X-Ray Compatible Cell for Liquids and Gases

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The development of advanced X-ray instrumentation provides transformative opportunities for investigation of phenomena at solid-liquid interfaces and many other dynamic processes at the frontiers of materials science. In recent years, there have been many experiments utilizing Polarized Resonant Soft X-ray Scattering (PRSoXS) to gain information on the molecular orientation in solid films. [1, 2] While very successful for solid films, this promising technique has largely been out of reach for applications such as biological molecules, structural nanocomposites, and liquid crystals. These applications typically require liquids or gas environments which create complexities for vacuum and soft X-ray compatible liquid cells.

Commercial devices are available consisting of two chips sealed together with silicon nitride membranes forming the liquid cell. However, their membranes either bulge out, causing too much absorption, or they collapse, resulting in insufficient scattering. In both cases, the actual volume of liquid being probed is unknown, making quantitative measurements impossible. An alternative solution is required to create a robust platform to enable PRSoXS for liquids and gases.

Our solution is to use a single chip with two nitride membranes supported with pillars. Membrane thickness of 50 nm with regularly spaced pillar supports result in membrane deflections in the range of 50 nm to 100nm for pressures ranging from 20 MPa to 100 MPa [3]. However, the regular spacing of the pillars leads to a strong scattering signal which can obscure the signal from the sample (Figure 1a). If we randomize the pillar spacing, shape, and orientation independently, the resulting diffraction pattern is effectively that of an amorphous material, comprising a few diffuse concentric rings as shown in figure 1b for a single membrane. To further mitigate such effects, we decreased the overall pillar density and increased the variation in both the size and shape of the pillars. Figure 1c shows the data from two connected membranes supported by the new random pillar design. This design will be used for real liquid samples. We observe an essentially featureless scattering pattern where any structure factor features are absent. The randomized pillar supported double membrane will make the data analysis more straightforward and will help improve the robustness of the devices.

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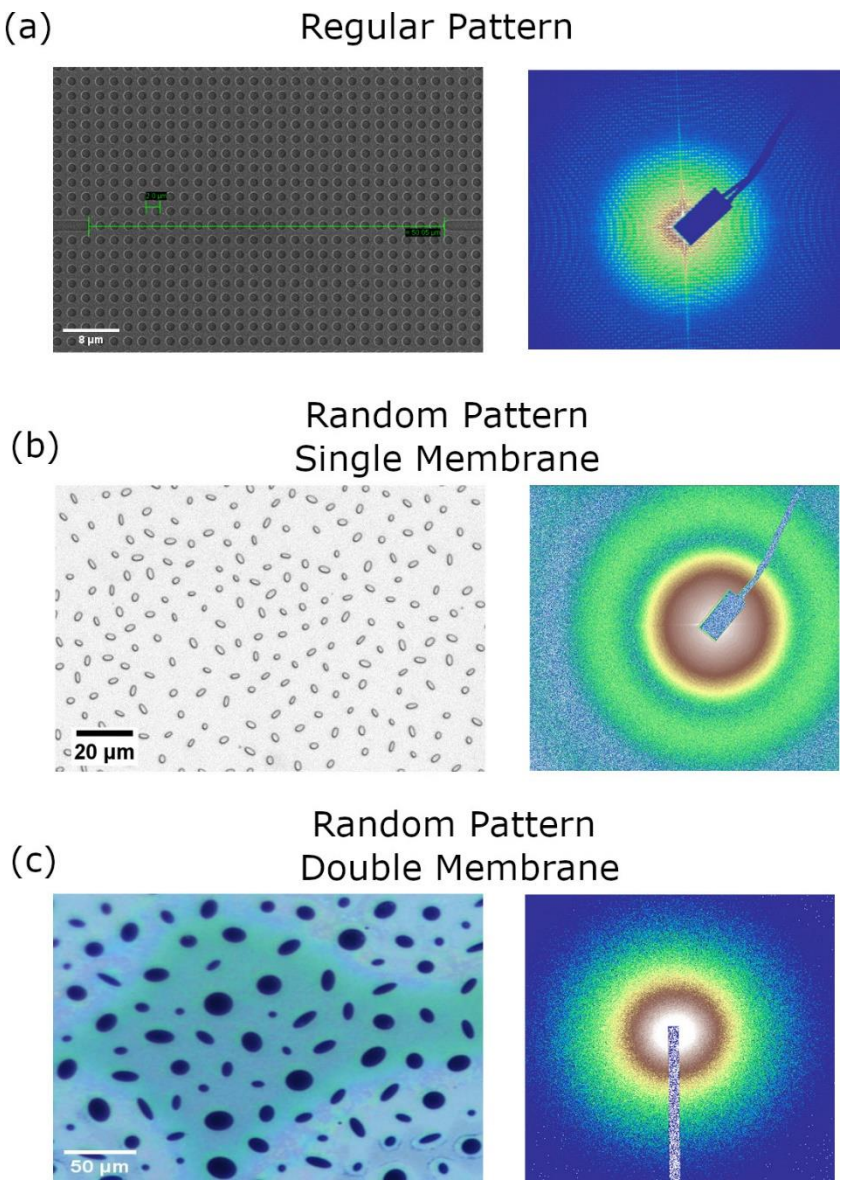

Figure 1 – Micrographs and the corresponding raw scattering data of the (a) regular pillar pattern, (b) random pillar pattern on a single membrane, and (c) enhanced random pillar pattern in a double membrane structure. The features are reduced to concentric rings (b) with the random pattern and are further reduced (c) by decreasing the overall pillar density and increasing the variation is both the size and shape of the pillars.