First application experiments with the Stockholm compact soft x-ray microscope

M Bertilson¹, O von Hofsten¹, J Thieme², M Lindblom¹, A Holmberg¹, P Takman¹, U Vogt¹, H Hertz¹

¹ Biomedical and X-Ray Physics, Royal Institute of Technology/Albanova, SE-10691, Stockholm, Sweden
² Institute for X-Ray Physics, University of Göttingen, Friedrich-Hund-Platz 1, D-37077 Göttingen, Germany
michael.bertilson@biox.kth.se

Abstract. Most soft x-ray microscopes operating in the water window (λ = 2.3 - 4.4 nm) rely on synchrotron radiation sources. In the future we believe scientists will use soft x-ray microscopes as one imaging tool among others in their own laboratory. For this purpose we have developed a full field soft x-ray microscope with a laser-plasma source compact enough to fit on an optical table. In this contribution we describe the current status of this microscope now featuring stable operation at λ = 3.37 nm or λ = 2.48 nm. In-house fabricated single element zone plates offering the possibility to perform phase contrast imaging have been implemented. We also report on the first application experiments for compact soft x-ray microscopy, including results from studies of clay minerals and colloids existing in nature and results from phase optics experiments. Planned upgrades of the microscope include increasing the source brightness, implementing more efficient condenser optics, and installing a cryo sample stage for tomography. These improvements will open up for further applications, especially in the field of biological imaging.

1. Introduction
Soft x-ray microscopy is now a successful imaging technique. Soft x-rays in the water-window (λ = 2.3 – 4.4 nm) offer a natural contrast mechanism, which opens up for applications in, e.g., bio imaging and soil science. Since the early days of soft x-ray microscopy synchrotron radiation sources have been used. The limited accessibility of these large facilities prevents this imaging technique from being one of the standard tools among scientists. For this reason we developed the first full-field soft x-ray microscope based a compact liquid-jet laser-plasma source making the entire system fit on an optical table [1, 2]. Brighter compact sources together with more efficient condenser and objective optics have made compact soft x-ray microscopes able to capture images with a quality comparable to synchrotron-based microscopes within reasonable exposure times. With these improvements the commercial interest has increased and a few companies are now developing microscopes based on compact sources.

In this paper we report on the current status, recent applications and future plans of the Stockholm compact microscope.
2. The microscope

The microscope has demonstrated stable operation at two wavelengths within the water-window, \( \lambda = 3.37 \text{ nm} \) [1] or \( \lambda = 2.48 \text{ nm} \) [2]. The microscope consists of a high-brightness liquid-jet-target laser-plasma source, condenser optics, the sample environment, an objective zone plate and a soft x-ray sensitive CCD detector. The arrangements for the two operation modes are illustrated in figure 1.

![Figure 1. Illustrations showing the basic components of the microscope for operation at \( \lambda = 3.37 \text{ nm} \) (above) and \( \lambda = 2.48 \text{ nm} \) (below)](image)

The soft x-ray emitting plasma is generated by focusing a pulsed high power laser (~3 ns, 100 Hz, 100 mJ) onto a liquid jet target. For operation at \( \lambda = 3.37 \text{ nm} \) a methanol is used and for operation at \( \lambda = 2.48 \text{ nm} \) liquid nitrogen is used. The generated photon flux is \( 10^{11} - 10^{12} \text{ ph.}(\text{pulse} \times \text{sr} \times \text{line}) \) with a line monochromacy of \( \lambda / \Delta \lambda > 500 \). A 300 nm chromium filter blocks the green light scattered from the source. For details on the sources see refs. [3, 4].

The condenser optics images the source directly onto the sample (critical illumination). A Cr/Sc multilayer mirror, 58 mm in diameter with an average reflectivity of 3.14\%, is used for operation at \( \lambda = 3.37 \text{ nm} \) [5]. When operating at \( \lambda = 2.48 \text{ nm} \), a 49 nm outer zone width condenser zone plate, 4.53 mm in diameter, is used [6].

Samples are kept in a helium atmosphere at normal room pressure. Two types of sample holders can be used. The first is a \( \pm 8^\circ \) tiltable sample stage for silicon nitride membranes which offers the possibility to do stereo imaging of dry and wet samples. Here, the wet samples are sandwiched between two membranes. The other holder is a rotatable capillary holder, shown in figure 3, which makes the collection of tomographic data of wet and dry samples possible.

In-house fabricated high-resolution nickel zone plates [6] are employed as objectives. So far zone plates with outer zone widths down to 25 nm have been used resulting in a measureable resolution better than 30 nm half-period [2]. Single element differential interference contrast (DIC) zone plates [7] and Zernike-like phase contrast [8] zone plates have been developed and offer contrast enhanced imaging.

The magnified image of the sample is detected by a cooled, back-illuminated and thinned x-ray sensitive CCD with \( 2048 \times 2048 \) pixels, each pixel 13.5 \( \mu \text{m} \times 13.5 \mu \text{m} \) in size.

3. Applications

Soft x-ray microscopy is the only full-field microscopy technique capable of imaging several \( \mu \text{m} \) thick wet samples with spatial resolutions down to tens of nanometer. This makes it an interesting technique for studying soil particles in their aqueous environment. Figure 2 shows one example from the first
experiment where the compact soft x-ray microscope was used for imaging colloidal particles in an aqueous soil suspension [9, 10].

Figure 3 shows one projection image of a diatom mounted on a capillary. The stereo and capillary holders open up for new applications where more depth information is needed. A projection series of 50 images for later tomographic reconstruction could be recorded in ~5 h.

The microscope also serves as a test bench for in-house fabricated zone plates. For example, DIC [7] and Zernike phase contrast [8] zone plates have been tested and characterized in the microscope. The microscope can easily be rebuilt to a compact instrument for diffraction efficiency measurements of zone plates, which gives important feedback to the fabrication process [11].

![Figure 2. Colloidal Chernozem soil particles in a 5-8 µm thick aqueous suspension between two Si₃N₄ membranes. The image was obtained in a 600 s exposure @ λ = 3.37 nm with a 25 nm outer zone width zone plate.](image)

![Figure 3. A diatom mounted on a glass capillary (capillary provided by S. C. Gleber and J. Sedlmair). The image was obtained in a 120 s exposure @ λ = 3.37 nm with a 30 nm outer zone width zone plate. The rotatable capillary holder enables stereo microscopy and tomography.](image)

4. Outlook

Future upgrades of the microscope include a new laser (Diode-pumped Nd:YAG, 2 kHz, 150 mJ, <1 ns pulses) for plasma generation, a new multilayer mirror condenser for the λ = 2.48 nm operation mode and a cryo sample holder with a motorized goniometer. These upgrades will reduce the exposure time. This is important for thick wet samples, for dynamic studies of soils, for recording tomographic data and for reducing problems with drift during exposures. Furthermore, the cryo sample stage will make it possible to record projection series of bio-samples without loss of detail due to radiation damage, which is important for high-resolution tomographic reconstructions.

References

[1] Berglund M, Rymell L, Peuker M, Wilhein T, Hertz H M 2000 J. Microsc. 197 268
[2] Takman P, Stollberg H, Johansson G A, Holmberg A, Lindblom M, Hertz H M 2007 J. Microsc. 226 175
[3] de Groot J, Hemberg O, Holmberg A, Hertz H M 2003 Appl. Phys. 94 3717
[4] Jansson P A C, Vogt U, Hertz H M 2005 Rev. Sci. Instrum. 76 043503
[5] Stollberg H, Yulin S, Takman P A C, Hertz H M 2006 Rev. Sci. Instrum. 77 123101
[6] Holmberg A, Rehbein S, Hertz H M 2004 Microel. Engin. 73-74 639
[7] Bertilson M, von Hofsten O, Lindblom M, Wilhein T, Hertz H M, Vogt U 2008 Appl. Phys. Lett. 92 064104
[8] von Hofsten O, Bertilson M, Lindblom M, Holmberg A, Vogt U 2008 Opt. Lett. 33 932
[9] Thieme J, Sedlmair J, Gleber S C, Bertilson M, von Hofsten O, Takman P, Hertz H 2008 XRM conf. proc.
[10] Gleber S C, Sedlmair J, Bertilson M, von Hofsten O, Heim S, Guttman P, Hertz H, Fischer P, Thieme J 2008 XRM conf. proc.
[11] Bertilson M, Takman P, Holmberg A, Vogt U, Hertz H M 2007 Rev. Sci. Instrum. 78 026103