

# ENGINEERING CHALLENGES ON THE I14 NANOPROBE BEAMLINE

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## Abstract

An overview of the double branch 185m I14 Nanoprobe beam-line under construction at DLS will be presented together with the end-station design in further detail. The end station consists of a split vacuum vessel containing a KB mirror configuration (at UHV) and the sample environment (at HV) which is just 50mm from the end of the final KB optic. An in-vacuum detector is mounted between the KB and the sample whilst two externally mounted detectors will operate between 0.25m & 3m from the sample. Four cryogenic samples can be brought into the vessel at a time and transferred remotely to the sample position with cooling provided by a Helium pulse tube cooler. With an initial 50nm size beam, stability is absolutely critical and careful attention has been paid in the design to mitigate any thermal and structural sources of vibration. An array of interferometers reference the KB mirrors and sample position and will be used to actively correct for any drifts. The very tight space constraints involved have greatly increased the complexity and duration of the design but testing of prototypes is now underway. The system is scheduled for build and test through the Autumn 2016.

## INTRODUCTION

The long Nanoprobe Beamline currently under construction and commissioning at Diamond Light Source features both a Nanoprobe & Mesoprobe branch. The Nanoprobe branch will be accepting first users in Spring 2017 whilst the Mesoprobe branch will be completed later in a 2<sup>nd</sup> build phase.

Each branch has a separate experiments hutch within the newly constructed external building, which also features a suite of Electron Microscopes laboratories.

Due to the arrangement of the Mirrors and Double Crystal Monochromator (DCM) it will be possible to deliver pink or mono beam to the end station of either branch.

## BEAMLINE CHALLENGES

The goal of the beamline is to provide a sub-50nm X-ray beam through which samples can be scanned in 2D and 3D under various conditions whilst collecting data from several detectors. In particular a nano-KB system was selected to achieve nano-focussing with a cryogenic system able to handle frozen hydrated samples for biological imaging. To achieve this we need:-

- Beam stability of < 50 nrad RMS before the nano-focus endstation and stability at the sample of <3 nm

- A KB mirror and sample stage system designed for flexible sample conditions in air or vacuum whilst maintaining the KB system under UHV at all times.
- Long term thermal stability to minimize thermal drift.
- A detector layout for multi-modal experiments.
- An interferometry system capable of sample rotation.
- Cryogenic cooling for 2D and 3D scanning with associated sample handling and sample staging for frozen hydrated samples.

## BEAMLINE LAYOUT

The beamline is 185m long to improve demagnification of the beam at the sample, whilst maintaining a reasonable working distance. This also provides a coherent beam for diffraction limited focussing and coherent imaging applications.

The beamline was designed for a cryogenic in-vacuum undulator, with the main optics installed inside two hutch- es within the main synchrotron building. The beam propagates through a long vacuum pipe to an external building some 90 m away from the main synchrotron which houses a third Optics Hutch together with the Nanoprobe and Mesoprobe Experiments concrete hutch- es.

### Hutch 1&2 Key Components

Optics Hutch 1 contains the primary optics including the mirrors and DCM.

Nano-focussing and coherence experiments require high stability and the beamline targeted < 50 nrad rms beam movement in both the vertical and horizontal direction from 1-100 Hz.

With the demands for vertical beam stability at the sample ultimately to within a few nanometres, the optical arrangement was carefully considered and an all horizontally deflecting geometry was used.

The Mirrors M1 (with bender) & M2 (flat) use stripes of Si, Rh and Pt to provide harmonic rejection and horizontal beam shaping with respect to the secondary source.

The horizontally deflecting monochromator is a fixed exit system using a pair of Si(111) crystals to provide continuous energies from 4.5 to 26 keV with much attention paid to the mechanism stiffness and arrangement of the cryogenic cooling pipework which has been proven to provide vibration free performance below 100 Hz and less than 50 nrad RMS up to 200 Hz.

A fast feedback system operating at 500Hz will be used to actively control the pitch and roll and suppress low frequency vibrations based on beam position measurements. As nano-probe experiments are flux hungry, a filtered pink beam option was considered by using mirror M3 (to be installed at a later date) with space also available to consider a multilayer monochromator.

As I14 is on a short straight at Diamond, only a single undulator can be used. However, swapping between endstations for the nano-probe or meso-probe will be achieved with the further future addition of M4 & M5 switching mirrors which would allow either mono or pink beam to be delivered to the Mesoprobe branch.

Whilst the DCM is cryo-cooled, the mirrors employ a gravity fed water cooling system to minimise vibration.

In Optics Hutch 2 at around 50 m from the source a double set of precision slits (S3) define a secondary source for each branch. Beyond this point each branch occupies its own vacuum space through to the external building.

At the end of Optics Hutch 2 each branch has its own independent photon shutter allowing beam to be maintained on the upstream optics, whilst the external hutches are accessed thus assisting thermal stability.

### *Beam Duct*

The Ø100 mm beam pipes to the external building are housed in a concrete beam duct which passes under two roads.

Due to the harsh environment and maintenance access difficulties there are no pumps in the duct although there are vacuum gauges to monitor pressure. Despite a distance of around 113 m between the pumped ends of we have achieved vacuum pressures in the region of  $1 \times 10^{-8}$  mBar at the midpoint.

### *External Building*

The 1500 m<sup>2</sup> footprint external building is a purpose built facility for the I14 Nanoprobe beamline and the complimentary Electron Bio-Imaging Centre (eBIC) and Electron Physical Sciences Imaging centre (ePSIC)

The beamline and electron microscopy (EM) laboratory floors are constructed on 800 mm thick concrete supported on 14 m concrete piles for stability in the same manner as the main Diamond synchrotron hall floor.

Excellent thermal management is essential to maintain stability of the sample. Consequently where forced air conditioned ventilation is traditionally used, we have opted for radiant panels mounted on the ceilings and walls to remove short term thermal oscillations and limit air pressure fluctuations.

Further to aid thermal stability, each experimental hutch is constructed from 300 mm thick concrete and is accessed via a vestibule with self-closing door to minimise the volume of air exchange when opening the hutch door.

### *Optics Hutch 3*

Optics hutch 3 serves to provide pumping at the external end of the beam duct and also the first external diagnostics. Sufficient space is provided for future installation of optics (such as Compound Refractive Lenses) on the Mesoprobe branch.

### *Experiments Hutch 1 (Mesoprobe)*

The Nanoprobe beam pipe is brought through the Mesoprobe Experiment Hutch inside a lead shielded box thus allowing access to the room whilst the Nanoprobe branch is taking beam. The Mesoprobe endstation is yet to be designed. The switching mirrors in Optics Hutch 1 together with the length of the beamline means that a 12 mrad deflection of the beam results in over 1.5 m separation of the beam pipes at this position.

### *Experiments Hutch 2 (Nanoprobe)*

A series of components on the “Pre Table” include an XBPM and a set of precision slits to finally define the beam before the endstation and control beam position onto the KB. These are mounted on a common granite block.

The rest of the hutch contains the KB mirror system, sample station and detector table.

## **KB MIRRORS / NANOSTAGE**

The KB/Nanoprobe endstation is shown in Fig.1. It will be used for 2D scanning and 3D tomography both at cryogenic and room temperatures. With experiment times ranging up to 24 hours and a beam size of 50nm (reducing by more than a factor of 2 in the future), much effort has been made to achieve the best possible thermal and mechanical stability within the space available.

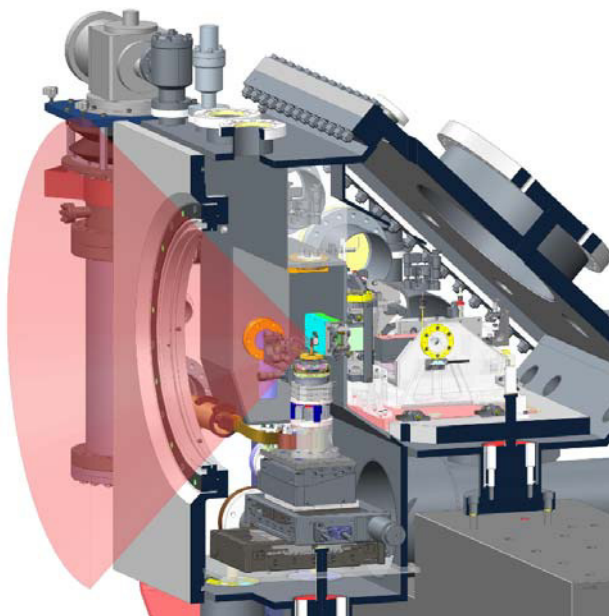


Figure 1: KB/Nanoprobe endstation.

With the sample point only 50 mm away from the end of the last KB mirror optic and an XRF detector required in the space between them, it has proved a considerable challenge to arrange and best support each mechanism within separate vacuum environments.

With no space for separate vessels they are mounted in a common vessel separated by a thin dividing wall housing a diamond window for passage of the beam on

through the XRF detector to the sample. The KB mechanism will operate at UHV whilst the sample side will either be at HV or used in air. This is to protect the KB mirrors from contamination and allow greater flexibility with the sample environment.

The vessel and internal mechanisms will be supported initially by a granite block directly fixed to the floor during commissioning and first use. However the intention is to upgrade this in the near future with an air bearing slide arrangement designed to easily swap the end station for other alternative setups.

The design can be broken down into the following key areas:-

- KB mechanisms
- Nanoprobe sample stage
- Interferometry for active corrections
- Sample mount and cooling
- Sample docking and transfer
- On axis sample viewing camera
- Vessel and support structures

### *KB Mirror Mechanism*

The fixed focus KB mirrors consist of the Vertical Focussing Mirror (VFM) first followed by the Horizontal Focussing Mirror (HFM) mounted onto a common base plate which is directly supported by the granite block through the vessel.

Flexure mechanisms provide pitch and roll (VFM roll only) adjustment with a motorised coarse adjustment provided for initial setup and fine piezo adjustment with <50 nrad resolution when in use. Each axis is directly encoded with a resolution of around 35 nrad to assist initial setup and commissioning. This could be improved further to <5 nrad using a different encoder if deemed useful to use as part of the position monitoring system.

The mirror optics are of an extremely high specification and have been measured and verified by the Diamond NOM facility to have slope errors <50 nrad with a figure error of <0.2 nm.

The mechanisms have been designed and built for some time. Whilst metrology tests have not been able to reveal any weakness in the design, limitations in the stability of the test set up has not allowed us to measure down to the levels of stability we will actually need at the endstation.

### *Nanoprobe Sample Stage*

The sample stage stack features coarse stepper motor driven XYZ stages at the base with piezo XYZ, rotation and tip tilt stages mounted above. With a target of < 5 nm linear repeatability and a radial error < 30nm required at the sample we have bought the best commercial stages we can source to fit within the space envelope. The repeatable and non-repeatable system motion error imposes a limitation on performance which will need to be addressed by active corrections using look up tables and feedback from the interferometers.

The stage assembly is mounted on a base plate directly supported through the vessel to the granite support block below.

Working upwards from the base, the stepper motor XY stages offer 50mm travel range whilst the vertical stage offers 13mm total range. These large translations are needed for experiments at positions other than just the focal point of the KB mirrors to allow for variations in sample mounts as well as reaching all areas on a sample TEM grid.

The custom piezo driven combination tip/tilt/height stage provides +/- 2 mrad tip/tilt and up to 200  $\mu\text{m}$  vertical fine adjustment along with a combination Piezo XY stage offering a 200  $\mu\text{m}$  range.

The piezo driven sample rotation stage mounted at the top provides up to +/- 180° rotation with a resolution of around 5  $\mu\text{rad}$ .

The fine piezo stages are needed to correct wobble / runout arising from the rotation stage and also to fine scan the sample.

### *Interferometry for Active Corrections*

In order to maintain the position of the beam and the sample to < 3 nm we need to actively correct the mirrors and sample stages to account for drifts and other sources of error and instability.

The arrangement has been the source of considerable iteration during the design process made all the more challenging by the space constraints involved. The final concept is shown in Fig. 2.

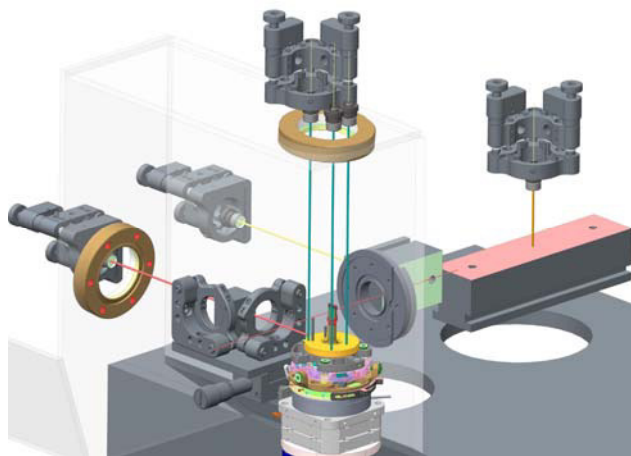


Figure 2: Interferometry arrangement.

All interferometers are mounted inside the KB vacuum space and supported from the mechanism base plate which should provide a good support and common reference of the sample stage relative to the KB optics themselves. Nonetheless it will be a challenge to finalise a structure which will provide sufficient stability within the space allowable. An interferometer references each KB optic whilst the others reference the sample stage through viewports in the chamber wall.

A gold coated Be Cu disk mounted below the sample has a highly polished top surface and edge which acts as a 3D mirror for the interferometers. It was designed to

reach cryogenic temperatures to maintain alignment with the sample mount.

Up to three interferometers will point vertically down at the surface of the disk to monitor pitch, roll and height variations whilst one pointing at the side of the disk monitors XY variations. The horizontal interferometer will also be mounted on motorised XY stages in order to maintain a perpendicular reference to the radius of the disk for maximum signal. To assist with this a beam splitter and diode will also be mounted in the path of the laser beam to indicate intensity as the interferometer falls off the peak reflection of the disk.

The disk allows for around 25 mm stage movement along the beam before the interferometers referencing the surface of the disk start to fall off the edge.

Although not allowing for the full movement of the nano-stage the aim is to provide active interferometry at positions other than the nominal sample position.

### *Sample Mount & Cooling*

Whilst the power in the beam is only around 0.01 mW the small beam size gives rise to a significant power density. With the need to keep cryo samples to <100 K with the beam on, FEA confirmed that LN<sub>2</sub> cooling would not be sufficient. Therefore we opted for a closed circuit gaseous Helium two stage pulse tube cooler system capable of achieving 4 K on the primary stage. The chosen unit is specifically an ultra-low vibration design.

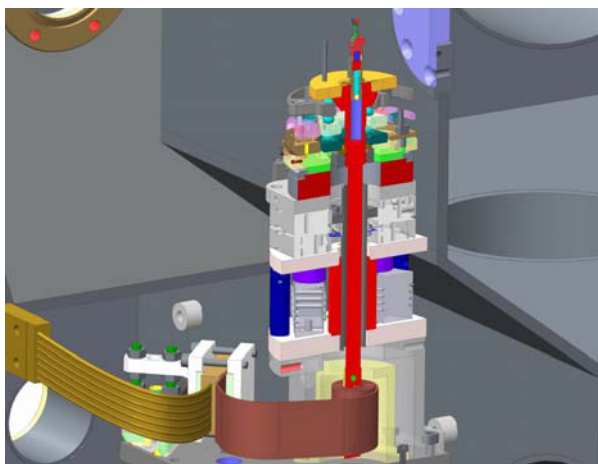


Figure 3: Cooling to sample.

In order to minimise radiation losses, the thermal link to the cooler primary stage is kept as short as possible whilst still allowing sufficient flexibility for the motions of the stage. Various arrangements were considered to transfer the cooling to the sample mounts allowing for the movement ranges of the stages whilst minimising potential strain on the piezo stages.

The final design shown in Fig. 3 brings the cooling to an anchor point on the stepper stages and then on axis through the stage stack via a Be Cu shaft which is attached to the sample mount tip.

To allow for virtually strain free movement of the sample rotation, the bottom of the shaft is thermally linked to the anchor point via a laminated soft copper coil “clock spring” design.

The sample mount tip is centred on axis by adjusting its mounting plate relative to the stage stack with 3 levels of thermal separation provided via zirconia balls in a kinematic arrangement. This minimises cold leakage into the stage stack which would give rise to thermal instability.

Tests on an earlier prototype setup revealed an 80 K gradient between the cooler at around 4 K and the sample tip. The goal is to achieve a temperature of around 20 K at the sample tip which is a considerable challenge. Minimising losses at such low temperatures is extremely difficult however we anticipate a significant improvement with the final design which contains some major enhancements including better clamping of the cooling at the base of the shaft, thermal isolation and less joints in the thermal link to the cooler. An array of PT 111 temperature sensors will be fitted around the structure to monitor temperatures and losses.

As well as the cooling aspects, the mass and height of the parts making up the overall stack above the stepper stages were minimised as far as possible to arrive at a stiff and light structure to facilitate the best dynamic performance from the piezo stages.

### *Sample Docking & Transfer*

We have adapted a commercially available sample transfer arm system to manually place loaded cryo samples into the vacuum chamber via a load-lock. Up to four sample mounts can be loaded at once on copper docking block (with cryogenic radiation shield) using magnets to retain them in place. There is also a magnet in the cooled sample stage tip which helps to positively locate the transferred sample mount and provide some preload to aid thermal transfer between them.

The transfer arm itself is a source of thermal gain for the cryogenic samples and is the subject of a separate Diamond project to improve its performance.

Once inside the chamber the sample loading block shown in Fig. 4 is docked on a rotation stage to allow full access for the internal XYZ piezo driven sample transfer shuttle.

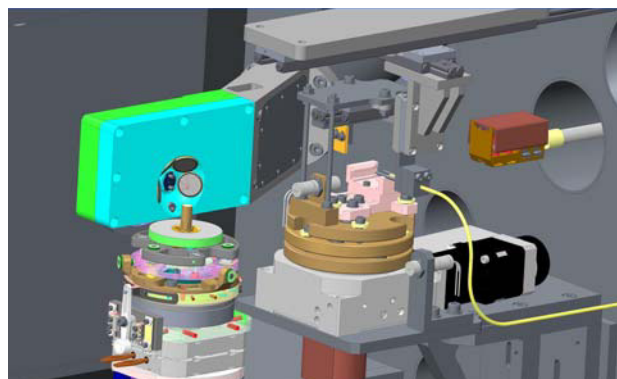


Figure 4: In vacuum sample transfer.



The docking block is maintained cold in a similar way to the sample stage but made off the cooler's second stage at around 20 K.

The design of the internal sample transfer mechanism along with variations of cryogenic sample mount is currently being refined further but a prototype arrangement has proved the principle works.

Alternative types of sample mount for non-cryogenic use are also being developed. A feature of the design allows the sample mount tip on the sample stages to be removed without disruptive dis-assembly to allow the fitment of different styles and sizes of samples.

### *On Axis Sample Viewing Camera*

An on axis sample viewing camera is currently being developed which will work around the limited space available in the vessel.

### *Vessel and Support Structures*

The vacuum vessel to house the KB and sample stage mechanics along with their associated sub systems and space constraints is complex. It has undergone many iterations arriving at the final design. The vessel is mounted on a granite block with internal posts supporting the mechanisms also grounded to the support block.

The lid on the UHV side has an aluminium wire seal whilst the sample side at HV is sealed via a hinged door and Viton™ O-ring. This door will allow the passage of a full 90° cone signal from the sample through to the air-side detectors via a large laminated carbon fibre reinforced sheet window. This window minimises deflection under vacuum which would otherwise infringe upon the working envelope of the vessel. An ion pump is fitted on the HV side to achieve the best vacuum possible when in cryogenic operation.

In the dividing wall between the two vacuum spaces a 50 µm thick single crystal diamond window has a clear diameter of 4mm for the passage of beam. It will be sealed using a Helicoflex® seal to maintain UHV on the KB side.

Initially the whole assembly will be mounted on a granite block for commissioning and first users. Once all aspects the system have been fully proved in use we will develop the air bearing slide system as an upgrade for rapid exchange of alternative setups whilst maintaining high stability support.

## **DETECTOR TABLE**

As well as the in vacuum XRF detector there is an air side Excalibur detector and an X-Ray Eye camera set up mounted on a common granite base block. Each one can be placed between 3 m and 250 mm from the sample using independent motorised drives. Each detector also has a vertical lift stage whilst the X-Ray Eye has an additional fine lateral stage. The general arrangement is shown in Fig. 5.

The key to getting the arrangement to work was how to provide a very stable 200 mm range vertical lift for the Excalibur detector in a very compact space envelope. An

“aggressive” wedge angle stage design was chosen. Tests proved the stage was very stable even at the extreme positions and could still achieve full range in 45 seconds.

A motorised beam stop is mounted before the Excalibur detector and travels with it.

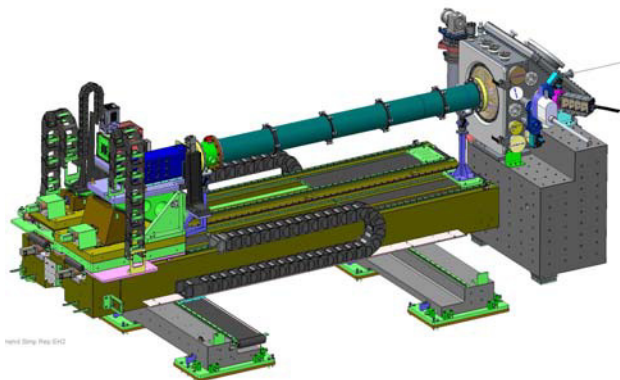


Figure 5: Detector Table.

A Ø200 mm vacuum pipe can be fitted between the endstation and the Excalibur detector which can be extended as required to the detector position.

Motorised lateral motion of the base block is provided to swap between either detector systems in less than a minute.

The detector table is currently being assembled in the hutch.

## **CONCLUSION**

I14 will be taking first light into the external building during September 2016.

A temporary setup in air for this milestone will utilise the KB mechanism and the upper piezo sample stages along with a representative interferometry set up to gain an understanding of the initial conditions and learn lessons for the final design.

Alongside this we will continue to test and refine elements of the cooling and sample transfer systems as well as the sample viewing camera design.

The vacuum vessel is due to be delivered in September 2016, when assembly of the final system will commence. Commissioning and extensive testing will follow in readiness for first users in Spring '17.