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METROLOGICAL CHALLENGES OF SYNCHROTRON RADIATION OPTICS*

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Metrological challenges of Synchrotron Radiation Optics*

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ABSTRACT

Modern third generation storage rings, require state-of-the-art grazing incidence x-ray optics, in order to monochromate the Synchrotron Radiation (SR) source photons, and focus them into the experimental stations.

Slope error tolerances in the order of 0.5 µRad RMS, and surface roughness well below 5 Å RMS, are frequently specified for mirrors and gratings exceeding 300 mm in length.

Non-contact scanning instruments were developed, in order to characterise SR optical surfaces, of spherical and aspherical shape. Among these, the Long Trace Profiler (LTP), a double pencil slope measuring interferometer, has proved to be particularly reliable, and was adopted by several SR optics metrology laboratories.

The ELETTRA soft x-rays and optics metrology laboratory, has operated an LTP since 1992. We review the basic operating principles of this instrument, and some major instrumental and environmental improvements, that were developed in order to detect slope errors lower than 1 µRad RMS on optical surfaces up to one metre in length.

A comparison among measurements made on the same reference flat, by different interferometers (most of them were LTPs) can give some helpful indications in order to optimise the quality of measurement.

Keywords: profilometry, optical metrology, surface metrology, x-ray optics, LTP

1.INTRODUCTION

One of the major advantages of Synchrotron Radiation (SR) sources, is their ability to provide high brilliance photons in the soft x-ray region. This part of the electromagnetic spectrum is of great interest, because many materials have strong absorption edges at these energies.

If one is able to monochromate these photons, the chemical state of atoms and molecules can be studied in depth, and consequently their interaction behaviour understood.

Unfortunately, from the point of view of the optics, this strong absorption is a disadvantage. As a matter of fact, the absorption properties of the materials make impossible the use of ordinary normal incidence optics schemes. Therefore one is forced to work in grazing incidence configurations. Mirror reflectivity decreases dramatically with increasing photons energy and incidence angle. For this reason, grazing incidence angles of 2 degrees or lower, are usually adopted by the SR beamline designers.

The typical shapes for synchrotron mirrors and gratings vary from plane to the more exotic aspherical forms (e.g. paraboloid, ellipsoid, toroids, and so on). Moreover, the parameters of these mirrors are rather variable. Ellipsoidal and toroidal optical surfaces, with radius (or equivalent radius) of curvature from 10 or 20 metres, up to some kilometres in the tangential direction, and close to a few centimetre in the sagittal one, are typically specified.

On the other hand, for such small incidence angles, every imperfection on mirrors and gratings optical surfaces, will result in drastically reduced overall performance of a multi-component beamline, designed to monochromate and focus synchrotron light.

Deviations from the ideal slope of a few µRad RMS, and surface roughness exceeding few Å RMS, might be sufficient to reduce substantially both, the energy resolution and the photon density required for the experiments.

Precise characterisation of each mirror or grating component to be installed at the beamline is therefore mandatory. Different solutions and different instruments have been adopted (and developed) by the optical manufacturer, as well as the x-ray optical laboratories to test, and consequently improve, the quality of the optics.

2.OPTICS METROLOGY AT ELETTRA

In order to verify the compliance of the delivered mirrors and gratings before their acceptance and subsequent installation at the Trieste *ELETTRA* SR source beamlines, an optics metrology laboratory was set up at our storage ring, and has operated some non-contact interferometers since 1992[1]. A ZYGO Mk.IV Fizeau-type interferometer was installed in a clean room. It is

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able to test flats up to 100 mm in length (direct view configuration), while in grazing incidence (autocollimation mode with a precision plane mirror) it is possible to test flats up to 500 mm in length. The accuracy for such instrument is of the order of $\lambda/20$, while the measurement repeatability is close to $\lambda/100$ (λ=633 nm). The current set up prevents us from measuring surfaces other than flat (or very large radius of curvature), but the instrument could be implemented for analysis of spherical or cylindrical shapes at any time, if needed. Another instrument, also available in the clean-room, is the MICROMAP Promap-512 phase measurement microscope. It is able to provide 3D surface roughness measurements, with a repeatability better than 1 Å RMS, and a lateral resolution close to 0.5 µm. It is mainly used for mirror surface finish characterisation. Moreover, it is also helpful in order to check the groove density of gratings up to 1000 lines/mm, or to characterise steps or defects on the optical surface. Sometimes it is also used to determine, in first approximation, the sagittal radius of curvature for cylinders or toroids, usually specified to be in the 30 mm to 100 mm range. The current configuration at our laboratory allows us to view sampled areas on the mirror surfaces up to 2.7 mm per 2.5 mm.

The most important interferometer we are operating at Elettra, is the Long Trace Profiler (LTP). This instrument was developed at the Brookhaven National Laboratories[2, 3] by Takacs et al., and marketed by Continental Optical Corporation. It is basically a double pencil, slope measuring interferometer, able to measure the slope error and radius of curvature for optical surfaces up to 1 m in length. Appropriately operated, it allows precise data acquisition, with a repeatability in the order of 10 nm P-V (or 0.2 µRad RMS).

3.LTP HARDWARE IMPROVEMENTS

As with many other commercial instruments, sometimes it is necessary to "personalise" the as-supplied set up, in order to improve the capabilities of the instrumentation to match your requirements. Some major hardware improvements were undertaken, to effectively increase the final accuracy of our LTP for SR optics metrology[4]. It was found that measurement noise was strongly reduced by substituting the solid-state laser source with a He-Ne laser tube, connected to the optics head by means of a polarisation preserving optic fibre. Following an idea developed by S.N.Qian et al.[5] at the Elettra laboratory, a new set up for the LTP were developed: the penta-prism LTP, with stationary optics head and scanning penta-prism. The main advantages of such a configuration were the introduction of an angle-maintaining penta-prism (less sensitive to the vibrations and the tilting errors of the scanning translation stage), a significant weight reduction of the movable part of the interferometer (with an obvious decrease of mechanical flexure of the scanning slide), and a side-mounting configuration for the surface under test (that greatly reduces the gravity induced deformation on the optical element under test).

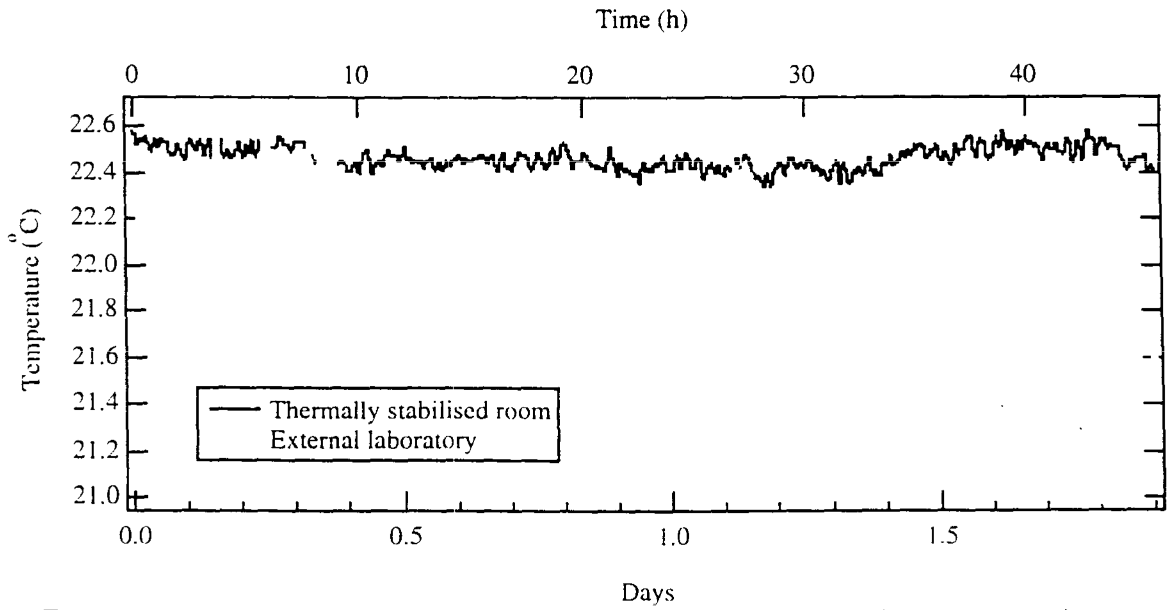

Fig.1 Temperature stability measurements inside the LTP interferometer hutch. On a two days timespan, the external environment has shown a temperature variation of almost 2 °C, while the LTP surroundings has varied for only 0.2°C peak-to-valley.

After these significant hardware improvements, the other parameters to be optimised were related to environmental changes while measuring (mainly temperature stability and air turbulence along the laser beam path). In our experience, the temperature changes that occur on the brief to medium time scales (i.e. comparable to the time required to collect a set of data) have a direct influence on the He-Ne laser source stability (due to slight changes in the laser cavity tube), in the polarisation-preserving optics fiber that feeds the laser head, in the LTP optics head mechanical assembly, and in the thermal expansion of the scanned surface blank.

For this reason we undertook a series of experiments, aimed at determining the acceptable temperature variation while measuring. It turned out that for the optics normally examined by us, a satisfactory repeatability on a series of subsequent scans was reached for a temperature stability close to +/- 0.2 °C on a time scale of one day. For this reason, in early 1995 we commissioned the construction of a temperature controlled room to host our LTP. This facility was ready at the end of that year, and has regularly operated since then. It consist of a double walled room, with adaptive temperature control by means of a circulating fluid system surrounding the hutch hosting the interferometer. Air circulation in the environment between the two walls ensures no thermal gradients from point to point of the inner hutch. Precise thermal monitoring at different points inside this inner room, has shown that the achieved temperature stability is of the order of +/- 0.2 °C, on time scale of several days (Fig. 1). The same shielded environment, greatly reduces air turbulence in the laser optics path, enhancing the final measurement accuracy.

An estimation of the overall stability of our set up, is achieved with an accurate monitoring of the temperature changes in the immediate surroundings of the LTP, by means of four thermocouples. Some "stability scans" (i.e. stationary beam data acquisition of a reference surface by means of the LTP) are also taken regularly along with the "real" data scans, in order to identify any undesired variation of the overall set up during the data collection.

4. CALIBRATION PROCEDURES

Some standard checks are usually scheduled to optimise the instrumental capabilities of our LTP. Among these, a scale-factor calibration is normally performed by measuring a spherical reference mirror (radius of curvature 10 m, and 100 mm length) that exploits the whole CCD aperture used for the data acquisition. A cross-check calibration is also done by means of a precise theodolite, in autocollimation mode. In this way it is possible to obtain an accurate scaling at the detector placed at the focus of the Fourier transform lens. Prior to each measurement session, a calibration of the optical alignment of our LTP with respect to the moving pentaprism is performed. From time to time, we also check the He-Ne laser source stability, by means of calibrated photodiodes.

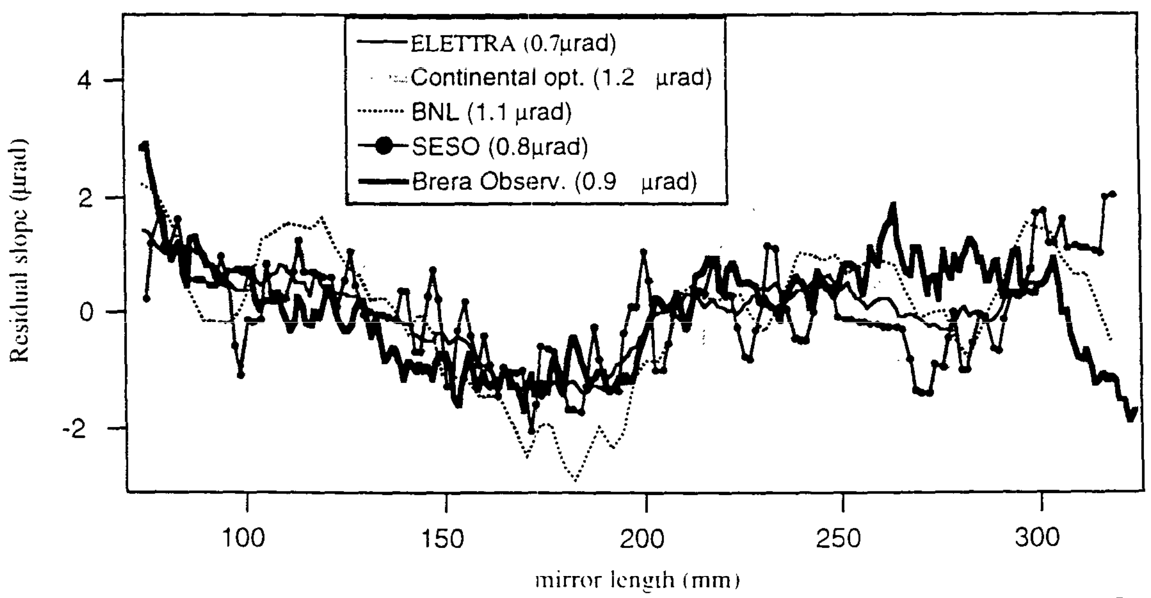

Fig. 2 Slope profiles as measured by different interferometers on the central 250mm of a reference flat mirror.

Further reference optics were provided to be used as standards to be tested with the interferometer. These are a plane mirror, a toroidal mirror, and a spherical mirror, all 400 mm long. Also a pair of convex and concave test plates are available for more tests.

A good source of information about the overall stability of our interferometer, is obtained scanning a precise reference flat, well characterised with a number of measurements made at different laboratories in Europe and the United States (Fig. 2).

This mirror was commissioned from the French company SESO, whose optics workshop delivered us a ZERODUR® reference flat, with a useful optical surface of 400x60 mm^2 and 80 mm thickness, with a planarity of $\lambda/10$ for the whole length of 400 mm, and of $\lambda/15$ for the central 250 mm. The slope error was specified to be 2 µRad RMS along the whole length.

Once the "real" shape of the optical surface was reliably established, periodic scanning of this mirror provides some helpful data in order to establish the stability of a given experimental set up. Usually there are two main sources of errors in each set of measurements: a random factor, due to instrumental and environmental noise, and a systematic deviation due to some residual miscalibration of each instrument. Comparing the scan results at different laboratories, it is clear that, as pointed out by Irick et al.[6], a frequent and random fluctuation in the slope profile will be more or less filtered out while integrating the data in order to obtain the corresponding height profile; vice versa a more gentle and continuous trend in the slope profile (like that introduced by a temperature drift) will result in a spurious radius of curvature in the height profile obtained from this data.

Only when mechanical and thermal equilibrium are reached, will a minimum in the value of these drift-induced radius of curvature errors be obtained. Even a small change in the temperature while scanning will provide a fictitious spherical profile. Leaving aside the radius of curvature, the residual profile gives information on the background noise influence at the laboratory.

5.FROM THE OPTICS LABORATORY TO THE BEAMLINE

All the considerations mentioned above, apply only to the optics laboratory procedures. Passing to the real operational situation, some additional issues should be considered. First, each mirror or grating is to be mounted on an appropriate holder. Typically it will be also directly connected to a cooling system and, finally, it will be positioned in Ultra High Vacuum.

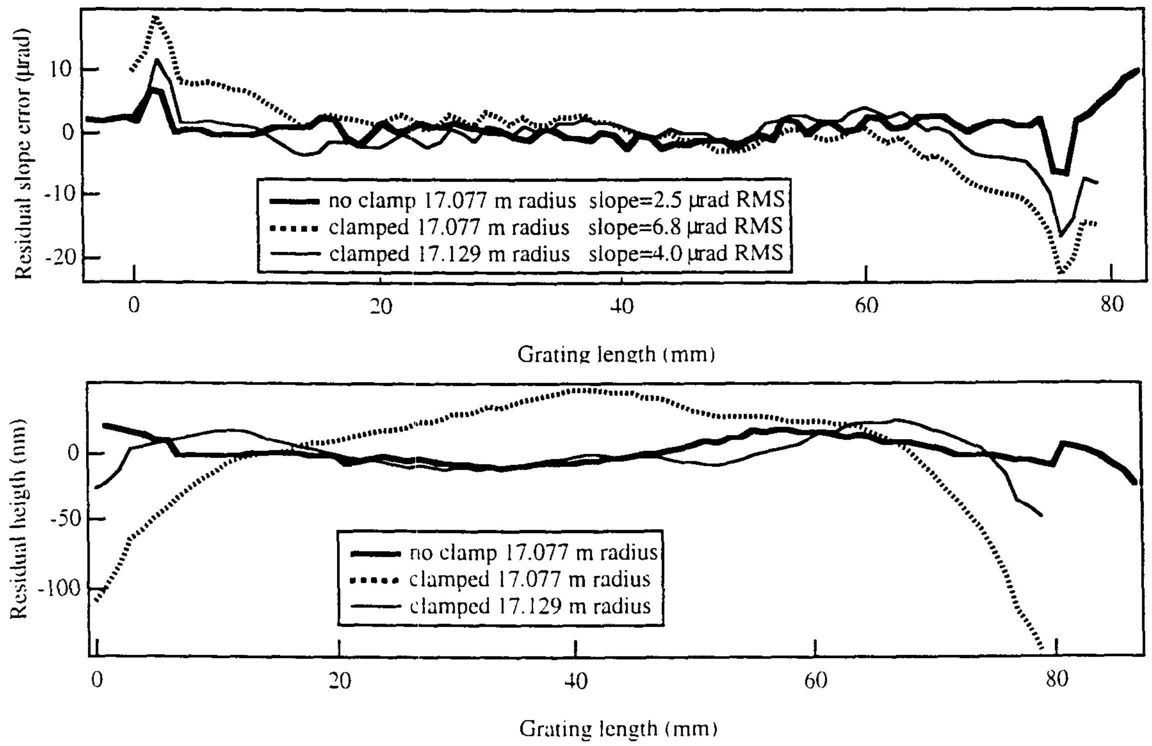

Fig. 3 Fixing an optical element onto its holder, sometimes provide a troubling distortion of its surface, due to the clamping force. Here is shown this effect on a SR spherical grating

This clamping operation will introduce some unavoidable deformation of the blank. and thus also in the optical surface of interest. It is very difficult for designers to calculate in advance the final distortion introduced by the clamping and cooling systems. but in most cases. is possible to scan the optics while ready to be mounted at the beamlines. As will be described by Cocco et al. elsewhere[7], we have undertaken some experiments to evaluate the extent of this clamping-induced deformation.

According to our measurements (Fig. 3), for a silicon carbide spherical grating of groove density 800 l/mm (size: 100 mm long, 40 mm wide and 30 mm thick), the top-end holding introduced a significant distortion in the optical surface. The residual height (peak to valley) after the subtraction of the specified 17 m sphere. passed from 30 μm to 150 μm, while the slope error passed from 2.5 μRad to 6.8 μRad RMS. The result was that, with the clamping, this optical element was significantly outside the given specifications. Even if the subtracted sphere has instead a radius of curvature of 17.129m (the best fit of the data file), the residual slope error will be 4 μRad RMS, also outside the given specifications. Some setting is therefore necessary before using this grating.

Another example of the deformation introduced by clamping, is shown in the case of a 250 mm long plane switching mirror, made with Glidcop. This item, is clamped in its final operating position by means of a joystick, fixed at its base with a flange having 6 screws. We measured its optical flatness with different locking force applied at the screws, and with a combination of screwing sequence at its base. From Fig. 4 it is clear that the specified slope error of 3.0 μRad RMS, is only matched for a certain force applied to the screws. This is another example that shows the utility of a precise metrological tuning of optical elements prior to their use.

One more issue that should be taken into account is the thermal-induced deformation by the synchrotron radiation beam, impinging onto the optical surface. As pointed out also with an experiment carried out at the Trieste light source by Qian et al.[8], this effect could not be completely compensated for in the design, and some in-situ interferometry should be considered, for the highest performance and most demanding beamlines[9].

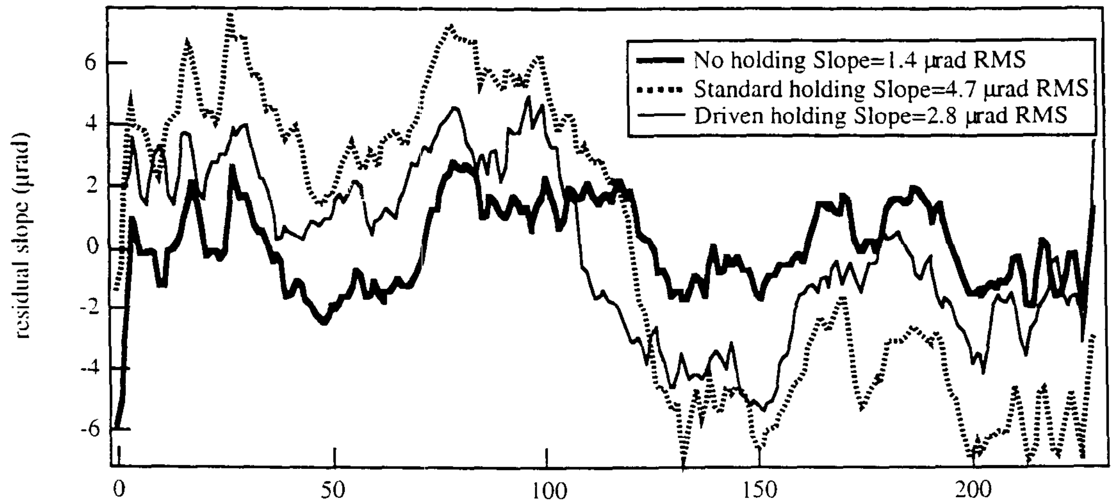

Fig. 4 Sometimes it's possible to optimise the figure of a given optics, just acting on its locking system. In this example, a GLIDCOP mirror, 250mm long, is driven into the specified flatness with a particular screwing force applied to its holding screws

6.DATA ANALYSING PROCEDURES

As pointed out by Ignette et al.[10], an agreed approach should be fixed, in order to evaluate the quality from a set of measurements. Sometimes, however, it is not possible to easily apply a standard analytical calculation to the data obtained with a long trace profiler, and some other appropriate procedure should be considered. In order to check the repeatability of our scans, usually we undertake a series of profile measurements on the mirror surface, taken along the same line, in backward and forward directions.

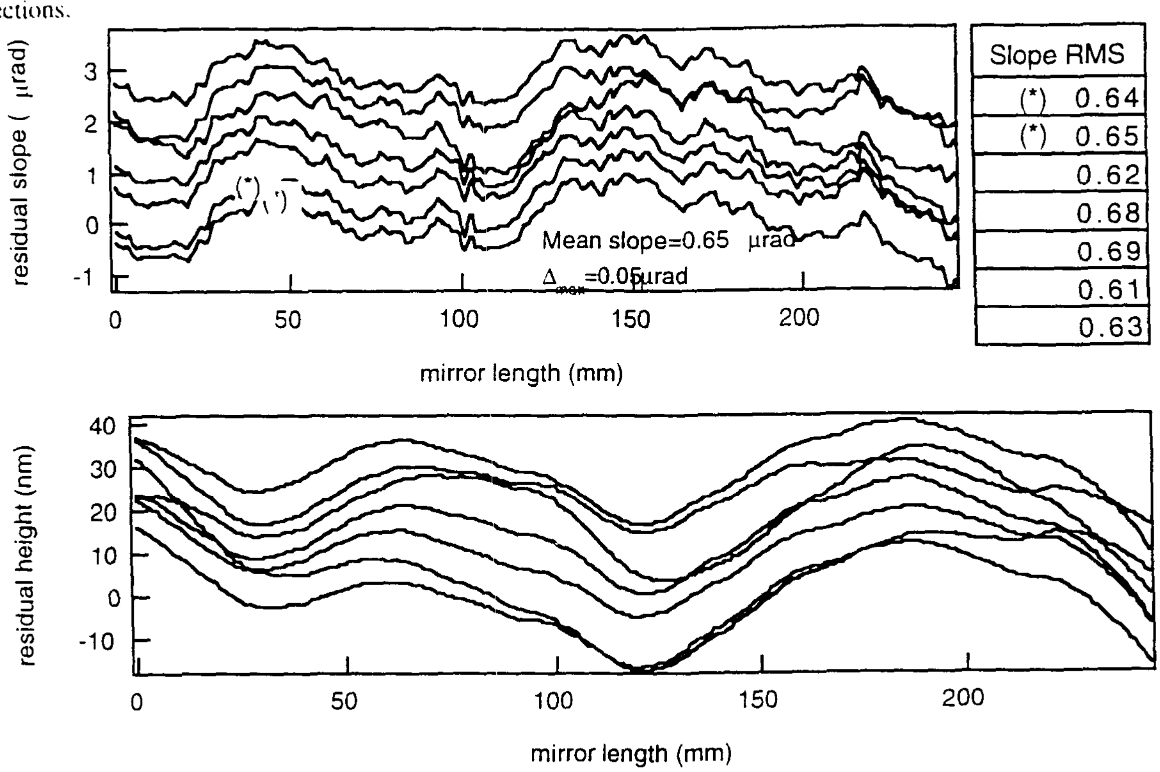

Fig. 5 Repeatability measurements on the central 250mm of a ZERODUR® reference mirror. A vertical shift was applied to the graphs, in order to separate each profile. The slope profiles marked with a (*) are made with the mirror rotated by 180° under the profilometer.

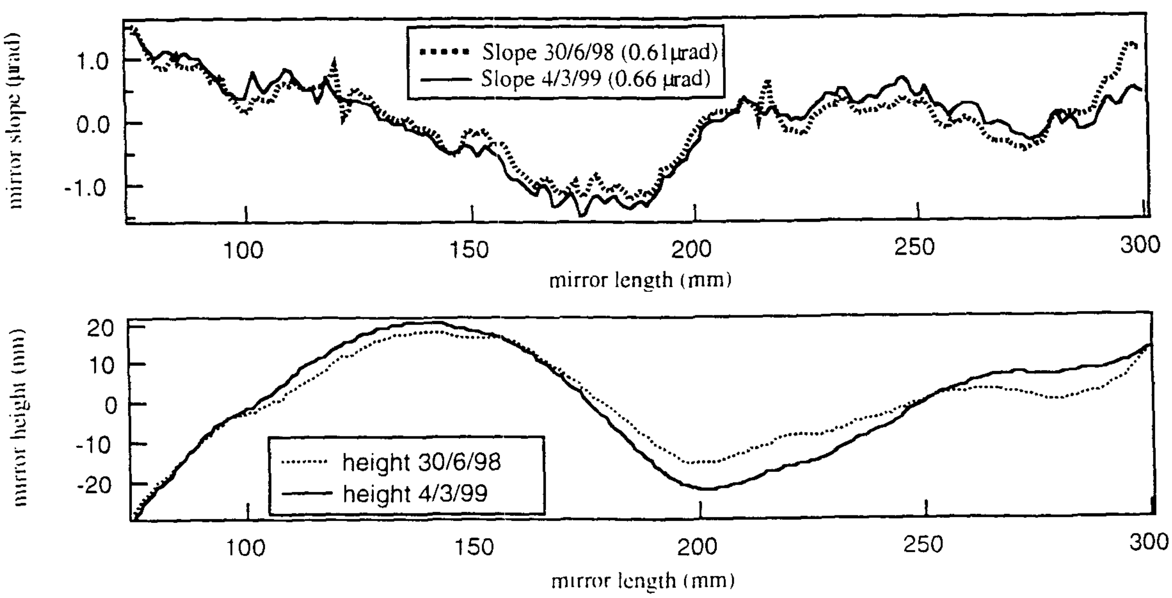

Fig. 6 Another example of the repeatability we can reach after the hardware and environmental improvements to our LTP: two scans, made on the same reference flat, shown an excellent agreement even if they were taken eight months apart.

Subsequently, we turn the mirror by 180°, and we repeat the same procedure for another series of measurements. Finally we compare the profiles obtained. This operation usually requires some days to be completed, and only if we find overall good agreement among our different data scans, can we assume that the set of measurements we collect is reasonably representative of the surface profile we examined. In other words, the repeatability we can reach is defined by the difference between the different scans. The lower this difference, the higher is the confidence in our measurement. This procedure was applied as well on the 400 mm reference flat above, usually employed for calibration purposes. As can be seen in Fig. 5, with the introduction of all the improvements listed above to our LTP, we were able to reach a fairly good repeatability level, across a set of measurements taken on a weekly basis. On these scans, as usual, the reference mirror was removed from its support, rotated by 180° under the LTP optics head, and again scanned back and forth. As shown in the same figure, the maximum difference in the residual slope error between each single measurement is lower than 0.05 μRad RMS.

The same good agreement could be seen also in two sets of data taken eight months apart from each other (fig. 6).

These two files give us another important information: due to the fact that the two sets was taken in different seasons (summer and winter, respectively) it is clear that our thermally stabilised room is also able to compensate successfully large external temperature variations, even over very long time-scales.

7.CONCLUSIONS

Current technology allows to accurately test the surface figure and finish of grazing incidence synchrotron radiation optics, down to 0.2 μRad RMS and 1 Å RMS repeatability. However, in order to match the required high degree of accuracy of most mirror and gratings used at third generation storage rings, some improvements and care are necessary to minimise the error sources, arising mainly from the hardware set up and the environmental conditions, sometimes found at metrological laboratories. Obviously, a calibration procedure should be established for the instrumentation in use. It is also worth considering providing some high-quality optics to be used as cross-check references between interferometers at different laboratories.

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