ESRF	**Experiment title:** Probing Tobacco Mosaic Virus Fibrillation by X-ray Nanodiffraction	**Experiment number**: LS-2298
Beamline:	**Date of experiment**: ID13 from:12.4.2014 to:14.4.2014	**Date of report**: 25.8.2014
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Report:

Experiments were performed using a solution with 405 mg/ml tobacco mosaic virus (TMV) particles in 1 mM EDTA solution + 0.1% azide, pH 7.2. The solution was diluted by factor 100 by deionized water. The deposited droplet volume was set to ~4 μL.

We deposited ~4 μL droplets by a manual pipette on a superhydrophobic chip based on a thin Si_3N_4 membrane and a SU-8 pillars pattern. Both periodic and non-periodic pillar pattern were tested; the latter allowing a precise droplet localization. The highly X-ray transparent chips had been developed in collaboration with the IIT-Genova for probing ultrasmall sample quantities by X-ray scattering techniques.[1] (Figure 1)

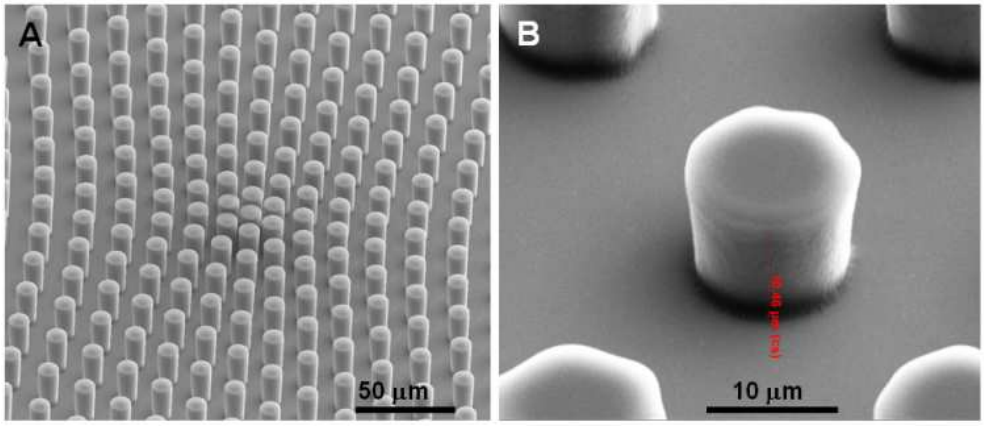

Fig.1 **A**: Scanning electron microscopy (SEM) image of central part of non-periodic SU-8 pillar lattice on Si_3N_4 substrate. **B**: Single SU-8 pillar of ~10 μm height.

We used a λ=0.08321 nm monochromatic X-ray beam, focused to a ~170(h)x130(v) nm^2 spot at the sample position. Diffraction experiments were performed in transmission geometry with the beam normal to the substrate. Data were collected by a Frelon CCD detector. Background scattering from the substrate was very small and did not contribute discrete peaks or short-range order to the XRD patterns.

We observed the formation of a coffee-ring type residue. [1] (Fig.2A) Raster-diffraction probing in transmission geometry revealed the presence of the hexagonal primitive TMV lattice.[2] A composite diffraction image with pixels corresponding to the strongest reflections of the TMV structure is shown in Fig.2B. An individual pattern is shown in Fig.2C. A zoom into the coffee-ring rim reveals a parallel orientation of the TMV nanorod axes to the rim as already observed on a wetting substrate.[2] We observe, however, local variations of the patterns depending on the angle of incidence as the residue layer is not flat. A more detailed analysis of the patterns is in progress.

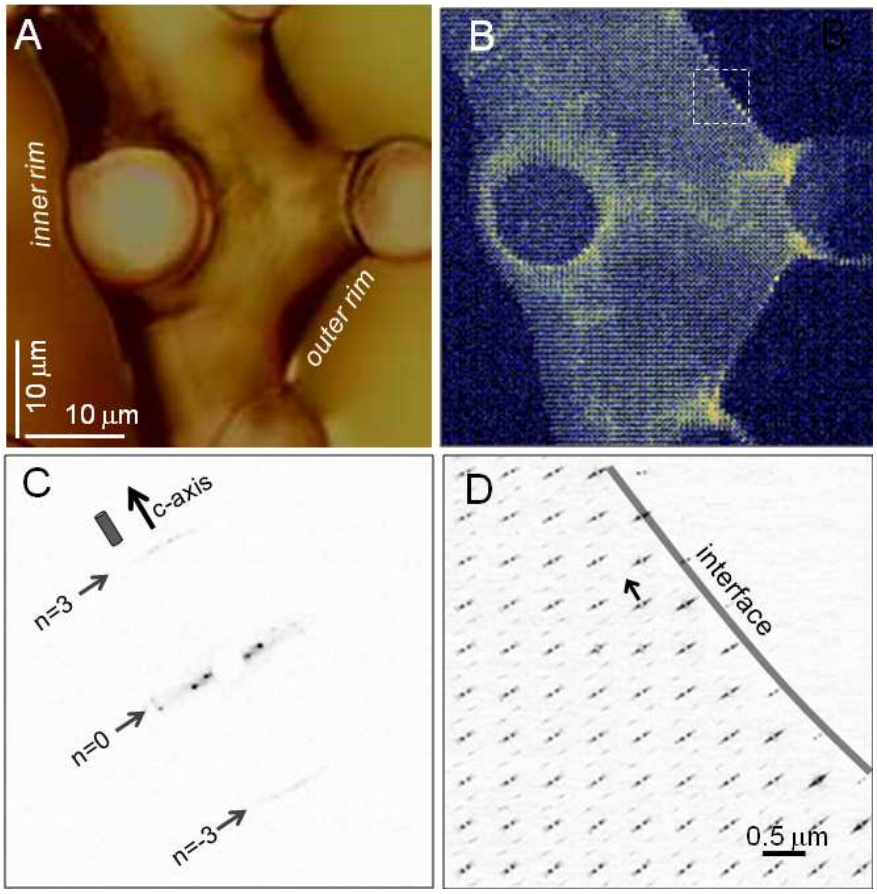

Fig. 2 **A**: Optical image of TMV residue on periodic pillared superhydrophobic chip. **B**: Composite diffraction image based on an 81x81 "pixels" mesh scan with 0.5 µm step size. **C**: Single pattern from outer-rim interface. The positions of equator (n=0) and n=3/-3 layer lines are indicated. The c-axis direction (arrow) corresponds also the direction of the long axis of the schematically depicted TMV nanorod (~300 nm long, ~18 nm diameter). **D**: Composite diffraction image for dashed square in (B) with schematic interface line. The upper display-range is limited to the n=3/-3 layer-lines although scattering from the n=6/-6 layers was also observed. The orientation of the local c-axis, corresponding to the TMV nanorod-axis, is indicated by an arrow. [1]

References

1. Marinaro, G., et al., *A superhydrophobic chip based on SU-8 photoresist pillars suspended on a silicon nitride membrane.* Lab on a Chip, 2014. Advance Article; **DOI:** 10.1039/C4LC00750F
2. Gebhardt, R., et al., *Virus particle assembly into crystalline domains enabled by the coffee ring effect.* Soft Matter, 2014. 10: p. 5458-5462.