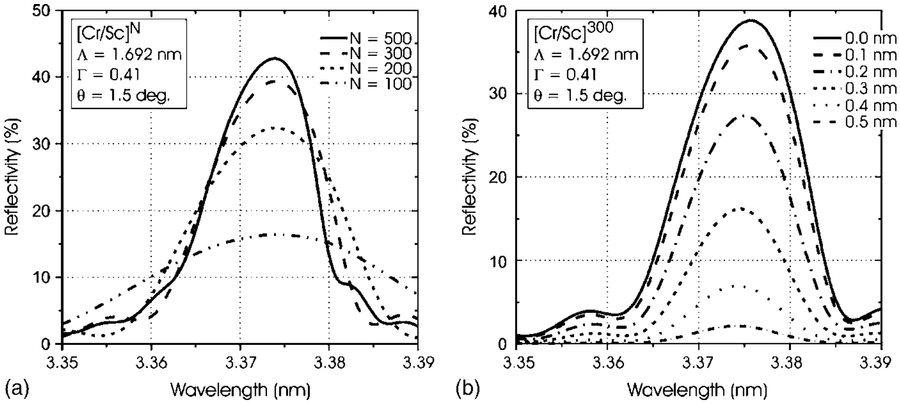

FIG. 2. Calculated reflectivity for Cr/Sc multilayers with a multilayer period of $\Lambda=1.692$ nm and a ratio of Cr thickness to total bilayer thickness $\Gamma=0.41$, optimized for an incident angle of 1.5°. (a) Reflectivity of the Cr/Sc multilayers depending on the number of bilayers. (b) Reflectivity for a stack of 300 Cr/Sc bilayers depending on the roughness.

large N since the interface quality may deteriorate with increasing number of layers. The optimum number of periods is determined by a compromise between the number of reflecting interfaces, required spectral bandpass, and sufficient interface quality.[14] Figure 2(a) shows a calculation for Cr/Sc multilayers, optimized for an incident angle of 1.5° and $\lambda=3.374$ nm. The ratio of Cr thickness to total bilayer thickness Γ is taken to be 0.41, since this ratio has been found to produce minimum roughness. In the present work Cr/Sc multilayers with $N=300$ bilayers were chosen as a compromise between high reflectivity ($R_{\mathrm{theor,max}}=39\%$) and a sufficient spectral bandpass [full width at half maximum (FWHM)=0.014 nm] to accommodate for minor variation in d-spacing when the mirror is used with the fixed-wavelength line source.

The Cr/Sc multilayers were deposited by dc-magnetron sputtering using an industrial sputtering system Kenotec MRC 903. This system is equipped with three rectangular magnetrons with a size of 120×360 mm^2. The Cr and Sc targets were operated at a power of 150 W. The system base pressure is $\sim1\times10^{-7}$ mbar which is achieved by a cryopump. The deposition is performed under a low-pressure argon atmosphere of 1×10^{-3} mbar. During the deposition process the axial-revolving substrates ($\Omega=500$ rotations/min) are moved beyond the targets and the film thickness scales with the inverse velocity. A high accuracy speed control ensures a good reproducibility of the film thickness even at high speeds, and the load/unload system provides the thickness repeatability on the level of 0.005 nm.

An important aspect is the requirement for very smooth interfaces to obtain a high reflectivity. The effect of the interface roughness on the reflectivity grows in importance with decreasing film thickness. Therefore the challenge in the fabrication of highly reflective Cr/Sc multilayer structures is the task to deposit a large number of layers with extremely small thickness and low interface roughness. Figure 2(b) illustrates the point by showing the calculated reflectivity of the $N=300$, $\Gamma=0.41$ Cr/Sc mirror as a function of roughness. Improvements in the deposition technology for reduced roughness, especially an optimization of the bias voltage, have been presented in a previous paper.[15] Also Cr and Sc targets with high density and a low Ar gas working pressure during the sputtering process were used to improve the performance of Cr/Sc multilayer mirrors.[16]

Due to the strong influence of initial substrate roughness on interface roughness inside Cr/Sc multilayer structures, the surface roughness of the substrates were investigated by atomic force microscopy. A NanoScope III, Dimension 3000 (Digital Instruments) was operated in the tapping mode with <10 nm radii silicon tips. To analyze the microstructure of the substrates, the atomic force microscopy (AFM) topographic images were recorded over scan areas of 1×1 μm^2 and 10×10 μm^2 with a resolution of 512×512 data points. The comparative investigation of the surface morphology by atomic force microscopy shows surface roughness of about 0.06–0.20 nm for the substrates.

In order to obtain a uniform high reflectivity on the spherical substrates, accurate control of the Cr/Sc lateral thickness period distribution was necessary since the bandwidth of the multilayers was extremely narrow ($\Delta\lambda\sim0.01$ nm). Furthermore, the required thickness period depends on the incidence angle, which changes along the surface from 0.57° to 1.47°. The desired lateral period distribution of Cr/Sc multilayers along the surface was achieved by using an appropriately designed mask. In our case the spherical substrate was spun by $\Omega=500$ rotations/min around its axis and the fixed mask was positioned above the rotating substrate. Figure 3 shows the comparison between the calculated lateral thickness distribution[21] and the best ex-

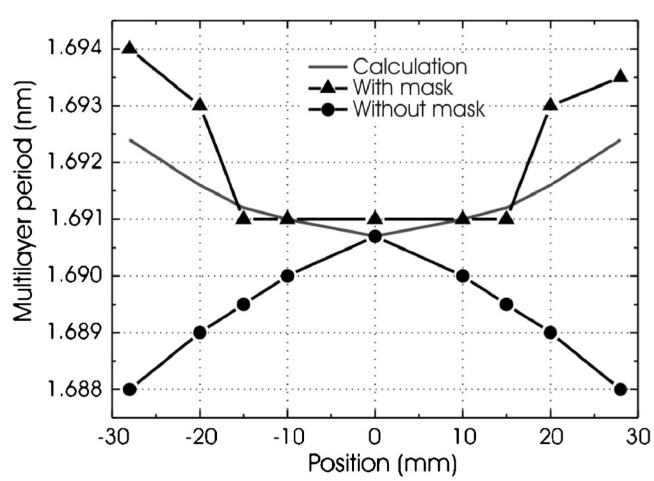

FIG. 3. Comparison between the calculated lateral thickness distribution using a mask during the deposition process and the best experimentally achieved one (measured by the Bruker diffractometer). To illustrate the effect of the mask the thickness distribution without the mask is shown.

TABLE I. Results of the hard and soft x-ray measurements for three Cr/Sc multilayer mirrors. Hard x-ray measurements [small angle x-ray reflection (SAXR) measurements] were performed using a Bruker D5005 diffractometer operated with Cu $K\alpha$ radiation (λ=1.54 nm). Soft x-ray measurements were done at the PTB reflectometer at BESSY II.

Mirror	Measured period (by SAXR) (nm)	Predicted wavelength (nm)	Reflectivity at λ=3.374 nm (average, %)	Uniformity $\frac{R_{max}-R_{min}}{R_{max}+R_{min}}$
1	1.693±0.002	3.375±0.004	2.58±0.80	0.55
2	1.693±0.002	3.375±0.004	3.14±0.10[a]	0.06
3	1.694±0.002	3.377±0.004	2.90±0.38	0.16

[a]Soft x-ray measurements are shown in Fig. 5.

perimentally achieved one. A multilayer period error less than 0.002 nm was achieved prior to real deposition of Cr/Sc multilayer condenser optics.

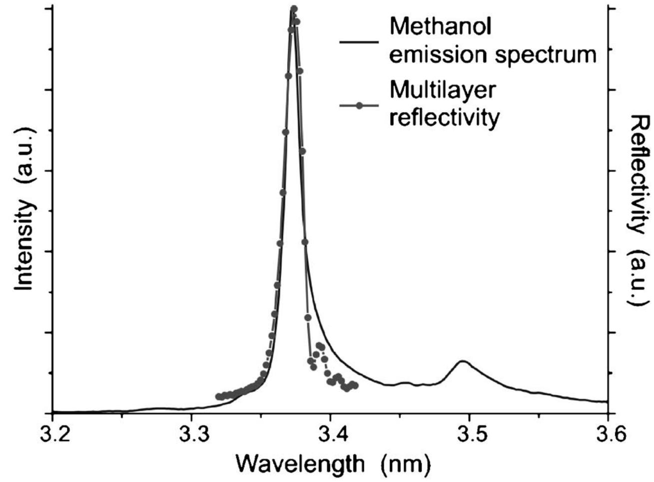

FIG. 4. Emission spectrum of the methanol-jet laser-plasma source in the water window and the reflectivity of the Cr/Sc multilayer mirror 2, matching the 3.374 nm emission line (source data from Ref. 19).

IV. RESULTS AND DISCUSSION

In this section we characterize the fabricated mirrors with respect to reflectivity, uniformity, and spectral response. Furthermore, a first image from the compact x-ray microscope equipped with one of the mirrors is shown.

A first characterization of the multilayer mirrors was performed by small angle x-ray reflection (SAXR) measurements with a Bruker D5005 diffractometer, operating with Cu $K\alpha$ radiation (λ=0.154 nm) in a symmetrical Θ-2Θ geometry. The measured bilayer period gives information about the optimum reflection wavelength. Table I summarizes the results for three mirrors. The two first columns show the measured multilayer period and the wavelength for which maximum reflectivity is predicted from the measurement. Note that the laser-plasma emission line at λ=3.374 nm is only 0.001–0.003 nm from the predicted wavelength and thus is well within the mirror bandwidth of 0.01 nm.

The absolute soft x-ray reflectivity was measured at the PTB reflectometer at BESSY II in Berlin, Germany. The wavelength was scanned between 3.32 and 3.42 nm with a resolution of 0.002 nm. The relative uncertainty of the measured reflectivity was 0.1%. The incidence angle was fixed at 1.5°, which corresponds to the maximum incidence angle in the current x-ray microscope arrangement. Figure 4 shows one example of such a wavelength scan for mirror 2. Superimposed on the mirror reflectivity profile is the emission spectrum from the laser-plasma source.[22] The data confirms the hard x-ray measurements, and it is clear that the peak reflectivity of the mirror matches the emission wavelength excellently. Note that the apparent linewidth of the source is larger than its real linewidth due to the resolution of the spectrometer used for the source measurement.

In order to determine the uniformity of the mirrors, soft x-ray reflectometer measurements were performed at nine points along a full diameter of the mirrors. Figure 5(a) shows such a measurement for mirror 2. For each of the mirrors the reflectivity in different points on the surface was determined for an incidence angle of 1.5°. This corresponds well to the 0.5°–1.5° angle of incidence used in the microscope. Calculations show that for this angular range the peak reflectivity changes <0.3% and the peak reflectivity shifts <8% of the mirror bandwidth compared to the values at 1.5°. Note that the peak reflectivity exceeds 3% on all measured positions, which compares well with previous results on small substrates[15] considering that the mask necessary for the uniformity reduces the achievable reflectivity. Furthermore, the variation in the wavelength of peak reflectivity is small which, in combination with the bandwidth, results in an average reflectivity of >3% at λ=3.374 nm. Thus, from Fig. 2(b), the average overall interface roughness is approximately 0.45 nm. Table I summarizes the results for three mirrors. In the third column the average reflectivity at the wavelength λ=3.374 nm is given and the fourth column lists the uniformity over the reflecting area, defined as $(R_{max}-R_{min})/(R_{max}+R_{min})$. To determine the performance of the full multilayer mirror, the image of the reflected radiation

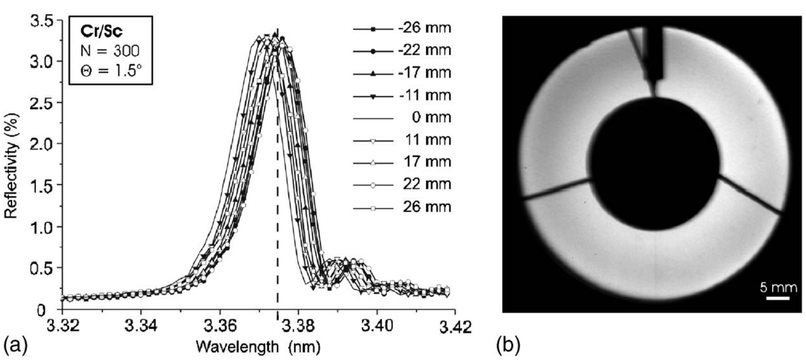

FIG. 5. Soft x-ray measurements of mirror 2. (a) Soft x-ray reflectivity for different measurement points on the mirror, measured by the PTB reflectometer at BESSY II. The position of the emission line at λ=3.374 nm is marked (dashed line). (b) Reflected x-ray radiation detected on the CCD camera. The shadows in the image are due to the nozzle and the central stop.

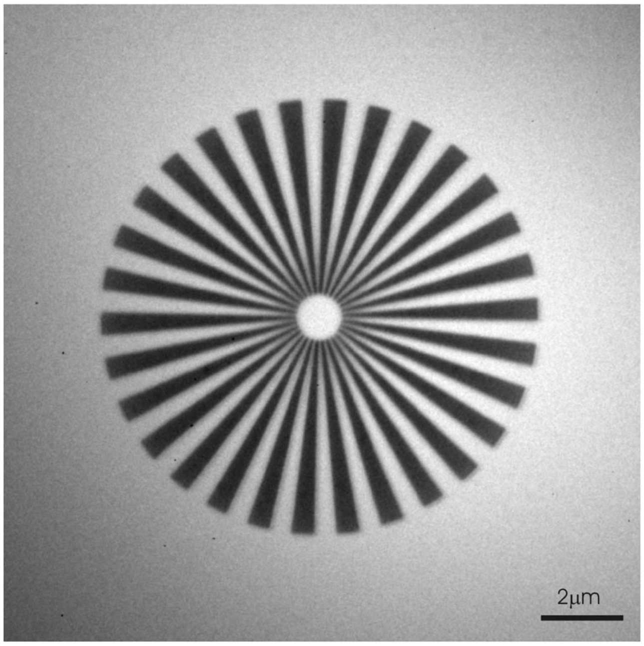

FIG. 6. Image of a Siemens star, taken by the compact x-ray microscope with 1000× magnification and a pixel element size of $26\times26\ nm^2$. The exposure time is 120 s.

from condenser mirror 2 was recorded by the CCD camera in the compact microscope illumination arrangement. The result is shown in Fig. 5(b) which demonstrates that the overall uniformity is very high. In summary, the mirrors combine excellent uniformity with high reflectivity at the correct wavelength.

We showed that Cr/Sc multilayer condenser mirrors fulfill the requirements for sufficiently high and uniform reflectivity at the methanol emission line $\lambda=3.374$ nm. In Fig. 6 the image of a test sample, a Siemens star pattern, recorded by the compact x-ray microscope with an exposure time of 2 min is shown. The resolution element in this experiment is $26\times26\ nm^2$. The signal-to-noise ratio in the image is about 22, which is high for compact soft x-ray microscopy. The number of photons in the sample plane is with this microscope arrangement about 500 photons per pixel in 30 s. This is an improvement of about ten times compared to the previous multilayer mirrors. With these high-reflectivity mirrors imaging of dry samples with subminute exposure times is possible, and also imaging of wet samples becomes practicable with exposure times of about 300 s.[23]

V. DISCUSSION

The critical issue for compact x-ray microscopes is the exposure time. Previous systems typically operated with exposure times of a few minutes for dry samples and approximately 10 min for wet samples. This is too long for a routine instrument. Unfortunately, the potential for reduction in exposure time due to increased efficiency in the CCD detector or the micro-zone-plate is small. The power of the laser-plasma source can be increased significantly but it scales linearly with laser power. Thus, a significant improvement would require an expensive new laser. Thus, the condenser was the component in the x-ray microscope with the largest potential to contribute to shorter exposure times. The tenfold increase in average reflectivity resulting in subminute exposure times demonstrated in this article illustrates the point. We note that there is room for another order of magnitude improvement in multilayer condenser efficiency since the theoretical reflectivity is more than ten times higher than that of the present condenser. On small substrates such high reflectivities have already been demonstrated.[16]

Another issue of large importance is the operating wavelength of the microscope. Operating in the lower wavelength range of the water window (e.g., at $\lambda=2.4$ nm instead of $\lambda=3.374$ nm) offers higher transmission and is therefore more appropriate for imaging thicker objects. Suitable sources exist at these wavelengths, e.g., the liquid-nitrogen-jet laser-plasma source with line emission at $\lambda=2.48$ nm.[24] High-resolution micro-zone-plates operate equally well here as for the longer wavelength. However, fabrication of large-diameter uniform multilayer condensers is significantly more difficult due to the shorter d spacing, making reflectivity losses due to roughness and intermixing more severe than at $\lambda=3.37$ nm. Encouraging attempts on small substrates[16] show promise for the future. With a stable deposition process the fabrication of multilayer condenser mirrors for compact x-ray microscopy also for this wavelength will be possible.

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