UPGRADING A TRANSMISSION SAX/WAX BEAMLINE TO ALLOW HIGH QUALITY GISAX/GIWAX EXPERIMENTS FOR SOFT MATTER THIN FILM

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Abstract

The project required a sample environment to deliver experiments in vacuum or helium, with high humidity, including capacity to use aggressive solvents. The compact, transportable system incorporates a high precision in-vacuum manipulator/ positioning stage (with repeatability better than 1 µm/ 1 mdeg) allowing for multiple sample configurations. Current sample mounts include in-situ film formation (Doctor Blade), thermal annealing/drying heater stage, sample cooling and multiple sample stages; the system has been designed to accommodate many sample substrate formats. The existing end station camera system has been upgraded to include two, in-vacuum, WAXS and SAXS area detectors, which are custom builds based on the Pilatus 6M. The SAX detector module includes three in vacuum, independent ,configurable SAXS beam stop manipulators to block GISAXS transmitted, reflected and specular flare as well as isotropic and anisotropic SAX, a photon sensitive detector shutter plate is included. The 4 mm diameter tungsten beam stops each include a miniature photodiode to measure beam intensity and can be positioned to within 10 µm precision in X and Y over 300 mm x 250 mm motion range.

I22 EXISTING CAPABILITIES

I22 [1] is an insertion device beamline operating at diamond since 2007. The beamline (see Fig.1) is dedicated to Non Crystalline Diffraction including small angle and wide angle x-ray scattering. The beam transmitted through samples in bulk form. Small angle X-ray scattering (SAXS) is a technique that is used to probe the internal structure of condensed matter systems at the micron and nanometre length scales. The Wide Angle X-ray scattering (WAXS) technique measures length scales on the order of Angstroms, and is typically dominated by diffraction processes, suited to study the close chain packing of semi-crystalline polymer chains within polymer blend films. The end station consisted of a 1D WAX camera and a 2D SAX camera of variable length from 800 mm up to 10 m. A single fixed beam stop, with built-in diode, measures beam intensity and blocks the transmitted beam. This paper discusses the changes made and features added to add high quality GISAXS functionality to this beamline.

THE GISAXS UPGRADE

The scope of the project was to replace hardware and reuse existing infrastructure. This limited the work to a GISAXS sample environment and positioning system, and

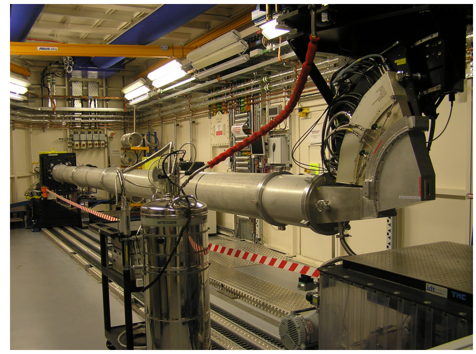

Figure 1: Original camera.

a new SAXS detector module to deliver the greater motion ranges required by GISAXS, with a new motorized beam stop module. The upgrade was combined with replacing the ageing 1D WAX detector with a 2D in vacuum WAX detector.

GISAXS Functionality

In transmission Small Angle X-ray Scattering (SAXS) the data is based around the axis of the transmitted beam with the elastic scattering pattern perpendicular to, and centred, on the axis of the primary beam. The technique is used to measure bulk samples where the interaction of the beam with the sample is determined by the size of the beam (≈300 µm × 100 µm) and the sample thickness which range from hundreds of microns to several millimetres. Grazing Incidence

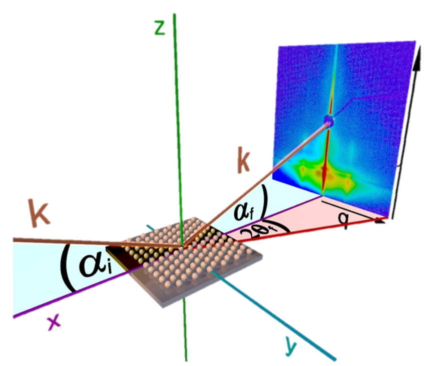

Figure 2: GISAXS theory,GISAXS Image courtesy of Dr Andreas Meyer [7].

Small Angle X-ray Scattering (GISAXS) is based on reflection geometry with the scattering pattern based around the reflected beam axis. It is intended to measure thin films cast on flat substrates. Typical film thicknesses are in the nanometer range and operating at the grazing incidence angles allows

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for increased length scales of beam/sample interaction, additionally other signals are generated that provide information about sample surface to environment and sample surface to substrate interfaces.

Sample System Design

It was chosen to design a specific, self-contained, GISAXS sample system. This has the capability of positioning GISAXS sample surfaces, oriented vertically (horizontal plane reflection) or horizontally (vertical plane reflection). The vertical orientation allows for in-situ dip cast films, the horizontal allows for ex-situ cast films and in-situ spread and spin coated samples. Sample environment is designed to allow for medium vacuum (1×10^{-4} mbar), a helium atmosphere or helium with humidity and helium with solvent vapour, the helium and vacuum being necessary to reduce parasitic scatter of the beam. To accommodate the vacuum function with such flexible sample arrangements it was felt necessary to enclose the entire system in a vacuum vessel. To prevent cross contamination between the sample manipulator stack and the sample volume the vessel was split into two chambers with a flexible motion feedthrough linking the two. The differential pressure between the two chambers is controlled to within 2 mbar by the pumping system, with pressure relief valves that vent the chambers if higher pressures (0.4 bar) occur. Note that system sat on the existing sample table, limiting the maximum mass of the system to 150 kg.

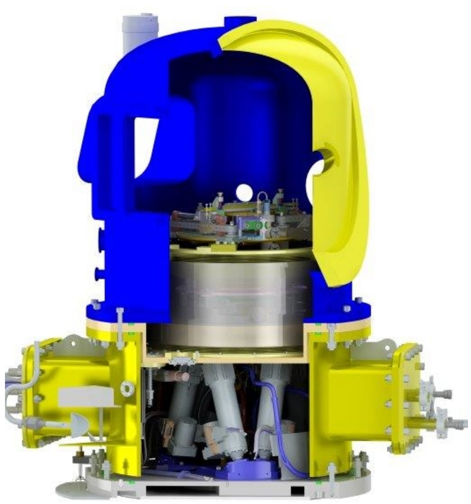

Figure 3: Sample System concept.

Motion Stack

For GISAXS measurement the sample must be positioned very precisely to the beam; once aligned, the sample has to be rotated about a point on its surface. The motion requirements are shown in table 1.

The entire sample manipulator stack is based on a custom built medium vacuum compatible (1×10^{-4} mbar) hexapod which is topped with a linear lateral stage which moves the sample surface across the beam to scan the surface in case of beam damage. The motion is transferred into the upper vessel via a support post. The flexibility for the six degrees of motion is provided by a pair of bellows mounted on bearings with rotary seals. At the top there is an interface for mounting various sample holder arrangements, including a breadboard.

Table 1: Motion Specifications

Translation	**range**	**resolution**	**repeatability**
Height	±20 mm	1.0 µm	0.5 µm
Lateral	±2.5 mm	1.0 µm	1.0 µm
Longitude	±2.5 mm	1.0 µm	1.0 µm
Lat.scan	±20 mm	1.0 µm	1.0 µm
Rotation	**range**	**resolution**	**repeatability**
Pitch	±140 mrad	17.4 µrad	17.4 µrad
Roll	±140 mrad	17.4 µrad	17.4 µrad
Yaw	±140 mrad	17.4 µrad	17.4 µrad

Hexapod was designed and built by Symetrie [4] delivering

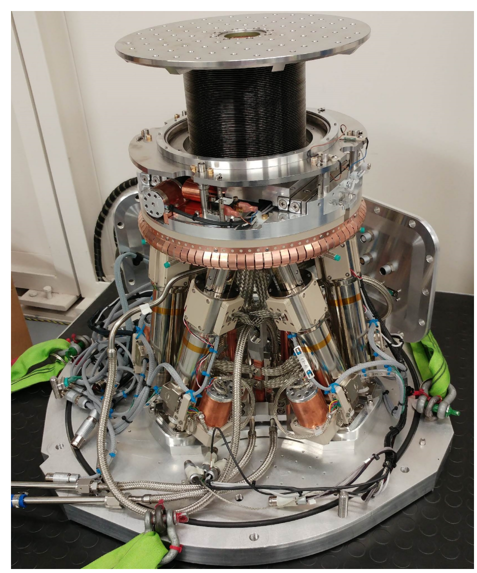

Figure 4: GISAXS ManipulatorStack.

sub-micron and millidegree precision at the sample position. The control system has two fixed machine coordinate systems plus two programmable coordinate systems, this allows the sample surface to align precisely to the beam and users to control the position and orientation at the sample surface relative to the beam. The additional linear stage placed on the moving table of the hexapod allows the sample surface to move laterally without losing alignment and maintaining the programmed coordinate systems.

Motion Stack Cooling

Temperature stability was addressed using invar for the hexapod legs and the sample support pillar, the hexapod drive motors are thermally isolated from the legs internal water cooling circuit, all seven motors and encoders plus each part of the support structure above the hexapod are thermally linked to the cooling system using copper braids or copper foil laminations. The cooling system goes on to a heat exchanger at the top of the sample manipulator stack in the upper vessel. Enabling a thermal anchor for the different sample mounts. Additionally the vessel baseplate

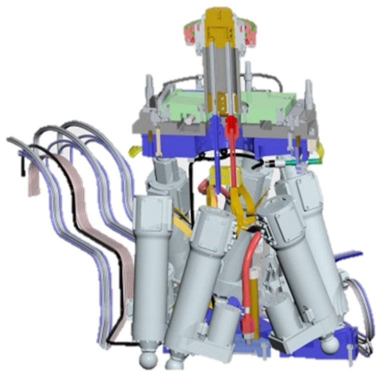

Figure 5: Manipulator Stack Cooling.

was designed carefully to avoid passing on vacuum forces to the manipulator stack by having a thin membrane to distort freely without disturbing the hexapod mountings

Sample Substrates

The sample substrate defines the sample quality; surface roughness causes diffuse scattering which affects the signal data significantly and inadequate flatness causes serious problems with alignment and contamination of data
Silicon wafers were specified for standard substrates as they are easily available with good flatness and surface roughness. Various formats are accommodated in the design, ranging from 25 mm rounds and squares, up to 100 mm rounds, with thickness ranging from 0.5 mm to 5.0 mm (some surface film coating techniques such as spin coating can lead to a thin substrate distorting as the film contracts). Additionally other materials are possible such as glass, although the critical angle of the material should be greater than that of the sample. In each case the substrate mounting must be designed to accurately locate the substrate without distorting it, and be simple to operate. Typically all the samples sit on flat surfaces with lateral position defined by two points of contact and a very light spring load. As well as substrates a sample alignment tool has been made to assist with initial beam alignment, it uses the same location features as the samples.

Sample Holder – Doctor Blade, Annealing Stage

The doctor blade annealing stage was designed to position the sample substrates in the beam and allow for fresh films to be applied in situ. The system uses spreader bar to cast the film onto the substrate, and can heating the substrate up to 250 °C to anneal or dry the sample. Substrates sit on an 85/15

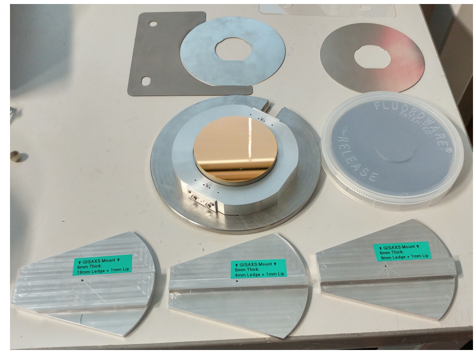

Figure 6: Sample holders and alignment tool.

tungsten copper plate, which combines high conductivity with stiffness, hardness and stability. The substrate mounting surface is lapped flat to 1 micron and Nickel plated. Beneath is clamped a 1.8 kW silicon nitride ceramic heater with a gas cooling matrix [5], the heater has excellent temperature uniformity and is larger than the maximum substrate size in order to keep the sample temperature uniform. The whole assembly sits on three height adjustable pillars topped with drilled Zirconia balls resting in Vee grooves, the interface point is located to neutralise heater plate expansion. The heater plate is very well insulated from baseplate by the balls (small contact area and low conductivity) this reduces the heat load to the internal cooling, and keeps the heat change in the support structure to a minimum. The baseplate assembly is thermally linked to manipulator stack cooling circuit. The Doctor Blade is a wire wound bar which spreads liquids into a uniform film across the substrate in a single stroke; a ramp lifts it clear of substrate at the end of stroke. The bar sits on a self-aligning carriage which runs along two stainless steel shafts using Iglide X bearing bushes. The Carriage is driven via a continuous stainless steel belt drive running around pulleys. A motor drive is encased in vacuum vessel to protect it from solvents and humidity and to prevent contamination of the sample volume.

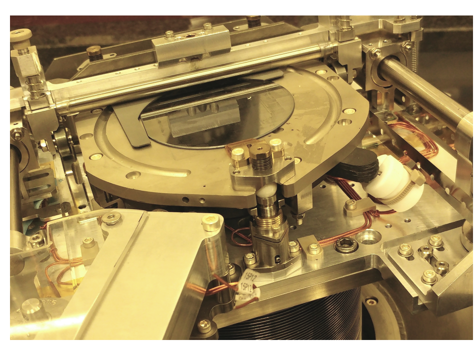

Figure 7: Dr Blade System.

IN-VACUUM AREA WAXS DETECTOR

The new WAX detector provides 2D WAX data, it is based on the Pilatus 2M [6] modified for use in vacuum (modules moved into a vacuum vessel, electronics remain in air). Additionally the detector has three modules removed to allow the SAXS data to pass through to the SAXS detector further downstream. The system is mounted to the original gantry

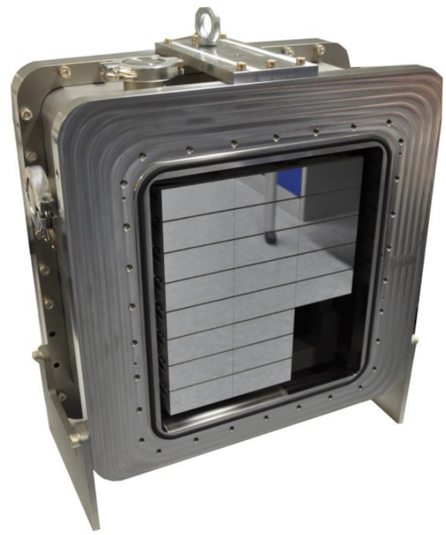

Figure 8: WAXS Detector [3].

system but has X and Y motions to position the detector in the appropriate position relative the data being collected. The rear of the detector interfaces to the SAX camera tube via a large gate valve (essential to maintain vacuum during camera length changes) and the bellows support by a gimbal which allows independent SAXS camera articulation. For GISAXS the detector is fitted with a large reinforced Kapton window [2] – otherwise there are various nosecone adapters for transmission SAXS/WAXS experiments.

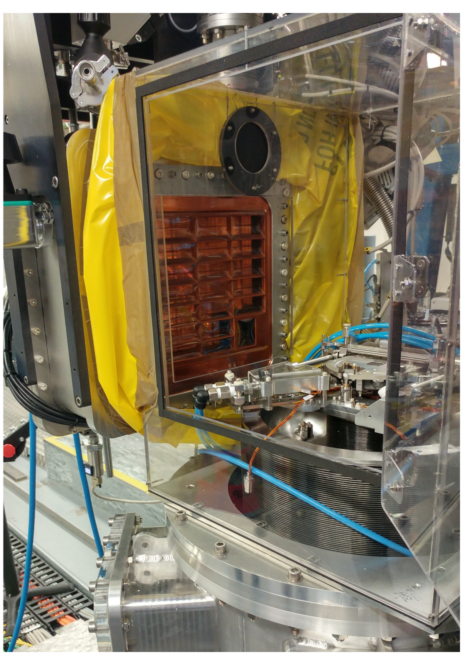

Figure 9: WAXS Detector in He.Trials.

GISAXS BEAMSTOPS

For the GISAX detector up to three beam stops are required to block transmitted beam and overspill , the reflected beam and specular flare, which would otherwise damage the detector. Each beam stop is required to be positioned at any point over the SAX detector area. The stops can have a silicon PIN diode fitted, or are isolated, shielded and connected to current amplifiers, so that photon/drain current can be measured. This generates useful transmitted beam intensity information that can be useful in data analysis and greatly assists beam stop alignment. the specular flare stop comprises a tungsten rod mounted on a rotary stage which allows for angular adjustment. The beam stop must be big enough to stop the transmitted beam and small enough to maximise the observed scattering intensity at very low angle, in this case the beam stops are 4 mm diameter10. Each tungsten beam stop is suspended by a combination of polyamide filaments and thin Kapton foils that are near-invisible on the detector and giving 360° of data collection. A pair of gold conductors connects the diode to the instrumentation.

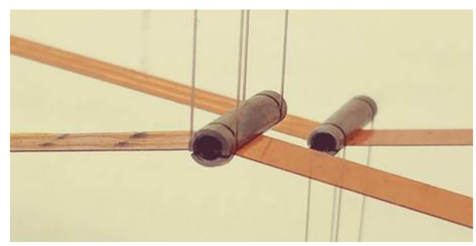

Figure 10: Beam Stops.

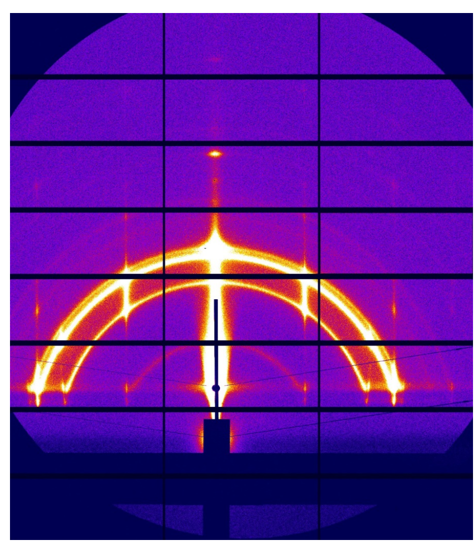

Figure 11: GISAXS image showing beam stop shadows.

Beamstop Vessel Assembly

The beam stops are mounted in module frames that locate inside a vacuum vessel (1×10^{-4} mbar) which is located in front of the SAX detector. There are three independent X Y stages inside the vessel that accommodate a module

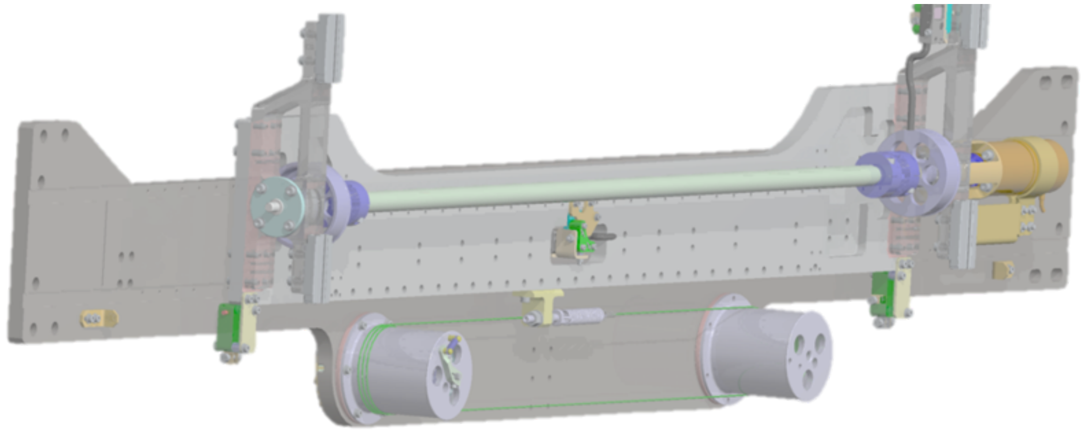

Figure 12: Beamstop actuator.

each. Each beam stop module can be loaded from outside the vessel through a rectangular flange. For GISAXS the beam stops include a large transmitted beam stop, a small reflected beam stop and rod specular reflection stop, for transmission SAXS only one beam stop is necessary. Also mounted on the vessel back plate is a photon shutter plate designed to isolate the SAXS detector face until the beam stops have been aligned. The plate is also configured to collect drain current, this signal is interlocked to prevent the shutter opening when the SAXS detector is in danger of damage. The shutter motion uses a crank slider mechanism actuated from outside the vessel using a pneumatic actuator via a magnetic rotary feed through.

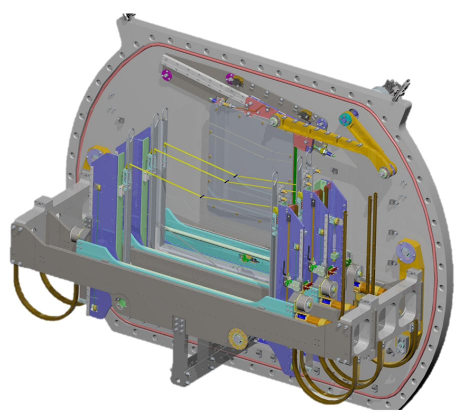

Figure 13: Internal mechanisms on vessel baseplate.

Beamstop Drives

The X Y drives have a range of 300 mm vertical by 252 mm horizontal to enable a beam stop to be positioned anywhere in front of the detector active area. The motions are actuated by capstan driven cable drives, where a preloaded drive cable is wound around a motorised cylindrical drum. The friction of the cable around a cylindrical drum T_2 is calculated using the capstan equation $T_2 = T_1 e^{\mu\beta}$ where T_1 is the tension in the cable, μ is the coefficient of friction and β is the total angle of cable contact.

For the X drive (horizontal) the cable attaches to the driven stage, runs 2.5 times around a driven drum then back around an idler drum before attached to the driven stage via a preload spring. For the Y drive (vertical) uses twin cable drive to prevent crabbing, the cables attach at the top and bottom of driven stage, with a spring preload and run 2.5 times around driven drums mounted on a shaft. See Figure 12 for arrangement. Each drive is equipped with linear encoders and a static low speed positioning accuracy of 0.01mm. However the low actuator stiffness limits the dynamic precision to around 0.1mm. These drives are low cost, light weight and compact and vacuum compatible.

SAXS DETECTOR TABLE

The beam stop vessel and Pilatus 2M SAX detector are all carried on a mobile support table. The table weighing 2600 kg runs along the floor under the power of a single driven wheel with very simple guidance this allows a SAX camera length (sample to detector distance) adjustment from 1.6 m minimum up to 10 m. The SAX table also allows 300 mm horizontal and 1000 mm vertical adjustment of the camera axis to align with the reflected beam from the sample. Additionally there are 3 linear motions to adjust the detector and added to the beam stops this amounts to 14 axes on this table. Instrumentation including photo diode amplifiers and remote I/O plus power cabling, compressed air and cooling water make for a complex and substantial cable management system.

CONCLUSION

The design project is complete and built with some beamline commissioning work to be done;

- GISAX sample environment assembled and tested for use in air and under helium, full vacuum operation function not completed.
- WAX Detector and system installed and fully commissioned and operational.
- SAX table installed and fully commissioned and operational.

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