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FAST TRACK COMMUNICATION

New imaging technique based on diffraction of a focused x-ray beam

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Abstract
We present first experimental results from a new diffraction depth-sensitive imaging technique. It is based on the diffraction of a focused x-ray beam from a crystalline sample and recording the intensity pattern on a high-resolution CCD detector positioned at a focal plane. Structural non-uniformity inside the sample results in a region of enhanced intensity in the diffraction pattern. The technique was applied to study silicon-on-insulator thin layers of various thicknesses which revealed a complex strain profile within the layers. A circular Fresnel zone plate was used as a focusing optic. Incoherent diffuse scattering spreads out of the diffraction plane and results in intensity recorded outside of the focal spot providing a new approach to separately register x-rays scattered coherently and incoherently from the sample.

X-ray diffraction imaging is a powerful tool for studying the structure of crystalline objects. Diffraction of x-rays in a crystal lattice is affected by defects, inclusions, interfaces and other irregularities. The perturbations are visualized by recording the intensity distribution on a film or a CCD detector. This is the goal of x-ray topography [1] which was developed a few decades ago and has been successfully utilized in such important areas as crystal growth and semiconductor technology. Driven by remarkable progress in x-ray focusing [2], new possibilities based on the diffraction of a focused x-ray beam have been theoretically explored recently and a new imaging technique has been proposed [3]. It is based on the diffraction of a focused beam from the sample located between the focusing optics (refractive lenses or zone plates) and the focus and recording a diffraction pattern at the exact focus position by a high-resolution 2D detector. This technique has the potential of bringing x-ray diffraction imaging to a submicrometre level opening up new opportunities in the diagnostics of crystalline structures produced by modern micro- and nanotechnology. In this letter, we report the first experimental realization of this technique.

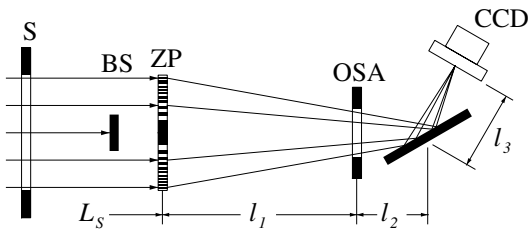

Figure 1. Experimental setup at the APS beamline 2-ID-D. Monochromatic x-ray beam from the undulator is focused by the Fresnel zone plate (ZP). BS is the beam stop; OSA is the order sorting aperture located at the distance l_1 from ZP. The focused beam is diffracted by the sample located at the distance of $l_1 + l_2$ from ZP and recorded by the CCD camera positioned at the focal length $L_F = l_1 + l_2 + l_3$.

The experiment was performed at the diffraction microscopy beamline 2-ID-D at the Advanced Photon Source [4]; the experimental setup is illustrated in figure 1. An x-ray beam with an energy of 9.5 keV was selected from the undulator by an upstream double-crystal Si(1 1 1) monochromator. A gold circular zone plate (ZP) with an outermost zone width of 0.1 μm and a focal length of

$F = 12.18$ cm at 9.5 keV was located at a distance of $L_s = 74$ m from the source. The slit S limited the beam to the size of the zone plate aperture of 160 μm. The 35 μm diameter beam stop (BS) and the 25 μm diameter order sorting aperture (OSA) located at the distance l_1 from the ZP are the important parts of the setup as they reduce the background radiation intercepted by the sample. The typical vertical size of the beam produced by this ZP was 0.2 μm. A high-resolution CCD camera with a spatial resolution of about 1.5 μm was mounted on the detector arm of the six-circle diffractometer at a total distance of $l_1 + l_2 + l_3 = L_F = F/(1 - F/L_s) = 12.20$ cm from the ZP. Samples were mounted on a sample stage with the axis of rotation at a distance of $l_3 = 8$ mm from the detector. To record diffraction patterns, the detector was rotated at an angle of $2\theta_B$ keeping the detector plane perpendicular to the diffracted beam.

The samples were thin silicon-on-insulator (SOI) layers with (1 1 1) and (1 1 0) orientations and thicknesses of 4.5, 10 and 25 μm [5]. The top layer was bonded to the substrate of a different orientation with a thin buried SiO_2 layer in between [6, 7]. To inspect the perfection of the layers, high-resolution x-ray rocking curves were measured at CHESS at the energy of 25.9 keV. The measurements showed significant broadening of the diffraction curve and a manifold increase in the integral intensity which are characteristic of a gradual variation of the lattice constant with depth into the layers.

CCD images of the focused beam diffracted from the samples were taken at different angles as the crystal was rotated through the angular aperture of the zone plate. Typical images recorded from all three layers and the cross-sections of these images by the scattering plane are shown in figure 2. One can clearly distinguish three intensity peaks: a bright spot on the left corresponds to the strong reflection from the front surface of the layer, a much weaker spot on the right is the reflection from the back surface and the intensity in between indicates structural imperfection of the layer. The zero point in the x-scale is chosen at the front surface. The peaks which correspond to the back surface are shown by arrows. By measuring the distance between the peaks from the front and the back surfaces, which is equal to $2 \cdot t \cdot \cos(\theta_B)$ where θ_B is the Bragg angle, the local thickness t of the layer can be immediately determined. The experimental thickness values for our samples are in good agreement with the values specified by the producer.

Every peak has the shape of an elongated streak extended in the scattering plane with the size in the plane perpendicular to the scattering plane of about 1.5 μm which is determined by the resolution of the CCD camera. This observation demonstrates the fact that diffraction spreads the intensity in the scattering plane while in the perpendicular direction perfect focusing still takes place. For a perfect crystal of thickness t, the size of the beam in focus is determined by the extinction effect if $t > L_{ex}$ where L_{ex} is the extinction length [3].

As we demonstrated, the new technique can provide immediate qualitative evaluation of the local structural perfection of a thin crystalline layer. To identify what kind of imperfections can generate experimentally recorded intensity patterns we performed computer simulations for a 15 μm thick Si (1 1 0) layer and two strain models. We consider a linear

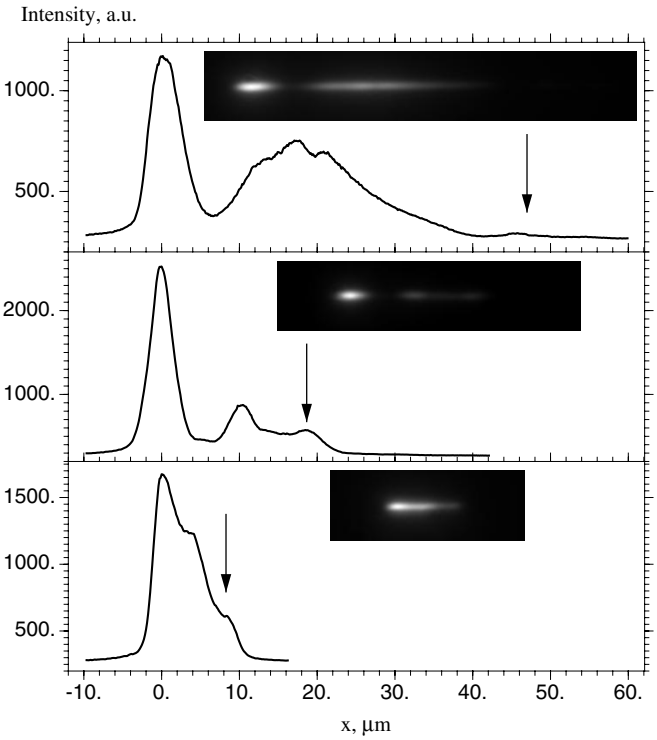

Figure 2. CCD diffraction images and their cross-sections by the diffraction plane recorded from the SOI layers of different thicknesses: 25 μm, (2 2 0) reflection, upper panel; 10 μm, (1 1 1), centre and 4.5 μm, (2 2 0), bottom. The arrows show the position of the back surface.

ZP with the parameters equivalent to the ZP used in the experiment. Propagation of the x-ray field from the ZP to the OSA and through the OSA was taken into account. The details of the algorithm can be found in [3].

The results are shown in figure 3. The intensity patterns in the upper panel are from a step-like interface between two layers of different lattice constants $\Delta d/d$ in the range from 0 to 10^{-4}. The interface is located at a depth of 8 μm below the surface. As one can see, the sharp interface generates a well-defined intensity peak with the position of this peak directly indicating the depth location of the interface. A second model is a linear $\Delta d/d$ profile which is characteristic of elastic bending. The values of the maximum $\Delta d/d$ (at the bottom surface) were chosen the same as for the step-like profile. The results are shown in the bottom panel. If the gradient is small, the intensity is spread through the layer with a slight increase at the bottom surface. With increasing gradient a broad peak starts forming, its integral intensity increases and the maximum shifts towards the front surface, in qualitative agreement with the results obtained with a narrow beam [8].

None of the above models alone can explain our experimental results, thus indicating a complicated strain distribution within the layers. In addition, we revealed in-plane structural inhomogeneities by scanning the sample laterally within the area of 50 μm. It is known that bonding of two surfaces is never perfect: flatness non-uniformity of two mating surfaces [9] generates a strain pattern within the layers, which results in a waviness contrast observed in standard (macro) x-ray topographic techniques [10]. We may

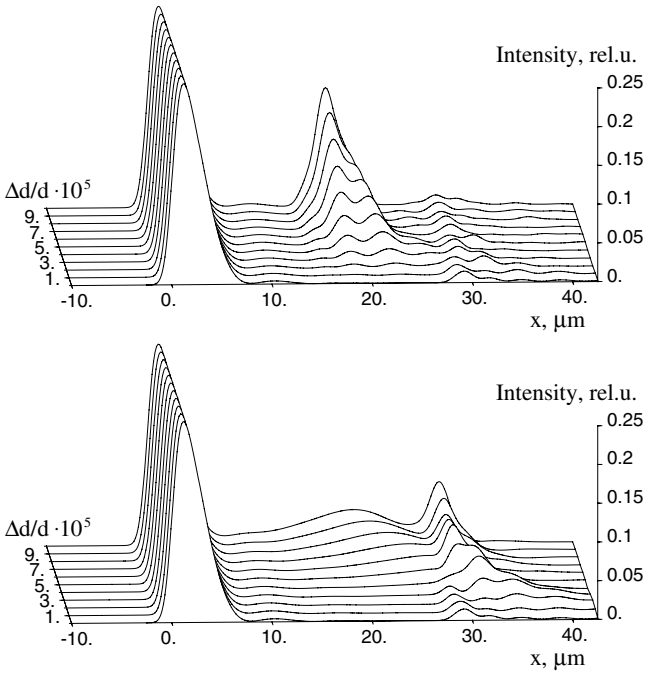

Figure 3. Theoretical simulations of the diffraction pattern from the 15 μm thick Si layer (220 reflection, 9.5 keV) having the strain of a different origin. Top: a step-like interface between two layers of different lattice constants, located at a depth of 8 μm below the surface. Bottom: linear $\Delta d/d$ profile representing elastic bending of the layer.

speculate that the superposition of the elastic strain fields from adjacent areas of opposite bending can create local regions with stronger gradients which show as more distinct peaks in our images.

So far we have considered only coherent x-rays. If the crystal causes decoherence of x-ray waves (e.g. due to defects and surrounding strain fields) we may expect that the incoherent part of x-rays will not focus perfectly and could be detected outside the focal spot. Indeed, careful examination of recorded patterns shows a weak halo extending further from the centre. Slowly decaying intensity tails (figure 4) characteristic of diffuse scattering can be clearly observed. The experimentally measured point spread function is shown as open circles for comparison. Since a lens creates a Fourier image of the scattered radiation at the focal position, each pixel in this image corresponds to a certain scattering vector defined by the pixel coordinates and the sample-to-focus distance. Thus the proposed technique offers a new method to map incoherent x-rays in a single shot allowing for fast time-resolved studies, e.g. with a single XFEL pulse.

In conclusion, we report first experimental results obtained with a new imaging technique based on the diffraction of a focused beam. The technique is capable of obtaining local in-plane and depth-sensitive structural information. Application of this technique to SOI layers revealed a complex strain

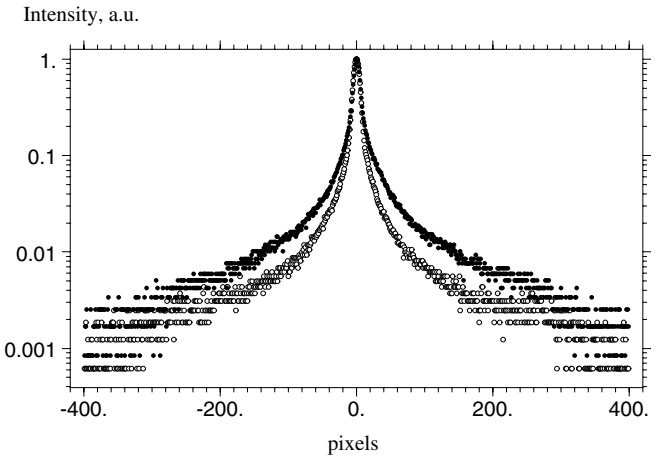

Figure 4. Intensity distribution perpendicular to the diffraction plane for the 25 μm thick SOI layer (solid circles); experimentally measured point spread function is shown for comparison (empty circles).

profile, which is most likely the result of a superposition of elastic strain fields from areas with opposite bending due to the flatness non-uniformity of bonded surfaces. The technique can be applied to study confined layered crystalline structures such as quantum wells, quantum dots and other optoelectronic structures. We found that using a circular focusing optic in this setup offers a new approach to recording incoherent scattering in a single shot simultaneously for all scattering angles. Future work will be directed towards expanding this technique to other focusing optics and increasing its depth sensitivity.

Acknowledgments

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