XVI IMEKO World Congress
Measurement - Supports Science - Improves Technology - Protects Environment ... and Provides Employment - Now and in the Future
Vienna, AUSTRIA, 2000, September 25-28

NANOMETROLOGY USING X-RAY INTERFEROMETRY

D.G. Chetwynd and N.O. Krylova
Centre for Nanotechnology and Microengineering
School of Engineering
University of Warwick, Coventry CV4 7AL, U.K.

Abstract: X-ray interferometers are now a recognized tool for sub-nanometre precision metrology in National Standards Laboratories. Used in slightly different ways, they have potential for wider application as the demands for greater precision in industrial metrology increase. After reviewing some of the key ideas about using monolithic devices in this manner, it is shown that a 'portable' system intended for industrial standards rooms can perform well. There is a brief discussion of some issues such as vibration isolation. Details are given of a novel experimental verification that a separated-blade interferometer could operate on a state-of-the-art kinematic slideway, although for reasons of cost, a full implementation has not been attempted. An interferometer to measure angular displacements to nanoradian precisions is demonstrated. Finally, there are a few comments on where the technology is likely to develop.
Keywords: X-ray interferometry, nanometre metrology, calibration

1 INTRODUCTION

The x-ray interferometer was developed in the early 1960s [1] and it was soon proposed as a length measuring device of sub-ångström accuracy [2,3]. However, the first practical characterization of a nano-displacement gauge by an x-ray interferometer was in the early 1980s [4]. From this demonstration it seemed that the method might act as a 'portable' secondary standard of length for the most demanding industrial metrology problems on the micrometre to nanometre scale. Since then, the emphasis in National Standards Laboratories has been towards highly sophisticated combined optical and x-ray interferometers (COXI), see for example [5], for fundamental metrology, especially for better determinations of Avogadro's Number. Complementary work at the Warwick Centre for Nanotechnology and Microengineering has concentrated on developing instruments and techniques to satisfy a wider range of extreme precision linear and angular measurements while working in good, but not ideal, environments. Currently, the technical performance is beyond most industrial needs so the relative disadvantages of using x-ray methods mitigate against its immediate introduction. This may soon change: many industries now require routine measurements precise to fractions of a micrometre so calibration systems fully traceable to better than 1 nm and resolving considerably more will increasingly be required.

The purpose of this paper is to show that monolithic x-ray interferometer technology is practical under industrial standards-room conditions. It reviews some important concepts, discusses a specific linear design and introduces variant methods applied to nanoradian angular calibration and alignment.

2 PRINCIPLES OF X-RAY INTERFEROMETRY

There is not space here to review the relevant theory of x-ray interferometry, which is covered elsewhere [6]. The design of immediate concern is the Laue (or LLL) interferometer in which x-rays are diffracted in transmission through thin pieces (or blades) of single crystal material. If such a blade is illuminated by an x-ray beam at the Bragg angle, two symmetrically disposed (forward and back diffracted) output beams will emerge. The crystal acts as a beam splitter and a second similar blade (the 'mirror') intersecting these beams splits each again, producing two converging beams. When these beams meet they interfere and produce a standing wave electric field that encodes the geometry of the crystals. If the interference occurs at a third (analyser) blade, the standing wave interacts with the crystal such that a strong output beam parallel to the original illumination is produced when all three blades are aligned as if part of a single crystal. However, a minimum intensity is produced if, say, the analyser blade is displaced laterally by half a lattice spacing, other conditions remaining the same. Furthermore, slightly rotating the blade causes moiré fringes in the output field. We may regard the x-ray interferometer as equivalent to a moiré fringe incremental encoder with a grating pitch equal to the lattice parameter (typically a few ångströms). Silicon is available in large, extremely pure single crystals, and has strong diffraction planes at spacings of about 0.2 and 0.3 nm, each known against the laser realization of the metre to a small fraction of a picometre. Silicon interferometers can therefore provide very high sensitivity to displacement with extremely good traceability and at moderate cost. Provided the output intensity is determined by photon counting, almost all the error sources that limit the accuracy of optical fringe interpolation are negligibly small.

The first step in making an x-ray interferometer is to machine three blades on a suitably crystal-oriented single crystal of silicon, usually by gentle diamond grinding followed by an isotropic etch to removed the crystallographically damaged surface layers. One blade can then be separated and the two parts remounted onto a high-stability flexure mechanism to allow the translation of the analyser blade with respect to the others. It is very costly to re-align the blades to the milli-arc-second precision necessary to reproduce the effect of a single crystal and give useful fringe contrast. Thus, from the earliest times, an alternative strategy has been to use a monolithic construction, with a flexure mechanism machined into the single crystal. This introduces significant limitations in displacement range and motion precision, but alignment is much easier and the overall cost of production tends to be lower. For nanometre displacement gauge calibration, the bulk of the monolith can also provide a metrological reference base. Typically, the analyser blade would be driven by an electromagnetic or piezo-electric actuator while the gauge measurement of the blade motion is compared to the fringe count. Figure 1 shows an example design. For best stability the blades would move in the horizontal plane, but often the operation of sensors being calibrated requires a vertical motion. The positioning of actuator, blade and measurement point represents a compromise between satisfying the Abbe alignment principle and avoiding complex machining on the monolith. Small Abbe offsets are tolerable because minute blade rotations cause large, easily detected changes of fringe contrast so potential errors are diagnosed automatically. Two parallel force actuators, offset either side of the required line of action, can be used to correct for such twisting by applying a small torque as well as a thrust.

The x-ray wavelength does not directly affect the fringes, which depend only on the lattice. Shorter wavelengths suffer less absorption by the blades but give smaller Bragg angles, which can make some forms of operation more difficult to set up. A good compromise for silicon, used in most of our systems, is molybdenum K_α radiation from a fixed-anode x-ray tube excited at around 40 kV and 40 mA. The output is measured either by a photomultiplier with a scintillator coating (for photon counting total intensity) or by an x-ray TV camera. The latter is a conventional low-light CCD device with a scintillator-coated coherent fibre optic faceplate that tapers to provide a suitable magnification.

The fringe contrast will inevitably vary over any reasonably large displacement, especially if a gauge probe is imposing forces on the monolith. The simplest forms of fringe interpolation then become inaccurate, so a phase-step method has been devised [7] to exploit algorithms well known from optical analysis. Placing a small, uniform piece of polymer in one beam of the interferometer,

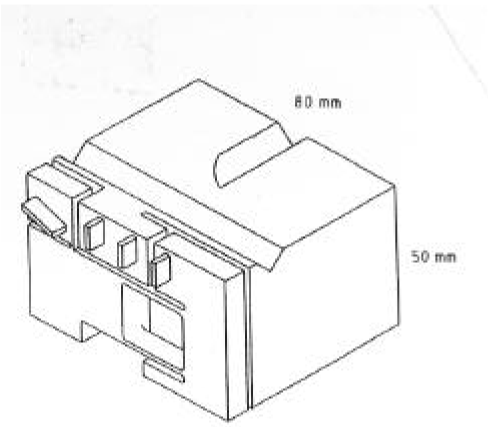

Figure 1. A silicon x-ray interferometer monolith for nanodisplacement calibration.

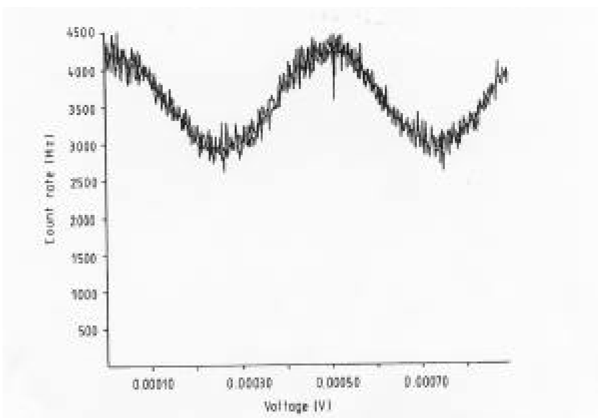

Figure 2. X-ray intensity from interferometer over a 0.5 nm scan and return

between the blades, changes the x-ray optical path length and so alters the phase of the output fringe relative to the lattice. Switching it sequentially into each beam then gives outputs phase advanced and retarded by equal amounts (ideally 120°) from the original. The intensity of these three signals at any point allows the local amplitude and phase of the fringe to be calculated unambiguously. The disadvantage is that three readings must be taken at each point, so the total measurement takes more than three times as long. It is desirable to use low x-ray intensities for safety and to reduce thermal loads and we have generally worked at detected rates of a few thousand photons per second. The Poisson statistics of photon counting then require significantly long counting times for good fringe definition. Broadly, 1 – 5 s is needed for each reading if fringe division to better than 0.1 fringe (around 20 pm) is wanted. A 1 µm scan will contain about 50000 fringes. The only way to guarantee traceability is to examine at least one point in every fringe, so the method is slow in operation. Practical applications involve a compromise between theoretically achievable precision and effects such as thermal drift in the systems under calibration. The interferometer can calibrate its own drives (a sort of 'bootstrap' operation) allowing multi-fringe steps to be made with good confidence, but the method is likely to remain one for calibration and characterization rather than for direct application in displacement control.

3 A DESK-TOP NANOMETRIC CALIBRATOR

The methods described above have been used to build a desk-top size instrument [8] for calibrating sensors over a range of ±5 µm with a sensitivity to about 10 pm (dependent on counting times, etc.). The x-ray tube, monolith assembly and detectors are all mounted onto a 25 mm thick aluminium plate that forms the base of a box approximately 1.2 m long, 0.7 m high and 0.5 m deep. The box is constructed from plywood on a wooden frame and then lined with 4 mm lead sheet to give both a radiation enclosure and a highly damped acoustic environment. The x-ray tubeshield is fixed horizontally onto a subsidiary aluminium plate, along with a beam colimator that points downward at approximately at the Bragg angle. The plate rests kinematically on three ball-ended screws in the base-plate to allow final adjustment of the Bragg angle at the monolith. TV and photomultiplier detectors are provided on a motorized column so that alignment and counting modes can be selected with minimal disturbance to the local environment. The monolith, of a design very similar to Figure 1, rests kinematically upon an aluminium column that contains a pair of electromagnetic force actuators for the drive and twist-correction. The column sets adequately the position of the monolith without need of adjustments. It also acts as a heat sink for the actuators. Sensors under calibration are normally attached to a suitable carrier that rests kinematically located on the top surface of the monolith. Most can be contained within the box, with access for setting up through a small door in its top, but there is provision for an extension tower of the enclosure above the monolith for handling larger systems.

Any relative lateral vibration of the interferometer blades of more than 0.1 nm will smear out the fringes over the counting time, so control of vibration levels is critcal. All internal protection against noise and vibration is passive, generally by friction damping at butt-joints, some with entrapped thin rubber sheets. For example, rubber inserts in brackets and friction at the kinematic mounts of the tubeshield sub-assembly help to reduce the transmission of noise from the x-ray tube cooling water. Soft internal baffles absorb sound and also reduce convection currents. The monolith is a highly tuned mechanical system, and not overly sensitive to modest vibration levels at frequencies well away from its resonances. Thus, a few mm thick pad of polypropylene 'wool' placed between the base and the column supporting the monolith has proved very effective. The total vibrational energy transmitted is hardly reduced but it is smeared more uniformly over the sonic and sub-sonic frequency range. The slight reduction in mechanical stability is acceptable since the x-ray beam is not completely collimated and the crystal is always illuminated by an aligned fraction of it.

Providing good positional control within one fringe demands an actuator resolution equivalent to a few picometres displacement, so to also provide a range of some micrometres requires a controller of at least 20-bit precision. Although not trivial, especially since complete monotonicity and a high degree of linearity is needed, current servos of this quality are well within the capability of modern electronics at the relatively low bandwidth required. The stability of the drive and the mechanical systems are illustrated by figure 2. This shows the output count as the monolith is driven very slowly by stepping the drive signal up and then down again at a constant rate. The monolith uses the (111) silicon planes and so one complete fringe is covered in 0.316… nm. The whole trace covers approximately 30 minutes and the maximum displacement is about 0.5 nm, so the scan speed is about 2 nm h^{-1}. The whole enclosure was placed on four partially inflated tyres on a reasonably robust desk in a basement laboratory, temperature controlled only to about 2 K. The forward and reverse traces are virtually indistinguishable except, briefly, when the signal is lost. This event is known to correspond to a person entering the laboratory and the drop of the signal to the mean intensity is consistent with a vibration of the analyser blade. There is no memory of this event in the later signal.

4 PRECISION SLIDEWAYS AND SEPARATED BLADE INTERFEROMETRY

All the well-documented separated blade x-ray interferometer systems have depended for blade translation on linear flexure mechanisms. Suitably deep leaf-springs or notch-hinges provide high resistance to disturbances in axes other than the drive axis and their residual errors are highly repeatable. However, linear flexures have a very limited range compared to their own size. In principle, devices could be made having millimetres or more of total range with nanometric precision anywhere within it, but a slideway would be required for the translation axis. The plausibility of this approach has been assessed by constructing a translation mechanism that adheres strictly to the principles of kinematic design [9]. It uses five thin-film PTFE bearing pads running on a polished 'Zerodur' prismatic guideway and driven through a 10:1 reduction lever by a commercial feedback-controlled piezo-electric actuator. Levers and wedges allow the alignment of the slideway axis in the other five degrees of freedom. All the elements that define positions are made of 'Zerodur', except for the small sapphire balls used for the kinematic locations. The final performance was such that the available commercial laser interferometers could not detect the residual error motions.

A separated blade interferometer was set up using established processes, see [10]. A small silicon blade set was cut as a monolith. A slightly larger base was cut from silicon (to match thermal coefficients). The base has v-grooves in its top surface and the side parallel to the interferometer axis is optically polished. A slot was machined vertically almost all the way through the base. It was then glued onto the slide with the slot positioned above the gap between the moving and static parts. Six sapphire balls were placed in the grooves, held temporarily by small spots of lacquer, at positions suitable for kinematic mounts for both parts of the interferometer. A small amount of a special very slow curing epoxy adhesive was placed on top of each ball and the blade set rested on top of them in the correct position. The slow setting ensures minimum strain in the crystal. Once the adhesive was fully set, the lacquer was dissolved, the blade set lifted off and the analyser separated by sawing and etching. The remaining depth of the slot in the base plate was removed by gentle hand filing to allow the slide to move. The blade set could then be relocated kinematically on the base in almost its original position as a single piece. Drift in the slideway alignment can be adjusted using the mirror regions of the base and an autocollimator.

Final alignment of the blades, sufficient to generate fringes, can be achieved using the transmission Bragg 'rocking curve' [10]. The mean output intensity first rises and then falls away again as the incident x-ray beam is 'rocked' about the true Bragg angle. There will be a feature at its peak (the exact form depending on the blade thickness) that is no more than 75 milli-arc-seconds wide. Our system had a trough where the count rate was more than 1 kHz below the peak value of about 4 kHz. After alignment, low-contrast moiré fringes could be seen from time to time in the output beam when using the x-ray TV camera. There was some evidence that the fringe pattern moved with small translations of the stage, but the contrast was not stable even when the stage was static. The contrast loss is attributed to a combination of vibration and stick-slip at the many kinematic location points. The system was operated on a heavy, active air-supported optical bench in a radiation and thermal enclosure that has regularly been used satisfactorily for monolithic interferometers. Extra isolation was required before any fringes could be found with this system. The whole stage was supported on small, partially inflated pneumatic tyre inner tubes and foam polymer acoustic lining sheets were hung on wires around it in order to reduce the coupling of higher frequencies into the blade region. Tyre inner tubes are designed to have external support and the ones used here (around 200 mm diameter) tended to creep unless some reinforcement was added. Wapping them with a layer of self-adhesive 'pressure sensitive' tape (also called 'masking tape') has proved very effective. The mean intensity, observed by photon counting, with the Bragg angle set to the centre of the trough showed negligible change over a two-hour period and so the angular stability is good to a few milli-arc-seconds over such periods. Figure 3 shows the mean intensity while the stage was moving and again the angular alignment remains more than adequate for maintaining fringes. The slideway motion is satisfactory for use with a separated blade interferometer but its vibration performance is not. In the absence of a specific application, it was uneconomic to pursue this work beyond the initial demonstration of feasibility. Clearly slideway technology could be used but the bulky kinematic alignment mechanisms are not the best approach. Although requiring trailing wires to the stage, it would be preferable to trim the alignment of the sensitive axes by a small elastic mechanism mounted to the interferometer base.

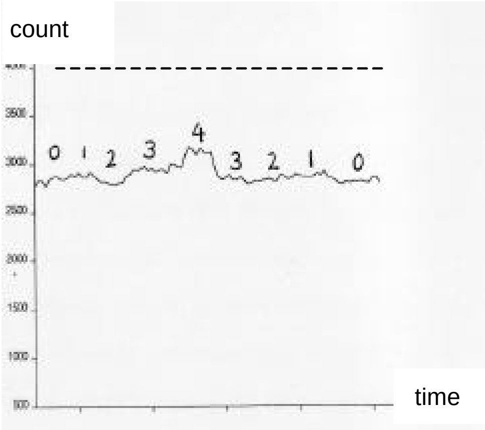

Figure 3. Intensity variation in the rocking-curve trough. Dotted line is peak level. Numbers show displacement in units of 100 nm

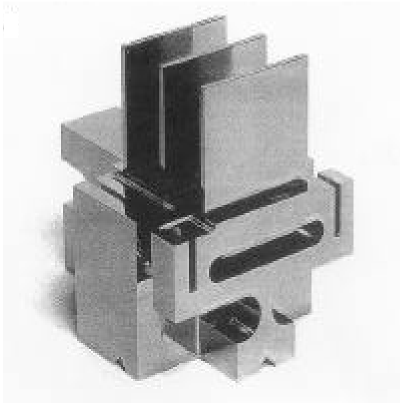

Figure 4. A silicon monolith for x-ray moiré fringes angle measurement. The blades are approximately 30 x 35 mm across.

5 AN ANGLE-READING X-RAY INTERFEROMETER

For small rotations, a, the moiré fringe spacing from a grating of pitch d is d/a. For a lattice parameter of 0.2 nm, a blade twist of only 0.2 µrad is needed to create x-ray moiré fringes as close as 1 mm pitch. In photon-counting mode, at most a small fraction of a moiré fringe can be tolerated within the photomultiplier window before contrast of the displacement fringes is seriously reduced, which is why so much control of parasitic motions is needed. However, with cost-effective TV x-ray imaging, we can exploit this effect by recording and measuring the fringes in order to assess very small angular displacements. Because of the reciprocal relationship between rotation and moiré pitch, the error associated with measuring that pitch has a proportionately smaller influence for nanoradian-level movements that are difficult to measure by other means. Figure 4 shows a silicon monolith specifically designed for angular measurements, with a notch hinge acting as a pivot for the rotation of the analyser blade. A very stiff hinge simplifies control of nanoradian rotations by relaxing the resolution demanded of the drive force transducers, at the cost of restricting the range.

Very good contrast moiré fringes have been obtained. Since they derive from an almost perfect, unstrained crystal, the fringes are straight and almost ideally sinusoidal with linear phase. The TV image can then be averaged line-by-line to reduce statistical noise. Also, a linear least-squares estimate of the change of phase with position provides a good estimate of the fringe pitch even when the field of view contains only a fraction of a fringe. Using a 6 mm wide x-ray beam allows determination of fringes of up to at least 60 mm pitch and the (220) lattice planes used by this device are spaced a 0.192… nm, so it resolves to better than 5 nrad. The largest rotation is limited by the image resolution to around 4 µrad for this device. In practice the fringes often rotate from their theoretical orientation, caused by the superposition of a Vernier type of fringe when the lattice parameters vary minutely between the blades. This will happen if there is a temperature gradient across the device, providing a very effective self-diagnostic against one of the principal error sources. The moiré fringe spacing can be extracted accurately even in the presence of this secondary rotation, so it can be used to monitor stability during normal operation.

6 CONCLUDING REMARKS

Demonstrations of x-ray interferometry over the last 15 years indicate that it could well become an important practical tool for meeting the steadily increasing demands for precision and traceability in industrial metrology. Operational speeds are likely to remain low and x-ray generators and associated safety systems are bulky, so it will be effectively restricted to off-line calibration. Future x-ray lithographic steppers for microelectronics fabrication might use optical metrology on their axes for speed but incorporate an x-ray interferometer for regular ‘compensation’ of those axes to match the silicon lattice. For example, a simplified COXI type of geometry could be associated with axis reference mirrors. Calibration of short-range precision gauges might use a transfer device based on, say, capacitive micrometry that has an in-built interferometer blade set. The whole device would be checked in an x-ray metrology system before and after each use to provide very good traceability to the lattice. This approach would allow higher speed, *in situ* calibration of instruments. Monolithic devices should suffice for all the applications envisaged. Long-range devices could be built, but would be very expensive. All the basic technologies now exist, but interesting challenges remain in producing adequate secondary gauging and control to make the calibrators easy to use. Industrial metrology systems must be referred to the monolith without introducing forces that cause error motions large enough to reduce fringe contrast unacceptably. How do we best create a 'non-influencing' coupling between mechanical systems that maintains sub-ångström traceability? High-gain linear gauging and 'null-servo' strategies can provide sufficient capability, but it is far from trivial. X-ray interferometry can certainly contribute to industrial metrology but perhaps only in major standards rooms.

REFERENCES

[1] U. Bonse, M. Hart, An X-ray interferometer, *Appl. Phys. Lett.*, **6** (1965) 155-156.
[2] M. Hart, An ångström ruler, *J. Phys. D: Appl. Phys*. **1** (1968) 386-388.
[3] R.D. Deslattes, Optical and X-ray interferometry of a silicon lattice spacing, *Appl. Phys. Lett*. **15** (1969) 386-388.
[4] D.G. Chetwynd, D.P. Siddons, D.K. Bowen, X-ray interferometer calibration of microdisplacement transducers, *J. Phys. E: Sci. Instrum*. **16** (1983) 871-874.
[5] G. Basile, A. Bergamin, G. Cavagnero, *et al*., A combined optical and x-ray interferometer for the calibration of high precision displacement transducers, in: H. Kunzmann *et al*. (eds) *Progress in Precison Engineering and Nanotechnology – Proc. 9th IPES* (Braunschweig 26-30 May 1997), PTB 1997, p. 292-294.
[6] D.K. Bowen, Applications of x-ray interferometry, in: A. Authier, B.K. Tanner (eds) *Dynamical X-ray Diffraction Theory and its Applications*, Plenum, New York, 1996.
[7] D.R. Schwarzenberger, D.G. Chetwynd, D.K. Bowen, Phase measurement x-ray interferometry, *J. X-ray Sci. Technol*. **1** (1989) 134-142.
[8] D.G. Chetwynd, N.O. Krylova, S.T. Smith, Metrological x-ray interferometry in the micrometre region, *Nanotechnology* **7** (1996) 1-12.
[9] A.F. Monteiro, S.T. Smith, D.G. Chetwynd, A super-precision linear slideway with anular correction in three axes, *Nanotechnology* **7** (1996) 27-36.
[10] P. Becker, Justierelemente und Justierungsmassnahmen, in: E-R Mewes *et al*. (eds), *Berichte uber Arbeiten am Röntgen-Verschiebinterferometer*, *Report Aph-8* PTB, Braunschweig, 1974.

ACKNOWLEDGEMENT

Parts of this work have been generously supported by the UK Department of Trade and Industry, the Engineering and Physical Sciences Research Council and the Paul Instrument Fund of the Royal Society. We also acknowledge with gratitude the large contributions of colleagues in the Centre to this body of work, especially Professor Keith Bowen, Dr Stuart Smith and Dr André Monteiro.

AUTHORS: Professor Derek G CHETWYND and Dr. Nina O. KRYLOVA, School of Engineering, University of Warwick, Coventry CV4 7AL, U.K., Phone Int. +44 2476 523121, Fax Int. +44 2476 418922, E-mail: dgc@eng.warwick.ac.uk