JOURNAL OF
SYNCHROTRON
RADIATION

Volume 25 (2018)

Supporting information for article:

Flexible sample cell for real-time GISAXS, GIWAXS and XRR: design and construction

M. Berlinghof, C. Bär, D. Haas, F. Bertram, S. Langner, A. Osvet, A. Chumakov, J. Will, T. Schindler, T. Zech, C. J. Brabec and T. Unruh

S1 Technical Drawings of the In Situ Cell

In this chapter technical drawings of the in situ cell are presented (see figures S1 to S4), which should be viewed in conjunction with figure 1 of the main article. This chapter is aimed to help interested researchers to recreate the cell and to design experiments using it. In addition the technical drawings, CAD-files, and specifications for duplication of the cell can be provided.

(a) 3D sketch

(b) Front view

(c) Top view

(d) Side view

Figure S1: Technical drawings of the outside of the in situ cell. All drawings – except a) – are in the same scale and all dimensions are in millimeter.

Figure S2: Technical drawings of the bottom cover of the in situ cell. All drawings are in the same scale and all dimensions are in millimeter. The dashed structures are buried inside of the cover and the meander like structures are channels for the flow of the heating / cooling water. The light gray part in a) is a cover plate made of polyether ether ketone (PEEK) for thermal isolation and can be replaced by plates with different thicknesses to adapt the cell to spatial requirements of different beamlines and instruments.

Figure S3: Technical drawings of the sample stage inside of the in situ cell. All drawings are in the same scale and all dimensions are in millimeter. The dashed structures are buried inside of the sample stage and are channels for the flow of the heating / cooling water. The stage is made of copper and is covered with a glass-ceramics pane. In d) the connections for the heating / cooling water and the Pt100 thermistor are shown.

Figure S4: Technical drawings of the top cover of the in situ cell. All drawings are in the same scale and all dimensions are in millimeter. The dashed structures are buried inside of the cover and the meander like structures are channels for the flow of the heating / cooling water. The light gray part in a) is a cover plate made of polyvinyl chloride (PVC) for thermal isolation. The top cover can be exchanged with customized top covers possessing e.g. optical probes.

S2 Pictures of the Experimental Setup of the In Situ Sample Cell Installed at Different Instruments

This section gives an impression of the feasibility to use the cell at multiple instruments and beamlines (KOHZU, P08, PETRA III, DESY; ID10 EH1, ESRF; VAXSTER, ICSP, FAU, see figure S5).

(a) KOHZU diffractometer at the P08 beamline (PETRA III, DESY).

(b) ID10 EH1 beamline (ESRF).

(c) VAXSTER (ICSP, FAU).

Figure S5: Photographs of the in situ sample cell installed at different instruments.

S3 Temperature Homogeneity and Stability

To evaluate the temperature homogeneity and stability of the in situ cell, the cell's temperature was controlled using a water thermostat (F32-HE, Julabo GmbH, Seelbach, Germany) in conjunction with two Pt100 thermistors. One Pt100 thermistor is permanently installed inside of the sample stage (referred to as internal Pt100) and is connected with the temperature control loop of the thermostat. Two temperature maps along the sample stage were measured (see figure S6) using an additional Pt100 (external Pt100) which was fixed at multiple positions on the top of the sample stage using adhesive tape. For the first map, the cell was cooled down (temperature setpoint = 5.18°C). The average temperature along the sample stage was $4.51^{\circ}\text{C} \pm 0.02^{\circ}\text{C}$. A second map was measured at a temperature setpoint of 61.15°C . Here, the average temperature was $60.04^{\circ}\text{C} \pm 0.06^{\circ}\text{C}$. For both temperatures a remarkably high degree of homogeneity could be demonstrated. The small deviation from the temperatures measured with the internal and external Pt100 are due to the two different methods used to measure the resistance of the thermistors. The resistance of the internal Pt100 was measured via a two-wire-setup, whereas the external one used a four-wire-setup. Therefore, temperatures measured with the internal Pt100 are slightly offset to higher values. This effect can typically be neglected since the deviation from the real temperature value is only in the range of 1°C and since the reproducibility of the temperature control is not effected. The cell was heated to 60°C to analyze its temperature stability. Afterwards, the temperature was kept constant for half an hour. The temperature was continuously measured during both periods using the internal Pt100 and the two-wire-method (see figure S7). The average temperature after the initial stabilization time of 10 minutes was $60.001^{\circ}\text{C} \pm 0.007^{\circ}\text{C}$, which is below the precision of the thermostat. The temperature stability can thus be graded as magnificent.

a) Setpoint 5.18 °C

b) Setpoint 61.15 °C

Figure S6: Temperature maps of the sample stage of the in situ cell at two different temperature setpoints. The temperature maps were measured using a Pt100 thermistor which was connected to the top of the glass ceramic plate of the sample stage. The positions were measured from left to right and from top to bottom. To control the temperature a Pt100 thermistor installed inside of the sample stage was used (marked with a white asterisk).

Figure S7: Temperature stability of the in situ cell. The cell was heated to and kept constant at 60.0°C. The time after which the temperature is stabilized is arbitrarily set to 0 and is marked with a dashed line. The temperature was measured using a Pt100 thermistor installed inside of the copper sample stage (see figure S6).