Design Requirements for X-Ray Compatible Liquid Cell

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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. Polarized Resonant Soft X-ray Scattering (PRSoXS) is sensitive to orientation and structure of organic as well as organic-inorganic composite systems. The sensitivity of resonant polarized X-rays to molecular orientation is now well understood and utilized frequently in Near Edge X-ray Absorption Fine Structure (NEXAFS) measurements. Transition dipole moments from core electron promotion into molecular orbitals make the interaction of X-rays resonant with these transitions highly anisotropic. In recent years, there have been many experiments utilizing this effect to gain information on the molecular orientation in solid films of materials [1, 2]. However, no direct quantitative morphological information has been obtained in case of liquid or gas environments that will enable resonant scattering studies on a variety of systems such as biological molecules, structural nanocomposites, and liquid crystals. This is largely due to the complexities in designing vacuum and soft X-ray compatible liquid cells.

The requirements for a PRSoXS compatible liquid cell are: (1) The thickness of the membranes of the liquid cell should be optimized such that they are robust but still reduce their absorption. (2) The scattering volume of the cells should be characterizable. (3) The cell should be able to maintain a positive pressure inside the liquid cavity. Commercial devices are available consisting of two chips sealed together with silicon nitride membranes forming the liquid cell. However, they do not meet all the requirements for PRSoXS. Their membranes either bulge out, causing too much absorption or they collapse, which can create issues in knowing the actual volume and can also result in not enough scattering.

Our solution is to use a single chip with two nitride membranes supported with pillars. It has been shown that using regularly spaced pillars with membrane thickness of 50 nm will result in membrane deflections in the range of 50 nm to 100nm for pressures ranging from 20 MPa to 100 MPa [3]. However, attempts to use regularly spaced pillars resulted in unacceptably large scattering amplitudes (Figure 1a). Here we present a new design approach, in which pillar spacing, shape, wall angle, and orientation are all independently randomized. The random pattern reduces the scattering intensity by an order of magnitude as shown in figure 1b and greatly simplifies its structure, making data analysis more straightforward.

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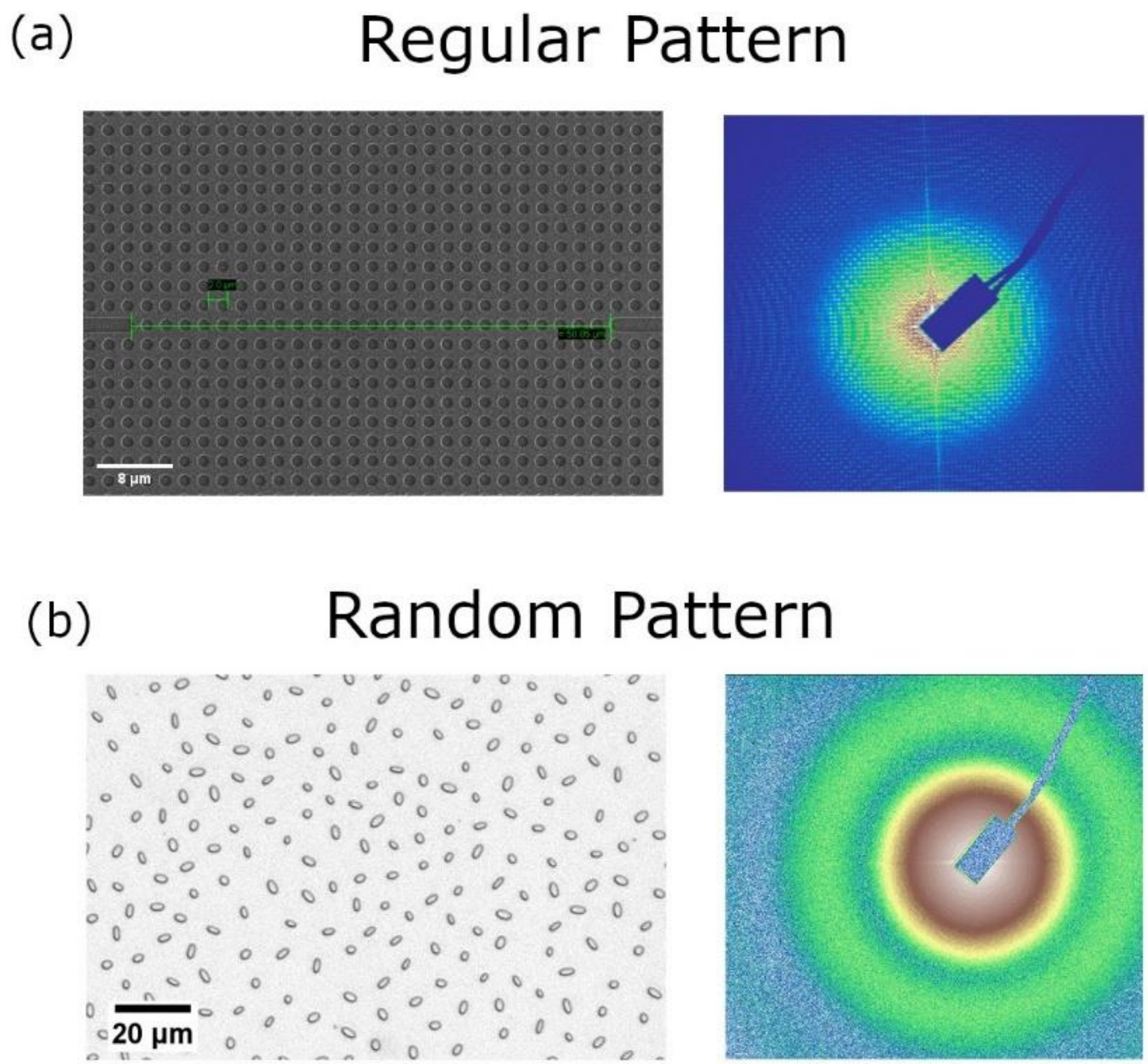

Figure 1 – Micrograph and the corresponding raw scattering data of the (a) regular pillar pattern and the (b) random pillar pattern. The scattering intensity is reduced by an order of magnitude with the random pattern.