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Supplementary material – Xurography for microfluidics on a reactive solid†

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Practical advises for the interferometer operator

On the Veeco NT1100 interferometer, two light passband filters are available for the PSI mode: "High Mag" filter (orange light, band of $\Delta\lambda = 40$ nm centered on the wavelength $\lambda = 605$ nm) and "Low Mag" filter (red light, band of 40 nm centered in 630 nm). The High Mag filter is chosen in this work due to the need of high enough intensity. The in situ measurements are done with objective x2, and field of view (FOV) x1.

The fringes have a very low contrast so they might be difficult to find, especially if the horizontal position of the mirror in the TTM is not correctly adjusted. First the TTM mirror position should be adjusted (using the small knob on the TTM casing) by doing a calibration on a standard mirror without compensation glass. When the TTM mirror is correctly located the fringes are visible at best focus.

Calcite is initially flat, but the different areas of the cell are visible due to different reflection coefficients on glue, photoresist and air. Birefringence of calcite both in the sample and reference arms, and the geometry of the chip itself results in multireflections in the chip. The light beam propagating perpendicular to the channel area images three different real interfaces, depending on the focus: i) plexiglas – water, ii) water – calcite, iii) calcite – air. Both i) and ii) interfaces are in principle visible twice when the orientation of the orientation of optical axis of the calcite differs from that of the light beam. In practice they are also additional multireflections of the beam in the microfluidic cell and/or the compensation window, resulting in additional virtual interfaces difficult to formally identify.

Before starting injecting water it is then necessary to investigate different focuses in order to identify a set of fringes that gives information on calcite dissolution and adjust at best the mirror position. This set is not unique. The fringes that show the calcite topography should initially be flat except at the photoresist emplacement. The fringes at the water – calcite interface should still be visible (however weak) when water flows, and deform consequently in the channel area. Due to change of reflectivity when water arrives, small readjustments may however be needed at that particular time. If focus readjustment is needed while water, precision of the measurement will most probably decrease. Confusion may happen with the fringes showing the Plexiglas® – water interface. Indeed the channel geometry can also be deduced from this interface since the difference of light paths depends on all the indexes and thickness of materials that transmitted light goes through [1]. In this case, the optical path difference is (simplified expression assuming no aberration) $\delta_w = 2(n_c d_{\text{ref}} + d_e - n_c d_s - n_w l_z)$ where d_e is the extra distance performed in the air in the reference arm by moving the mirror to compensate from the distance l_z in water in the sample arm. If the TTM mirror is re-adjusted so that $d_e = n_w l_z$, then this is equivalent to measuring at the water – calcite interface. To find the proper focus and refine the mirror position, one can also refer to the fringes in the tape area. Double-sides tape where the channel is cut consists actually of three layers: a discontinuous glue layer, a continuous PVC layer (carrier), and another glue layer. Outside the channel light reflection is in theory possible at each of the interfaces (plexiglas-glue, glue-PVC, PVC-glue, glue-calcite, calcite-air).

Detail of the topography data treatment

Here is the detail of the height analysis, obtained from the PSI measurements by white light interferometry.

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† Electronic Supplementary Information (ESI) available: Detail of the topography data treatment, and practical advises for the interferometer operator are available. See DOI: 10.1039/b000000x/

- Exclude unreliable measurements in time (ex: measurements while the focus was readjusted).
- Exclude at each time the point positions where the measurements are not enough steady up to this given time (see Fig. 5) i.e. compute a time dependent spatial mask where the analysis will be done. The analyze done at time t is done at the same pixels than at $t+\Delta t$ plus in additional pixels. Locations where NaN data are measured at t can be kept in the analysis providing that these points are steady measured before time t.
- Readjust the vertical 0 height reference for all data. Here it is done by taking the median values of valid data in the photoresist area – which is a well stable measured area along the time.
- Choose as reference data, measurements done at the beginning of the experiment (here: the reference is taken when air is in the channel), interpolate the data in space, and remove clear outliers on this picture.
- Correct the height signal of the phase incertitude using the equation:
  $h(t+1) = h_\phi(t+1) - \text{round}\left[\frac{h_\phi(t+1)-h(t)}{\lambda/(2n_c)}\right]\lambda/(2n_c)$.
  Interpolation of not at number (NaN) data is done using only data where the analysis should be done, using the time dependent mask previously defined.
- In the reference area where no dissolution is expected: an additional correction of the phase shift is done, using spatial and temporal continuity criteria, and a priori knowledge: we know that the depth of dissolution should be close to zero. Since noise may lead to a local height decrease, phase correction incertitude may still remain for few data.
- Correct the data from the spatial tilt measured in the reference area where no dissolution is observed (here this is done using data at the interface tape – calcite). This tilt is due to the focus readjustment and/or to small relative movements of the microfluidic cell compared to the mirror in the objective (for our setup we get a progressive deviation which ends up to $0.2\,\mu$m vertical shift).
- In the reference area where dissolution is expected: additional correction of the phase shift using spacial and temporal continuity criteria, and a priori knowledge (notably we know that the depth of dissolution increases with time)

The data analysis was run several times with various criteria of interpolation, average filter by sliding window, and different ways of taking the 0-height reference in each picture. Average filters spread NaN values and outliers, therefore they were finally not used in the retained data analysis. The code is written with the commercial software MATLAB [2].

References

1 J. Reed, M. Frank, J. J. Troke, J. Schmit, S. Han, M. a. Teitell and J. K. Gimzewski, *Nanotechnology*, 2008, **19**, 235101.

2 MATLAB, *version (R2014b)*, The MathWorks Inc., Natick, Massachusetts, 2014.