Developments at SSRF in soft X-ray interference lithography (postprint)

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Abstract

The soft X-ray interference lithography (XIL) branch beamline at Shanghai Synchrotron Radiation Facility (SSRF) is briefly introduced in this article. It is designed for obtaining 1D (line/space) and 2D (dot/hole) periodic nanostructures by using two or more coherent extreme ultraviolet (EUV) beams from an undulator source. A transmission-diffraction-grating type of interferometer is used at the end station. Initial results reveal high performance of the beamline, with 50 nm half-pitch 1D and 2D patterns from a single exposure area of 400 m$×$400 m. XIL is used in a growing number of areas, such as EUV resist test, surface enhanced Raman scattering (SERS) and color filter plasmonic devices. By using highly coherent EUV beam, broadband coherent diffractive imaging can be performed on the XIL beamline. Well reconstructed pinhole of 20 m has been realized.

Full Text

Preamble

Developments at SSRF in Soft X-ray Interference Lithography

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This article briefly introduces the soft X-ray interference lithography (XIL) branch beamline at the Shanghai Synchrotron Radiation Facility (SSRF), which is designed for obtaining one-dimensional (line/space) and two-dimensional

(dot/hole) periodic nanostructures using two or more coherent extreme ultraviolet (EUV) beams from an undulator source. A transmission-diffraction-grating interferometer is employed at the end station. Initial results demonstrate high beamline performance, achieving 50 nm half-pitch patterns in both 1D and 2D configurations across a single exposure area of 400 µm × 400 µm. XIL is increasingly applied in areas such as EUV resist testing, surface-enhanced Raman scattering (SERS), and color filter plasmonic devices. By utilizing a highly coherent EUV beam, broadband coherent diffractive imaging can also be performed on the XIL beamline, with successful reconstruction of a $ $20 µm pinhole already realized.

Keywords: Extreme ultraviolet, Soft X-ray interference lithography, Periodic nanostructures, Grating, Half-pitch

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Introduction

Large-area, high-resolution periodic nanostructures are essential for many scientific research areas, including spectroscopy gratings, bio-sensor arrays, surface plasmonics, photonic crystals, and magnetic recording. Various lithography methods can prepare such structures, including electron beam lithography (EBL), nanoimprint lithography (NIL), scanning probe lithography (SPL), and laser interference lithography (LIL) [1-4]. While EBL and SPL offer high resolution, they are too slow for large-area fabrication. Although NIL is cost-effective, it suffers from particulate contamination and residual polymer layer issues [5]. LIL can also print large-area periodic nanostructures at low cost, but its resolution is limited to approximately 100 nm by the laser wavelength [6].

Based on coherent radiation from an undulator source, soft X-ray interference lithography (XIL) has been developed to prepare large-area periodic nanostructures with resolutions below 100 nm [7]. Modern synchrotrons can provide highly coherent radiation up to about 100 eV [8]. Several XIL beamlines have been constructed at different synchrotron facilities, including the XIL II beamline at SLS [9], the EUV-IL beamline at NewSUBARU [10], the EUV-IL beamline in Wisconsin-Madison [11], the EUV-IL beamline at NSRRC (Taiwan) [12], and others. The highest resolution achieved to date using XIL techniques is line structures below 8 nm [9].

This paper reports the latest developments and potential applications of the XIL branch beamline (BL08U1B) at the Shanghai Synchrotron Radiation Facility (SSRF). Using diffraction (grating) optics, 50 nm half-pitch 1D and 2D periodic patterns have been obtained with a single exposure area of 400 µm × 400 µm. Additionally, broadband coherent diffractive imaging has been successfully implemented on the XIL experimental setup.

II. Beamline Layout

The XIL beamline is a branch of the soft X-ray spectromicroscopy beamline (STXM, BL08U1A) [13] that uses an elliptically polarized undulator (EPU). The energy range of the XIL beamline is 85-150 eV. In accordance with next-generation EUV lithography technology requirements, the EPU gap is typically set at 37.47 mm, corresponding to an energy of 92.5 eV [14]. The beamline layout is shown in Fig. 1 [Figure 1: see original paper].

A four-knife slit (Slit1) located 20 m from the source point defines the acceptance angles to $±$0.04 mrad in both horizontal and vertical directions. Slit1 also absorbs most of the heat load and protects downstream optical elements. Following Slit1, two gold-coated, side water-cooled cylindrical mirrors deflect and focus the beam. When Mirror 1 is inserted into the main beam, it diverts the beam into the XIL branch, deflecting it horizontally by 1.5° while maintaining vertical parallelism. This mirror also filters out higher-energy radiation (>2000 eV). Mirror 2 deflects the beam by 10°, which is sufficient to eliminate photons above 150 eV, thereby removing higher harmonics. It further deflects and focuses the beam onto a second four-knife slit (Slit2), which serves as a high-quality spatial coherence secondary source for the transmission-diffraction grating at the end station. The acceptance test measurement results for the beamline are summarized in Table 1 .

The XIL chamber, equipped with a transmission-diffraction-grating interferometer (mask) [15, 16], is installed in a 100-class clean room at the beamline terminus. The spatially coherent incident beam is diffracted by linear gratings patterned on a Si_3N_4 membrane (Fig. 2 [Figure 2: see original paper]). The resulting mutually coherent beams interfere at a specific distance along the beam direction, with exposure dose monitored by a photodiode (PD) installed before the mask. Two linear gratings create line patterns (Fig. 2(a)), while hole/dot patterns can be fabricated in a single exposure through the interference of three or more diffracted beams (Fig. 2(b)).

For two-beam interference under normal incidence, the interference fringe pitch is expressed as $p = d/2$, where d is the grating pattern pitch. For multi-beam interference, the relationship between diffraction grating period and fringe pattern varies depending on grating design [16, 17]. For example, in a four-beam scheme, a demagnification factor of 2 or $\sqrt{2}$ (in terms of pattern period) can be achieved when the phase factor difference of interfering beams is $n\pi$ or $(n + 1/2)\pi$, where n is an integer [17]. Thus, the fringe pattern period depends only on the grating period and is independent of the illuminating wavelength (Fig. 2). This allows efficient utilization of high-power broadband radiation from undulator sources, as a larger spectral width of the illuminating source yields higher flux, reducing exposure time and minimizing vibration issues. The beamline' s spectral width of 1/40 enables high flux (Table 1), allowing hole and line patterns in polymethyl methacrylate (PMMA) resist to be exposed within 30 seconds.

Examples of line and hole patterns with various periods fabricated on the XIL beamline are shown in Fig. 3 [Figure 3: see original paper]. Figures 3(a), 3(b), and 3(c) display 1D periodic line structures and 2D periodic holes with half-pitches of 50 nm and 100 nm, respectively. Figure 3(d) shows an SEM image of a single 400 µm exposure area. Depending on the grating structure, exposure times ranged from 10–30 seconds, significantly faster than conventional EBL techniques. All structures were exposed in 70 nm-thick positive PMMA resist spin-coated on Si wafers, then developed in a 1:3 methyl isobutyl ketone (MIBK):isopropyl alcohol (IPA) solution for 30 seconds.

The exposure structures in Fig. 3 appear as dot arrays rather than hole arrays. This anomaly arises from the non-conductive nature of the resist and the SEM scanning mode. SEM (U) mode is typically used to obtain clear images (Fig. 3) of non-conductive resist. Hole array images can be captured using SEM (L) mode (Fig. 4 Figure 4: see original paper), though with reduced clarity. However, hole arrays are clearly visible in AFM images (Fig. 4(b)). All SEM images were obtained using field-emission scanning electron microscopy (FE-SEM, Hitachi S-4800). The gratings were purchased from Eulitha AG.

III. Transmission Grating Fabrication

The grating is a critical component of XIL techniques [16–20]. Prior to acquiring our EBL machine (Crestec CABL-9500C) in 2013, we purchased gratings from Eulitha AG. Since then, we have fabricated and used a two-beam grating with a 300-nm period.

Dry-etching and lift-off processes are commonly employed in grating fabrication [16–18]. While smooth, steep Cr line structures can be fabricated using dry-etching, this process typically requires toxic gases such as Cl_2. Zhu et al. [19, 20] reported fabricating smooth Cr line structures through electron beam evaporation and lift-off processes. Based on rigorous coupled-wave analysis, they fabricated a Cr grating on a 100 nm Si_3N_4 membrane using EBL lithography and lift-off, achieving a first-order diffraction efficiency of 4.43%. Using the same process, we fabricated a Cr grating with a 300 nm period and obtained exposure results on the XIL beamline.

The grating fabrication process is schematically illustrated in Fig. 5 Figure 5: see original paper. A low-stress 100 nm Si_3N_4 layer serves as the transparent membrane. A Crestec CABL-9500C e-beam lithography system performed the patterning, after which a 50 nm Cr layer was deposited via electron beam evaporation. A 300 nm Au layer was deposited onto the outer grating region to function as a central photon stop layer for XIL. The backside silicon substrate was removed through wet etching in aqueous KOH solution at 80°C. Figures 5(b), 5(c), and 5(d) show SEM images of the Cr grating and its interference patterns (half-pitch = 75 nm) on PMMA resist and on a molecular glass (MG) resist developed by one of our users. Grating and PMMA exposure result images were captured using the SEM function of the EBL system (CABL-9500C), while the user’ s exposure result was imaged using FE-SEM (Hitachi S-4800).

IV. Applications

The XIL beamline began commissioning in September 2011, started operation in January 2012, and has been open to users since October 2012. Thanks to its excellent performance in pattern resolution, uniformity, and pattern area size, the system has been applied in numerous fields, including EUV resist testing, surface-enhanced Raman scattering, nanometer magnetism, biological self-assembly, nano-photoelectron devices, and high-density large-area gratings. To date, 10 patents and 7 articles have been published. Here we report applications in EUV resist testing and surface-enhanced Raman scattering.

A. EUV Resist Test with High Resolution [21]

EUV lithography using 13.4 nm irradiation is the most promising candidate for next-generation lithography and represents a key technology for continuing Moore’ s Law [22]. Challenges in EUV lithography include the lithography system, mask, and photoresist [23]. Yang et al. [21] conducted EUV resist testing on the soft XIL beamline, obtaining line structures of varying widths by adjusting exposure time. They achieved 30 nm resolution with line width roughness (LWR) of 2 nm (Fig. 6 [Figure 6: see original paper]).

B. Surface Enhanced Raman Scattering (SERS) with High Sensitivity and Uniformity [24]

Au nanodisk arrays exhibit significant, uniform, and reproducible surface enhancement of Raman scattering signals, enabling detection of R6G (Rhodamine 6G) at concentrations as low as 10^{-8} M with an enhancement factor of 10^{6} (Fig. 7 Figure 7: see original paper). Importantly, these SERS-active substrates demonstrate high reproducibility and stability alongside uniform high sensitivity.

The intensities of main Raman vibrations of R6G methanol solution were collected from 32 spots on the Au nanodisk arrays. The relative standard deviation (RSD) values for vibrations at 1313, 1366, and 1512 cm^{-1} are 18.1%, 15.5%, and 13.4% (Fig. 7(b)), respectively, providing vigorous evidence of substrate uniformity [24]. XIL nanofabrication thus appears to be a feasible approach for preparing uniform, reproducible, high-sensitivity SERS-active substrates for practical applications.

V. Latest Development—Broadband Coherent Diffractive Imaging

Broadband coherent diffractive imaging (CDI) represents the latest development on the XIL beamline. CDI is a powerful microscopy technique for two- and three-dimensional imaging of materials and biological specimens [25]. Since it does not rely on X-ray optics, its spatial resolution is limited only by wavelength. Significant resolution improvements require brighter sources, longer exposure times, or increased bandwidth acceptance. Using broadband X-ray beams offers a promising route to enhanced spatial resolution [26].

Utilizing the full spectral width of undulator radiation enables broadband XIL lithography. The beam' s spatial coherence and coherent photon flux can be precisely controlled, satisfying the conditions for broadband coherent diffractive imaging. Consequently, broadband CDI can be implemented on the XIL beamline. Typical results are shown in Fig. 8 [Figure 8: see original paper]. Broadband X-rays with the distribution shown in Fig. 8(a) irradiated a \$ \$20 µm pinhole at normal incidence. Fig. 8(b) shows the diffraction pattern collected by CCD, while Fig. 8(c) displays the pinhole image reconstructed after sufficient iteration using a broadband phase retrieval algorithm. Compared with Fig. 8(d), the reconstructed pinhole image matches the real image, with aperture edge details clearly visible (indicated by arrows). To avoid CCD saturation, a beamstop was used, causing black artifacts inside the reconstructed pinhole image due to missing central signals in Fig. 8(b). These missing central data can be estimated via Fourier transform of lower-resolution images from soft X-ray microscopes [27]. These results demonstrate the feasibility of broadband CDI on the XIL beamline, providing a prerequisite for developing rapid high-resolution three-dimensional imaging techniques.

VI. Conclusion

This study introduces a new branch beamline at SSRF for XIL exposure using a transmission-diffraction grating interferometer. The XIL beamline fabricates 1D periodic line structures and 2D periodic hole patterns through EUV exposure with two- or multi-beam gratings. A two-beam Cr transmission diffraction grating with 300 nm pitch was fabricated via lift-off process, producing high-quality exposure line patterns with 75 nm half-pitch on both PMMA and MG resists. Users have successfully performed EUV resist testing with high resolution and surface-enhanced Raman scattering detection with high sensitivity and uniformity, demonstrating the beamline' s excellent performance. Furthermore, by leveraging the highly coherent EUV beam, the XIL beamline enables broadband CDI, with successful reconstruction of a \$ \$20 µm pinhole already achieved.

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