

Thin-foil reflection gratings for Constellation-X

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ABSTRACT

The Reflection Grating Spectrometer (RGS) on Constellation-X is designed to supply astronomers with high spectral resolution in the soft x-ray band from 0.25 to 2 keV. High resolution, large collecting area and low mass at grazing incidence require very flat and thin grating substrates, or thin-foil optics. Thin foils typically have a diameter-to-thickness ratio of 200 or higher and as a result very low stiffness. This poses a number of technological challenges in the areas of shaping, handling, positioning, and mounting of such optics. The most minute forces (gravity sag, friction, thermal mismatch with optic mount, etc.) can lead to intolerable deformations and limit figure metrology repeatability. We present results of our efforts in the manipulation and metrology of suitable grating substrates, utilizing a novel low-stress foil holder with friction-reducing flexures. A large number of reflection gratings is needed to achieve the required collecting area. We have employed nanoimprint lithography (NIL) - which uses imprint films as thin as 100 nm or less - for the high-fidelity and low-stress replication from 100 mm diameter saw-tooth grating masters.

Keywords: x-ray optics, Constellation-X, reflection gratings, thin-foil optics, blaze, sawtooth, replication, nanoimprint lithography, figure metrology, Shack-Hartmann

1. INTRODUCTION

X-ray telescope designs generally employ sets of grazing-incidence mirrors such as in Wolter I type or Kirkpatrick-Baez optics. The ideal grazing-incidence optic would be "all surface, no volume", thereby allowing dense stacking of optics, no intensity loss due to photons impinging on the "side" of the optic, and thus maximizing collecting area. Real optics, of course, have a finite thickness. Due to the small grazing-incidence angles required for efficient reflection of x rays the resulting optics are often many cm long along the optical axis. The "low mass/high collecting area" requirement for satellite-based x-ray optics leads to the desire to make them as thin as possible. However, thinner optics have lower stiffness, which makes it more difficult to give them a desired shape and mount them with minimal distortion, and which leads to lower angular resolution for the x-ray telescope. Well known examples for this effect are the differences in the angular resolution, often quoted as half-power diameter (HPD), achieved by the Chandra (HPD = 0.5 arcsec, optic thickness $t = 30$ mm), and XMM-Newton (HPD = 15 arcsec, $t = 0.9$ mm) missions, and expected for the ASTRO-E2 (HPD = 1.8 arcmin, $t = 0.2$ mm) mission.

The moderate angular resolution demonstrated so far with thin-foil optics requires high dispersion to allow spectroscopy with high energy resolution. This requirement favors reflection gratings as dispersive elements over transmission gratings or calorimeters in the soft x-ray band. Since x-ray reflection gratings also operate at small angles of grazing incidence the same considerations as outlined above for x-ray mirrors apply. (One might be tempted to say that thin-foil grazing incidence optics beget more thin-foil grazing incidence optics.)

The design for the future Constellation-X¹ mirror assembly - the Spectroscopy X-Ray Telescope (SXT)² - calls for a Wolter I optic with an angular resolution of 15 arcsec (HPD) made from 0.4 mm-thick shell segments. For spectroscopy in the 0.25 - 2 keV photon energy range an array of flat, thin-foil-like reflection gratings, the Reflection Grating Spectrometer (RGS) - is to be placed into the converging beam downstream of the SXT. The current design assumes the RGS to consist of thousands of gratings of size 100×140 - 200 mm² and of thickness

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around 0.5 mm. Flatness of individual gratings has to be better than 2 arcsec. This requires challenging low-spatial frequency figure errors below 0.5 micron over the surface of a grating.

We have previously developed a Shack-Hartmann (SH) wavefront sensing configuration that allows us to measure the figure of such large area optics with sub-50 nm accuracy.^{3,4} In the next Section we briefly describe an extension of the dynamic range of our SH instrument for the measurement of optics with deviations from flatness on the order of hundreds of microns.

A crucial step towards achieving submicron figure errors on foil-like optics is to build a setup that allows measurement of their *stress-free* shape repeatably. In Section 3 we discuss the requirements and design for such an ultra-low distortion, thin-foil metrology holder that we recently completed in our laboratory and that so far achieves 50 nm figure measurement repeatability.

Much work will go into shaping the thousands of grating substrates required for Constellation-X. Further processing is required to supply the substrates with a surface relief grating pattern of high diffraction efficiency. These processing steps should be fast, economical, and cause only minimal distortion to the figure of the optic. In the past we have demonstrated the fabrication of high-efficiency reflection gratings for both the in-plane^{5,6} and off-plane⁶⁻⁸ geometry. Using those gratings as masters we have investigated nanoimprint lithography (NIL) as a promising replication technique to achieve the above goals. We present recent results in Section 5.

2. DYNAMIC RANGE OF A SHACK-HARTMANN WAVEFRONT SENSOR

We have previously described our deep-UV Shack-Hartmann wavefront sensor metrology setup for figure measurement of thin transparent optics.⁴ It was designed to have a large viewing area ($> 140 \times 100 \text{ mm}^2$), high angular resolution ($10 \mu\text{rad}$), and sufficient dynamic range (0.3 mrad). Light from a broadband mercury arc lamp is collimated, reflected from the optic under test (OUT), and demagnified for input into the Shack-Hartmann wavefront sensor, which consists of a 64×64 lenslet array. Each lenslet focuses its segment of the wavefront onto a CCD detector in the lenslet focal plane. By comparing focal spot locations between those reflected from a reference flat and those reflected from the OUT a slope difference map is calculated, which is then integrated to reconstruct the wavefront after reflection from the OUT. When the surface slope of the OUT gets too large the corresponding focal spot can move into an area on the detector associated with a different lenslet (area of interest, or AOI), which leads to errors in the original wavefront reconstruction algorithm and limits the dynamic range to about $350 \mu\text{rad}$. This limits our setup to optics with a maximum slope of less than about $50 \mu\text{m}$ over 140 mm. Grating substrate candidates such as off-the-shelf flat panel display glass or inexpensive silicon wafers often exceed this range, limiting our choices to more expensive preselected substrates. Ideally we also want to be able to follow progress in the shaping or flattening of substrates with a single instrument over the whole range of figure changes.

We have since developed our own software that allows the mapping of focal spots to lenslets even when spots wander into neighboring AOIs, as long as they do not cross each other. Theoretically this extends the dynamic range of the instrument by up to a factor of 64, depending on the actual shape of the OUT. Our method is qualitatively similar to the one described by Groening *et al.*⁹ The original reconstruction algorithm often failed for silicon wafers with a bow around $10 \mu\text{m}$. With our new software we so far have obtained reliable surface maps for optics with deviations from flatness up to $100 \mu\text{m}$.

Repeated measurements of the same unmoved object result in surface topography differences on the order of 5 - 20 nm. After removing and replacing either a stiff optical flat or a 100 mm diameter silicon wafer surface maps differ by roughly 36 nm peak-to-valley or 14 nm rms. The accuracy of surface maps is estimated to be better than 17 nm.⁴

3. THIN OPTIC DEFORMATION

The goal of the technology development for the Constellation-X RGS is the fabrication of light-weight and high-efficiency reflection gratings with sub-micron flatness in their stress-free state. However, due to their large length-to-thickness ratio of 200 or higher even very small applied forces might lead to deformations that exceed the flatness requirement. In our 1-*g* environment these thin optics need to be constrained by some sort of holder

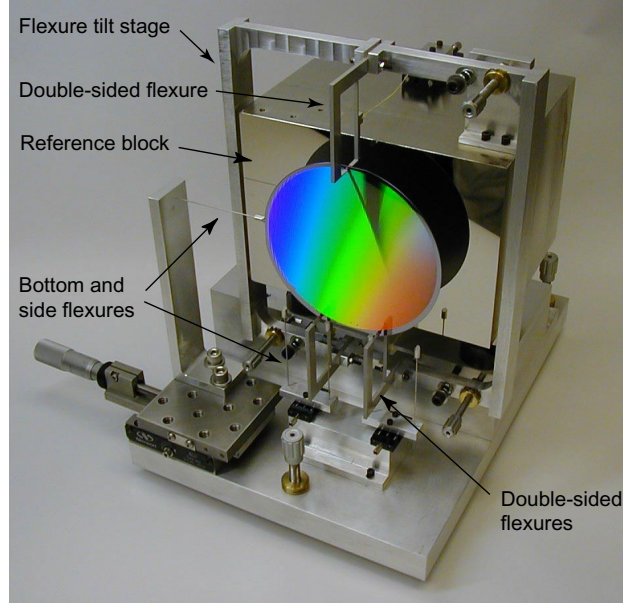


Figure 1. Ultra-low-stress thin-foil optic metrology holder, holding a 100 mm diameter silicon wafer that serves as grating substrate.

or mount, be it for figure measurement, alignment, or assembly. The main sources of stress that affect the figure of such thin-foil optics are gravity, thermal mismatch between optic and holder or mount, and friction between optic and holder during optic placement.¹⁰

3.1. Gravity sag

A thin-foil optic with the above dimensions made of glass or silicon will sag due to its own weight by many microns when supported horizontally at three points. For flat optics the effect of gravity sag can be practically eliminated through vertical orientation of the optic (with the normal of the reflecting surface pointing in the horizontal direction). For an ideally flat thin foil optic of length L and thickness t supported at its edges one can derive the maximum deviation from flatness δ_{max} due to gravity sag to be¹⁰

$$\delta_{max} = \frac{\rho g \sin \theta L^4}{6.4Et^2}, \quad (1)$$

where g is acceleration due to gravity, ρ is the mass density of the optic, E the Young's modulus of its material, and θ is the angular deviation from the vertical orientation. In order to keep gravity sag below 50 nm for a 140 mm long and 0.4 mm thick glass foil ($\rho = 2.53 \text{ g/cm}^3$, $E = 72.9 \text{ GPa}$) we therefore need θ to be below 80 arcsec.

3.2. Thermal mismatch

Differences in the rate of thermal expansion between the optic and its holder can lead to deformations in the thin foil optic. For example if we consider a 140 mm glass foil constrained at its ends by an aluminum holder a change in temperature by 1 degree C would lead to a difference in expansion of about $2 \mu\text{m}$. Following a simple geometrical argument that assumes the worst-case scenario of an incompressible and infinitely soft optic as well as fully constrained endpoints the optic will bow close to its midpoint by about $325 \mu\text{m}$, which exceeds our tolerances by far.¹⁰ In reality we expect deformation due to differential thermal expansion to be negligible as long as the resulting forces remain below the buckling limit of the optic.

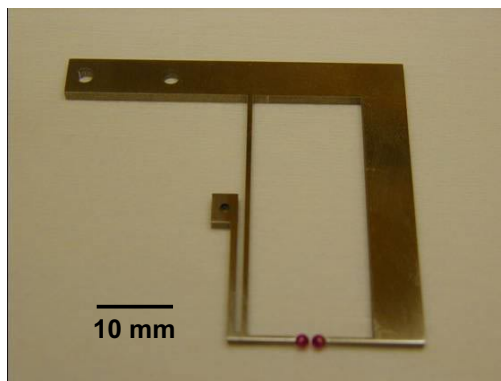


Figure 2. One of three double-sided flexures for the thin foil holder.

3.3. Friction

Our optics need to be supported against gravity, whether we want to perform shape metrology, alignment, or assembly. In the vertical orientation the optic is standing on its thin side, and its weight is easiest supported from below. The optic will need to be actuated horizontally for placement in a metrology apparatus or mounting module. If this actuation takes place some distance from the contact point between the optic and its supporting surface any friction at the contact point might prevent the optic from sliding, and the actuation force can lead to distortions in the optic. Even if the actuation point is only 1 mm from the point of friction we expect worst-case deformations on the order of $2\ \mu\text{m}$ towards the center of our optic for a previously proposed mounting scheme.¹⁰

4. THIN FOIL METROLOGY HOLDER

Based on the above sources of deformation and other considerations we designed a thin foil metrology holder with the following functional requirements: The holder must be able to hold thin foil optics (up to 1.6 mm in thickness) repeatably in an orientation that does not deviate from vertical by more than 70 arcsec. Thermal expansion and friction effects are mitigated to the extent of being negligible compared to the repeatability of our measurement system.

The holder is shown in Fig. 1. It consists of an aluminum reference block that has an optically polished, nickel coated front reference surface with $0.1\ \mu\text{m}$ flatness. The reference block has an inclinometer mounted to it with 14 arcsec ($\sim 68\ \mu\text{rad}$) resolution and 36 arcsec repeatability. The reference block rests on a base with $2\ \mu\text{rad}$ tilt resolution to allow for accurate alignment of the reference surface to the gravity vector.

The thin foil optic rests on long and narrow bottom flexures with very small lateral stiffness which minimize deformation due to friction between flexure and optic as described in Section 3.3 above. A similarly designed side flexure serves as a stop for repeatable horizontal positioning in the direction parallel to the reference surface. In order to reduce friction each bottom flexure has a small sapphire rod embedded in its top surface that contacts the optic and that is oriented with its axis parallel to the optic normal.

Positioning a thin foil optic on these bottom and side flexures minimizes deformations due to friction, but is inherently unstable. The foil optic is aligned to the reference flat and held in place at three points by double-sided flexures that pinch the optic between two small, precisely juxtaposed ruby balls (see Fig. 2). These three flexures in turn are rigidly mounted to a flexure tilt stage with 2 degree adjustment range in pitch and yaw and 1.8 arcsec adjustment resolution.

The monolithic double-sided flexure design is realized via wire electrical discharge machining of stress-relieved aluminum. The part of the flexure that runs quasi-parallel to the optic surface provides the necessary small preload to keep the optic in place, allows for the insertion and removal of optics, and adjusts to a range of foil optic thicknesses (from 0.3 to 1.6 mm). Misalignment of the ruby balls is designed to be small enough as to only cause negligible distorting moments. The part of the flexure that runs normal to the optic is designed to

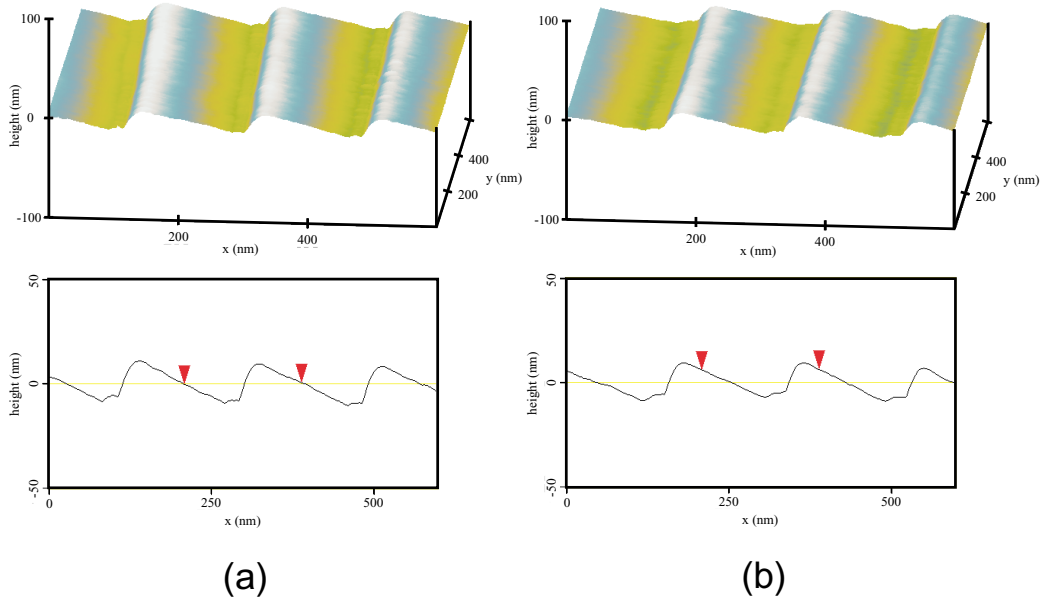


Figure 3. Atomic force micrographs of 200 nm period sawtooth grating replicated via nanoimprint lithography. (a) Thermal-cure NIL. (b) UV-cure NIL.

be compliant enough in the direction along the optic surface to allow for differential thermal expansion between optic and holder up to a temperature difference of 1 deg C before slipping occurs.

We have so far conducted a number of tests with the SH sensor to evaluate the performance of the optic holder. Repeated removal and replacement of a 100 mm diameter and 400 μm thick silicon wafer in the holder shows a repeatability of 55 nm (peak-to-valley) in the reconstructed surface-height maps. This puts an upper limit on the effects of friction and random errors in the placement of the optic. Changes in temperature during these tests were negligible. Placing the optic into the holder and remeasuring its figure over three hours during which the temperature changed by 1 deg C shows figure changes of less than 50 nm.

So far the optic holder seems to perform as expected, allowing reliable figure metrology on 400-500 μm thick thin-foil optics up to $100 \times 140 \text{ mm}^2$ in area. Further systematic tests are underway.

5. GRATING REPLICATION WITH NANOIMPRINT LITHOGRAPHY

There are currently two grating mounts under discussion for the RGS.¹¹ The in-plane mount has the plane of incidence normal to the grating grooves, and the dispersion direction is along the grating normal in the plane of incidence. In the off-plane mount the incident beam is quasi-parallel to the grating groove direction, leading to conical diffraction. The main difference in terms of grating fabrication is that the latter geometry requires an order of magnitude higher line densities and blaze angles than the in-plane mount. We have previously fabricated blazed reflection gratings from miscut silicon wafers for both diffraction geometries with $\approx 0.2 \text{ nm}$ roughness and very high - in some cases close to theoretical - diffraction efficiency.⁵⁻⁸ However, due to the considerable time and effort involved in patterning and chemically processing these silicon gratings it is very desirable to use them as masters in a suitable replication process.

In the past surface relief gratings were typically replicated using many micrometer-thick layers of epoxy on a thick blank. For our stringent figure requirements on thin-foil substrates this technology would lead to intolerable optic distortions due to thin-film stress, epoxy shrinkage during curing, and epoxy film thickness variations. In contrast, NIL uses polymer or monomer films less than 100 nm thick, which greatly reduces the potential for the above problems and minimizes possible outgassing effects. We recently demonstrated good replication of groove profiles with an in-house NIL setup and high diffraction efficiency for the replica.⁶⁻⁸

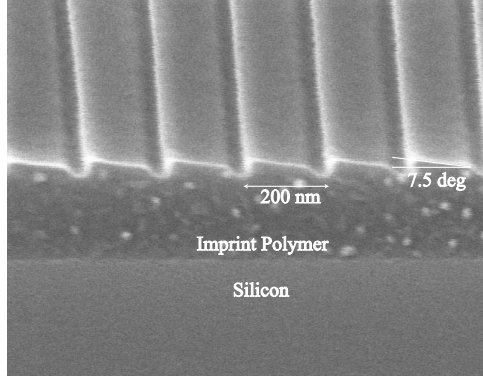


Figure 4. Scanning electron micrograph of a grating replica made by thermal-cure NIL. The groove profile is sharper than in Fig. 3(a) due to the absence of AFM tip size artefacts.

We are currently collaborating with a commercial NIL tool vendor and have investigated two different NIL techniques.¹² Our imprint master is a 100 mm diameter silicon wafer that has a 200 nm period sawtooth grating with a 7.5 deg blaze angle etched into it.^{7,8} In the case of thermal-cure NIL the master is pressed into a thin layer of thermoplastic (NXR-1020) at elevated temperatures (120 deg C). The replica substrate is a 100 mm diameter, double-side polished, $\sim 500 \mu\text{m}$ thick silicon wafer. Master and replica are separated at room temperature. In the case of UV-cure NIL the template is pressed into a liquid layer of UV curable polymer (NXR-2010/NXR-3020) at room temperature. The polymer is cured via UV exposure through the transparent replica substrate, a 100 mm diameter, $500 \mu\text{m}$ thick fused silica wafer.

Using the same master as in previous studies we have obtained replicas with sharper groove profiles (see Figs. 3 and 4). However, after coating the replicas with evaporated Cr/Au or Ti/Au for better x-ray reflectivity, AFM data show some degree of degradation in the groove profile, probably due to stress in the metal film. Nevertheless, very recent synchrotron measurements that are still being analyzed indicate higher diffraction efficiencies from the commercially imprinted replicas (43% peak absolute efficiency in first order versus $\sim 32\%$ in previous work⁶ at a wavelength around 2.5 nm, where the reflectivity is predicted to be $\sim 65\%$). Preliminary analysis seems to indicate a groove efficiency (sum of all diffracted orders divided by reflectivity) well above 90%, which agrees well with the low level of scatter observed between orders. Low-stress sputter deposition of the metal layers or the use of a stiffer material as the imprint layer might reduce rounding of the groove profile and could lead to even better x-ray performance.

A key question is whether the NIL process itself will degrade the figure of our thin-foil substrates. We therefore measured the shape of the replication substrates with the above UV-Shack-Hartmann wavefront sensor,⁴ with the substrates being carefully mounted in our thin optic metrology holder. Since we are only interested in changes in the figure of the substrate we actually measure the back surface before and after NIL. This eliminates bias due to possible changes in reflectivity from the imprinted grating layer on the front surface.

The figure of the optics is analyzed in terms least-square fits to Zernike polynomials. For small distortions in the linear-elastic regime of the material, out-of-plane distortion induced by thin films can be shown to be related only to the change of the Z_{21} Zernike coefficient,¹³ which is often referred to as defocus. The radius of curvature R of a round optic of diameter d is related to Z_{21} via¹³

$$\frac{1}{R} = \frac{16Z_{21}}{d^2}, \quad (2)$$

i.e. the often used term wafer bow¹⁴ corresponds to $2Z_{21}$. We find that changes in Z_{21} , as well as in other relevant Zernike coefficients, due to both NIL processes remained within the repeatability of the measurement, which was on the order of 40 nm, and which is an order of magnitude smaller than the flatness tolerance for

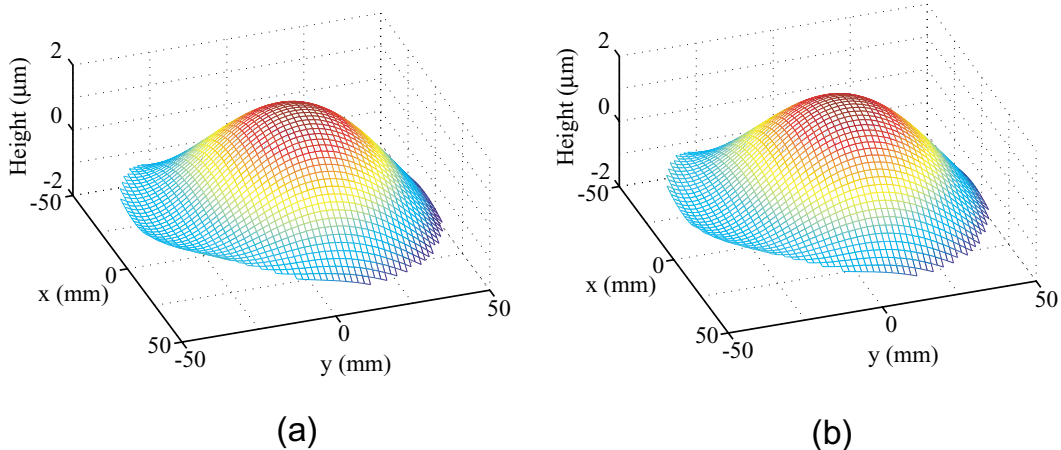


Figure 5. Silicon wafer surface map reconstruction from Shack-Hartmann data. (a) Before NIL ($3.601 \mu\text{m}$ P-V). (b) After thermal-cure NIL ($3.625 \mu\text{m}$ P-V).

Constellation-X (see also Fig. 5). Combining these results with Stoney’s equation gives an upper limit for the thin-film stress σ in the polymer layer:

$$\sigma = \frac{8 E_{bi} t_s^2}{3 t_f d^2} \Delta Z_{21} < 10 \text{ MPa}, \quad (3)$$

where E_{bi} is the biaxial elastic modulus of the substrate, t_s its thickness, and $t_f \approx 85 \text{ nm}$ the thickness of the thin film. We also tested our method on a silicon wafer with a high-stress, 20 nm thick evaporated chrome film. ΔZ_{21} was on the order of $2 \mu\text{m}$, from which we derive a thin-film stress of 1.7 GPa , which is in agreement with previous results obtained with a different technique.¹⁴

Our tests show that both thermal and UV-cure NIL are techniques well suited for the high-fidelity, low-distortion and low-cost replication from sawtooth grating masters for the fabrication of reflection gratings for Constellation-X.

6. SUMMARY

We are developing technology for the fabrication of x-ray reflection gratings that fulfill the challenging requirements for future x-ray telescopes such as Constellation-X. These requirements result in thin-foil-like dimensions for the grating substrates and therefore easily distorted optics. We have identified the main sources of distortion in the manipulation of such thin optics and designed and built an ultra-low distortion holder that - in combination with our Shack-Hartmann setup - allows us to perform highly repeatable figure metrology on these optics. Repeatable metrology is an important first step in the shaping of thin-foil optics to figure tolerances below $0.5 \mu\text{m}$. The lessons learned during this project will be helpful in the design of low-distortion schemes for the alignment and assembly of many thin-foil optics into grating modules.

We have investigated the high-fidelity replication of blazed reflection gratings with nanoimprint lithography. Both the thermal-cure and the UV-cure process resulted in excellent replication of the master groove profile. Preliminary x-ray measurements on metal coated replicas indicate very good diffraction efficiency. Thin film stress from the sub-100-nm thick polymer films used in NIL leads to negligible substrate distortion. Thus grating replication via NIL presents itself as a very promising technology for the economical fabrication of the large number of reflection gratings needed for missions such as Constellation-X.

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