Design of a full-filed transmission X-ray microscope with 30nm resolution

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Abstract

A full-field transmission hard X-ray microscope (TXM) with 30nm resolution was designed and its prototype was constructed. The TXM relies on a compact, high stiffness, low heat dissipation and low vibration design philosophy and utilizes Fresnel Zone plate (FZP) as imaging optics. The design of the TXM was introduced in detail, including the optical layout, the parameters of the FZP, the mechanical design of the TXM instrument. Preliminary imaging result with 52nm spatial resolution was achieved.

Full Text

Design of a Full-Field Transmission X-Ray Microscope with 30 nm Resolution

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Abstract

A full-field transmission hard X-ray microscope (TXM) with 30 nm resolution has been designed and its prototype constructed. The TXM adopts a design philosophy emphasizing compactness, high stiffness, low heat dissipation, and low vibration, utilizing a Fresnel Zone Plate (FZP) as the imaging optics. The design of the TXM is introduced in detail, including the optical layout, FZP parameters, and mechanical design of the instrument. Preliminary imaging results have achieved a spatial resolution of 52 nm.

Keywords: X-ray imaging, zone plate microscopy, precision instrument

1. Introduction

The microscope serves as humanity's eye for observing the microcosm. Throughout the long evolution from light microscopy to electron microscopy, each significant advancement in microscopy has profoundly impacted scientific and technological progress. X-ray microscopy represents another milestone, offering a new window into the microcosm and becoming a focal point for scientific development. Compared to visible light, X-rays have wavelengths three orders of magnitude smaller, enabling a corresponding three-order-of-magnitude improvement in diffraction-limited resolution [1-3]. Relative to electron beams, X-rays possess powerful penetrating capabilities, allowing non-destructive acquisition of density distribution information inside samples with nanoscale spatial resolution.

Full-field transmission X-ray microscopy (TXM) enables observation of sample three-dimensional structures at the nanoscale [4-7]. TXM holds enormous application prospects and has the potential to become as ubiquitous as optical and electron microscopes in the laboratories of scientists and engineers across fields such as life sciences, materials science, energy science, information science, and environmental science. Recognizing the importance and significant application potential of TXM, substantial breakthroughs have been achieved over the past decade. Developed countries worldwide have established X-ray TXM beamlines and experimental stations at synchrotron facilities, including ESRF, APS, NSLS, and Spring-8 [3, 8]. China has also established several X-ray TXM beamlines and experimental stations at synchrotron facilities [9-11].

Beyond synchrotron-based TXM, significant progress has been made in TXM systems based on X-ray tubes. As one of the world's most advanced scientific instruments, TXM integrates multiple cutting-edge technologies, including high-brightness X-ray sources, high-resolution X-ray optical components, high-precision CT sample stage systems, and high-precision environmental control systems. The TXM industrial project conducted at Jinan Hanjiang Opto-electronics Technology Ltd. (HJ OPTECH) is driven by the needs of a diverse industrial community seeking non-destructive high-resolution microscopy, aligning well with several key industries in Shandong Province. This work introduces the design of a full-field TXM with 30 nm resolution based on a micro-focus X-ray tube.

We first discuss the optical layout, corresponding optical design, and mechanical design of the TXM. Second, we report preliminary imaging results from the TXM instrument, including the condenser's focusing performance and spatial resolution testing. We conclude with an outlook for future work.

2.1 Optical Layout

[Figure 1: see original paper] Optical layout of hard X-ray nano-CT system.

The TXM instrument operates on the principle of full-field microscopic imaging using an FZP as the magnification lens, as illustrated in Fig. 1. The instrument comprises several key components: an X-ray source, condenser, beam splitter, sample, FZP, beam analyzer, Bertrand lens, and imaging detector.

X-rays emitted from the X-ray tube are focused by the condenser. As shown in Fig. 1, a beamstop (BS) positioned at the front center of the condenser absorbs central X-rays, providing hollow-cone illumination for the TXM. This dedicated hollow-cone beam design separates the illumination beam from the image at the imaging plane. The illumination beam passes through the beam splitter and irradiates the sample. The modulated X-rays containing sample information are then magnified by the FZP. The detector records the amplified projections modulated by the sample at the image plane. The beam analyzer works together with the beam splitter in phase contrast imaging mode.

Unlike X-ray nanoprobe systems that acquire sample information point-by-point, TXM can capture the entire sample image simultaneously. When combined with CT technology, TXM equipped with FZPs of different outermost ring widths and diameters can achieve multi-scale non-destructive three-dimensional imaging of samples ranging from 5 m to 100 m in diameter with resolutions from 30 nm to 150 nm.

In theory, with sample modulation, five major imaging contrasts can be provided in the X-ray imaging regime: traditional absorption imaging, phase imaging, differential phase contrast imaging, phase Laplacian imaging, and scattering imaging. This TXM instrument is primarily developed for absorption imaging, differential phase contrast imaging, and scattering imaging.

2.2 Parameters of FZP

The imaging schematic of the FZP is shown in Fig. 2(a). The FZP functions as a lens operating according to geometrical optics principles, where d_0 denotes the distance between the object and the FZP, and d denotes the distance between the FZP and the image. The FZP is a diffractive optical element with a series of zones satisfying the zone plate law, as depicted in Fig. 2(b).

An X-ray tube with a chromium (Cr) target was utilized. The Cr target has a characteristic peak energy of approximately 5.4 keV. The FZP has an outermost zone width of 28.5 nm and a focal length of 8.7 mm. The zone number (N) of the FZP was designed as follows, yielding a corresponding FZP diameter of 616 m.

For the TXM instrument, a gold-structure zone plate with an outer ring width of 28.5 nm was designed. Due to manufacturing process limitations, the maximum aspect ratio of the gold grating bars on the zone plate is approximately 20 to 30, with a designed thickness of 800 nm. The FZP parameters are listed in Table 1 , and an SEM image of the gold FZP is shown in Fig. 2(c).

$$D = 2\sqrt{N\lambda F} = 2 \times \sqrt{616 \times 0.23\text{nm} \times 8.7\text{mm}} = 70\mu\text{m}$$

2.3 Imaging Parameters

[Figure 2: see original paper] The principle of Fresnel zone plate as an imaging lens.

For the TXM instrument, the imaging resolution is 30 nm, and the depth of field (DOF) of the zone plate is calculated accordingly. The detector features a pixel size of 7.52 m and 2084 pixels. An optical coupling system with approximately 15× total magnification is placed between the scintillator screen and the detector sensor. The FZP magnification is chosen to be 50×, resulting in a total magnification of 750× for the TXM. Under this configuration, the 7.52 m pixel size of the detector sensor corresponds to a pixel size of 500 nm on the scintillator screen. At the object plane, the corresponding pixel size is 10 nm, yielding a field of view of 20.8 m. Table 2 lists the relative magnifications for the TXM instrument.

Referring to Fig. 2 and assuming M is the magnification factor of the FZP, the object distance can be determined as follows:

$$d_o = \frac{f(M+1)}{M} = \frac{8.7\text{mm} \times 51}{50} = 8.874\text{mm}$$

And the imaging distance is:

$$d_i = f(M+1) = 51 \times 8.7\text{mm} = 443.7\text{mm}$$

2.4 Mechanical Design

[Figure 3: see original paper] The schematic and photograph of the TXM. (a) The 3D model of the major modules. (b) The 3D schematic of the TXM. (c) The photograph of the TXM instrument.

The TXM prototype shown in Fig. 3 has been constructed and tested. For the first phase of the TXM industrial project, two complete prototype sets were delivered. The TXM comprises several key modules: the X-ray tube, condenser module, sample module, optical chamber, and detector module, as clearly labeled in Fig. 3(a). All major TXM modules are mounted on a dedicated marble supporting base. Furthermore, the major modules and marble supporting base are mounted on a high-rigidity welded supporting frame, as shown in Fig. 3(b). This TXM instrument weighs over 4 tons, with its photograph depicted in Fig. 3(c).

3. Preliminary Imaging Results

Preliminary imaging results with approximately 52 nm spatial resolution have been achieved, including characterization of the condenser's focusing performance and imaging tests of tungsten nanoparticles.

First, as shown in Fig. 4(a), a scintillator plate was placed near the condenser focus to convert X-ray beams to visible light. A simple imaging system comprising a Navitar Zoom6000 module and a Hamamatsu CMOS detector was used to observe the two-dimensional intensity distribution of the condenser's focus. By scanning this imaging system along the optical axis, the focal point was precisely determined. The intensity distribution of the focus is depicted in Fig. 4(b), while Fig. 4(c) shows the one-dimensional profile of the focus in the horizontal direction, fitted with a Gaussian function to calculate a focus size of 26.1 m FWHM.

[Figure 4: see original paper] Characterization of the illumination beam focused by the condenser. (a) The photograph of the experimental setup. (b) The 2D intensity distribution in the focal plane. (c) The 1D profile of the focus in the horizontal direction.

Second, the optical chamber containing the mounted FZP and the high-resolution optical coupling X-ray detector were carefully aligned into the optical path, as shown in Fig. 5(a). For demonstration imaging experiments, tungsten nanoparticles approximately 50 nm in diameter were randomly sprayed onto a 10 m-thick Kapton membrane, which was then mounted on the sample holder shown in Fig. 5(b). A typical random distribution of tungsten nanoparticles observed by SEM (provided by the supplier) is depicted in Fig. 5(c). After systematic adjustment of the sample and FZP positions, a clear absorption-mode image of the nanoparticles was acquired, as shown in Fig. 5(d). Numerous nanoparticles are visible, with several forming irregular nanoclusters, as magnified in Fig. 5(e). A typical nanoparticle marked by red line L1 exhibits a transmission profile along the horizontal direction shown in Fig. 5(f), consistent with a nanoparticle approximately 50 nm in diameter. Another profile along red line L2 is presented in Fig. 5(g).

[Figure 5: see original paper] Preliminary imaging experiment. (a) The photograph of the TXM instrument. (b) The sample and its holder. (c) A typical SEM image of the sample. (d) Imaging of nanoparticles. (e) The 1D transmission profile of a selected nanoparticle.

Finally, a more accurate calculation of the TXM's spatial resolution was derived using the radial power spectral density (RPSD) technique [9, 10]. As shown in Fig. 6, the sample's power spectrum cuts off at a spatial period of approximately 104 nm. Therefore, the TXM spatial resolution, typically represented as half the spatial period, is determined to be 52 nm.

[Figure 6: see original paper] The radial power spectral density (RPSD) curve.

4. Conclusion

A high-resolution full-field transmission X-ray microscope based on a microfocus X-ray tube has been constructed. The instrument design has been described in detail, including the optical layout, FZP parameters, mechanical design, and preliminary imaging performance. The mechanical design encompasses the X-ray source module, sample stage module, optical chamber module, and detector module. Preliminary testing results demonstrate an illumination beam of 26.1 m FWHM and a spatial resolution of 52 nm. This study reports only absorption-mode imaging results; phase contrast imaging and scattering imaging are currently under development and will be presented in future work.

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Note: Figure translations are in progress. See original paper for figures.