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Surface roughness characterization of soft x-ray multilayer films on the nanometer scale

J. Yu
Department of Mechanical Engineering, Chubu University, 1200 Matsumotocho, Kasugai, Aichi 487, Japan and State Key Laboratory of Applied Optics, Changchun Institute of Optics and Fine Mechanics, Chinese Academy of Sciences, Changchun 130022, People's Republic of China

J. L. Cao
State Key Laboratory of Applied Optics, Changchun Institute of Optics and Fine Mechanics, Chinese Academy of Sciences, Changchun 130022, People's Republic of China

Y. Namba
Department of Mechanical Engineering, Chubu University, 1200 Matsumotocho, Kasugai, Aichi 487, Japan

Y. Y. Ma
State Key Laboratory of Applied Optics, Changchun Institute of Optics and Fine Mechanics, Chinese Academy of Sciences, Changchun 130022, People's Republic of China

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The soft x-ray reflectivity of multilayer films is affected by the surface roughness on the transverse nanometer scale. Scanning tunneling microscopy (STM) is an ideal instrument for providing high-lateral-resolution roughness measurements for soft x-ray multilayer films that cannot be obtained with other types of instruments on the transverse nanometer scale. The surface roughnesses of Mo/Si, Mo/C, and W/Si soft x-ray multilayer films prepared by an ion-beam-sputtering technique were measured with a STM on the vertical and transverse attributes. The film roughnesses and average spatial wavelengths added to the substrates depend on the multilayer film fabrication conditions, i.e., material combinations, number of layers, and individual layer thickness. These were estimated to lead to a loss of specular reflectivity and variations of the soft x-ray scattering angle distribution. This method points the way to further studies of soft x-ray multilayer film functional properties and can be used as basic guidance for selecting the best coating conditions in the fabrications of soft x-ray multilayer films. © *1996 American Vacuum Society.*

I. INTRODUCTION

Soft x-ray regions usually cover a range of electromagnetic wavelengths approximately from 1 to 30 nm, corresponding to photon energies from 40 eV to 1.2 keV. Soft x-ray multilayer films are being made for critical applications such as high-reflection cavity mirrors for the experiments of soft x-ray laser double-pass amplification.[1,2] For soft x-ray multilayer structures, it is well known that two important factors affect the performance (specular reflectivity). One is the surface and interfacial roughness and the other is the transverse attribute of individual films. There is also an increasing interest in understanding roughness as an intrinsic dynamic behavior of growing surfaces and interfaces, with emphasis on soft x-ray multilayer structures.

Surface roughness can be measured with visible-light scattering instruments, optical interferometers, and mechanical stylus instruments, with the best vertical sensitivity of 0.1 nm. But it is difficult to measure surface roughness on a transverse scale of less than 100 nm with these methods. Scanning tunneling microscopy (STM) is a powerful supplement which can be used to measure surface roughness with 0.1 nm resolution on the transverse nanometer scale. Recently, STM (including atomic force microscopy) was used to investigate x-ray optics.[3–6] Most of this previous work was intended to prove good correlation between the STM results and direct x-ray measurements. This is the basis with which one uses a STM to measure the surface roughness of x-ray optics. In this article, surface roughnesses of Mo/Si, Mo/C, and W/Si soft x-ray multilayer films prepared with the ion-beam-sputtering technique were measured with a STM to determine the roughness and spatial wavelength the films added to the Si wafer substrates on which they were deposited. The goal of this article is to present a method to evaluate the performance of soft x-ray multilayer films from both vertical and traverse attributes with STM, which can be used as basic guidance for selecting the best coating conditions in the fabrication of soft x-ray multilayer films.

II. SURFACE ROUGHNESS AND REFLECTANCE

Characterization of optical surfaces frequently involves the power spatial density function, often called the power spectral density (PSD). If $z(x)$ is the surface roughness deviation as a function of distance x, the Fourier transform of the deviation $z(x)$ from its mean surface level defines the one-dimensional power spectral density

$$\mathrm{PSD}(f_{sp}) = \lim_{L\to\infty} \frac{2}{L} \left| \int_0^L z(x)\exp[-i(2\pi f_{sp}\, x)]dx \right|^2, \quad (1)$$

where L is the length of scans; f_{sp} is the surface spatial frequency, $f_{sp}=1/\lambda_{sp}$, where λ_{sp} is the surface spatial wavelength. All statistical properties of the surface finish can be obtained from the PSD(f_{sp}) of $z(x)$. The rms surface roughness is given by

$$\sigma^2 = \int_0^\infty \mathrm{PSD}(f_{sp}) df_{sp}. \quad (2)$$

From the height deviations $z(x,y)$ of the actual surface of a mirror from its perfect shape, one obtains the three-dimensional rms roughness

$$S\ \mathrm{rms} = \sqrt{\frac{1}{S}\int\int_s z^2(x,y) dx\ dy} \quad (3)$$

and the three-dimensional rms slope

$$S\Delta_q = \sqrt{\frac{1}{S}\int\int_S |\nabla Z(x,y)|^2 dx\ dy}, \quad (4)$$

where S is the area of the scans, and the rms slope is usually considered to be in a specific direction, the x axis or y axis direction. From Eqs. (3) and (4), one obtains the three-dimensional rms surface wavelength $S\lambda_q$ as

$$S\lambda_q = 2\pi\left(\frac{S\ \mathrm{rms}}{S\Delta_q}\right). \quad (5)$$

In these factors, the surface roughness strongly affects the reflectivity because the reduction of reflectivity is proportional to the exponent of the squared rms roughness as[7]

$$R_s = R_0 \exp\left[-\left(\frac{4\pi\sigma\cos\theta_0}{\lambda}\right)^2\right], \quad (6)$$

where R_s is the surface specular reflectance, R_0 is the total reflectance, $R_0 = R_s + R_d$, R_d is the diffuse reflectance, and θ_0 is the angle of incident of light on the surface. For example, if $\lambda = 2.362$ nm (O $K\alpha$ line), $\theta_0 = 0°$, $R_s/R_0 = 20\%$, σ should be less than 0.24 nm. On the other hand, the surface transverse attribute affects the scattering angle distribution. It can be calculated from the diffraction grating equation, where only the first diffracted order needs to be considered if the roughness amplitude is much less than the light wavelength λ,[8]

$$\lambda = \lambda_{sp}(\sin\theta_0 \pm \sin\theta_d), \quad (7)$$

where λ_{sp} is the groove spacing (surface spatial wavelength) and θ_d is the angle of diffraction (scattering angle). The shortest spatial wavelength occurs when $\sin\theta_d = 1$ or $\theta_d = 90°$. It is equal to the light wavelength λ, 1–30 nm for soft x rays. So the measuring instruments with ~0.1 nm vertical sensitivity on the nanometer scale (transverse direction) should be used for evaluating the soft x-ray multilayer films.

III. STM MEASUREMENT CONDITIONS

The soft x-ray multilayer films were investigated with our roughness software system based on a STM developed in the Beijing Laboratory of Electron Microscopy. Electrochemically etched Pt–Ir tips used in this study were characterized to an apex diameter of ~0.1 μm measured on the scanning electron microscope (SEM) photographs. Note that the intrinsic microscopic structure of the tip is not yet known; the interaction of a probe tip and a substrate surface is complex. As in electron tunneling, short-range forces are primarily sensitive to the atomic structure of the tip apex and the sample surface in a volume of atomic dimensions.[9] These suggest that the effective apex diameter of the tip is smaller than the nominal value ~0.1 μm. STM offers much higher performance than the traditional contact-stylus profilometer mainly because it has significantly effective sharper probe tips, although a shovel-shaped stylus with a sharp dimension of ~0.2 μm is also available for the stylus instrument.[10] The surface topographic maps were made at a tunnel current of 2 nA and bias voltage of 50 mV in a raster scan area 95 nm×95 nm.

TABLE I. Design parameters and fabrication conditions of various Mo/Si, W/Si, and Mo/C multilayer films.

Material combinations	Substrate	Number of layers	Individual layer thickness d_H (nm)	Individual layer thickness d_L (nm)	Work pressure (Pa)	Intensity of ion beam (mA/cm^2)	Accelerating voltage (V)
Mo/Si	Si	31 (Mo on top)	4.61 (d_{Mo})	8.01 (d_{Si})	1.2×10^{-3}	0.9	900
Mo/Si	Si	61 (Mo on top)	4.30 (d_{Mo})	5.00 (d_{Si})	2.8×10^{-3}	0.9	900
W/Si	Si	121 (W on top)	0.80 (d_W)	1.70 (d_{Si})	4.2×10^{-2}	0.9	900
W/Si	Si	61 (W on top)	0.80 (d_W)	1.70 (d_{Si})	4.2×10^{-2}	0.9	900
Mo/C	Si	34 (C on top)	2.62 (d_{Mo})	3.76 (d_C)	1.4×10^{-3}	1.0	900
Mo/C	Si	45 (Mo on top)	2.62 (d_{Mo})	3.76 (d_C)	1.4×10^{-3}	1.0	900

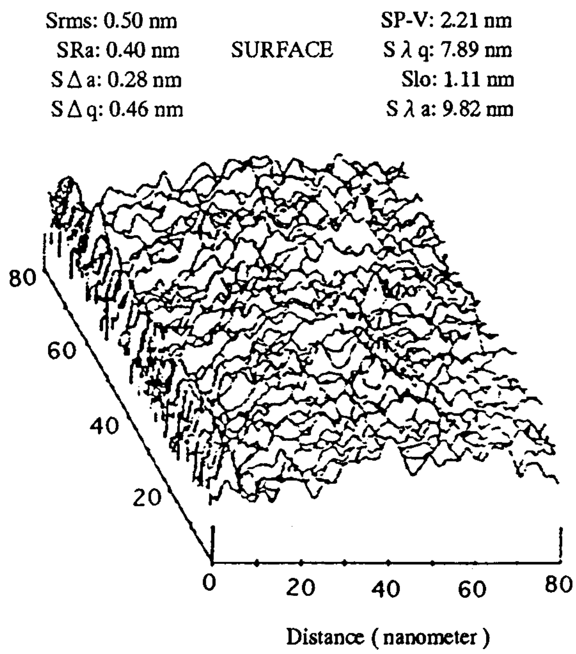


FIG. 1. A 3D STM plot of the Si wafer substrate on which 4-nm-thick molybdenum film was sputtered; 0.5 nm rms roughness; the area is 80 nm×80 nm.

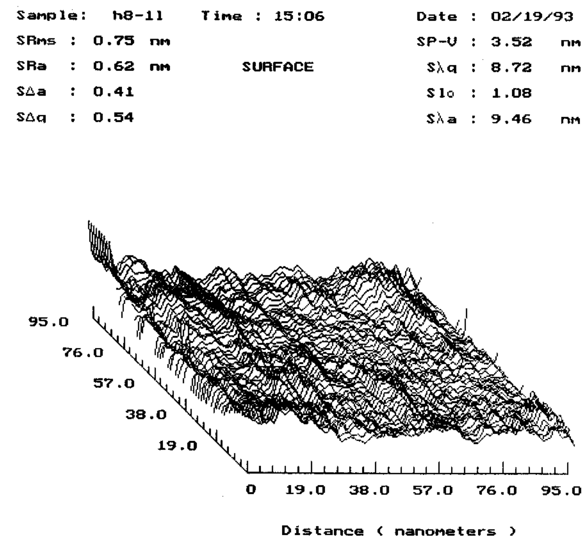


FIG. 2. A 3D STM plot of Mo/Si 31 layer films (Mo on top) of a 0.75 nm rms roughness area over a 95 nm×95 nm area.

IV. MULTILAYER FILMS AND SURFACE MEASUREMENTS

For this study multilayer films of three pairs of different materials, i.e., Mo/Si, Mo/C, and W/Si, were deposited at the ion-beam-sputtering (IBS) 60 system onto Si wafer substrates. Argon ions produced in a Kaufman ion gun chamber were accelerated and the argon ion flow rate was controlled by a mass flowmeter. The acceleration voltage V_a and ion current density ρ could be regulated continuously and uniformly in the range of 300–2000 V and 0–2 mA/cm^2, respectively. The diameter of the argon ions was 60 mm, and the uniformity was better than 95%. The variations of ρ, V_a, and working pressure P_w was controlled less than 1%. The ion-beam-sputtering system, composed of a turbo-molecular-pump and a front end of a mechanical pump, was evacuated to a base pressure of 10^{-7} Torr after liquid-nitrogen cooling off. The designed parameter values and fabrication conditions of the various Mo/Si, Mo/C, and W/Si multilayer films are in Table I. In order to investigate the effect of the film material combinations, number of layers, and the individual layer thickness (high atomic number and low atomic number materials) on superficial rms roughness, the same Si wafer was used as the substrates for all of the multilayer films, and the design parameter values were changed. For example, for the Mo/Si combination, the number of layers was 31 and 61, respectively, and the individual layer thickness was $d_{\mathrm{Mo}}=4.61$ nm, $d_{\mathrm{Si}}=8.01$ nm and $d_{\mathrm{Mo}}=4.30$ nm, $d_{\mathrm{Si}}=5.00$ nm. Other material combinations are as shown in Table I.

To measure the rms roughness of the Si wafer with STM, a 4-nm-thick molybdenum film was deposited carefully. It was believed that the roughness at the single layer Mo film, 4 nm thick, was exactly reproduced from the substrate. The single molybdenum film can be sputtered uniformly with the IBS 60 system. Figure 1 shows a three-dimensional (3D) STM plot of the Si wafer substrate adjacent to the abovementioned multilayer films, 0.50 nm rms, 80 nm×80 nm scan area. The obvious scratch structure on the surface was avoided and quite uniform. Figures 2–7 are 3D STM plots of the six multilayer films in Table I. The surface of Mo/Si (31 layers), W/Si (121 layers), W/Si (61 layers), and Mo/C (34 layers) films (Figs. 2, 4, 5, and 6, respectively) are quite uniform as well as is the Si wafer substrate. The Mo/Si (61 layers) and Mo/C (45 layers) films (Figs. 3 and 7) have obvious streak features. For Mo/Si and Mo/C films, it seems clear that the topographic differences are caused by the dif-

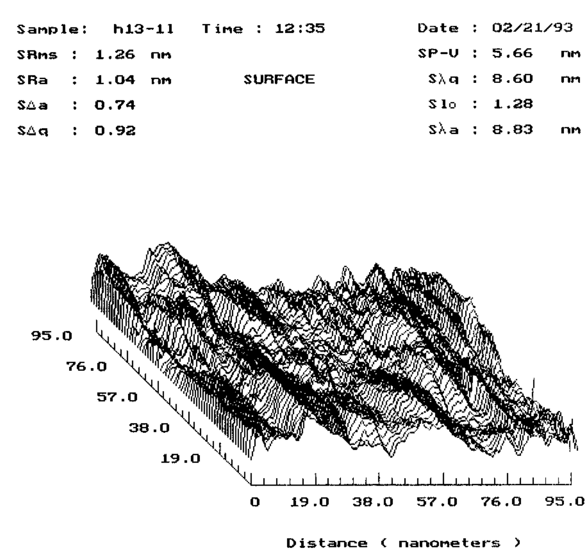


FIG. 3. A 3D STM plot of Mo/Si 61 layer films (Mo on top) of a 1.26 nm rms roughness area over a 95 nm×95 nm area.

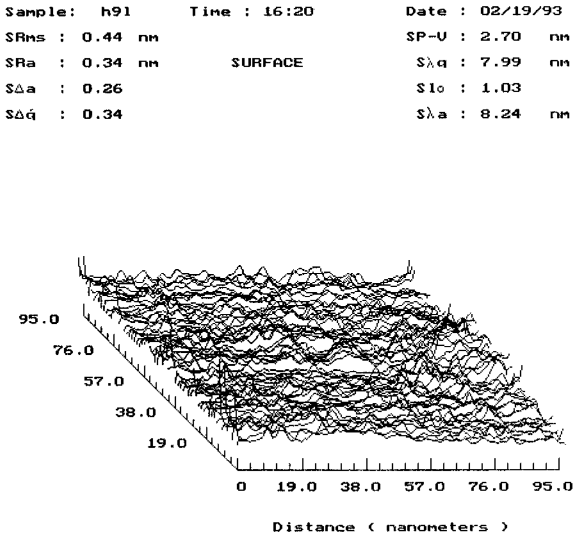


FIG. 4. A 3D STM plot of W/Si 121 layer films (W on top) of a 0.44 nm rms roughness area over a 95 nm×95 nm area.

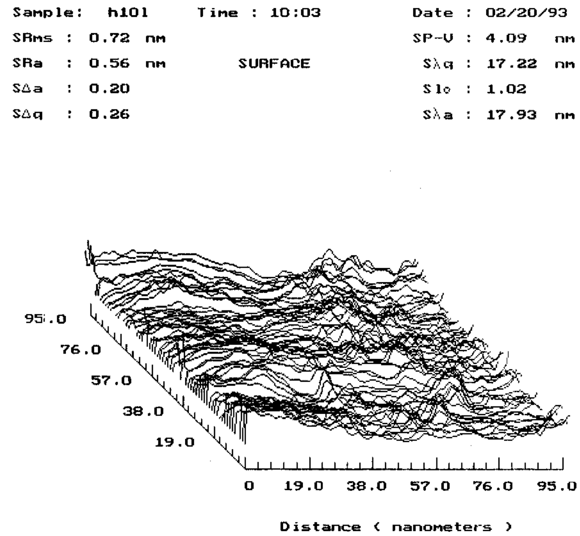


FIG. 6. A 3D STM plot of Mo/C 34 layer films (C on top) of a 0.72 nm rms roughness area over a 95 nm×95 nm area.

ferences of the number of layers or by the different individual layer thicknesses shown in Table I. To evaluate the quality of multilayer films quantitatively, rms roughness measurements were made at three different spots on each multilayer film and Si wafer substrate. These rms roughness values were summarized as shown in Fig. 8(a). The left column represents the minimum of the three rms roughness measurements, and the right represents the maximum. The added film roughness were calculated by taking the difference between the average film roughness and the average substrate roughness; these values are in Fig. 8(a). These results suggest that the added film roughnesses of Mo/Si (61 layers) and Mo/C (45 layers) are present to a significant extent, increasing from 0.46 nm rms (Si wafer) to 1.37 nm rms and 1.14 nm rms, respectively, and those of Mo/Si (31 layers), W/Si (61 layers), and Mo/C (34 layers) films are small from a statistical point of view; the W/Si (121 layers) film surface is amazingly smooth and has a roughness nearly identical to that of the Si wafer substrate on which it was deposited. The added film roughnesses were expected to lead to a loss of specular reflectivity. Figure 8(a) also shows the calculated relative reflectivity of normal-incidence C $K\alpha$ (4.47 nm), Be $K\alpha$ (11.4 nm) and neonlike Ge (23.2 nm) characteristic soft x-ray lines. With increasing rms roughness, the relative reflectivity decreases, shown in Fig. 8(b). For C $K\alpha$, the rate of decrease is much larger, from 1 to 0, in the rms roughness range of [0.46,1.14] nm. For the neonlike Ge line, the reflectivity changes gently in the same range of rms roughness.

To make further investigations of the multilayer film transverse attribute, the rms average spatial wavelength λ_q was calculated on all samples as a length characteristic pa-

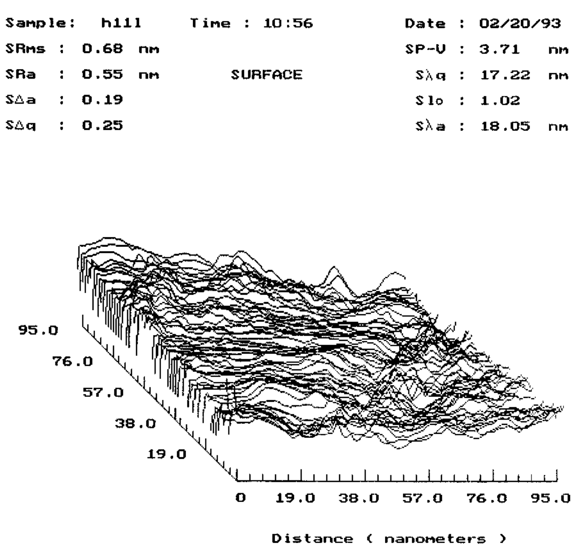


FIG. 5. A 3D STM plot of W/Si 61 layer films (W on top) of a 0.68 nm rms roughness area over a 95 nm×95 nm area.

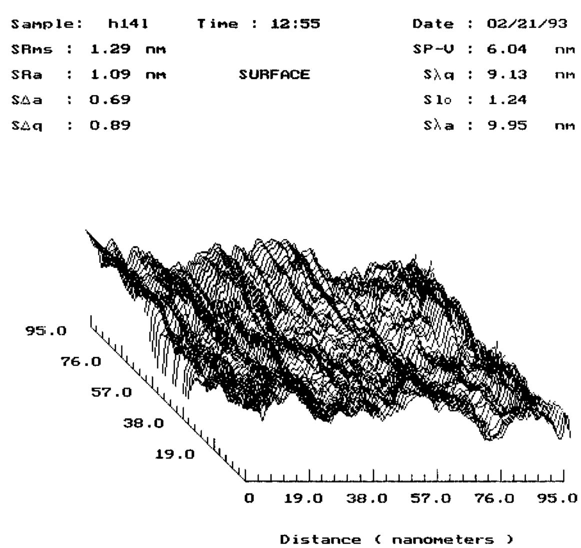
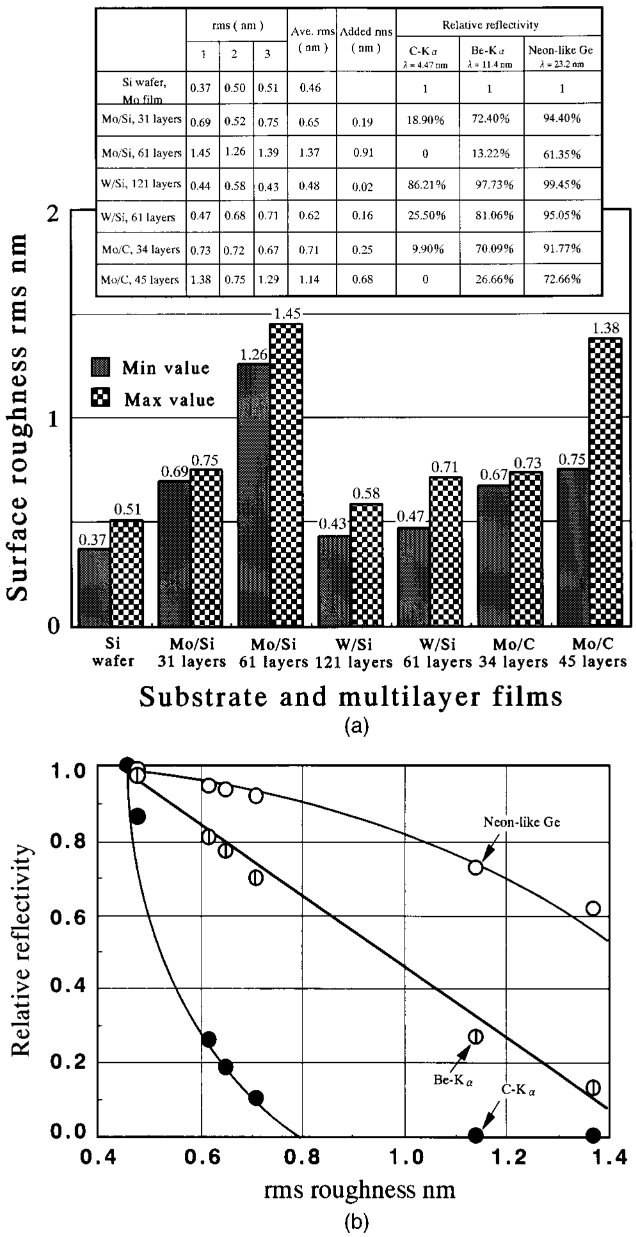


FIG. 7. A 3D STM plot of Mo/C 45 layer films (Mo on top) of a 1.29 nm rms roughness area over a 95 nm×95 nm area.



	rms (nm) 1	rms (nm) 2	rms (nm) 3	Ave. rms (nm)	Added rms (nm)	Relative reflectivity C-Kα $\lambda = 4.47$ nm	Relative reflectivity Be-Kα $\lambda = 11.4$ nm	Relative reflectivity Neon-like Ge $\lambda = 23.2$ nm
Si wafer, Mo film	0.37	0.50	0.51	0.46		1	1	1
Mo/Si, 31 layers	0.69	0.52	0.75	0.65	0.19	18.90%	72.40%	94.40%
Mo/Si, 61 layers	1.45	1.26	1.39	1.37	0.91	0	13.22%	61.35%
W/Si, 121 layers	0.44	0.58	0.43	0.48	0.02	86.21%	97.73%	99.45%
W/Si, 61 layers	0.47	0.68	0.71	0.62	0.16	25.50%	81.06%	95.05%
Mo/C, 34 layers	0.73	0.72	0.67	0.71	0.25	9.90%	70.09%	91.77%
Mo/C, 45 layers	1.38	0.75	1.29	1.14	0.68	0	26.66%	72.66%

FIG. 8. (a) Added film roughnesses of Mo/Si (31 layers), Mo/Si (61 layers), W/Si (121 layers), W/Si (61 layers), Mo/C (34 layers), and Mo/C (45 layers) compared to a Si wafer substrate. (b) The added film roughnesses were estimated to lead to a loss of specular reflectivity with the three kinds of characteristic soft x-ray lines of C $K\alpha$, Be $K\alpha$, and neonlike Ge. The reflectivity was normalized by that of the Si wafer to relative reflectivity for the compared purpose.

rameter. λ_q can be considered as λ_{sp} of Eq. (2) to estimate the scattered angle θ_d for comparative purposes. θ_d was evaluated with three kinds of soft x rays of C $K\alpha$ (4.47 nm), Be $K\alpha$ (11.4 nm), and neonlike Ge line at 23.2 nm, as shown in Table II. For normal-incident light of 23.2 nm, the shortest spatial wavelength that produces scattering for correlated roughness occurs when $\sin \theta_d = 1$ or $\theta_d = 90°$ [from Eq. (2)]; it is equal to the incident light wavelength 23.2 nm, and this light is scattering at a grazing angle along the surface. The rms average spatial wavelengths of all multilayer films studied here (second column of Table II) are less than 23.2 nm, so no scattered light occurs from the roughness on the characteristic wavelength scale, suggesting that all of these multilayer films are suitable for the 23.2 nm wavelength optical system. For the Be $K\alpha$ (11.4 nm) x ray, 17.22 nm characteristic wavelength features may produce scattered light on $\theta_d = 41.5°$ on W/Si (61 layers) and Mo/C (34 layers) films shown in the sixth column of Table II, suggesting that they are not suitable for the Be $K\alpha$ line system. For the C $K\alpha$ (4.47 nm) x ray, scattered light occurs on the corresponding characteristic wavelength on all samples, shown in the fourth column of Table II. The biggest θ_d is 34° for W/Si (121 layers) films and the smallest θ_d is 15° for W/Si (61 layers) and Mo/C (34 layers) films, suggesting that all of these multilayer films are not suitable for the 4.47 nm wavelength soft x-ray optical system. Figure 9 shows the θ_d distribution

TABLE II. Calculated θ_d as a function of the normal-incidence light wavelength λ and average spatial wavelength λ_q with three kinds of characteristic soft x-ray lines of C $K\alpha$, Be $K\alpha$, and neonlike Ge.

Films	λ_q (nm)	C $K\alpha$ λ (nm)	C $K\alpha$ θ_d (deg)	Be $K\alpha$ λ (nm)	Be $K\alpha$ θ_d (deg)	Neonlike Ge λ (nm)	Neonlike Ge θ_d (deg)
Si wafer Mo film	7.89	4.47	34.5	11.4	⋯	23.2	⋯
Mo/Si 31 layers	8.72	4.47	30.8	11.4	⋯	23.2	⋯
Mo/Si 61 layers	8.60	4.47	31.3	11.4	⋯	23.2	⋯
W/Si 121 layers	7.99	4.47	34.0	11.4	⋯	23.2	⋯
W/Si 61 layers	17.22	4.47	15.0	11.4	41.5	23.2	⋯
Mo/C 34 layers	17.22	4.47	15.0	11.4	41.5	23.2	⋯
Mo/C 45 layers	9.13	4.47	29.3	11.4	⋯	23.2	⋯

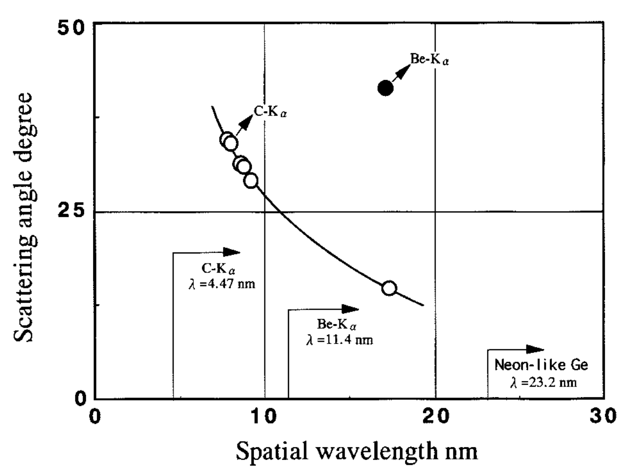


FIG. 9. θ_d distribution vs average spatial wavelengths for the soft x-ray multilayer in Table I. Horizontal arrows mean the begin points of producing scattering for the three kinds of soft x-ray characteristic lines.

versus the average spatial wavelengths for the soft x rays and the horizontal arrows show the shortest spatial wavelengths that produce the scattering.

As analyzed from the above consideration of vertical and transverse attributes, we knew that the Mo/Si (31 layers), W/Si (61 layers), and W/Si (121 layers) films have better performance for the neonlike Ge line at 23.2 nm and Mo/Si (31 layers) and W/Si (121 layers) for the Be $K\alpha$ line. The quality of substrates is important for determining the final performance of soft x-ray multilayer films. All of the rms roughnesses and average spatial wavelengths of multilayer films discussed here are larger than those of Si wafer substrates, shown in Fig. 8(a) and Table II. For high performance, roughnesses of substrates should be very little, and average spatial wavelengths should be less than the wavelength of incident light. As an example, one of these kinds of Mo/Si multilayer film mirrors (31 layers, 23.2 nm design wavelength) has been used in experiments of x-ray laser double-pass amplification in the neonlike Ge line,[2] and the reflectivity of normal incidence light ($\lambda=23.2$ nm) achieved 20%.

V. CONCLUSION

The STM is an ideal instrument for providing high-lateral-resolution roughness measurements for soft x-ray multilayer films that cannot be obtained with other types of instruments on the transverse nanometer scale. We have presented a method using the STM for evaluating the performance of the soft x-ray multilayer films not only from the vertical attribute, i.e., rms roughness, but also from the transverse attribute, i.e., rms average spatial wavelength λ_q. This method points the way to further studies of soft x-ray multilayer film functional properties.

Six kinds of Mo/Si, Mo/C, and W/Si soft x-ray multilayer films were examined using this method. For the vertical attribute, we found that the added film roughnesses of Mo/Si (61 layers) and Mo/C (45 layers) were present to a significant extent compared to Si wafer substrates; those of Mo/Si (31 layers), W/Si (61 layers), and Mo/C (34 layers) films were small from a statistical point of view; the W/Si (121 layers) film surface was amazingly smooth and had a roughness nearly identical with that of the Si wafer substrate on which it was deposited. These suggest that in general ion-beam-sputtered multilayer film structures are not perfect. The film roughnesses added to the substrates depend on the multilayer film fabrication conditions, i.e., material combinations, number of layers, and individual layer thickness. These were expected to lead to a loss of specular reflectivity, which is detrimental for soft x-ray imaging applications. For the transverse attribute, we found that in general the rms average spatial wavelengths of ion-beam-sputtered multilayer films were enlarged by the growing films, which depend as well on the multilayer film fabrication conditions. The rms average spatial wavelengths of W/Si (61 layers) and Mo/C (34 layers) were present to a significant extent compared to Si wafer substrates; those of Mo/Si (31 layers), Mo/Si (61 layers), and Mo/C (45 layers) were small from a statistical point of view; the W/Si (121 layers) film had an amazingly complete correlation with the Si wafer substrate. Its rms average spatial wavelength was almost the same as that of the Si wafer substrate, implying that the roughness at the layer boundaries was exactly reproduced from layer to layer. The increments of λ_q in the film growing process were expected to lead to the variation of the soft x-ray scattering angle distribution. As might be expected, usually the quality of the substrate is important in determining the final performance of soft x-ray multilayer films, no matter what attribute (vertical or transverse direction) is considered.

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