Optical setup for tabletop soft X-ray microscopy using electrical discharge sources

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Abstract. We report on an optical setup for compact full-field transmission microscopy developed for imaging with laboratory scale X-ray sources such as gas discharge plasmas. The optical setup consists of a reflective condenser and a zone plate objective imaging in the water window region (wavelength 2.3 - 4.4 nm). The microscope is able to visualize object structures in the sub-40 nm region. The radiation for the microscope is generated by a gas discharge in nitrogen with an input power of 10 kW and few Watt/(2π·sr) narrowband line emission. A gold coated axially symmetrical elliptical mirror is used to collect the radiation and forms a focused spot on the sample. Having a diameter of several 100 µm the spot is larger than the utilized imaging field, which leads to uniform illumination of the object. The mirror can be adjusted by a 5-axes piezo driven translation stage to optimize the flux in the object plane. The He-α line emission (2.88 nm) is selected of the nitrogen emission spectrum by means of a titanium thin film filter providing monochromatic sample illumination. A micro zone plate generates a magnified image detected by a back illuminated TE-cooled CCD camera. At high repetition rates the source is suitable for imaging test objects like Siemens stars, latex spheres and diatoms with magnifications of 1000 at exposure times in the range of a few seconds.

1. Introduction
Nowadays soft X-ray microscopy is a promising technique providing information of samples not easily obtainable with visible light or electron microscopy [1]. Especially biological specimen in their aqueous environment show a high natural contrast in the water window spectral region (λ = 2.3 nm to 4.4 nm) due to the high absorption differences between carbon (protein) and water. With the availability of table-top X-ray sources such as laser induced plasma [2] or gas discharge plasma [3], X-ray microscopy becomes independent of large facilities like storage rings. However, reducing exposure times to a few seconds for thick biological samples is still a task.

In this publication we present a compact full field transmission X-ray microscope based on a gas discharge light source using a grazing incidence collector for sample illumination and a micro zone plate as imaging optic.
2. Experimental setup

The schematic of the compact soft X-ray microscope is shown in figure 1. A gas discharge with pseudo spark like electrode geometry and 10 – 20 Joule pulse energy is used as X-ray source [4, 5, 6]. It is operated at repetition rates of up to 1 kHz and has a lateral source diameter of 1175 µm (FWHM). The resonant 1s²-1s2p transition of Helium like nitrogen at 2.88 nm wavelength is used for imaging which is realized by means of a titanium metal foil (thickness 200 nm) to provide monochromatic sample illumination. With calibrated grating spectrographs a photon number for the He-α line emission of 2·10¹³ photons/(pulse·sr) is measured at 10 J pulse energy.

To mitigate debris and separate the vacuum between the source (~1·10⁻² mbar) and optic chambers (~1·10⁻⁶ mbar) a capillary consisting of several glass channels with 50 µm diameter is mounted in front of the optical setup. The structure of the capillary is matched to the aperture of the collector and has a measured transmission of 33% (theoretical value 80%). The transmitted light is focused on the sample by a grazing incidence gold coated elliptical collector (ZEISS, axes: 600.27 mm and 18 mm). The collector has a gain of about 100 and demagnifies the source to a focal spot of about 800 µm diameter providing uniform sample illumination.

Figure 1. Schematic drawing of the optical setup.

A central stop is mounted in front of the collector to avoid direct light from the source passing through the sample reaching the detector. The sample is dissected on a Si₃N₄-membrane closely behind an aperture. A micro zone plate generates a magnified image of the sample on a TE-cooled, back-thinned CCD-detector with 1340 x 1300 pixels and a pixel size of 20 µm squared (Princeton Instruments). The zone plate has 468 zones and a diameter of 56 µm with an outermost zone width of 30 nm. The focal length at 2.88 nm wavelength is 585 µm. The diffraction limited spatial resolution of the zone plate is 37 nm.

All optical components are mounted on motorized stages and can be positioned independently. The collector and the focusing direction stage of the zone plate are piezo driven for fine adjustment.

3. Experimental results

First experiments were done with lithographic test structures to show the performance of the microscope. In figure 2 (left) a microscopic image of a Siemens star is shown. It was exposed for 60 s at 300 Hz repetition rate with a magnification of 1057x. The inner structures with 100 nm feature size are clearly resolved. To quantify the achieved resolution a high contrast region in the image has been examined. On the right of figure 2 the intensity plot of the marked area in the image of the Siemens star is illustrated. The plot is averaged over a width of 5 pixel and demonstrates an intensity rise from 10% to 90% within 37 nm, which corresponds to the theoretical diffraction limit of the zone plate.

To present the applicability of the microscope for biological research dried cells have been examined. Figure 3 shows rat embryonic fibroblast cells (left 2 min, 400 Hz; middle 5 min, 200 Hz; both 1057x). Inside the left image filament structures of about 50 nm size are visible. The picture on the right illustrates a HeLa TK-cell (5 min, 300 Hz, 1057x), a human epithelial cell line derived from a cervix carcinoma stopped during cell division.
Figure 2. Microscopic image of a Siemens star (left) and a plot of the marked area demonstrating an edge slope (10% to 90%) below 40 nm (right).

Figure 3. Dried biological cells imaged with a magnification of 1057x. Details see text.

4. Conclusion and Outlook
The developed optical setup is capable of resolving structures below 40 nm at exposure times of a few ten seconds with a magnification of 1000x. Better resolution is attainable by using a zone plate with smaller outermost zone width. The results we achieved show that using this concept laboratory X-ray microscopy of biological samples is within reach. To reduce the exposure time to the range of a few seconds several ideas will be realized in the near future. First, the source will be improved for more brilliance and stability at repetition rates above 1 kHz. Second, a new collector with matched geometry to the micro zone plate and a smaller focal spot will be integrated.

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