JOURNAL OF SYNCHROTRON RADIATION

Volume 27 (2020)

Supporting information for article:

The microfluidic laboratory at the synchrotron SOLEIL

Igor Chaussavoine, Anthony Beauvois, Tiphaine Mateo, Ramakrishna Vasireddi, Nadine Douri, Jordan Priam, Youssef Liatimi, Stéphane Lefrançois, Hervé Tabuteau, Mélanie Davranche, Delphine Vantelon, Thomas Bizien, Leonard, M. G. Chavas and Benedikt Lassalle-Kaiser

S1. µ-XRF and µ-XAS data collection

µ-XRF data were collected on the LUCIA beamline with a beam energy of 7300 eV, which was monochromatized with a double-crystal Si(111) monochromator. Fluorescence data were collected in back-scaterring geometry (45° incoming and outgoing angle) with a silicon drift diode Bruker detector. Fluorescence data were normalized to the intensity of the incoming beam, which was measured by current collection on an aluminium foil placed in the beam path upstream of the sample.

S2. Au NPs solution preparation

5 and 10 nm Au NPs were purchase from Sigma-Aldrich. The samples were diluted in citrate buffer 0.1 mM to obtain a 10 nM suspension of Au NPs (concentration obtained by UV-vis measurement). The two solutions were used within a week and gently centrifuged before injection to eliminate any aggregate and the resulting concentration was verified by UV-vis measurement. No decrease in concentration was observed.

S3. SAXS data collection

SAXS data were recorded at 12 KeV on the SWING(David & Perez, 2009) beamline of SOLEIL. The beam size was 25 x 25 µm² obtained using a Fresnel zone plate as focusing lense. Using a PCCD Aviex detector at 1 m from the sample, diffusion patterns were recorded for 1 second for reciprocal spacing $q = 4\pi \sin(\theta)/\lambda$ varying between 0.025 and 1 $Å^{-1}$, that is, repetitive distances $d = 2\pi/q$ ranging from 250 to 20 Å. 1D SAXS curves were obtained by circular averaging of the 2D images using the Foxtrot software. As no difference were observed between the 25 images recorded for each sample, the 1D curves were averaged in order to obtain a single curve for each sample and reference with better statistics.

S4. *In situ* SAXS data collection

In the PDMS chip, SAXS images were recorded along the channel every 500 µm. Sample and buffer where injected using the pressure-control system, with a steady flow rate of 10 $µl.min^{-1}$. 2D images were reduced to 1D curves and background was subtracted using the Foxtrot software (custom-made software). For the 3D-printed chip, all curves were similar along the liquid path, as no dilution was occurring. Curves were fitted using the Irina package,(Ilavsky & Jemian, 2009) giving NPs of 28.47±0.06 Å in radius (see figure S3).

S5. Lysozyme crystals preparation protocol

This protocol allows obtaining crystals of lysozyme with sizes ranging from 5 to 40 microns in the longest direction. Prepare first the following solutions:

• 120 mg/mL (w/v) Lysozyme (HEWL, Sigma-Aldrich) dissolved in 50 mM acetate buffer (pH 3.5)

• Mother liquor with: 1 M NaCl, 35% ethylen glycol, 12.5% PEG-3350, 50 mM acetate buffer (pH 3.5).

Cool both solutions in ice. Mix in a 1:3 ratio the lysozyme solution with the mother liquor solution (*e.g.* 1 mL lysozyme with 3 mL mother liquor). Vortex 2 min. After incubate at 4ºC for 24 hours, protein crystals appear with sizes ranging from 5 to 40 microns. Aliquots of this crystal suspension can be stored at -20ºC.

Table S1 Crystallographic data for lysozyme crystals. Values in parentheses are for the highest resolution shell.

Number of data merged	3	crystal 1	crystal 2	crystal 3
Resolution range (Å)	35.63-1.60 (1.64-1.60)	35.63-1.60 (1.64-1.60)	35.63-1.60 (1.64-1.60)	35.63-1.99 (2.11-1.99)
Unit-cell parameters (Å)				
$a = b$	79.67	79.67	79.68	79.77
c	37.90	37.90	37.92	37.79
Total reflections	119 489 (5 636)	55 360 (8 417)	32 770 (5 028)	28 987 (4 394)
Unique reflections	16 529 (1 196)	15 890 (2 502)	14 455 (2 084)	6 758 (1 100)
Multiplicity	3.7 (2.4)	3.5 (3.3)	2.2 (2.4)	4.2 (3.9)
Completeness (%)	99.6 (99.4)	95.5 (94.8)	86.8 (79.1)	76.6 (79.4)
Mean $I/$□	15.7 (1.3)	12.3 (1.2)	9.1 (1.5)	16.6 (7.1)
CC (1/2)	0.996 (0.641)	0.997 (0.667)	99.5 (77.0)	99.7 (97.1)
Wilson B factor (Å^2)	34.046	34.3	32.2	33.0
R_{merge}	0.071 (0.808)	0.059 (0.825)	0.063 (0.567)	0.058 (0.218)

Figure S1 3D scheme of the mixing chip used on the LUCIA beamline for *in situ* observation of Fe nanoparticles formation.

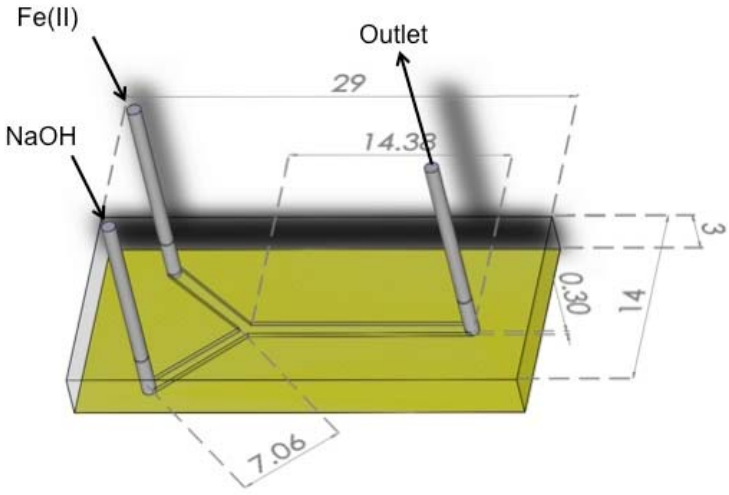

Figure S2 Schematic description of the fabrication process for the SAXS/WAXS PDMS chip reinforced with a 3D-printed backbone.

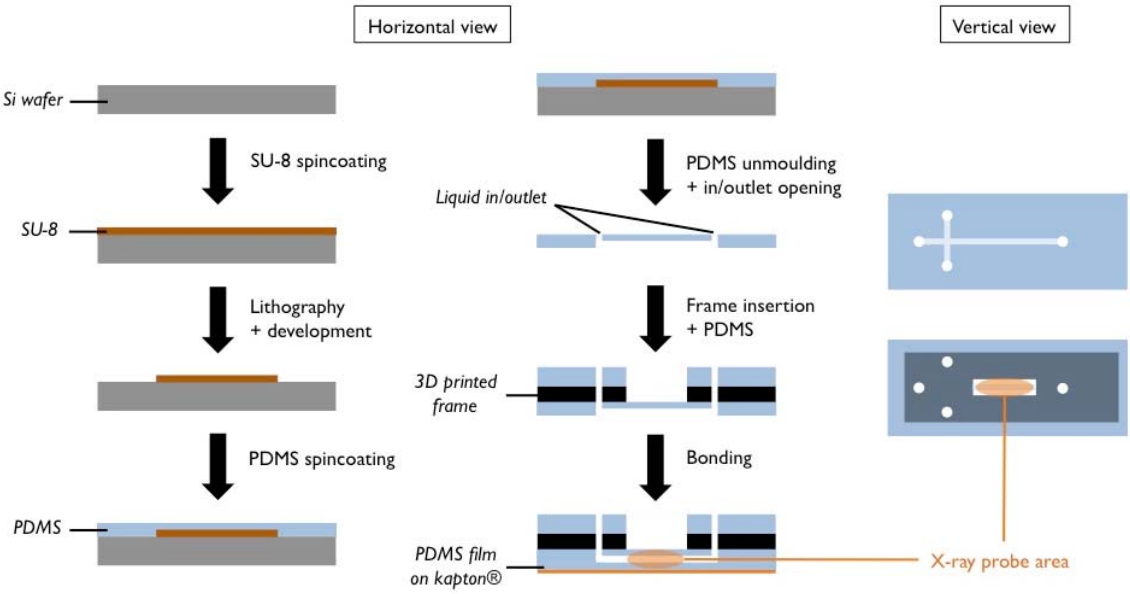

Figure S3 Pictures of the 3D-printed, single block flow-cell for *in situ* SAXS/WAXS data collection.

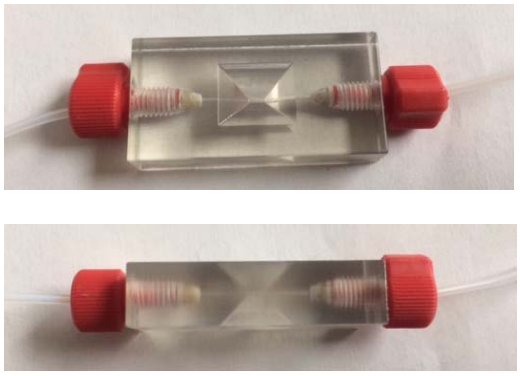

Figure S4 SAXS diffusion curves of Au NPs of 5 nm diameter in the 3D printed channel (left) and in the PDMS channel (right).

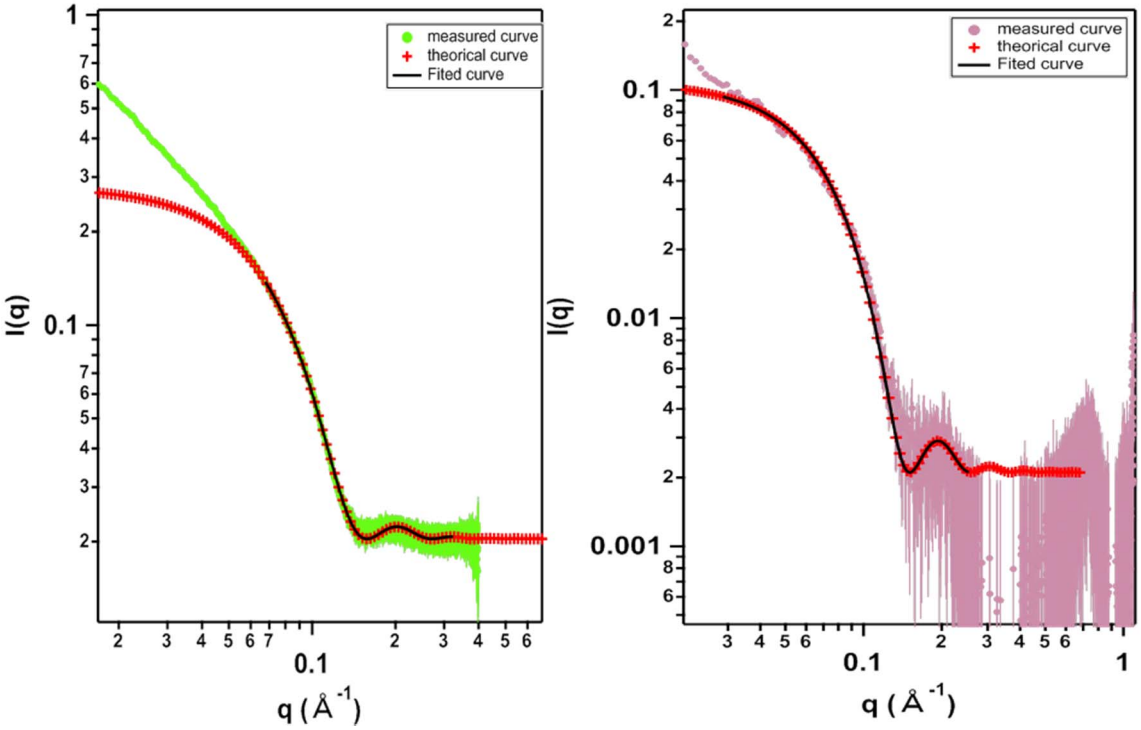

Figure S5 Schematic view of the microfluidic chip used to trap micro-crystals for *in situ* X-ray diffraction experiments.