Status of the Transmission X-ray Microscopy

The NSRRC transmission X-ray microscope, exploiting the light source generated from an advanced superconducting wavelength shifter, was being stationed at BL01B beamline since September 2004. It was designed and been experimentally shown to provide 2D imaging and 3D tomography in energy range 8-11 keV with a spatial resolution of 25-60 nm, and with the Zernike-phase contrast capability for imaging light materials. This article will present the design concept of the microscope, construction progress, as well as several recent commissioning results of the NSRRC X-ray microscopy.

The X-ray has long been employed as an excellent imaging agent ever since the very day when Wilhelm C. Röntgen discovered X-rays in 1895. Because of the deep penetration depth of X-rays in matter, the X-ray microscopy is expected to benefit those researches that prefer a non-destructive probe, for example, the analysis of failure mechanisms in microelectronic devices due to electro-migration, thermal breakdown or inhomogeneity and the characterization of porous materials such as soils and rock as well as the investigation of the transportation behaviour in these porous structures. Furthermore, the X-ray microscopy can be applied in the researches of cells in life science with the 3D "virtual sectioning" capacity, with which one can view either a single cell, cell clusters, or any region of a tissue in a much natural state. With labelling contrast agents, such as gold, in-situ imaging for specific cellular functions is feasible.

The development of X-ray microscopy is rather lagging than light and electron counter ones because of the late availability of highly brilliant X-rays sources and high performance X-ray optical elements. The modern synchrotron light source providing more than six orders of magnitude of brilliant X-rays than that of in-house rotating-anode generator has drastically fulfilled the requirement for photons. The invention and fabrication of high performance X-ray optical elements was however only starting as late as in the 90s, in that several focal components suchlike zone-plate, tapped capillary, compound refractive lens, Fresnel-Bragg diffractive lens, Kirk-Baez mirrors, etc., were introduced. Among them, the zone-plate and the tapered-capillary are exploited by the NSRRC X-ray microscopy.

Figure 1 depicts the schematic optical layout of the NSRRC X-ray microscope. At a first glance, the X-ray microscope is quite similar to a visible light microscope in the sense that the incident X-rays are condensed (by tapered capillary) onto sample and thereafter magnified by an objective lens (zone-plate), and the ultimate images are formed onto a two-dimensional area detector (CCD).

The NSRRC X-ray microscope is designed to exploit the hard X-ray source generated by a newly installed 5-Tesla superconducting wavelength shifter, an insertion device that shifts the intrinsic synchrotron spectrum toward higher energy X-rays. In the present case, the device promotes the critical energy of synchrotron spectrum from 2.14 keV to 7.5 keV, giving a photon flux 5×10^{12} photons/sec/0.1%bw

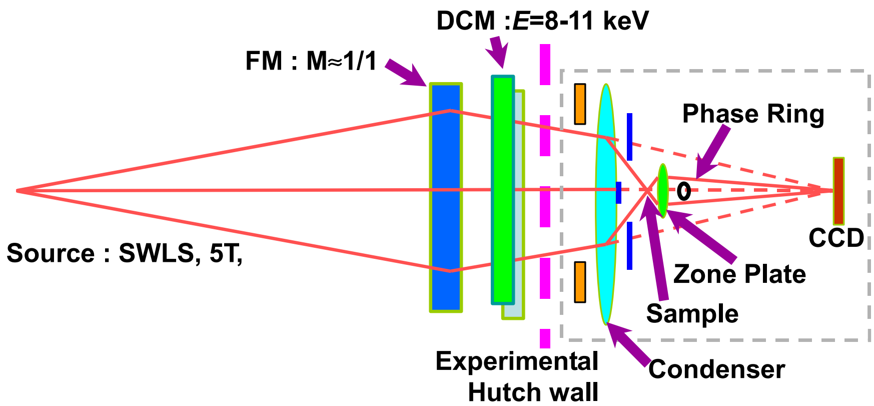

Fig. 1: Schematic optical layout of NSRRC transmission X-ray microscope.

in energy range 5-20 keV. The X-rays generated by wavelength shifter is actually a good source for full-field type transmission X-ray microscope (TXM) in the respect that the large illuminating phase space can efficiently match with the numerical aperture of the objective zone-plate, an key optical component for the transmission X-ray microscope, such that the X-rays lost in imaging process can be reduced to a manageable level. The beamline construction and the installation of the microscope were done in 2002 and 2004, respectively. There are three operational modes for the microscope, namely, first order diffraction, third order diffraction and phase contrast mode, respectively, as will be described shortly. The operational modes and the corresponding parameters are tabulated in *Table 1*.

The X-rays generated by wavelength shifter are primarily focused at the charge coupled detector (CCD) by a toroidal focusing mirror (FM) with focal ratio nearly equal to 1:1. A double crystal monochromator (DCM) exploiting a pair of Ge (111) crystals selects X-rays of energy 8-11 keV, with energy resolving power better than 1000 in match with the zone number of the objective zone-plate, in the present case ~ 400. The energy range 8-11 keV is thus chosen in order to cover the absorption edges of Cu, Zn, Ga, Ge, As, Ta, W, Au, Hg, Pb, etc., the most useful elements for semiconductor industries.

The condenser with a glass tapered-capillary intercepts the impinging X-rays and further focuses the X-rays onto the sample. It is remarkably noticed that the focusing efficiency of the tapered capillary condenser is as high as 90% due to the internal totally reflecting nature inside capillary, in contrast to that of zone-plate condensers adapted by other existing X-ray microscopes with focusing efficiency usually less than 10% for hard X-rays. There are three tapered-capillary condensers for the three operational modes.

Table1: Performance of NSRRC transmission X-ray microscope

Energy 8-11 keV		Spatial resolution (nm)	Phase contrast	2D Field Of View (μm)	3D Tomography volume (μm)
2D		60	Yes	15x15	-
		25	(Yes, 2005)	5x5	-
3D		60x60x80	Yes	-	15x15x15
		(30x30x40)	(Yes, 2005)	-	(5x5x5)
Material analysis capability		Cu, Zn, Ga, Ge, As, Ta, W, Au, Hg, Pb, etc.			

A zone-plate is a circular diffraction grating consisting of alternating opaque and transparent concentric rings (zones). The zones function in such way that the phases of the X-rays passing through sequential transparent zones change by 2π each and interfere in phase at the image point. Being that the numerical aperture is much less than one, mostly in X-ray case, a zone-plate can be adopted like an ordinary refractive lens in visible light microscope in that the geometrical optics can be simply applied. In the present microscope, the zone-plate is being used as an objective lens magnifying the images 44× and 132× for the first order and third order diffraction mode, respectively. Conjugated with 20× downstream optical magnification, the microscope provides total magnification of 880× and 2640× for first order and third order mode, respectively.

The spatial resolution δ of the microscope is set by $\delta = 1.22\ \Delta r/m$, where Δr is the outermost width of the zone-plate and $m=1,3,5,..$ the diffraction order. Accordingly, one can achieve m times better in spatial resolution by using higher diffraction order m but with the expense of downgrading focusing efficiency proportionally to $1/m^2$. The 50 nm outermost zone width of the present zone-plate thus suggests spatial resolutions of 60 nm and 20 nm for the first order and third order diffraction mode, respectively. The fabrication challenge of a high performance zone-plate is set by the high aspect ratio of the outermost zone, in this case ~19, a challenge as is seen in the measured first order diffraction efficiency of about 15% at 8 keV compared to 40% of an ideal phase zone-plate. There are total six zone-plates, each three serving for first order and third order diffraction mode, respectively, covering the energy range 8-11 keV.

The interaction of X-rays with sample can be represented by the complex refractive index of the sample, $n(\lambda)=1-\delta(\lambda)-i\beta(\lambda)$, where λ is the wavelength of the incident X-rays, $\delta(\lambda)$ represents the phase shift and $\beta(\lambda)$ the absorption properties of the sample. The outgoing wave front of X-rays is distorted by sample both in amplitude and phase. In a conventional X-ray microscope, only the change of amplitude can be recorded, named absorption contrast mode, whereas the phase term is being lost in imaging process. The phase term in fact can be retrieved by the Zernike's phase contrast method being introduced in light microscopy since 1930s. Similar to light microscope, the gold-made phase ring positioned at the back focal plane of objective zone-plate retards or advances the zeroth-order diffraction phase by $\pi/2$ resulting a recording of the phase contrast images at detector. The enhancement in phase contrast mode can be evaluated from the ratio $\delta(\lambda)/\beta(\lambda)$ of the sample, being more pronouncing for light elements than heavy ones. For comparison, the typical phase contrast enhancement for copper in 8-11 keV is 10-50, whereas for polyimide, almost transparent to X-rays in the same energy range, the enhancement is as high as 200–500. There are total twelve phase rings in the microscope with different thickness and diameters for employing different photon energies.

The resolution of microscope was tested by imaging an electroplated gold spoke pattern in first order and third order diffraction mode (*Fig. 2*). The filed of view of the images were 15µm×15µm and 5µm×5µm for first order and third order diffraction mode, respectively. The visibly resolved 50 nm finest line widths imaged in first order diffraction mode indicates the achievement of theoretical 60 nm spatial resolution. A further modulation transfer function (MTF) test (not shown in the figure) of the third order diffraction mode demonstrates a 25 nm spatial resolution.

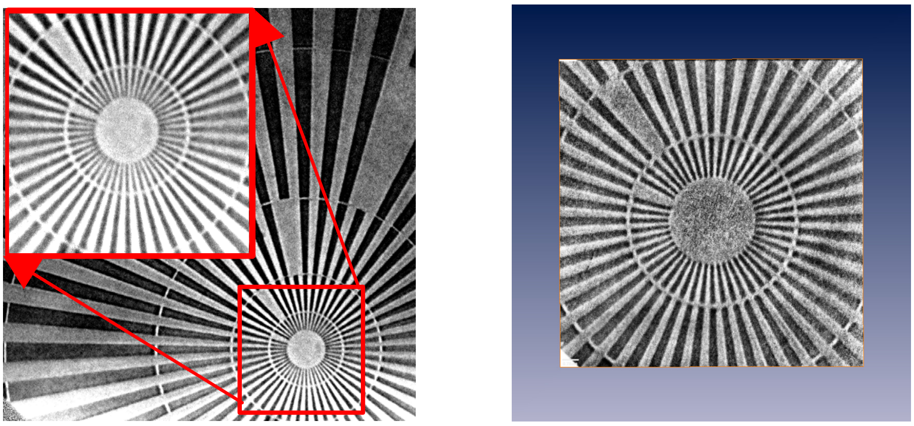

Fig. 2: An electroplated gold spoke pattern with 50 nm finest line width was adapted to test the resolution of the microscope, in first order (left) and third order diffraction mode (right). The filed of view of the images were 15µm×15µm and 5µm×5µm for first order and third order diffraction mode, respectively.

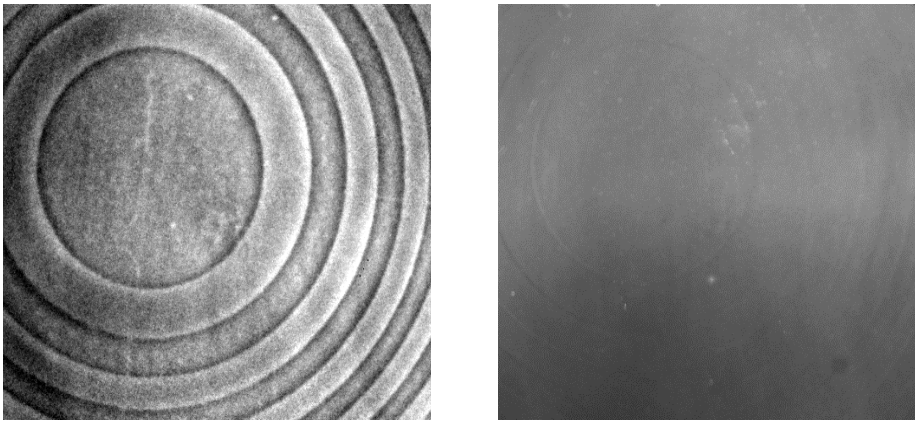

Fig. 3: The images of a one µm thick plastic zone-plate imaged with (left) and without (right) phase contrast enhancement. The contrast in the phase enhanced mode is estimated of 12%.

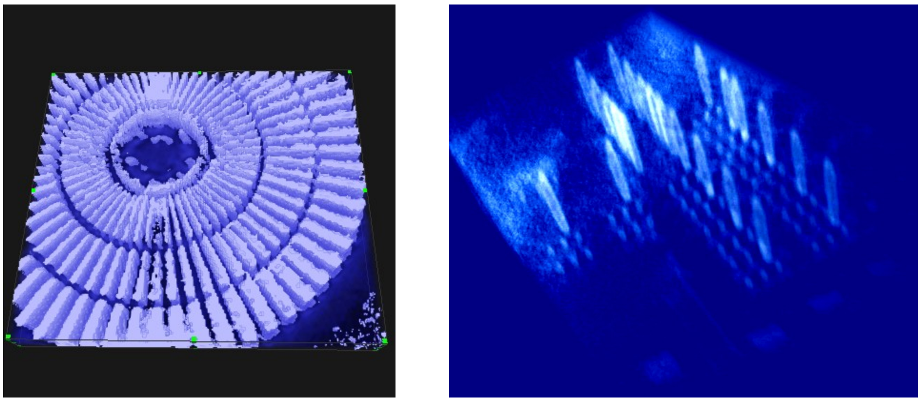

Fig. 4: 3D tomography resolution of 60nm×60nm×80nm by observing a gold spoke pattern.

Figure 3 shows the images of a one µm thick plastic zone-plate imaged with (left) and without (right) phase contrast enhancement. For one µm thick plastic, the absorption contrast is 0.01% almost invisible at 8 keV. Although the phase contrast in the test is estimated of 12%, lower than the calculated 33%, due mainly to pre-optimized phase ring thickness, one can clearly see the cracks formed on the plastic zone-plate surface with phase contrast enhancement, in contrast to the nearly vague one without phase contrast enhancement.

The tomography of the microscope was tested by imaging an electro-plated gold spoke pattern (Fig. 4). The tomography was reconstructed based on 141 sequential image frames taken in first order diffraction mode with azimuth angle rotating from -70^{o} to 70^{o}. The spatial resolution for the gold spoke pattern is estimated 60 nm × 60 nm × 80 nm.

In summary, the state-of-the-art transmission X-ray microscope has been installed at BL01B beamline since September 2004 and the performance of the microscope is being tested promisingly. The microscope can provide images with 60nm and 25nm for first order and third order diffraction mode, respectively, nowadays the world best results in 8-11 keV. The phase contrast mode tested by a polymer zone-plate, although not been optimized yet, has been demonstrated unambiguously. For IC investigation, the depth of interesting layer is about several microns, which is within the field of view of the microscope, and is well suitable for tomography. Associated with a new project subject to auto-alignment of sequential image frames, the routine tomography data collection for well-patterned samples such like IC devices can be optimistically expected.

For further evaluation, a number of sample systems from diverse disciplines are under preparation. For instance, in cooperated with a group from National Taiwan University, we have set to study the internal structure of fault zone gouges, which is thought a possible sake for earthquake instability, and was sampled in the fault zone of 1999 Chi-Chi earthquake via the Taiwan Chelungpu-fault Drilling Project (TCDP). It is expected that the non-destructive investigation of the gouge texture, grain size and 3D structure of the fault rocks from different depth away from the ground may eventually lead to a better understanding of the earthquake process. In life science, the X-ray microscopy has been successfully applied to investigate locations of specific proteins by means of immunolabeling interest proteins with contrast agent such as gold nano-particles. Trial systems suchlike the immunolabelled microtubules in Chinese Hamster Ovary tumour cells are being in processing.

BEAMLINE
01B1 SWLS - X-ray Microscopy beamline

EXPERIMENTAL STATION
Transmission X-ray Microscopy end station

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