

# FAST FIND FOR POLYMER GRATING ROUGH EDGES

Gratings are used to filter particles with dimensions from millimeters to micrometers; they are also used to split and diffract light in scientific instruments and security “holograms” on credit cards and products. Gratings are even used to lay out the basic nanoscale patterns for making the integrated circuits at the heart of every modern electrical device, from smart phone to smart refrigerator. Fitting more circuits in these devices requires high-fidelity gratings with smaller separations, even below 10 nanometers. A new approach to studying the imperfections in such gratings could be used to make the smoother and better at their job. That is the goal of new research carried out at the APS. The results point to a tried-and-true synchrotron x-ray research technique as a potentially effective tool for examining the surfaces and internal structures of gratings down to the nanoscale level.

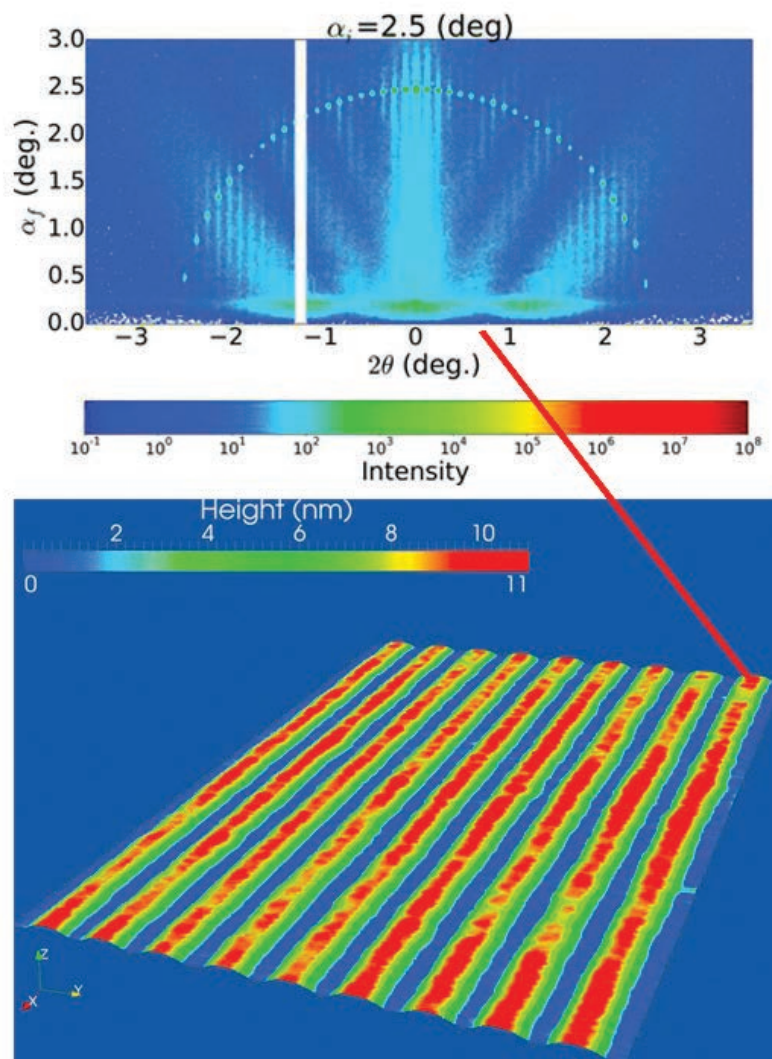
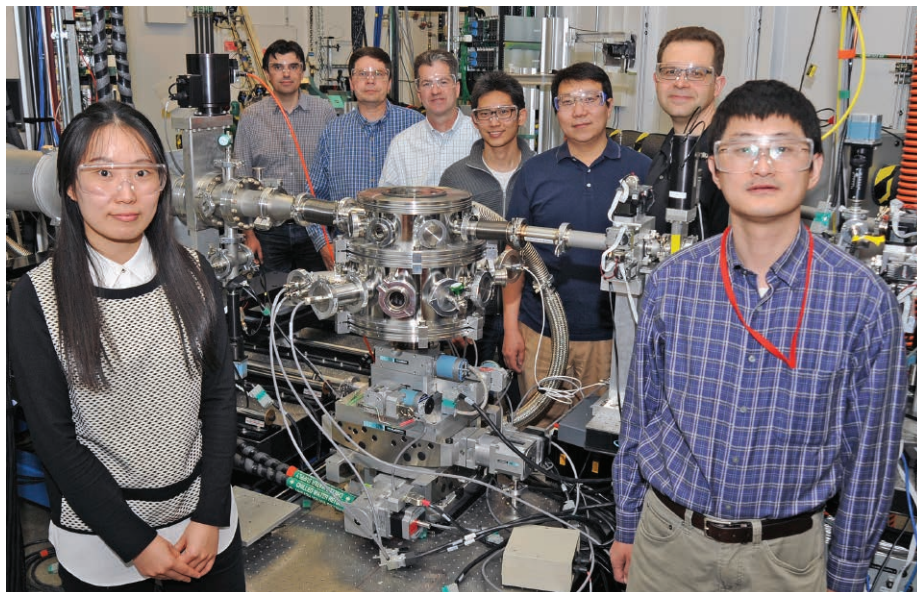


Fig. 1. GISAXS measurements probe quantitatively the fidelity of nanometer-sized polystyrene gratings. From H. S. Suh et al., *J. Appl. Cryst.* **49**, 823 (2016). © International Union of Crystallography

Characterizing imperfections at very small length scales in gratings used in a wide variety of technological applications is a challenging problem and one that is absolutely critical to many areas of science. Modern research in filtering processes, scientific measurements, and chip manufacturing could all be improved by a clearer understanding of gratings. Researchers from The University of Chicago and Argonne used grazing-incidence small-angle x-ray scattering (GISAXS) to examine polymer gratings manufactured by IMEC (Belgium). The researchers found that GISAXS can reveal flaws efficiently and even more accurately than state-of-the-art atomic force microscopy and so allow manufacturers to implement new schemes for quality control, and design new and improved products.

Polymer gratings used in a wide range of applications consist of regularly spaced arrays of shapes arranged in parallel. These patterns can have functional detail at a wide range of lengths, from visible spacing to microscopic and then nanoscale features. Checking that they are made with precision and suitable for a given job can be done with a microscope. Specifically, for the fine gratings used in scientific instrumentation, scanning electron microscopy, transmission electron microscopy, atomic force microscopy, and variations and combinations of those techniques can be used. These sophisticated techniques are costly to employ as well as suffering from a frustrating drawback: They can only examine a tiny area of any given grating at a time (Fig. 1). This limitation of microscopy means that the time required to check a complete grating can be prohibitive. In contrast, there are several x-ray techniques—such as x-ray scattering—that can probe with nanoscopic detail a large surface area very quickly at high resolution. Indeed, various x-ray scattering techniques have been used over the last decade or more to examine gratings. However, even these techniques have limitations.

But the reflection-mode technique GISAXS involves exposing the surface of the sample to a focused synchrotron x-ray beam at a grazing incidence angle rather than perpendicular. The large area of a sample in this setup dramati-



Several of the study's co-authors shown in the beamline 8-ID-E research station with the sample environment that was developed for this project under a partner-user proposal led by Paul Nealey and Wei Chen. From left to right: Xuanxuan Chen, Manolis Doxastakis, Mike Fisher (XSD, the sample chamber designer), Paul Nealey, Hyo Seon Suh, Wei Chen, Joseph Strzalka, and Zhang Jiang. Not pictured are co-authors Paulina A. Rincon-Delgadillo, Jin Wang, Roel Gronheid, Juan J. de Pablo, and Nicola Ferrier. This multi-sample vacuum environment has streamlined operations and is now used practically daily, by at least 75% of the 8-ID-E grazing-incidence scattering users.

cally enhances the intensity of the signal produced, which means measurements can be obtained very quickly compared to all other approaches. The technique also has the major benefit of extracting complete three-dimensional (3-D) information from the sample in a single step and requires none of the post-processing needed by other techniques to reconstruct the surface details in 3-D from what is essentially a two-dimensional scan.

A systematic study of GISAXS data acquired for polymer grating structures using different grazing angles at XSD beamline 8-ID-E at the APS shows just how well this technique works on a cross-linked polystyrene (xPS) nanoscale grating. Comparison with the much slower atomic force microscopy images from the same grating are coincident in the details they reveal, for instance showing the rough edges. This, the researchers suggest, points to GISAXS being a potentially useful technique for examining surface details of polymer gratings and even the internal structure of the components of the grating, which are all extracted in 3-D by the technique. — *David Bradley*

See: Hyo Seon Suh<sup>1,2</sup>, Xuanxuan

Chen<sup>1</sup>, Paulina A. Rincon-Delgadillo<sup>3</sup>, Zhang Jiang<sup>2</sup>, Joseph Strzalka<sup>2</sup>, Jin Wang<sup>2</sup>, Wei Chen<sup>1,2</sup>, Roel Gronheid<sup>3</sup>, Juan J. de Pablo<sup>1,2</sup>, Nicola Ferrier<sup>1,2</sup>, Manolis Doxastakis<sup>2\*</sup>, and Paul F. Nealey<sup>1,2\*\*</sup>, "Characterization of the shape and line-edge roughness of polymer gratings with grazing incidence small-angle X-ray scattering and atomic force microscopy," *J. Appl. Cryst.* **49**, 823 (2016).

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*Author affiliations:* <sup>1</sup>The University of Chicago, <sup>2</sup>Argonne National Laboratory, <sup>3</sup>IMEC

*Correspondence:* \* edoxasta@utk.edu  
\*\* nealey@uchicago.edu

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8-ID-E • XSD • Materials science, polymer science, physics • Grazing incidence small-angle scattering, x-ray photon correlation spectroscopy • 7.35-7.35 keV, 11-11 keV • On-site • Accepting general users •