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Capabilities of using white x-rays for the reconstruction of surface morphology from coherent reflectivity

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ABSTRACT

We present a new method to reconstruct the surface profile of a sample from coherent reflectivity data of a white x-ray beam experiment. As an example the surface profile of a laterally confined silicon wafer has been reconstructed quantitatively from static speckle measurements using white coherent x-rays from a bending magnet in the energy range between $5 < E < 20$ keV. As a consequence of using white radiation, speckles appear in addition to the Airy pattern caused by scattering at the entrance pinhole. Nevertheless, the surface profile of a triangularly shaped specimen was reconstructed considering sufficient oversampling between the beam-footprint and the effective sample width. For the profile reconstruction the Error-Reduction phase retrieval algorithm was modified by including the spectral illumination function and a Fresnel propagator term. The simultaneous use of different x-ray energies having different penetration depth provides information on the evolution of the surface profile from the near-surface towards the bulk. The limitations of present experiment can be overcome using white or pink radiation from a source with higher photon flux.

1. Introduction

Third generation synchrotron sources provide coherent photon flux to employ coherent diffraction as an imaging technique in various fields of science. After the first proof of the principle [1], coherent x-ray reflectivity from surfaces was not further pursued. In principle, the method yields surface profile information over a large lateral length of a sample surface with nanometer resolution in local height. Few attempts have been made to measure static speckles from various surfaces using monochromatic x-rays and to reconstruct the spatially resolved surface profile information at sub-nm resolution [2–4]. Here subsurface information is tailored by the wavelength used for the experiment. However, the method is not well established as a routine surface imaging technique yet, mainly due to the lack of routine in quantitative data analysis. Another application of coherent diffraction is the method of X-ray photon correlation spectroscopy (XPCS) where the speckle fluctuations are quantified to probe a certain dynamical process within the specimen [5]. Here the decision which physical process is behind the dynamics requires measurement at different values of momentum transfer [5]. In principle, energy-dispersive experiments with coherent white or pink x-ray radiation could provide the required additional information from one and the same experiment. In the following we present results of coherent reflectivity from a spatially confined sample surface using the white x-ray beam recorded at a bending magnet synchrotron source. We show that we can reconstruct the surface profile unambiguously and can provide additional subsurface information. The present limitations of a low flux bending magnet source are also discussed.

2. Experiment

The coherent reflectivity experiments are performed using white x-rays in contrast to the previous monochromatic experiments by others [2–4]. The measurements are performed at the bending magnet EDR beamline at BESSYII which provides polychromatic x-rays with the useful energy range between $5 < E < 20$ keV. The main advantage of energy-dispersive method compared to a monochromatic one is that surface speckles data are measured over the wide energy range simultaneously in a relatively short time interval without changing the illuminated area at the sample [6–8].

The coherent part of the incoming beam is selected by an entrance pinhole of size 10 μm (transverse coherence lengths $\xi_v \sim 85$ μm and $\xi_h \sim 70$ μm for 5 keV to $\xi_v \sim 21$ μm and $\xi_h \sim 17$ μm for 20 keV x-rays at ∼31 m from the source) at a distance of 0.3 m upstream the sample (see Fig. 1). Due to the white x-ray beam it

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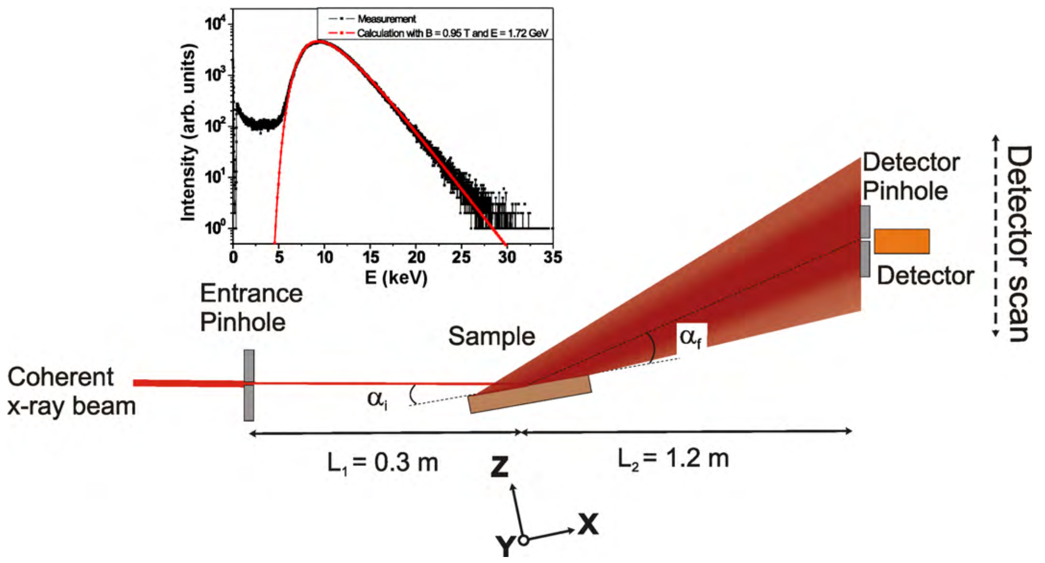

Fig. 1. Experimental set-up for the measurement of static speckles from surfaces at the EDR beamline at BESSYII. The inset shows the measured and calculated emission spectrum of the white x-ray beam.

is not possible to place an additional guard pinhole in the beam path to block the higher order fringes of diffraction generated by the entrance pinhole like in monochromatic set-ups. Consequently the beam profile at the sample position cannot be approximated by a Gaussian [8–9]. The illumination function is the energy-dependent complex Fresnel diffraction function of the entrance pinhole.

Specular and nonspecular scattering is measured by scanning an energy-dispersive point detector perpendicularly to the incident beam 1.2 m downstream the sample. The detector aperture is defined by a pinhole of size 10 µm that provides the spatial resolution to resolve the speckle pattern from the sample surface. In our method we measured the energy dependence of the scattering at a fixed incident angle, α_i. Reciprocal space mapping was realized by taking subsequent spectra at the different exit angle, α_f, in the detector perpendicular scans (see Fig. 1). Each measured intensity map $I(E,\alpha_f)$ for a given incident angle α_i taken as function of exit angle α_f, has been transformed into reciprocal space coordinates using the relation

$$q_x = \frac{2\pi E}{hc}(\cos\alpha_f - \cos\alpha_i) \tag{1}$$

$$q_z = \frac{2\pi E}{hc}(\sin\alpha_f + \sin\alpha_i) \tag{2}$$

where h is the Planck's constant and c is the speed of light.

The investigated sample was a Si (1 1 1) wafer with a triangular shape. As mentioned in Ref. [10], the sampling in the reciprocal space is equivalent to measurement from a region of nonzero density surrounded by zero density region. The non-zero density region is known as support region. The oversampling ratio is the ratio of the total illuminated area to the area of the support region. For the coherent reflectivity experiments, the oversampling ratio [10] (geometrical beam-footprint/sample length) can be varied by translating the sample perpendicular to the direction of the incident and the scattering beam. The surface scattering is measured from the part of the sample with lateral width 0.8 mm along the beam direction, thus giving the oversampling ratio of 3.5 (2.8/0.8).

The Fresnel number (N) decides the propagation region of the measurement [11]. It is defined as $N = (a^2/\lambda L)$, where a is the size of the aperture and L is the distance from the source to the observation plane. With an entrance pinhole of 10 µm and pinhole-detector distance of 1.5 m, here the Fresnel number (N) ranges from 0.6 at 5 keV to 2.4 at 20 keV. Hence the static speckle data are always measured in the near-field (Fresnel region) of the sample for the x-ray energies between 5 and 20 keV.

The measured reciprocal space maps in (q_x,q_z) space (Fig. 2) exhibit a series of lines of maximum and minimum intensity parallel to the q_z axis. These extrema are due to the constructive and destructive interference of the x-rays scattered from different positions on the surface of the sample having different local height. In addition one finds certain regularity in the pattern along q_x originated by diffraction from the collimating pinhole that coherently overlaps the speckle features. For better clarity, lines of constant energy are drawn in Fig. 2.

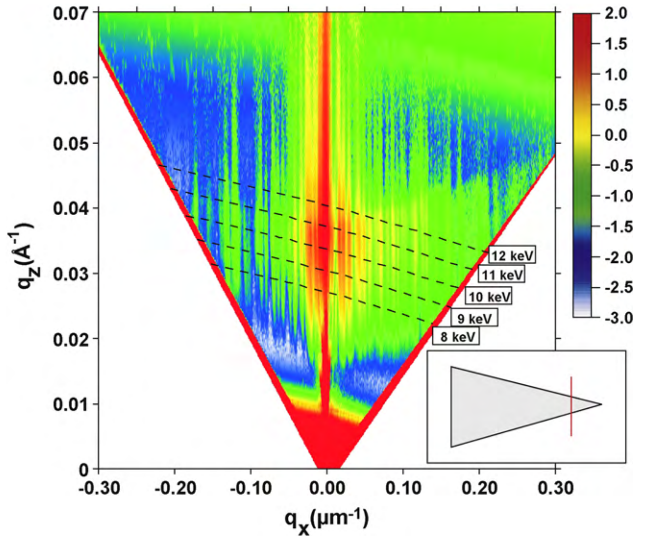

Fig. 2. Measured coherent reflectivity maps in the ($q_x - q_z$) space for a sample width (~0.8 mm) smaller than half of the beam-footprint (~2.8 mm), satisfying the support constraint with over-sampling ratio $\sigma = 3.5$. The dashed lines mark the scans at selected energies E = 8, 9, 10, 11, 12 keV. The inset shows the position of beam-footprint (red vertical line) on the sample surface.