SURFACE CHARACTERIZATION OF SUPERPOLISHED SUBSTRATES FOR X-RAY AND NEUTRON MULTILAYER OPTICS

D. Vernani, G. Pareschi, F. Mazzoleni, D. Spiga, R. Valtolina

Brera Astronomical Observatory, E. Bianchi 46, 23807 Merate, Italy
email: Dervis.Vernani@media-lario.com

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Abstract
Experimental results from surface characterizations of substrates employable in X-ray, and neutron multilayer optics are presented. A selection of materials, fused silica, silicon, borofloat and nickel, have been prepared with a super-polished surface roughness by different techniques such as: lapping powders; chemical etching; floating and electroplating process. The surfaces have been studied with topographic techniques of optical profilometry and Atomic Force Microscopy (AFM) in addition to X-ray scattering measurements (XRS). Fractal behavior of these samples was analyzed by using an inverse power-law into a wide spatial frequency bandwidth. Fractal parameters and rms roughness are quantities utilized in order to exhibit and compare the substrates quality.

Introduction

In recent years the development of modern microtechnologies and nanotechnologies in microelectronics, micromechanics, X-ray and neutron optics has generated in increasing attention towards methods of controlling ultra smooth surfaces. For x-ray and neutron optics a suitable way to analyse problems of low reflection efficiency of the incident beam at greater than critical angles, consists on the use of interferential multilayer mirrors rather than the more common single-layer. The final surface finish of such multilayer coatings is known to be strongly influenced by the starting roughness of the substrate on which the films are to be deposited and depends also on both the deposition techniques and the physical properties of the materials involved. Surface finish requirements for X-ray, and neutron optics that are capable of maintaining high reflectance have been the subject of research of several authors in the last decade [1, 2]. In 1998 D. G. Stearns, in an in depth study concerning the non-specular X-ray scattering in a multilayer-coated imaging system [3], has clearly demonstrated how the final topography of a multilayer coating can be predicted by a linear growth model that takes into account the initial topography of the substrate and the mechanisms of relaxation of the materials deposited on it. In such a works the presence of a correlation between the roughness of substrates and the roughness of multilayer-coated optics, especially within the spatial frequencies band extending approximately between 1 and 100 m^{-1}, has been demonstrated.

In experimental tests to evaluate the finishing level of optical surfaces several parameters are involved, however the root-mean-square (rms) roughness is undoubtedly the most significant parameter with respect to both reflectivity improvement and reduction of scattering effects. For a linear profile measured along a scan length L, the rms roughness $_{rms}$ is expressed as [4]

$$_{rms} \quad \sqrt{\lim_{L} \frac{1}{L} \int_{L/2}^{L/2} z^2(x)dx}, \tag{1}$$

with L being the scan length and $z(x)$ the height deviation from the mean value at the spatial coordinate x. It is common practice to treat the data obtained by different experimental techniques in terms of Power Spectral Density (PSD). PSD is defined as the squared absolute value of the height distribution Fourier transform, averaged on the scan length. For a linear profile, the PSD function is therefore described as [5]

$$PSD_1(f_x) \quad \left| \lim_{L} \frac{1}{L} \int_{L/2}^{L/2} z(x)\exp(\ 2\ ixf)dx \right|^2, \tag{2}$$

where f_x represents the linear spatial frequency of the height distribution. The linear PSD units are typically nm^3. For a given range f_x of spatial frequencies, the squared rms roughness of the measured profile is related to the PSD by the following equation [5]:

$$^2_{rms} \quad \int_{f_{\min}}^{f_{\max}} PSD_1(f_x)df_x, \tag{3}$$

The concept of roughness can, however, be replaced by an exponent that refers not the roughness itself but to the way in which it changes when correspondingly the observation scale changes. Fractal objects in nature are the same on different observation scales and super-polished optical surfaces show fractal behavior frequently. The corresponding PSD follows a power-law with a variable spectral index n [6]:

$$PSD_1(f_x) \quad K_n / 2f_x^n. \tag{4}$$

In the equation (4) $1<n<3$ and K_n is a constant with the units of $(length)^{3-n}$, numerically equal to $2\ PSD_1(1)$. This means that a log-log plot of the power spectrum is a straight line with slope ($-n$) and value $PSD_1(1)$ at $f_x = 1/length$. Fractals have the unique quality that their power spectrums can be characterized by only two quantities: n and K_n. The closeness to the upper limit of the exponent n will be a merit criterion since it will determine a PSD dominance at lower spatial frequencies which cause scattering away

from the specular direction at very small angles. It should, however, be noted also that the proportionality constant K_n plays a leading role, a greater value of n means that the amplitudes of roughness decrease more steeply for increasing spatial frequencies depending on the absolute values of these amplitudes.

The following characterizations are presented: a sample consisting of super-polished fused silica produced by GSI Group company (GSI Group Inc., Billerica, MA, USA); a monocrystalline Silicon substrate representative of mass-production for microelectronic applications manufactured by MEMC multinational (MEMC Electronics Materials Inc., St. Peters, MO, USA); a borosilicate glass substrate produced by SCHOTT company (SCHOTT AG, Mainz, Germany); a replicated electroformed Nickel substrate realized by MEDIA LARIO Tech. company (MEDIA LARIO S.r.l., Lecco, Italy).

Sample preparation

The first sample presented is a General Optics, which is the commercial name of the super-polished fused silica manufactured by GSI Group company (GSI Group Inc., Billerica, MA, USA) that are used in general as standard reference for the polishing degree of optical surfaces. Fused Silica is a high purity synthetic amorphous silicon dioxide that combines a low thermal expansion coefficient (CTE) with good optical qualities. The production of fused silica is an energy intensive process involving the electrofusing of silica to form a solid mass whose properties are enhanced as a result of the process. The surfaces of these samples are first machined and then polished by lapping with abrasive powders. The specimen considered in this work is a 2 in. diameter super-polished fused silica flat sample (thickness ~ 9 mm). It should be noted that the sample used in this case was not in its original factory supplied condition due to the fact that it had been used several times as master for the replication of substrates by means of a Nickel electroforming process. In the following we will refer to this sample with the acronym GO.

The second sample presented is a mono-crystalline Silicon wafer representative of a mass-production manufactured by MEMC multinational for microelectronic applications (MEMC Electronics Materials Inc., St. Peters, MO, USA). These Silicon wafers are sliced from monocrystalline Silicon “ingots” previously grown via the Czochralski (CZ) growing process, where a crystal seed is rotated and at the same time drawn upwards from a molten polysilicon material. Once sliced they are loaded into a precision lapping machine which uses pressure from rotating plates and an abrasive slurry to ensure a uniform removal of saw damage present on both front and backside surfaces. Chemical etching is then used for the removal of residual surface damage caused by lapping, and a thin layer of silicon is deposited on the polished surface, known as the epitaxial layer. The specimen considered in this work is a standard quality 4 in. diameter n-type monocrystalline (1,1,1) 0.5 mm-thick silicon wafer. In the following we will refer to it with the acronym SW.

The third specimen considered is a borosilicate glass substrate produced by SCHOTT company (SCHOTT AG, Mainz, Germany) whose commercial name is Borofloat 33. It is commercially available in sheets with a range of thickness from a few hundreds of microns to several millimeters. This float glass is manufactured by using the floating process, where molten glass flows continuously from the melting tank on a bath of liquid tin. There the molten glass spreads out uniformly and then undergoes mechanical treatment to produce the required thickness. When it reaches the end of the tin bath, the glass, which has now solidified, is removed from the surface of the metal and then annealed to relieve any internal stress. After it leaves the annealing oven, the glass ribbon is cut into the required sheet sizes. Borosilicate glass is a glass which contains a significant proportion of boric oxide B_2O_3 in addition to its main component which is quartz sand (silicon dioxide SiO_2). The specimen considered in this work is a standard quality 1 mm-thick 200 mm x 200 mm flat sample. In the following we will refer to it with the acronym BO.

The last sample characterized is a replicated electroformed Nickel substrate produced by MEDIA LARIO Tech. company (MEDIA LARIO S.r.l., Como, Italy). The concept of the replication technique is that the figure and the micro roughness of a specific component can be reproduced by exactly copying the surface of a given master [7]. This technique can be used for the volume production of high efficiency multilayer mirrors with by making use of a low-roughness master having the negative profile of the mirror that has to be fabricated. The electroformed Nickel sample characterized here has been produced by MEDIA LARIO Tech. labs with a proprietary deposition process in order to produce a bare nickel surface with a minimized crystallinity. This 0.5 mm thick specimen was replicated from a 2 in. diameter General Optics fused silica sample identical to the one described in the beginning of this section (sample GO). In the following we will refer to it with the acronym EN.

Experimental

All experimental measurements have been performed at INAF - Brera Astronomical Observatory by using different non-destructive metrological systems that allow topographic characterization of a surface in a very broad band of spatial frequencies (from tenths of cm^{-1} to few nm^{-1}). Atomic Force Microscopy (AFM) topography and optical profilometry allowed us to trace respectively 2-dimensional maps and 1-dimensional profiles of representative regions of the substrates surface, whilst with a triple-axis diffractometer X-ray scattering measurements (XRS) have been performed. These techniques have allowed independent estimates of spectral roughness distributions over the typical spatial frequencies band of every single instrument. The AFM adopted is a stand-alone Digital Nanoscope III (Digital Instruments Inc., Santa Barbara, USA) that can cover a spatial frequency range between 0.01 250 m^{-1} and that allows to trace 512 × 512 points digital maps with a lateral resolutions of few tens of nanometers and with vertical sensitivity of the order of 0.1 nanometers. For all the measurements reported in this work (taken systematically over scan areas of 100 × 100 m, 10 × 10 m and 1 × 1 m)

tapping-mode configuration was used which has a typical value of background noise lesser then 0.1 nm.

The surface optical profiler is a Wyko Topo-2D (Wyko Corp., Tucson, AZ, USA) that offers two different magnifications of 20× and 2.5× by means of Mirau-type objectives. It can perform measurements with a constant sampling of 1024 points over a scan length respectively of 660 m and 5280 m with a lateral resolution respectively of ~ 0.64 m and ~ 5.15 m. With this optical profiler is possible to cover a spatial frequency range between 0.2 $mm^{-1} \div 1$ m^{-1} but it should be noted that due to the low roughness samples handled in this work, the spectral analysis could be extended only up to 0.3 m^{-1}, at higher frequencies the sensitivity limit of the instrument was reached.

XRS tests have been carried-out by making use of a three-axis diffractometer Bede D1 Systems (Bede Scientific Instruments Ltd, Durham, UK). XRS diagram gives an immediate idea of the imaging degradation due to the surface micro roughness. Using a known relation between the XRS diagram and the surface PSD one can obtain an indirect PSD measurement [8] which is completely independent on the topographic methods previously listed. The requirement for XRS measurements of smooth surfaces is a finely collimated beam with sharp edges incident into the surface with a grazing angle lower than the critical angle of total reflection. A conventional x-ray tube with a Copper anode was used as radiation source ($_{CuK}$ = 0.154 nm). In the experimental setup used it was possible to obtain a monocromaticity E/E of the order of 10^{-4} and a divergence of ~ 20 arcsec with a maximum counting rate of 10^5 count/s. The intrinsic noise of the counting channel did not exceed 0.2 count per second as a result of a two threshold discriminator (upper and lower). By means of XRS experiments the PSDs functions were calculated in a range of spatial frequencies that spans the particular frequency domains of the optical profiler and AFM.

A detailed description of the workings of the metrological instruments used to investigate surface roughness is listed in the reference section [9].

Results and discussion

The first consideration to be noted in this section is that the experimental data concerning the substrates characterization are expressed in terms of mono-dimensional PSDs. Power spectra are then used to exhibit and compare the substrates quality in terms of rms roughness and fractal approximation.

Scaling of the mono-dimensional PSDs (in units of nm^3) versus spatial frequencies (in units of m^{-1}) obtained by measurements performed on the samples has been possible over about seven order of magnitude. For each sample good agreement was found in the overlapping regions, since the ratios of PSDs functions from different experimental techniques did not exceed a 1.5-fold factor.

Fig. 1 shows the experimental data concerning the silicon wafer sample. Each PSD is the average of a number of independent measurements performed on different regions of the sample. The low dispersion among the experimental data from the different topographical techniques comes out from the high statistic.

A way to compare optical surfaces morphologies, exploits the theory of fractal surfaces. It is the case to underline that from the theory of fractal surfaces is expected a spectral index n with values between 1 and 3. Moreover, for a given constant K_n, a steeper power law would be preferable because it would reduce the PSD amplitude in the high-frequency range, which is responsible for the x-ray/neutron large-angle scattering and for the amplification of the interfacial roughness in multilayer reflective coatings. Fig. 2 shows the comparison between the substrates in the fractal approximation: in the log-log plot all the power spectral distributions are well fitted by the in-

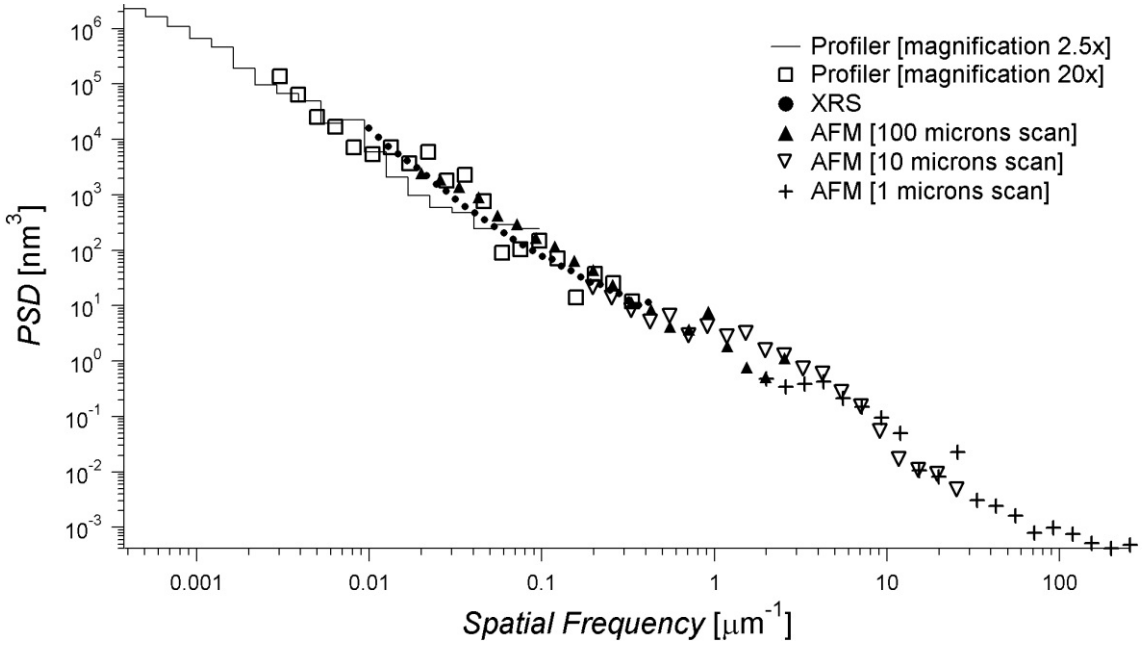

Fig. 1. Scaling of the mono-dimensional PSDs versus spatial frequencies obtained by the characterization performed on the silicon wafer sample.

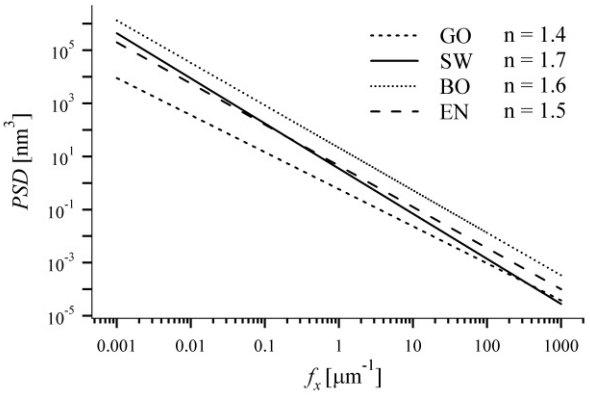

Fig. 2. Comparison between the fractal approximation of the samples. The constants *n* reported in the legend are the spectral indexes of the power laws.

verse power law expressed in equation (4) with spectral indexes standing between 1 and 2. The two quantities – *n* and K_n – that characterize the power spectra of the optical surfaces are reported in Tab. 1.

Tab. 1 reports the rms roughness values as calculated from the integration of PSD following the equation (3) where the spatial frequencies domain was conventionally divided in two separate spectral ranges: a medium frequency range (between 0.001 ÷ 0.03 m^{-1}) that corresponds to waviness error and a high frequency range (between 0.03 ÷ 1000 m^{-1}) that corresponds to micro-roughness error.

In spite of the quite flat PSD (n = 1.4), GO sample exhibits the lowest values of rms roughness in both spatial frequencies ranges ($_{rms}$ = 5.7 Å in the medium range and $_{rms}$ = 0.8 Å in the high range). This is due to the fact that its PSD has the normalization factor K_n with the lowest value (K_n = 1.2).

SW sample shows the steepest PSD (n = 1.7) and has values of rms roughness slightly higher than GO sample. In Fig. 2 it can be seen that, with the increasing of the spatial frequencies values, SW substrate tends to reach values of power spectrum similar to those of the GO sample. This is very likely due to the fact that GO was used several times as master for the replication of Nickel substrates by means of the electroforming process. This could have caused a partial deterioration of the surface.

BO sample has a PSD with a slope (n = 1.6) very similar to the one of SW sample, but the K_n constant (K_n = 40) determines the worst roughness level. It is true, however, that the rms roughness values found are still very likely consistent with the production of efficient multilayer optics. It should be noted that this kind of material is employed in neutron reactors as substrate for the multilayer neutron beam guidelines [10] and also the possibility to use this substrates to produce high throughput x-ray multilayer focusing telescopes is being examined [11].

The sample EN, that has been replicated from a General Optics superpolished fused silica by means of an electroforming process without any additional polishing, displays a surface finish quality comparable to the other superpolished samples. There is no significant difference with respect to the surface used as master for the replication. This result is important in both X-ray and neutron application fields, a feasibility study concerning high throughput X-ray multilayer focusing optics by means of the electroforming technique is under investigation [12] and implementing this technique also for the manufacturing of neutron beam guidelines could represent a feasible alternative to borofloat.

Conclusions

Roughness spectra of substrates consisting of different materials and superpolished/manufactured through different technological approaches have been evaluated in a wide spatial frequency bandwidth. All the substrates considered in this work have exhibited a surface quality consistent with the deposition of high efficiency x-ray/neutron multilayer films. GO sample has given the best experimental results, followed in descending order by SW, EN and BO. Since multilayer coatings deposited upon substrates in

Table 1. Summary of the experimental results obtained by the characterization of the samples: $_{rms}$ values are divided into two ranges of integration (medium and high spatial frequencies); n and K_n are the fractal parameters; materials and manufacturing/polishing techniques are also reported.

Sample	Material	Manufacturing technique	Polishing technique/s	$_{rms}$ [Å]		Fractal parameters	
				f_x [m^{-1}] = 0.001 ÷ 0.03	f_x [m^{-1}] = 0.03 ÷ 1000	n	K_n
GO	Silica	Electrofusing + machining	Lapping	5.7	0.8	1.4	1.2
SW	Silicon	Sliced from a ingot grown via CZ	Lapping + chemical etch.	6.9	1.1	1.7	7
BO	borosilicate	Classic floating	None	9.5	3.1	1.6	40
EN	Nickel	Electroplating	None	6.8	2.7	1.5	9

general tend to copy the roughness of the substrates and then add their own intrinsic roughness, the experimental results demonstrated in this work can be used as starting point in the study of multilayer-coated imaging systems performances.

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