Laboratory x-ray microscopy using a reflection target system and geometric magnification

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Abstract: This paper describes a laboratory X-ray microscopy setup, based on geometric magnification. The setup uses a sharp metal tip as a reflection target and a highly focused electron beam of a scanning electron microscope. Here we will describe the structuring process for these metal targets. To demonstrate the capabilities of our system, we show radiographs of test structures corresponding to resolutions below 100nm. There are abilities for 3D imaging in later updates of the system. Further we discuss the first imaging examples for high- and low-absorbing samples.

1. Introduction
The principal limit to the spatial resolution of conventional X-ray sources is set by the focal spot size. In dedicated laboratory setups it can be reduced to less than 1µm [1], e.g. by performing coherent diffraction imaging [2] or by using Fresnel zone-plates [3]. In contrast to these techniques and other which are restricted to synchrotron beamlines, the standard laboratory setup achieves high resolution by geometric magnification in analogy to early X-ray shadow microscopy. This method does not require any optical elements in the beam path, which are both expensive and sensitive to vibrations. Further there are little restrictions concerning X-ray wavelength bandwidth, and one can afford to increase the acceleration voltage of the electron beam up to 30keV without filtering. The physical limit for the resolution of such a system is mainly given by the size of the X-ray source spot. We present a laboratory based X-ray microscope with an ultra small source size and a resolution below 100nm.

2. General considerations
The concept of geometric magnification

\[ M = \frac{z_1 + z_2}{z_1} \]

with \( z_1 \) for the distance between source and object and \( z_2 \) for the distance between objet and a pixelated X-ray detector (shown in fig. 1), is resulting in two main resolution constrains, assuming there is no detector blur. On the one hand the resolution is limited by the physical pixel size \( P_p \) of the detector. This leads to a sampling size or an effective pixel size \( P_s \) with

\[ P_p = M \cdot P_s. \]

On the other hand the sharpness of the picture is limited by the projection of the source s on the imaging plane.
Putting these two together, plus assuming a spot size $s < 100\text{nm}$ as well as a physical detector pixel size of $P_p = 55\mu\text{m}$, this results into

$$P_{opt} = \frac{s \cdot P_p}{s + P_p} \approx s ; s \ll P_p.$$  

Hence the resolution is mainly limited to feature sizes that are in range of the focal spot size.

### 3. Setup

We modified a JEOL JSM7100-F SEM by replacing the original sample stage with a manipulator for both object and target, which results in a reflection target system (shown in fig. 2). The target is a pointy needle of tungsten or molybdenum with a tip curvature below 100nm, which results in a very small X-ray source spot. The acceleration voltage of the focused electron beam is set to 30keV. To optimize the detected photon flux, we used a 2x3 (hexa assembly) Medipix2 MXR photon counting detector [4]. The sensor layer is composed of 1mm thick cadmium telluride, with 768x512 pixels by a pitch of 55\(\mu\text{m}\). The detector is placed outside of the vacuum chamber. Therefor we used a X-ray window composed of 250\(\mu\text{m}\) thick beryllium. This setup allows to work with a small focal spot and achieves large magnifications combined with a source-object distance below 1mm in combination with a reasonable photon flux.

### 4. X-ray target preparation

Electrochemical etching turned is a fast and reliable method to produce sharp metal tips[5]. As electrolyte we used a 2N sodium hydroxide solution. The two electrodes are identical 500\(\mu\text{m}\) thick wires made of either Mo or W. When applying a DC voltage, the etching process is mainly controlled by the electrochemical potential gradient around the tip and the haphazard ionic motion of the electrolyte. With an AC voltage on the other hand the etching rate mainly depends on bubble growth rate and sliding along the tip. The bubble buoyancy during one half of the circle drives the recently etched metal oxides upwards, were they get partially absorbed during the second half of the circle. The result is a higher removal rate of the metal on the lower part of the wire (shown in fig. 3a), in this way the tip shape is formed.
Figure 3. a) Schematic of the electrochemical etching process. The wires made of Mo or W are etched in a 2N NaOH solution. By using an AC voltage the tip is mainly formed by the bubble float, this results in a higher removal rate on the lower part of the wire. b) SEM image of a tungsten tip manufactured with the proposed process, with a radius below 70nm.

Figure 4. Image of a test pattern (a) imaged with the Xradia UltraXRM L200, 5min exposure, monochromatic Cu Kα radiation courtesy of Peter Krüger, Fraunhofer IZZP, Dresden; b) imaged with our setup, 2min exposure, polychromatic 30keV tungsten spectrum; c) shows an image of a low absorbing 20µm Al₂O₃ fiber (3min exposure, polychromatic 30keV tungsten spectrum) with an intensity profile beneath. Edge enhancement due to inline phase contrast is clearly visible.

The etching rate can be controlled via AC frequency, peak-to-peak voltage and viscosity of the electrolyte. Therefore we added a small amount of DECON®90 to the solution. By applying a 10Hz sinusoidal ac voltage (20Vpp), the metal wire becomes a metal tip with a very small radius (down to 70nm) after 20 to 40 minutes of etching (shown in fig. 3b).
5. Results
To determine spatial resolution, a modified Siemens Star pattern with structures down to 50nm was imaged with the presented setup and with a commercially available X-ray microscope, the Xradia UltraXRM L200, which is based on Fresnel zone plates (shown in fig. 4a and 4b). It is clearly visible that with our setup a resolution of <100nm is possible and both setups are quite comparable, due to the fact that our setup is still in its early commissioning stage, there is plenty of room for improvements. The contrast in our image is lower due to the fact, that our spectrum has higher energies (30keV tungsten, contrary to the monochromatic Cu Kα of the UltraXRM). A low contrast image with a corresponding intensity profile of a 20μm Al₂O₃ fiber is shown in fig. 4c. It shows that with small spot sizes the spatial coherence of the source is given, and so inline phase contrast imaging is possible.

6. Summary and outlook
The presented laboratory X-ray microscope, using a reflection target source and geometric magnification, is able to achieve a resolution better than 100nm in 2D imaging, as well as inline phase contrast imaging, both at a photon energy up to 30keV. We produced sharp metal tips made of tungsten or molybdenum with a tip radius below 100nm by electrochemical etching, to achieve an ultra small X-ray spot size.

In further updates details study of 3D imaging is planned, as well as damping target and object vibrations, movement precision inside the chamber and focal point stability of the electron beam for longer exposure times.

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