EUV-FEL diffraction imaging of nanostructures at SCSS

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Abstract We report on a successful demonstration of coherent diffraction imaging (CDI) by using the EUV-FEL at the SPring-8 Compact SASE Source (SCSS). Strong speckle patterns were obtained from a holey carbon mesh, used as a test specimen, from a single pulse exposure. The reconstructed image, a complex density object, well reproduced the structural features of the test specimen at a few hundred nm resolution detail.

1. Introduction

The X-ray diffraction (XRD) is a well accepted technique in characterizing structures of crystalline specimens at atomic scale. However, it is not always amendable for the XRD technique to study biological specimens and nanostructures samples, especially when a crystal specimen is not available. CDI is a promising technique to investigate structures of non-crystalline samples[1–8]. It provides high resolution and high contrast images of non-crystalline specimens with particular advantages in studying biological single cells or organelles as well as functional nanostructures. To achieve high resolution, strong incident beam becomes indispensible, which accompanies significant radiation damages to the samples at the same time. As one way to deal with this x-ray radiation damage, acquiring a diffraction pattern by using intense and short pulse (<10 fs) x-rays was proposed[9]. X-ray free electron lasers (XFELs) provide bright, ultra-short pulse, coherent x-rays enabling high resolution 3D imaging of biological specimens with a single-particle approach. As a prototype XFEL source, the SCSS has been constructed. We have developed an EUV-FEL coherent diffraction microscope (CDM) and carried out CDI experiments for a test sample at the SCSS. We have successfully demonstrated that high quality speckle pattern can be obtained even from a single shot (10^{12} photons).

2. Experimental

The SCSS provides laser lights of the wavelength between 51 and 61 nm operating up to 60 Hz repetition rate. The SCSS delivers about 10^{12} photons per single pulse[10]. Higher photon density is achievable by using a pair of elliptical and cylindrical focusing-mirrors. The pair of focusing-mirrors is installed before the diffraction microscope delivering 10^{12} photons at 30 microns by 30 microns focal spot at 1 m downstream from the center of the second cylindrical mirror.

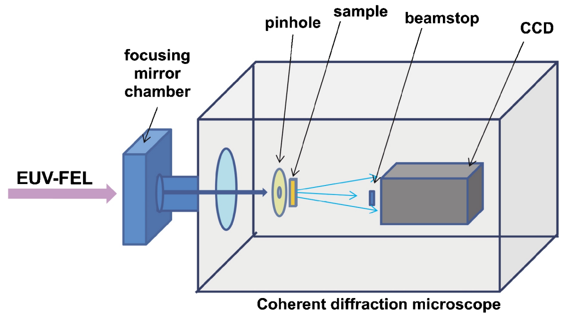
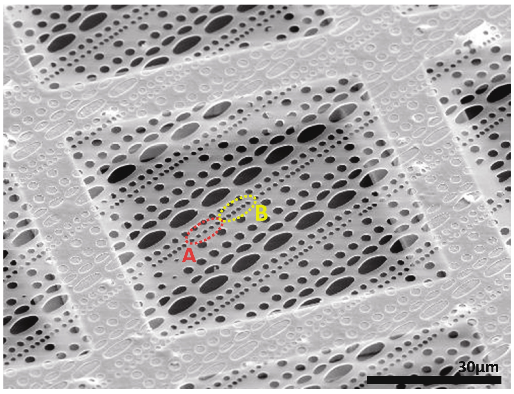


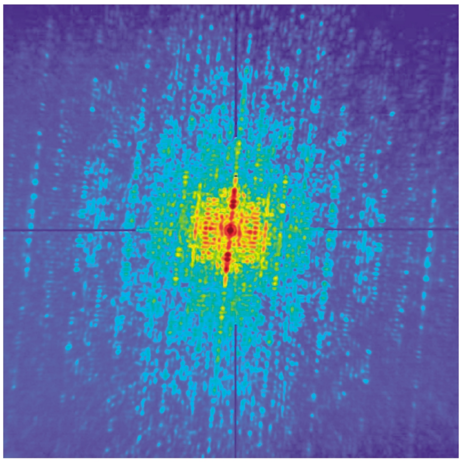
Fig. 1 Schematic drawing of coherent diffraction microscope.

We have built a CDM for EUV-FEL at the SCSS. The microscope hosts submicron precision linear motion stages to manipulate a pinhole aperture, a sample holder and a beamstop as shown in **Fig. 1**. The beamstop is made of 2 mm × 2 mm square tungsten block hold by 50 microns diameter tungsten wires. Speckle patterns are recorded on an in-vacuum CCD detector (PI-MTE 1300) housed inside the diffraction chamber. During the data acquisition, the CCD chip of the detector was kept at −40°C cooled by Peltier circuit reducing thermal noises.

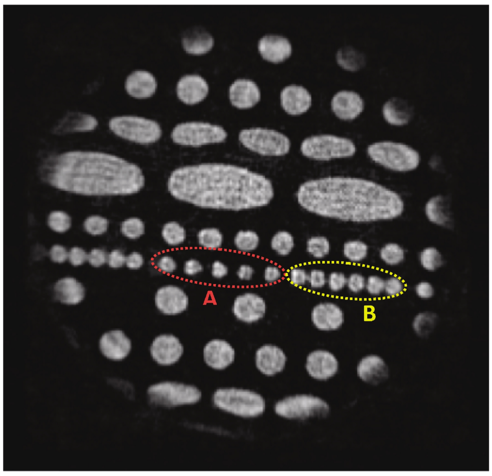
We have carried out CDI experiments by using a holey carbon mesh (SPI, Quantifoil). The thickness of the carbon film is 10 nm. The holey carbon mesh has a repeated pattern composed of circular and elliptical holes in various sizes as shown in **Fig. 2(a)**. The circular holes have diameters of 1.0, 1.4 and 2.0 μm. The elliptical holes measure 1 μm × 4 μm and 2 μm × 8 μm along the shortest and longest axes, respectively[11]. The mesh is attached to 12 micron thick Cu pinhole aper-



(a)



(b)



(c)

Fig. 2 (a) SEM image of holey carbon film[11], (b) Coherent diffraction pattern from the test sample, high q region is obtained by a single shot, low q region is obtained by 2000 pulses with gas attenuator (c) Reconstructed image.

ture with 30 μm in diameter defining a field of view. The wavelength of incident EUV-FEL beam was tuned at 51 nm. The distance between the sample and the CCD detector was 80 mm.

3. Results

Fig. 2(b) shows a measured speckle pattern from the sample. Non-negligible absorption of EUV lights by the carbon film results in non-centrosymmetric diffraction pattern. Focused single EUV-FEL pulse was sufficient to acquire a strong speckle with saturated pixels beyond the dynamic range of the CCD at a low Q region. During the acquisition, the beamstop was employed around the center of the pattern. The two dark blue lines of the vertical and horizontal directions are the regions that the diffraction signals have been blocked by the tungsten wires holding the beamstop. Later by removing the beamstop, we have also acquired the data for the low-Q region: portion of data blocked by the beamstop before. To avoid the damage on the CCD from the beam intensity fluctuation especially without using a beamstop, incident beam flux was reduced by using an Ar gas attenuator on the beamline. The data was taken by accumulating 2000 frames of single-shot data increasing the S/N ratio.

Fig. 2(c) shows an image reconstructed through iterative phase retrieval processes[12–18] from the oversampled diffraction pattern. Complex image reconstruction was carried out and only the magnitude is displayed in **Fig. 2(c)**. All the features of the mesh described above are clearly resolved in the obtained image. The field of view is 30 μm in diameter, the size of employed pinhole aperture. The pixel resolution of the image in **Fig. 2(c)** is 161 nm. We note that the 1.0 μm diameter circular holes are not equidistance in the region A and B indicated by red and yellow dotted circles in **Fig. 2(c)**, respectively, which is consistent with the SEM image of the mesh in **Fig. 2(a)**.

4. Summary and perspective

In this work, we have developed a CDM for EUV-FEL. We have obtained strong speckle patterns from a holey carbon film. The reconstructed image well reproduced the structural features in submicron scale of the film in detail. We have successfully demonstrated the CDI experiments via a single pulse exposure of EUV-FEL are feasible at the SCSS at a few hundred nm resolution. Since the resolution of CDM is limited only by the wavelength of incident light in principle, XFEL with wavelength of angstrom order will enable to get the 3D single particle image at near-atomic resolution based on this experimental scheme.

Acknowledgement

This study is collaboration with experimental facility group at XFEL project head office, RIKEN. We are particularly grateful to M. Nagasono, M. Yabashi, T. Ishikawa and the SCSS test accelerator operation group, RIKEN for their stimulating discussions and technical support.

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