| ESRF | **Experiment title:**<br>Characterization of Phase-type transmission Zone Plates for X-ray focussing in the 0.28-1.6 keV energy range. | **Experiment number**:<br>MI-305 |
|---|---|---|
| **Beamline**:<br>ID21 | **Date of experiment**:<br>from:25/03/1999 to:29/03/1999 | **Date of report**:<br>25/02/2000 |
| **Shifts:**<br>9 | **Local contact(s)**:<br>**Kaulich Burkhard (PLUO E)** | *Received at ESRF:* |

**Names and affiliations of applicants** (* indicates experimentalists):

**P. Charalambous *** King's College London

**J. Sussini *** ESRF

**R. Barrett *** ESRF

## Report:

### Introduction

The aim of the experiment was to characterize a number of Zone Plates (ZPs) over a wide X-ray energy range, from 280 eV to 1.6 keV. For various beam line operational reasons however, it was not possible to tune the energy to energies below aproximately 2.3keV, and consequently the experiment had to be tailored to the available energies. In the weeks leading up to the experiment, we abandoned zone materials optimum for water window optics (Ni, Cu, $Si_3N_4$, Ge), and concentrated on Tungsten ZPs which could give us useful diffraction efficiencies at energies > 2.3 keV, and at the same time provide the maximum absorbtion dominated efficiency of ~10% in the water window. (280-600 eV). A number of ZPs were fabricated, with Zone thicknesses ranging form 360 nm to 500 nm (see table ). Our fabrication technicques allow tailoring of the line : period ratio, which in turn facilitates the enhancement of higher diffraction orders, albeit at the expense of the 1$^{st}$ order. A number of single ZPs as well as ZP arrays were fabricated, and characterized in the available time.

**Experimental**

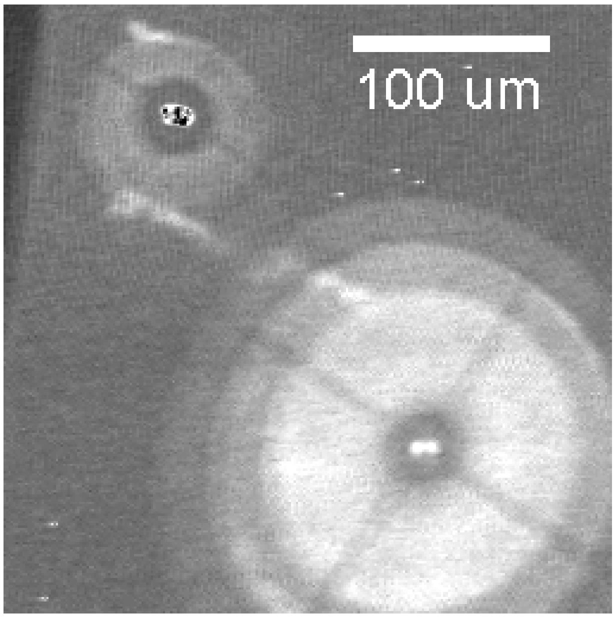
All the ZPs tested were of the *bilevel* variety, made using the "Subtractive" fabrication technique. The Zone material of the required thickness is predeposited on a suitable substrate (in this case $Si_3N_4$), followed by the deposition of multi level resist materials finishing with the resist itself at the very top, with a thickness rarely exceeding 100 nm. The obvious advantage of this, is that forward scattering of 30 KeV electrons during exposure is negligible, and the exposure of sub 20 nm lines is readily achieved. After exposure and development, the pattern is transferred into the underlying resist materials, until the eventual mask material is reached, which is usually Nickel. The final RIE stage is always fluorine based, and Nickel is an exceptionally resistant material to this chemistry. This approach, with Tungsten as the final zone material, has yielded the best high energy ZPs so far both in terms of thickness of zones, and resolution (outermost zone width, $d_{rn}$).

Although the beamline energy can be tuned down to ~2.3keV, the available flux at this energy is significantly reduced in relation to 3keV or higher (partly because of increased air absorbtion, especially for long focal paths). For this reason, all ZPs were tested at 3keV or more (see table 1). We concentrated on assessing the diffraction efficiency of as many ZPs as possible, in order to select viable ZPs to be used on the beam line for imaging experiments. It is hoped that the other critical aspect of ZP performance, resolution, will be assessed in time during these "real" imaging experiments. Assessing ZP resolution is considerably more time consuming than assesing their diffraction efficiency.

**Table 1. Sample of High Energy Tungsten Zone Plates tested**

| Diameter (μm) | Outermost $dr_n$ (nm) | Thickness (nm) | Energy (KeV) | Groove eff. (%) | COMMENTS |
|---|---|---|---|---|---|
| 50 | 125-160 | 450 | 3.5 | 7.5 | Array of four |
| 70 | 148 , 194 | 500 | 3.5 | 11 | Array of two |
| 70 | 185 | 500 | 6.0 | 8.8 | Single |
| 92, 200 | 90,200 | 360 | 3.0 | 10 | Array of two, PHOTO |
| 300 | 116 | 360 | 3.0 | 8.4 | Single |

Although only first order diffraction efficiencies are listed in the table, the presence and magnitude of higher orders was examined on a number of ZPs. The X-ray micrograph to the right, shows the pair of 92/200 μm ZPs listed above. It was recorded at 3 keV, and shows the first order focal spot of the small ZP, which almost coincides with the 5th order focal spot of the larger ZP. A number of "donuts" of intensity of other orders (including the –1st order around the ZP perimeter) are also clearly visible.




**Conclusions and future work**

We have made considerable progress towards the fabrication of Tungsten ZPs suitable for operation at high X-ray energies. The parameters of some of these ZPs are optimized for use with our Scanning Near Field X-ray Microscope (SNXM)(See MI348), which is also based at ID21 at the ESRF. The optical arrangement of the SNXM would be greatly simplified if we could eliminate the use of an Order Selecting Aperture (OSA). This could be possible if the zones of the (condenser) ZPs had a blazed profile, minimizing the presence of 0th order.

We are at present in the process of fabricating such *trilevel* ZPs, and we are planning to characterize them during our next beam time at the end of March 2000.

In addition we now have a number of ordinary *bilevel* ZPs transferred into 750 nm thick Tungsten, (See SEM micrograph to the right) which we are hoping to characterize at future beam times on ID21, which will potentially yield significantly higher diffraction efficiencies than the ZPs reported here.