Breaking the X-ray Resolution Limit

Laboratory X-Ray System with 50 nm Imaging Resolution

We have shown that 50 nm details can be distinguished in x-ray images using a compact laboratory set-up. A novel emitter design inside the x-ray source produces a very small, stable focal spot and prevents diffraction limiting effects. Due to unlimited depth of focus, the source doesn't impose a limit on the specimen size that can be reconstructed by 3D x-ray nano-tomography.

Introduction

In the quest to achieve nanometer scale resolution in x-ray images, x-ray optics such as Fresnel zone plates are often being employed. Despite the fact that they can achieve extreme high resolutions in x-ray projection images [1], the zone plates are not always suited for tomographic imaging due to their very limited depth of focus which restricts the size of the specimen to be imaged to a few microns [2]. Nano-focus x-ray sources on the other hand, do not pose this problem. However, resolving extremely small details in an object requires both a good image resolution and a high signal-to-noise ratio. In addition when structures with dimensions below a few hundred nm are being imaged using x-rays of a few keV, diffraction effects start to play a role.

To overcome these limitations, we developed a new set-up for nanotomography where both the target structure to generate the x-rays and the imaging configuration were tuned to achieve sub-100 nm resolutions with infinite depth-of-focus.

Experimental Setup

Figure 1 shows a schematic drawing how a scanning electron microscope (SEM) with a thermal field emission electron gun was used for x-ray imaging. The target is mounted at a 45 degree angle relative to the electron beam and can be coated with a thin layer of a specific material to generate characteristic x-rays with appropriate energies. The x-rays penetrating through a sample mounted on a rotation stage are acquired by a cooled X-ray CCD camera with direct photon detection.
To measure the resolution achievable with our setup, we used a resolution pattern with 180 nm thick Au structures varying in size down to 50 nm. The target substrate was coated with a 200 nm silver layer. Figure 2 shows an x-ray projection image of a part of the resolution pattern.

The enlarged portion of the resolution pattern shows that image contrast completely disappears at spatial frequencies corresponding to details of 300 nm. At even higher spatial frequencies the image contrast is inversed. This is due to Fresnel diffraction. Figure 3 shows the computed detector response when 1D grids with varying periods are irradiated with x-rays produced by a silver target substrate. The red lines show the ideal response while the blue lines correspond to the expected detector response with diffraction taken into account. The complete loss of image contrast for structures sizes around 290 nm corresponds well with what we observed in the measured x-ray projection image.

**Overcoming Diffraction Limit**

After analyzing calculations of diffraction effects we modified the set-up to push diffraction limiting effects to higher spatial frequencies by increasing the mean x-ray energy and reducing the defocusing distance. The former can be achieved by choosing a different target material which produces characteristic x-rays at higher energies, e.g. use a gold layer instead of silver. The defocusing distance was reduced by tilting the target from a 45° angle to a 75° angle such that the distance from the x-ray focal spot to the resolution pattern can be lowered from 0.7 mm to about 0.3 mm.

To improve contrast at these higher x-ray energies a resolution pattern with 500 nm thick Ta structures was used. Figure 4 shows a drawing of the central part of the resolution pattern (left) and the resulting x-ray projection image (right). Structures of 150 nm to 200 nm are now clearly visible.
X-ray Imaging with 50 nm Spatial Resolution

Using a thin planar target structure creates difficulties in generating very small (<150 nm) x-ray spot sizes with a significant x-ray flux due to the limited volume in the target material where x-rays are produced. Therefore a novel target structure has been invented. It consists of a 250 µm Pt/Ir wire with a sharp tip (fig. 5a). The axis of the wire is oriented towards the x-ray camera. The electron beam is positioned on the end of the tip and hence x-ray camera will see the emission volume as a small x-ray spot. Because the x-ray emission volume is now determined by the physical dimensions of the tip, the electron beam current can be increased and small movements of the electron beam during the exposure can be tolerated without any influence to the spatial resolution. Figure 5b shows an x-ray image of the central part of the resolution pattern. The ring with 100 nm structures is now perfectly resolved. The 50 nm structures in the inner ring are also visible.

Nanotomography: Non-destructive 3D Imaging

A microfossil sample was mounted on the rotation stage for tomographic imaging. Figure 6a shows a projection image of the microfossil acquired with a pixel size of 130 nm. To obtain a 3D tomographic dataset number of angular projections were acquired with a rotation step 1.8 degree. Due to limited mechanical accuracies of the rotation stage, sample movements have been observed. The random shifts were estimated by an iterative algorithm that fits measured projections to estimated projections from the reconstruction [4]. Once the magnitude of the X-Y shifts for each recorded projection is known, all projections are aligned and virtual slices can reconstructed using filtered back-projection algorithm. Figure 6b shows three orthogonal slices through the 3D reconstructed volume.

Conclusions

Imaging bulk samples at sub-100 nm resolution poses a number of challenges. The usage of X-ray optics such as zone plates with limited depth of focus can be applied to planar objects, but creates problems for proper imaging of bulk specimen and in acquiring datasets for tomographical reconstruction. To overcome these limitations we optimized an imaging/tomographical set-up based on a nano-focus x-ray source. The introduction of a novel target structure made of the appropriate material allows creating a very small but very stable x-ray focal spot whilst avoiding detrimental diffraction effects. Image blurring due to small random mechanical movements during tomographic acquisition is compensated
We have demonstrated that using this set-up 50 nm structures can be resolved in projection images. The infinite depth-of-focus allows any object to be completely in focus for all angular views, which is essential for 3D tomographic reconstruction. The 3D tomographic imaging capabilities were demonstrated on a microfossil sample, clearly showing details of a few hundred nanometers using a 130 nm image pixel size.

References:

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