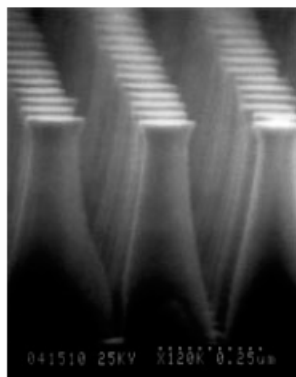


Probing surface grating structures of 300 nm periods with 1 Å X-rays

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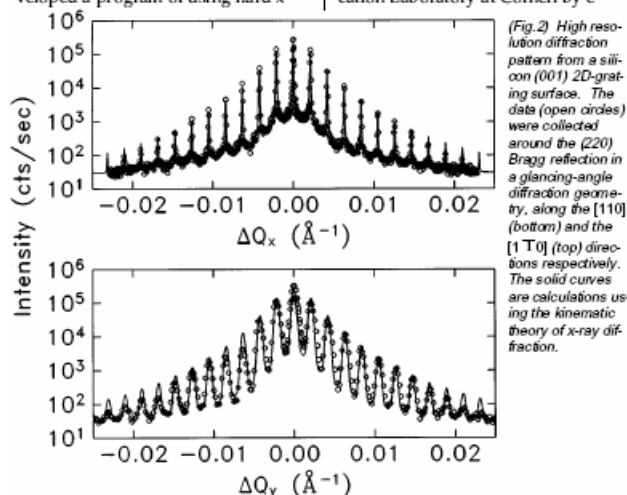
Periodic nanostructures of sizes 1 to 100 nm on semiconductor crystal surfaces have attracted great interests in recent years because of their unusual electronic band structures due to quantum confinements, and their potential applications in electronic and optoelectronic devices. The periodically structured grating surfaces also serve as model rough surfaces with a predominant spatial frequency for studying atomic kinetics involving surface diffusion and step arrangements. To date, optical diffraction using lasers and scanning electron microscopy have been the primary methods of characterizing surface grating structures. Compared to these methods x-ray scattering is a natural extension of optical diffraction for probing shorter wavelength gratings with higher spatial resolution.

In collaboration with Professor J.M. Blakely's group in the Department of Materials Science & Engineering here at Cornell, we have developed a program of using hard x-



(Fig. 1) A scanning electron micrograph of a 2D grating structure on Si (001) surface.

ray diffraction to study surface grating nanostructures and their evolutions when being annealed or etched. Two-dimensional (2D) grating structures (Fig.1) on Si (001) surfaces are fabricated at the National Nanofabrication Laboratory at Cornell by e-



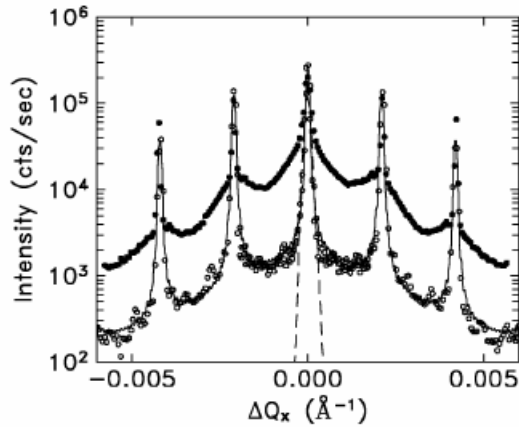
(Fig.2) High resolution diffraction pattern from a silicon (001) 2D-grating surface. The data (open circles) were collected around the (220) Bragg reflection in a glancing-angle diffraction geometry, along the [110] (bottom) and the [110] (top) directions respectively. The solid curves are calculations using the kinematic theory of x-ray diffraction.

beam lithographic techniques, and the x-ray experiments are performed at the F3 and the B2 stations at CHESS. Because of the long coherence length available with synchrotron radiation and perfect crystal monochromators, extremely sharp diffraction satellite peaks are observed around each bulk Bragg reflection. An example of such a diffraction pattern is shown in Fig.2. These satellite peaks are the result of interference due to the extra grating periodicity on top of the existing crystal lattice. In the language of crystallography, these grating pillars constitute "super molecules" that form a super lattice on the surface. The data illustrate that a mesoscopic scale (~3000 Å) periodic structure can be investigated in a straightforward way by diffraction of 1 Å x-rays!

The x-ray diffraction pattern from crystal surface gratings can be analyzed in analogy with optical Fraunhofer diffraction from multiple slits [see Shen, Umbach, Weselak & Blakely, Phys. Rev. B 48, 17967 (1993)].

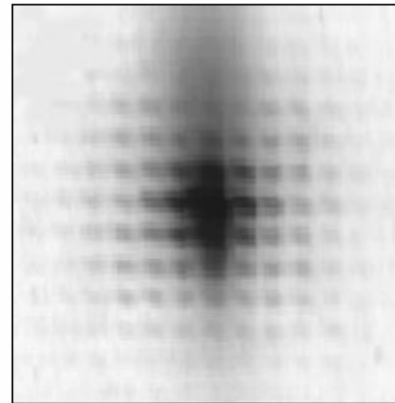
The grating peak positions are determined by the grating wavelength and their intensities are modulated by the diffraction intensity from a single grating period. By analyzing these quantities various grating parameters can be deduced. The intensity variations along a grating rod normal to the substrate surface provide information on the height of the grating pillars and their side wall slopes and shape. By comparing the grating reciprocal lattice with the crystal lattice, the orientation of the grating lines and their atomic registry can be obtained with respect to the bulk crystal.

One of the unique abilities of x-ray diffraction is to measure lattice strain, defects, and disorder on an atomic scale, and unevenness in the size of the grating pillars. These crystal and grating imperfections are



(Fig.3, left) Comparison of diffraction profiles from two different grating surface treatments. The data from a virgin sample are shown by open circles, which are part of Fig.2, and those from an oxidize-and-etched sample (needle-grating) are shown by filled circles. Note the line shape of the grating diffraction peaks, which contains information on the imperfections and the possible short-range-order in the grating pillar structures. The dashed line illustrates the experimental resolution, taken on a perfect crystal Si wafer.

(Fig.4, below) Laue diffraction image using white beam from a 2D Si (001) grating surface. The image is around the (004) Bragg reflection and is recorded on a Type 55 Polaroid film with a 3 min exposure time. The film to sample distance is 80 cm and the image border in the plot corresponds to roughly 5 mm x 5 mm in real scale.



important to the understanding of the physics in quantum confinements and to the growths of semiconductor overlayer structures. Figure 3 illustrates that these imperfections can produce interesting and distinct diffraction profiles (filled circles) on the grating reflections. In this study, the sample shown in Fig.1 was oxidized in a controlled fashion to grow SiO_2 on the pillar surfaces. The oxide was then removed by a subsequent HF etch. The treatment produced extremely sharp needle-like pillars, which may therefore contain some variation in their sizes, and some intrinsic lattice strain and short range order. An analysis of the x-ray diffraction data on the diffuse scattering

part will provide valuable information on these imperfections in the grating pillar structure.

The diffraction data from the surface grating structures can also be collected using a highly collimated polychromatic synchrotron beam, just like taking a Laue diffraction photograph. An example is shown in Fig.4, which is a magnified diffraction spot of the Si (004) Bragg reflection recorded on an x-ray film. With this technique a grating diffraction pattern of more than 100 peaks can be collected in just 2-200 seconds. We believe that the time

resolution can be further enhanced by using a CCD detector, and therefore Laue white beam diffraction is a promising technique for doing real time-resolved diffraction experiments on periodic surface grating structures.