The Optical Metrology Laboratory at Diamond: pushing the limits of nano-metrology

Ioana-Theodora Nistea*, Simon G. Alcock, Murilo Bazan da Silva, Kawal Sawhney
Optics & Metrology group, Diamond Light Source Ltd, Harwell Science and Innovation Campus,
Oxfordshire, OX11 0DE, United Kingdom

ABSTRACT

We present recent advancements in the Optical Metrology Laboratory (OML) at Diamond Light Source. Improvements in optical manufacturing technology, and demands from beamlines at synchrotron and free electron laser facilities, have made it a necessity to routinely characterize X-ray mirrors with slope errors < 100 nrad rms. The Diamond-NOM profiler can measure large, fully assembled optical systems in a sideways, upwards, or downwards facing geometry. Examples are provided of how it has recently characterized several challenging systems, including: actively bent mirrors; clamped monochromator gratings in a downward-facing geometry; and four, state-of-the-art, elliptically bent, long mirrors with slope errors < 100 nrad rms. The NOM's components and data analysis procedures are continuously updated to stay ahead of the ever-increasing quality of X-ray optics and opto-mechanics. The OML's newest instrument is a Zygo HDX 6" Fizeau interferometer. A dedicated support frame and motorized translation and rotation stages enable sub-aperture images to be stitched together using in-house controls and automation software. Cross-comparison of metrology data, including as part of the MooNpics collaboration, provides a valuable insight into the nature of optical defects and helps to push optical fabrication to a new level of quality.

Keywords: optical metrology, slope profilometry, Fizeau interferometry, X-ray mirrors

*ioana.nistea@diamond.ac.uk

1. INTRODUCTION

1.1 Diamond Light Source: present status and future plans

Diamond Light Source[1] is the UK's national facility for generating ultra-intense beams of synchrotron radiation. These photon beams are simultaneously utilized by multiple experimental stations (so-called beamlines). This enables research in a broad spectrum of scientific and technological fields: from viruses and vaccines, to fossils and jet engines. Since experiments began in 2007, many tens of thousands of researchers from academia and industry have used Diamond. Currently, 33 beamlines are operational at Diamond, many of which have been significantly upgraded since operations began.

We are now entering a new era of scientific opportunity with the advent of 4th generation X-ray sources, the so-called Diffraction Limited Storage Rings (DLSRs) and X-ray Free Electron Lasers (XFELs). For Diamond to continue to deliver world-changing science, its particle accelerators will be upgraded in the near-future. This proposed upgrade has just passed through the conceptual design review process[2]. The machine upgrade will be transformative in terms of X-ray data quality and speed. The new scientific opportunities afforded by the new Diamond-II machine will necessitate the renewal of beamline optics, sample environments, and detectors, as well as substantial enhancements to data storage and computation. The upgrade will also address emerging opportunities in areas such as nano-probe, high-spatial resolution, higher energy-bandwidth, maximum transverse coherence, or combinations of these.

The proposed Diamond-II source brings many exciting opportunities, but also challenges for beamline optics. For the experiments to stay at the forefront of research, the optical layout of every beamline, comprising multiple mirrors, monochromator crystals, and lens, will need to be individually optimized to benefit from the upgraded X-ray source. At a time when synchrotron and XFEL facilities around the world are poised to begin upgrades, and when there are only a few qualified suppliers of X-ray optics, it is mission critical that Diamond has the requisite infrastructure and expertise in optics and metrology.

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1.2 Motivation for Optics and Metrology group

The successful exploitation of the intense synchrotron light depends to a significant extent on the quality and performance of beamline optics. For each beamline, the Optics and Metrology (O&M) group at Diamond delivers a custom selection of optics to efficiently transport the synchrotron light to the sample and produce the desired focus and wavelength selection. In collaboration with each beamline team, the O&M group lead the design of the X-ray optics and liaise with manufacturers to specify and procure the necessary components. After acceptance testing and optimized assembly into their opto-mechanical holders, the optics are installed on the beamline. The O&M group then actively assists the beamline scientists with X-ray commissioning of the optics. To extend the capabilities of Diamond's beamlines, the O&M group is also actively pursuing multiple research activities including development of next-generation optics, instrumentation, and techniques. This is supplemented by collaborations with key external research institutes and industry.

Several beamlines at Diamond-II will require ultra-high-quality X-ray optics that are ~ 2 - 5 times improved in figure error compared to present-day X-ray optics. This will require enhancing the capabilities of our in-house metrology facilities so that X-ray optics with slope errors < 50 nrad rms can be reliably measured. Exploitation of Diamond-II for nano-focusing, coherence preservation, requires mirrors and high-resolution gratings with figure errors < 1 nm peak-to-valley. This is only possible via deterministic polishing techniques, such as ion beam figuring (IBF). Since there are only a few suppliers of the most technologically demanding optics, this represents a serious risk in terms of cost and time. To mitigate this risk, one of the O&M group's research projects is coordinating the build of an Ion Beam Figuring system at Diamond which aims to create improved quality X-ray mirrors[3].

2. OPTICAL METROLOGY AT DIAMOND

2.1 Optical Metrology Lab

Independent metrology is a necessary prerequisite for critical inspection and acceptance of procured X-ray optics. The O&M group have established a state-of-the art Optical Metrology Lab (OML) which is built over a floor area of ~ 100 m^2 and is a class 10,000 (ISO 7) cleanroom with a temperature stability of ~15mK over a 24-hour period. The OML has been operational for 12 years and during this time we have measured the vast majority of the X-ray optics on Diamond's beamlines, including hundreds of different mirrors, diffraction crystals, and internally cooled substrates. The optics range in size from a few millimeters to over 1.5 m. Fully assembled optical system can weigh over 100 kg. More than 70 optical systems are characterized in the OML every year. Such data also provides valuable inputs for ray-tracing or wave propagation simulations performed by the O&M group to predict how challenging optics are likely to perform on the beamline using X-rays.

To accurately predict the performance of a synchrotron optic it is important to characterize surface defects over a wide range of in-plane periods (from < 10 nm to over 1 m). To cover this large dynamic range, the OML is equipped with a suite of metrology instruments including: the Diamond-NOM slope profiler, a Zygo HDX Fizeau interferometer, an atomic force microscope, and a Bruker GTX stitching micro-interferometer[4]. Environmental enclosures and robust support platforms are used in the cleanroom to achieve the exceptional temperature, vibration, humidity, and air flow stability necessary to perform nano-metrology. Used in synergy, metrology data from the different instruments is intelligently combined to achieve reliable and accurate measurement.

Aside from using a high-grade substrate, it is vitally important to design and construct suitable opto-mechanics to securely hold (and often cool) the optic without adding significant distortions. In the early years at Diamond there were several occasions where suppliers installed optics into their holders on the beamline without metrology feedback. In many such cases, subsequent investigations revealed major optical deformations due to clamping, which compromised beamline performance. This motivated a procedural change at Diamond, and we have now adopted a holistic philosophy to X-ray optics and their opto-mechanics. As the group's expertise and instrumentation have evolved, we have progressed from basic acceptance testing of the bare substrate to routine optimization of fully clamped and mounted optics. Lessons learnt are applied to future projects, thereby evolving and improving performance. Beamline optical systems are now purposefully designed to be modular such that they can easily be dismounted from the beamline vacuum vessel and transported to the OML. Once built and optimized in the OML, each optical module is moved as a whole unit and installed directly onto the beamline, typically with support from the O&M group (Figure 1 and Figure 2). In this manner we ensure that the final optical system is fit for purpose and will perform well using X-rays. This approach has significantly reduced the risk of discovering a fault prior to X-ray commissioning.

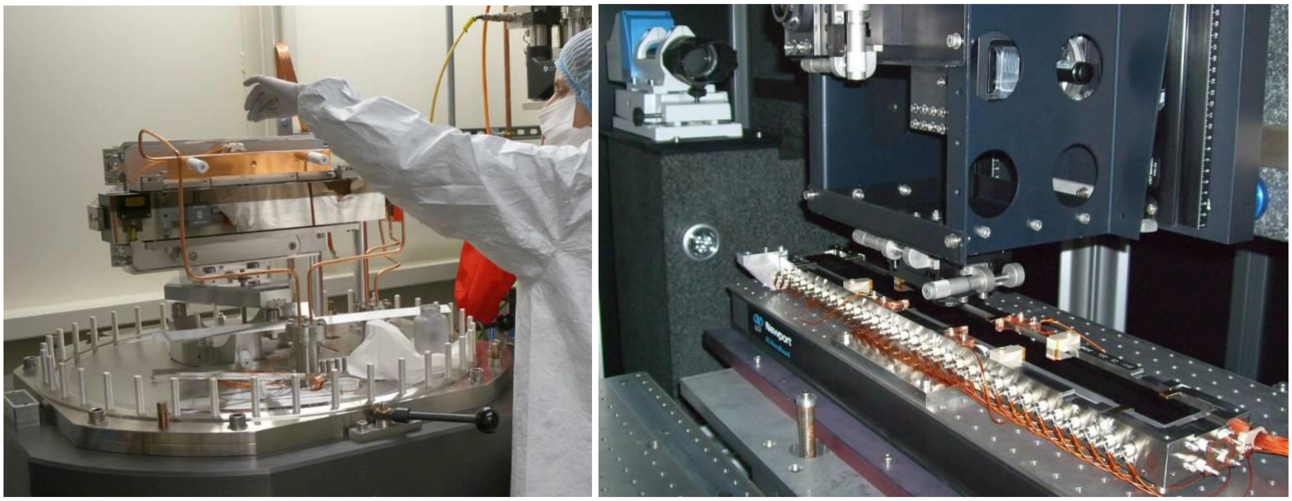

Figure 1. (a) Adding eutectic liquid to the cooling channels of a mirror installed on the beamline. (b) Characterizing a piezoelectric bimorph deformable X-ray mirror using the Diamond-NOM slope profiler.

Figure 2. An in-house designed, double toroidal mirror system with side cooling, as installed on the beamline.

2.2 The Diamond-NOM: measurements in any orientation

The Diamond-NOM is a non-contact, slope measuring profiler[5], capable of measuring long- to mid-wavelength features on the optical surface of synchrotron optics with sub-nanometer height resolution and repeatability. Surface errors can be recorded over a lateral scale of ~2 mm up to the full length of the mirror.

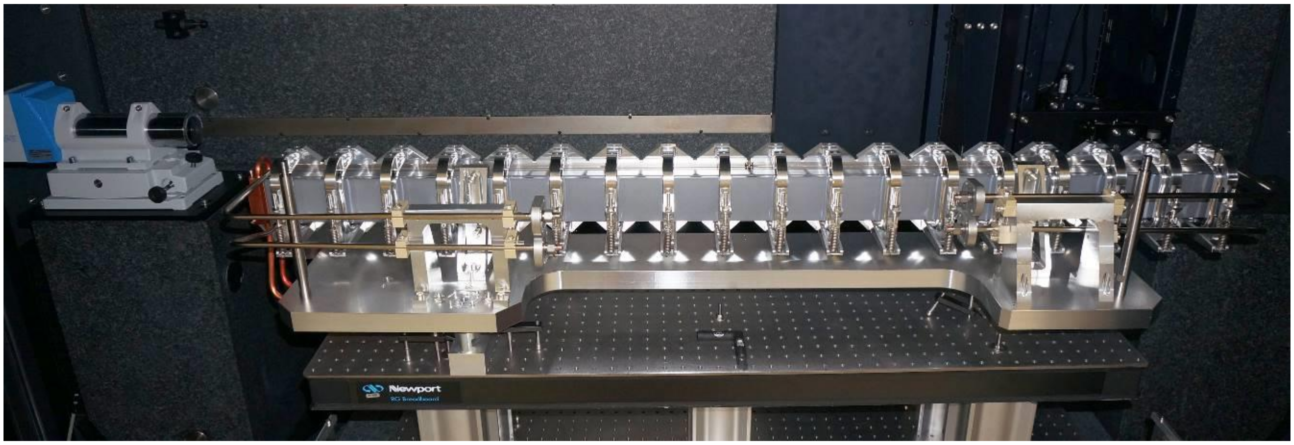

Figure 3. The Diamond NOM slope measuring profiler measuring a large and heavy, side-cooled mirror system.

The NOM is used almost continuously in the OML and there is an ongoing research and development program to improve its performance. Heavy duty platforms support and precisely align very large (1.5 m long) and heavy (> 100 kg) optical assemblies. The Diamond-NOM has been upgraded so that X-ray mirrors can be measured facing upwards, downwards, or sideways. This allows measurement of optics in their final beamline geometry, to help better understand the influence of gravity sag. This concerns not just the substrate, but also the opto-mechanics which can also deform under gravity and introduce strain into the optical surface. On the nanometer scale this is vitally important for enhanced optical performance.

One recent example is a collaboration with an optic supplier. The substrate was shipped to Diamond and was measured on the Diamond-NOM in its final beamline geometry of facing downwards (as shown in Figure 4). It was purposefully placed on support points which mimic exactly how the optic will ultimately be held on the beamline. This mirror was also measured facing upwards and sideways on the NOM to understand the influence of gravity for non-cuboid shaped substrates. The supplier will use this metrology data for IBF correction, such that when the mirror is held in the final holder it will sag into exactly the right elliptical shape required by the beamline geometry.

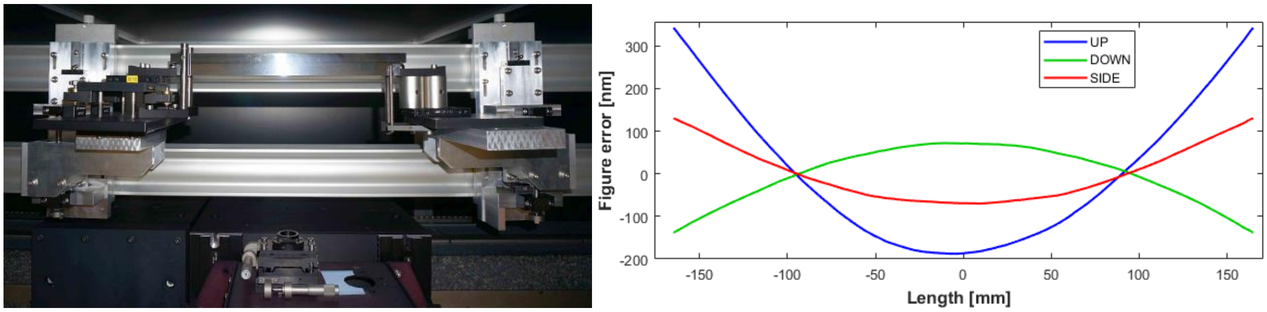

Figure 4. Diamond-NOM metrology of an X-ray mirror substrate: (a) measured facing downwards to mimic its final beamline geometry; (b) Figure error, relative to the specified ellipse, of the mirror measured facing upwards (blue curve), sideways (red), and downwards (green). Data will be used by the optical supplier for ion beam correction.

2.3 HDX Fizeau interferometer and stitching system

The newest addition to the suite of metrology instruments in the OML is a state-of-the art Fizeau interferometer. The Verifire HDX from Zygo has a 150 mm (6 inch) diameter, stabilized laser beam. A high-resolution camera with an 11-megapixel CCD sensor allows the HDX to resolve features in fine detail on the optical surface. It also has a fast frame rate of 96 Hz enabling multiple images to be acquired in a short period, thereby reducing the effect of random noise by suitable averaging. A comprehensive software package (MX) is provided with the instrument for acquisition control and data analysis. A custom-built support platform for the HDX has been designed and built at Diamond in collaboration with the Engineering team (Figure 5a). Finite element analysis was used to guide the design to minimize the impact of angular and linear vibrations. The support platform can be varied in height using a heavy-duty lifting jack. Once at the required height, spacer blocks are added, and the platform is securely locked down to minimize vibrations. This mechanism allows a single person to make a height change without the need for manual lifting.

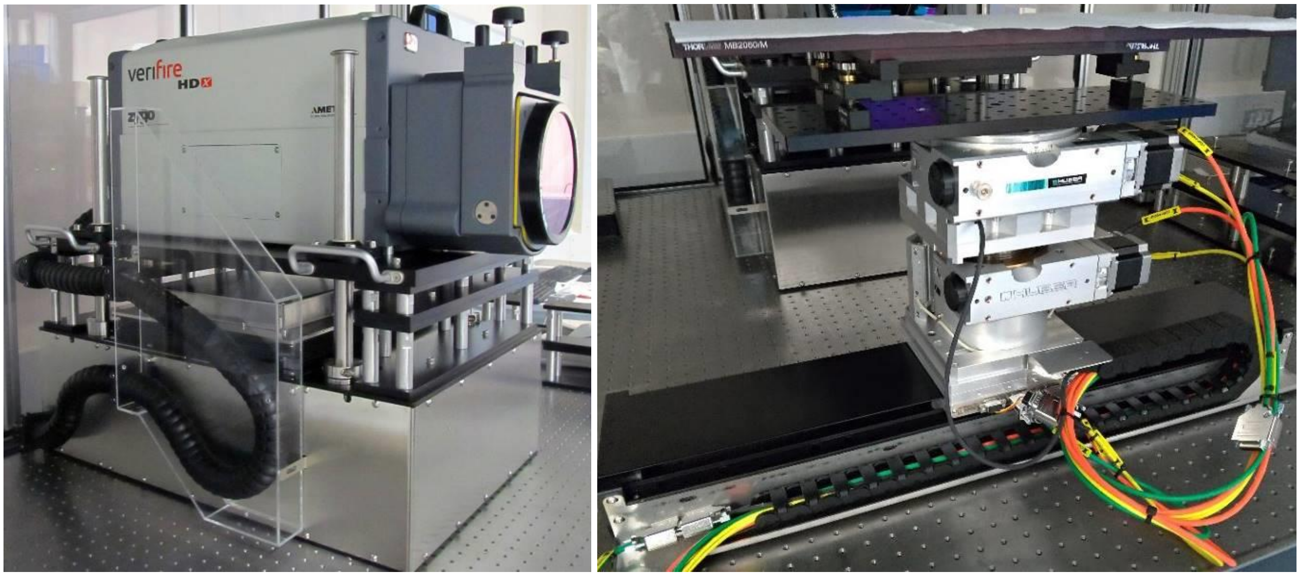

Figure 5. (a) Zygo Verifire 6” HDX Fizeau interferometer mounted on an in-house-built, modular support platform. (b) Motorized stages for the HDX stitching system which precisely translate and rotate the test optic in front of the Fizeau’s beam. A motorized roll stage will soon replace the current manual adjuster.

As with any Fizeau interferometer, measurement of the mirror under test is relative to the Fizeau’s transmission flat. The Fizeau’s reference flat ($\lambda/20$ quality) was mapped in the OML using an ultra-high-quality X-ray mirror from JTEC (Japan), which is now installed on the I21 beamline at Diamond. This state-of-the-art mirror has a slope error of < 100 nrad rms and a tangential radius of curvature of > 500 km, making it the flattest mirror we have ever measured on the Diamond-NOM! This ultra-flat mirror (450 mm long) was measured on the Fizeau at multiple positions and in different orientations. Averaging these scans together, to randomize minor surface defects on the test mirror, produced an accurate map of the Fizeau’s reference optic.

Currently, a stitching system is being designed and built for the HDX. All the necessary hardware has been purchased and recently commissioned. A stack of motorized stages from Huber and Newport (Figure 5b) provides fine pitch adjustment (< 1 µrad) and vertical and lateral translation (< 100 nm) of the test mirror. Instrumentation and controls software, using EPICS and MX, is being finalized. Synchronization of data acquisition by the HDX, and compound movement of the motion stages, is coordinated by Python scripts developed at Diamond. The goal for the system is to perform angular stitching (for highly curved optics) and linear stitching (for optics longer than 150 mm), and combinations of both. The first HDX metrology results for a beamline mirror are in excellent agreement with data from the Diamond-NOM (Figure 6).

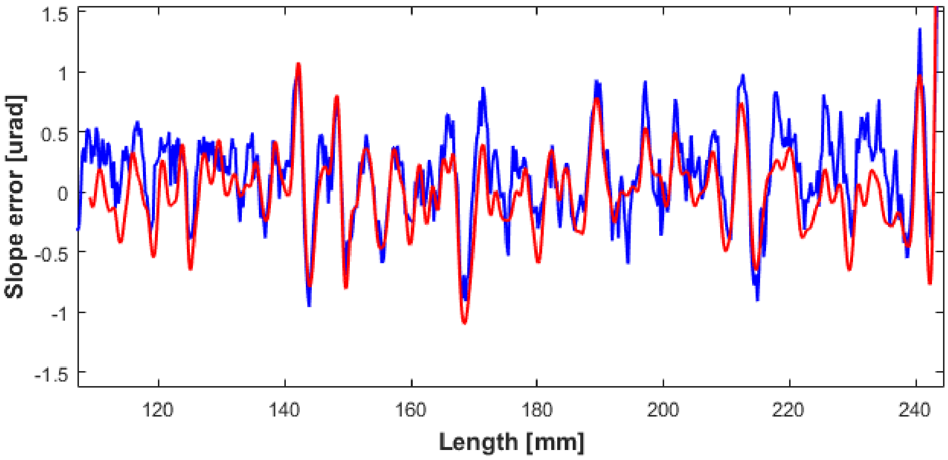

Figure 6. Comparison between the slope error of a beamline mirror as measured by the Diamond-NOM (blue curve) and the new HDX Fizeau interferometer (red).

This work is part-funded by the MooNpics collaboration[6], a European initiative between research laboratories at several synchrotrons, the European-XFEL, and selected X-ray optic suppliers. The aim is to push metrology in Europe to another level to be able to reliably measure X-ray mirrors with figure errors < 1nm peak-to-valley, and slope errors < 20 nrad rms. This quality needs to be achieved for a range of X-ray optic types, including: long (> 1 m) ultra-flat mirrors (radius > 3000 km) for X-ray beam transportation; and strongly curved mirrors for nano-focusing (typically radius < 100 m, but < 15 m in extreme cases). Such diffraction-limited optics can potentially be fabricated using deterministic polishing techniques such as IBF. However, highly accurate metrology is required to provide suitable inputs for deterministic correction of such optics. A major issue for metrology of long, highly curved mirrors is how to accurately stitch together multiple, sub-apertures to obtain a composite map of the entire optical surface. Great care needs to be taken to ensure that any errors introduced by the stitching hardware or analysis routines don't accumulate beyond the extreme accuracy requirements for diffraction-limited optics. At present, the hardware implementations and software algorithms for stitching vary between each metrology lab. It is hoped that the MooNpics collaboration can standardize such efforts. As such, algorithms are being developed by the collaboration to accurately stitch together metrology data from a variety of metrology instruments, including slope profilometers (such as long trace profilers or NOMs) and Fizeau and micro-interferometers.

2.4 MooNpics Round Robin: Diamond's 3-lane mirror

Another part of the MooNpics collaboration is a Round Robin exercise: three challenging X-ray mirrors (a long flat mirror, a highly curved mirror, and an elliptical mirror) are being circulated for independent measurement at each facility over a period of ~ 2 years. Every facility will measure each optic with all of their available instruments (and in some cases will also use X-ray metrology methods). Diamond is contributing a novel optic to the Round Robin: an elliptically curved mirror with 3 lanes, as described in[7]. Each lane has a series of parabolic arcs, of varying amplitude, polished into its surface. Each set of parabolic arcs is designed to purposefully change the size of the reflected X-ray beam. The X-ray beam size can then be rapidly modified (in < 1 second) by translating the mirror sideways to illuminate the chosen lane. A mirror with 7 such lanes, fabricated to our specifications by JTEC, is currently operational on the VMXm beamline at Diamond. The concept for such a mirror was developed by David Laundy and colleagues from the O&M group.

The MooNpics project was an excellent opportunity to characterize this 3-lane mirror using the Diamond-NOM, the HDX Fizeau interferometer, and the GTX micro-interferometer. Figure 7 shows the figure residuals (i.e. the deviation of the optical surface relative to the best fit ellipse) for each of the lanes, as measured by the Diamond-NOM (blue curves), HDX (red curves) and GTX (green curves). The figure residual for Lane 1 is effectively a figure error (no parabolic arcs, as this lane is simply designed to focus the X-ray beam). Whereas, the figure residuals for Lanes 2 and 3 represent the parabolic arcs described above (nominally with amplitudes of 25 nm and 50 nm peak-to-valley respectively). The metrology data from the three instruments is in excellent agreement. The small differences in the low frequency component of the profiles measured by the GTX micro-interferometer is a result of the stitching process, as expected for an instrument of this type.

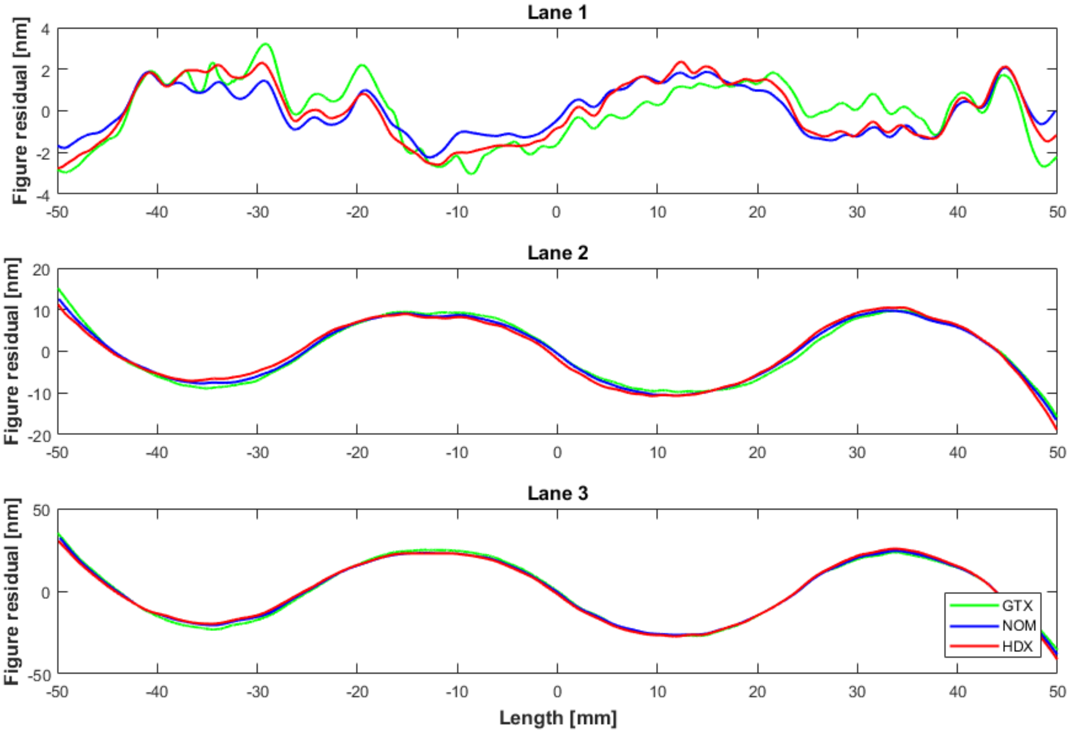

Figure 7. Figure residuals for all 3 lanes of the mirror, as measured by the Diamond-NOM (blue), GTX stitching micro-interferometer (green) and the HDX Fizeau interferometer (red).

A series of fiducial markers (including dots, lines, and cross-hairs) were inscribed onto the outer parts of the optical area of the 3-lane mirror to assist with measurement alignment and data analysis. This has proven extremely useful and has helped to carefully align the datasets acquired by the different instruments. When measuring at a nanometer level, even the slightest translation or angular misalignment can lead to errors which are larger than the required precision of the measurement. This form of cross-comparison is a very valuable method to learn more about each instrument's strengths and weaknesses, and to perfect procedures for comparing the datasets in a meaningful way. For example, determining how each dataset needs to be spatially filtered such that we can meaningfully compare instruments with different spatial sensitivities and measurement ranges.

2.5 Clamping of optics: RIXS beamline monochromator gratings

As discussed in Section 2.1, the manner in which optics are clamped and supported plays a major role in their overall performance. Therefore, clamping and optimization of optics into their opto-mechanical holders based on metrology feedback is a core activity in the OML for all beamline mirrors and diffraction crystals and gratings. The performance of the Resonant Inelastic X-ray Scattering (RIXS) beamline (I21) is extremely sensitive to slope errors on its monochromator systems, primarily due to its demanding requirements for X-ray energy resolution. The plane grating monochromator (containing an internally cooled mirror and three, side-cooled, diffraction gratings) is a critical part of I21. When they noticed a reduction in performance, they requested for the gratings to be removed from the beamline and investigated in the OML. Before making any adjustments to their clamping, the gratings were measured using the Diamond-NOM. It was found that that all three gratings had slope errors of ~ 250 nrad, and 3rd order polynomial slope profiles consistent with gravity sag (blue curve in Figure 8). Previous measurements of the unclamped substrates showed that they all had exceptional initial quality, with slope errors < 100 nrad. The challenge was to re-clamp the three original gratings (and also a new one) without adding significant distortion. Over a 3-week period, all gratings were iteratively clamped, measured, and reclamped based on metrology feedback from the Diamond-NOM. Optimization was performed with the optics facing upwards on the NOM. But after the final iteration of clamping, they were also measured facing downwards to mimic the beamline orientation. The end result (red curve in Figure 8) showed that we were able to repeatably clamp the gratings without adding any further slope error distortions. For all clamped gratings we achieved slope errors of ~100 to 150 nrad. For the grating shown in Figure 8, the slope error was improved from 250 nrad (blue curve) to 90 nrad (red curve).

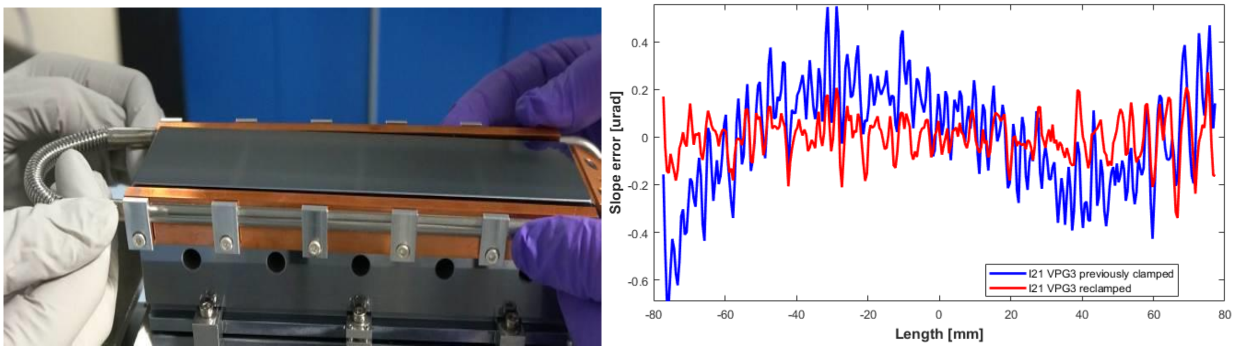

Figure 8. Iterative clamping procedure for the I21 diffraction gratings. (a) clamping the water-cooling manifold to the side faces of the silicon substrate. (b) Representative slope error measurement using the Diamond-NOM of before (blue) and after (red) reclamping one of the gratings.

2.6 Optimizing active X-ray optics

Another core activity of the OML is characterizing active X-ray mirrors. Such optics, which can be bent into a range of shapes using either mechanical or piezoelectric actuators, are highly complex systems which require significantly more effort to ensure correct operation. A protocol was developed to investigate the behavior of each system, and to highlight any possible issues. For example, misaligned clamps and actuators which cause the substrate to twist or roll during bending. Typically, a range of parameters are measured, including bending range, hysteresis, and curvature drift over extended periods of time. Systems also undergo basic testing to find any general faults, such as: electrical / wiring faults; checking that motor ranges and limits are correctly set; and confirming correct communication in EPICS with the actuators and any on-board metrology instruments (such as strain gauges or capacitive displacement sensors). The active optics system is then optimized to determine the motor or voltage settings which optimally bend the mirror to a range of specified cylinders, ellipses, or parabolas (Figure 9). Such data are provided to the beamline teams to assist with X-ray commissioning. This useful information means that only fine adjustments are needed to optimize the size and shape of the X-ray beam. On numerous occasions, these fault-finding and optimization procedures have saved significant amounts of X-ray commissioning time. Based on our experiences working with active optics, Diamond has an extensive research program which aims to improve the speeds and tuneability of bimorph mirrors. This work has recently led to improvements on one of Macromolecular Crystallography beamlines (I24), whereby large changes can rapidly be made to the size of the X-ray beam and stabilized in just a few seconds.

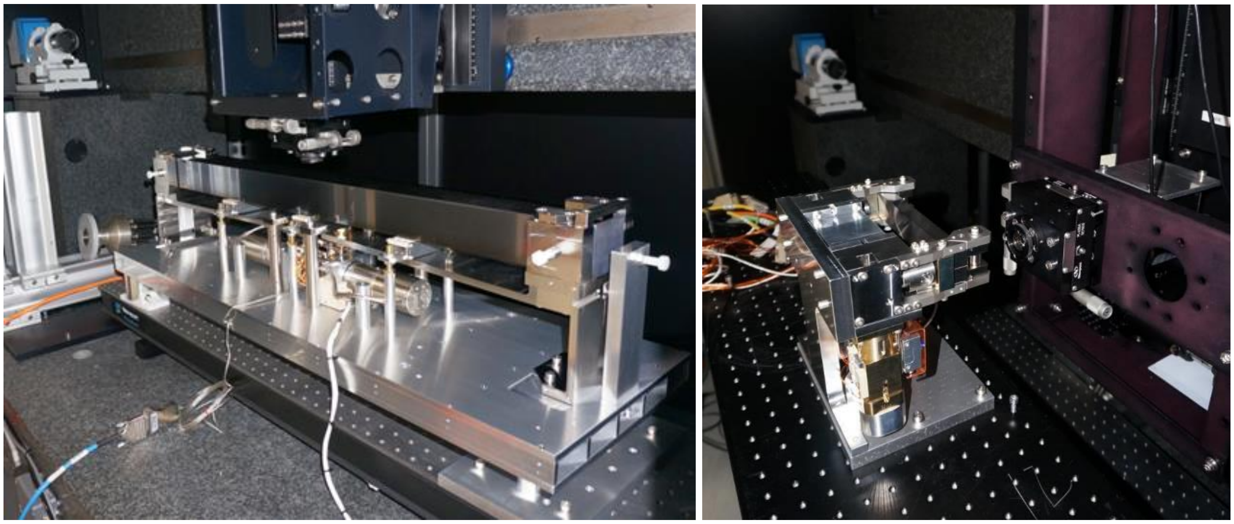

Figure 9. Examples of mechanically bent mirror systems being characterized and optimized on the Diamond-NOM.

2.7 Pushing the limits of optical metrology

As well as testing optics for Diamond's beamlines, the OML also tests substrates and optical systems on a commercial basis on behalf of manufacturers and other synchrotrons and XFELs. A recent highlight of such work was characterizing two pairs of X-ray KB mirrors on behalf of the European XFEL. These optics, manufactured by JTEC, are perhaps the highest quality optics we have ever measured at Diamond. Each single-crystal silicon substrate is between 600 and 1000 mm long and is polished to an elliptical figure with a slope error <<100 nrad. Elliptical bending systems were designed and built by FMB-Oxford[8] to bend each substrate into a range of ellipses. The unclamped substrates were measured on the Diamond-NOM before and after coating (Figure 10). After this, each substrate was mounted into its bender mechanics, and the clamping was optimized such that minimal tangential deformation or sagittal twist was introduced into the optical surface. The bending mechanisms were then fully characterized using the Diamond-NOM.

This project was particularly challenging due to the extreme quality of the optical surfaces. The polishing errors are approaching the current limits of the Diamond-NOM's measurement capabilities. Therefore, significant numbers of scans were needed to be averaged together to diminish the random errors to a suitable level. Similarly, each optics had be measured at multiple angles, translations, and orientations to pseudo-randomize the systematic measurement errors, such as the "pixel effect" and other such errors in the NOM's autocollimator. Also, great care had to be taken to the stabilize the environment around the measurement to diminish its influence on the mirror and the autocollimator. However, this type of challenge is what drives a metrologist, and is a sign of things to come. Over the next few years, optics with challenging aspheric profiles with slope errors of << 50 nrad will become commonplace. To be able to test such high-quality optics, it is imperative to future-proof the metrology instruments in the OML and corresponding acquisition and analysis procedures. We are currently developing strategies to try to stay ahead of the curve.

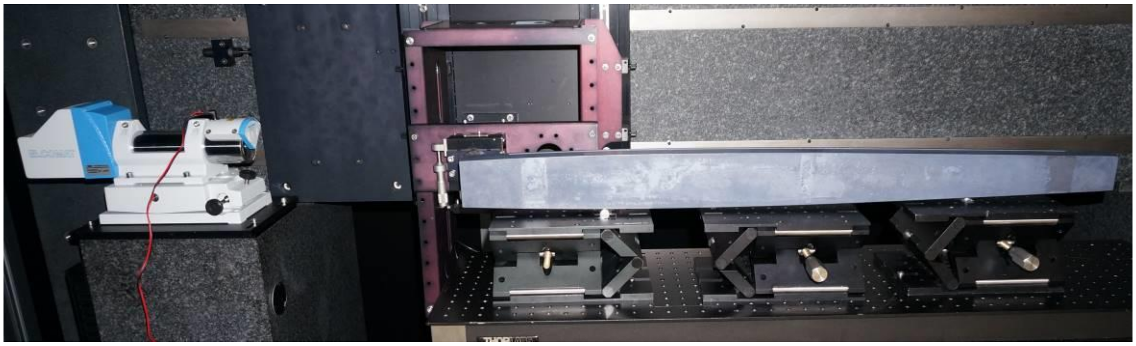

Figure 10. Horizontally focusing mirror substrate being measured on the Diamond-NOM on behalf of the European-XFEL.

3. CONCLUSIONS

In summary, the Optical Metrology Lab at Diamond has been in operation for 12 years, during which time hundreds of X-ray optics have been measured. We have provided several examples of research undertaken in the OML, including development of new metrology hardware and techniques to help to measure today's state-of-the-art X-ray optics. We are working closely with a range of optic suppliers and collaborators at other facilities to help to develop novel X-ray optics which are beyond today's state-of-the-art. As many other synchrotrons are currently doing, or are planning, the Diamond machine will be upgraded in the near-future. This will hopefully lead to an improved X-ray source, which in turn will lead to a new set of challenges for designing, measuring, and delivering X-ray optics of almost unimaginable quality to successfully utilize the intense photon beams for world class scientific research.

4. ACKNOWLEDGEMENTS

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