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Multilayer optics for the EUV and soft X-rays

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Abstract : The demand to enhance the optical resolution , to structure and observe ever smaller details , has pushed the way towards the EUV and soft X-rays. Induced mainly by the production of more powerful electronic circuits with the aid of projection lithography , optics developments in recent years can be characterized by the use of electromagnetic radiation with smaller wavelength. The good prospects of the EUV and soft X-rays for next generation lithography systems ($\lambda = 13.5 \text{ nm}$) , microscopy in the “ water window ” ($\lambda = 2.3 \sim 4.4 \text{ nm}$) , astronomy ($\lambda = 5 \sim 31 \text{ nm}$) , spectroscopy , plasma diagnostics and EUV/ soft X-ray laser research have led to considerable progress in the development of different multilayer optics. Since optical systems in the EUV/ soft X-ray spectral region consist of several mirror elements a maximum reflectivity of each multilayer is essential for a high throughput. This paper covers recent results of the enhanced spectral behavior of Mo/ Si , Cr/ Sc and Sc/ Si multilayer optics.

Key words : Mo/ Si ; Cr/ Sc ; Sc/ Si ; optics ; multilayer mirror ; magnetron sputtering ; EUV ; soft X-ray

1 Introduction

Engineers and scientists in fields as diverse as microlithography and space astronomy have a common need to use “ light ” ranging from EUV to the soft X-ray region. The extreme ultraviolet and soft X-ray spectral region lie between the ultraviolet and the hard X-ray regions of the electromagnetic spectrum. In spite of the smooth transitions between the spectral subdivisions it 's worth to define roughly their approximate borders^[1] :

extreme ultraviolet	EUV	$\sim 50 \text{ nm}$ to $\sim 5 \text{ nm}$
		$\sim 25 \text{ eV}$ to $\sim 250 \text{ eV}$,
soft X-ray region		$\sim 5 \text{ nm}$ to $\sim 0.2 \text{ nm}$
		$\sim 250 \text{ eV}$ to $\sim 6 \text{ keV}$.

The nanometer unit is widely used in the vacuum ultraviolet , while the photon energy units are frequently used for wavelengths near the

soft X-ray region. It must be understood that the above-named limits are not intended to be used rigidly.

Quite a large number of non-absorbing dielectric optical materials can be used in the visible spectral range for coatings and transmissive substrates. Anyhow , going to shorter wavelengths , the number of potential materials reduces drastically due to absorption resulting in a strongly limited choice of the optical materials. To get highest possible optical contrast in the EUV and soft X-ray region , a high electron density material (absorber) has to be paired with a low density electron material (spacer) . Here , only metals and semiconductors can be used. Very often , the optimal spectral working range of a layer pair is a few nanometers only. To outline some examples that have been successfully realized in the past , the following material combinations represent the absorber/ spacer : Sc/ Si (36 nm) ,

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Mo/Si (12.5 nm), Mo/Be (11.4 nm), Ru/B₄C (6.7 nm), Co/C (4.4 nm), Cr/Sc (3.2 nm) and W/Si (0.7 nm). The listed wavelengths define the absorption edge of the spacer material, representing the lower working range wavelength limit of the respective material couple.

2 Experimental setup

According to the optics requirements of an EUVL tool, the accurate deposition of high reflective and laterally graded multilayers on ultra-precise polished substrates can be regarded as one of the major challenges of EUV lithography development today. To meet these requirements, a New EUV Sputtering SYstem-NESSY has been developed (Fig. 1, 2).

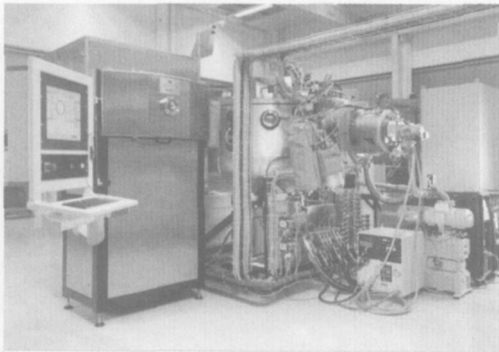


Fig. 1 DC magnetron sputtering system NESSY



Fig. 2 Substrate loading

The dc magnetron sputtering system is equipped with four rectangular magnetrons, 600 mm × 125 mm each (Fig. 3). The simultaneous coating of two 450 mm substrates or three 300 mm substrates can be realized (Fig. 4). The substrates can be spun up to 500 r/min during the deposition process. The target-substrate distance is variable from 50 mm to 150 mm allowing the installation of moving shutters to realize lateral thickness gradients of the sputtered multilayer. The system is load locked (Fig. 2) and cryopumped. The base pressure is well below 8×10^{-7} Pa.

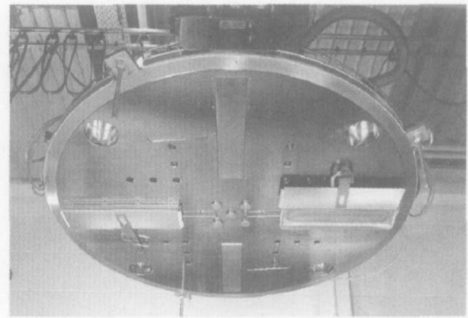


Fig. 3 Top cover with targets

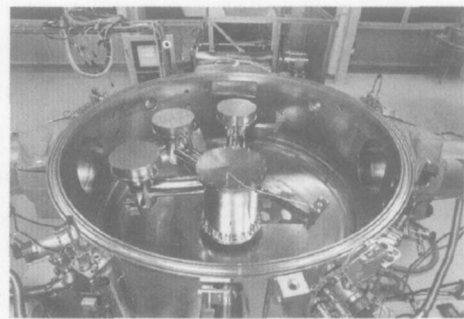


Fig. 4 Vacuum chamber with substrate holders

Special effort has been made to construct the cathodes. Different configurations of the magnets have been successfully realized in order to assure highest flexibility for different coating materials in terms of homogeneity requirements and target utilization. All magnetrons work stable at a working pressure of less than 7×10^{-2} Pa in argon atmosphere. A lateral period homogeneity of less than 0.1% on 300 mm has been

demonstrated recently with Mo/Si multilayers.

3 Experimental results

3.1 Mo/Si multilayers for $\lambda = 13 \dots 15 \text{ nm}$

The main application of Mo/Si multilayer mirrors in the EUV spectral range around 13.5 nm is the development of EUV lithography tools for the semiconductor industry. A good review of actual developments in this field can be found in the proceedings of the annual conference "Microlithography" [2]. In the past few years, the main focus of research has been the increase of reflectivity of EUV mirrors, because the designs for EUV lithography systems involve 9 mirrors and therefore the reflectivity of the whole system is proportional to R^9 .

A serious drawback of multilayer coatings for their application in EUV optics is their limited range of reflectivity in the spectral range; the spectral FWHM (full width at half maximum) of typically 0.5 nm covers only a small part of the output of some EUV sources, e. g. the spectrum of a broadband Xe source [3]. In all cases where maximum peak reflectivity is not required, e. g. in EUV metrology, astronomy and microscopy, broadband mirrors provide useful applications.

Whereas the tailoring of the spectral properties of optical components is developed to a high level and widely used for the hard X-ray range, the UV, VIS and IR, it is rarely used by now in the EUV range. However, some papers focus on the use of depth-graded multilayer mirrors in the hard X-ray region [4] and in neutron optics [5]. So-called "supermirrors" with a broadband reflectivity have been developed on the basis of a period thickness variation by a power law [6]. Specially depth graded multilayer mirrors [7] are used for telescopes, beam collimators, and X-ray scanners [8].

Mo/Si multilayers with different thin film designs were realized. Beside the maximization

of the peak reflectivity using a periodic multilayer design, the maximization and minimization of the FWHM were designed and realized using special broadband and narrowband multilayer designs, respectively.

Normal incidence reflection measurements were performed with synchrotron radiation at the PTB Berlin (BESSY II), Germany. All mirrors were measured in the wavelength range 12...15 nm with a wavelength resolution of 0.02 nm and an accuracy of $R = 0.5\%$. The incident angle of the beam was fixed at 1.5° , the spot had a diameter of 1.5 mm at the sample surface. Fig. 5 compares the measured reflectance of Mo/Si multilayer mirrors with a periodic, a broadband and a narrowband design.

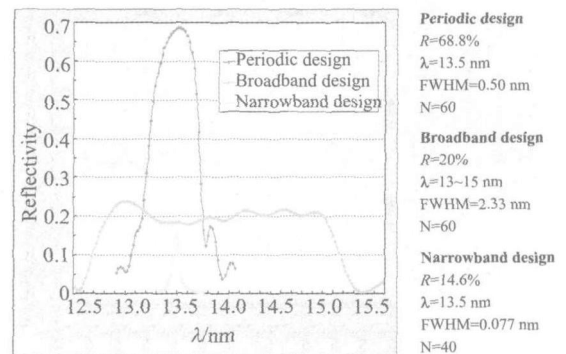


Fig. 5 Measured EUV reflectivity of Mo/Si multilayers reflectivity

3.1.1 Broadband design

A non-periodic design was used to obtain the broadband reflection in the EUV range. The design is based on a stochastic optimization process of all layer thicknesses. A desired spectral reflectivity $R_0(\lambda)$ in the wavelength range between λ_{\min} and λ_{\max} is used as a so-called "target function". Numerical calculations are used to optimize the design of a multilayer stack by a stochastic variation of each layer thickness to minimize the deviation between $R_0(\lambda)$ and the reflectivity $R(\lambda)$ of the multilayer design. For this purpose, the merit function

$$MF = \frac{\max}{\min} (R(\lambda) - R_0(\lambda))^2 d$$

is minimized. For all calculations, we used a commercial thin film design program (SCI Film Wizard™). The optical constants of molybdenum (Mo) and silicon (Si) from the Henke tables^[9] were imported to this program. Before the start of the calculation the number of layers and limits for the maximum and minimum film thickness have to be specified. The result of the optimization strongly depends as well on a reasonable choice for d_{\min} and d_{\max} as on a target function $R_0(\lambda)$ with reflectivity values that are in an achievable range.

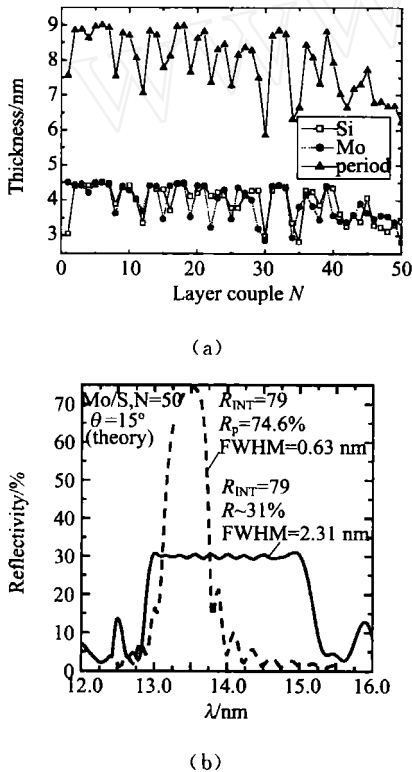


Fig. 6 Optimized thickness distribution (a) of a broadband multilayer mirror in the wavelength range from 13 nm to 15 nm and its theoretical performance (b) in comparison to a periodic multilayer mirror (dash line).

A mirror with a constant ($\pm 1\%$) and as high as possible reflectivity in the wavelength

range from 13 nm to 15 nm was designed. The design consists of 101 layers of Mo and Si in the thickness range between 2.8 nm and 4.5 nm. The optimized thickness distribution of this multilayer and the theoretical performance demonstrated in Fig. 6. An advantage of the nonperiodic design is the possibility to provide constant reflectivity over wide spectral range.

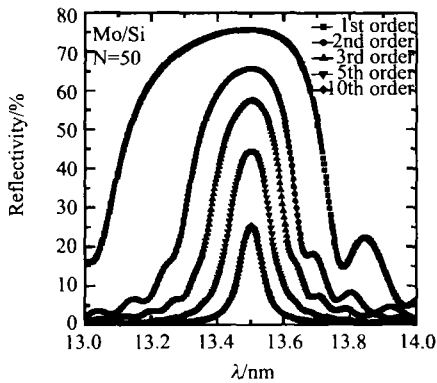
The integral reflectivity R_{INT} can be used to estimate the performance of a broadband mirror. It has to be noted that the measured integral reflectivity of the sputtered broadband mirrors was found to be considerably greater (10 % to 30 %) than the integral reflectivity of a periodic multilayer Mo/Si design.

3.1.2 Narrowband design

It has been shown that the FWHM of Mo/Si multilayer mirrors can be adjusted by variation of the absorber layer thickness ratio. Since this method is very limited the FWHM variation of Mo/Si multilayer mirrors was realized using high reflectance orders. Simulation results for Mo/Si multilayer mirrors with $N = 50$ periods used at normal incidence and optimized for maximum reflection at $\lambda = 13.5$ nm are presented in Fig. 7.

According to the Bragg condition ($2d \sin \theta \cong m \lambda$) for the application of high orders ($m = 2, 3 \dots$), the multilayer period d has to be increased as shown in Fig. 4, (b). The FWHM of the reflection is reduced by a factor that is almost equivalent to the order of reflection m . The calculated theoretical reflectivities of the multilayers decrease due to the higher absorption caused by the increasing overall thickness, but the decrease in their bandwidths is much stronger. For example, in the 3rd reflection order the FWHM is reduced by a factor of 2.89 in comparison to the 1st order mirror, whereas the peak reflectivity is only reduced by a factor of 1.32 compared to the 1st order mirror. In high reflection orders a strong reduction of the FWHM can be achieved, e.g. in the 10th reflection order a re-

duction by a factor of almost 8 is possible.



(a)

m	Period H , nm	Γ	N	R_{\max} , %	FWHM nm
1	6.92	0.43	50	75.6	0.631
2	13.7	0.22	50	65.7	0.327
3	20.5	0.17	50	57.4	0.220
5	34.0	0.096	50	44.4	0.137
10	67.8	0.046	50	25.2	0.080

(b)

Fig. 7 Theoretical performance (a) and corresponding design (b) of a Mo/Si multilayer with 50 periods optimized for high reflectivity in high reflection orders.

3.2 Cr/Sc multilayers for $\lambda = 3.1 \dots 4.4$ nm

Cr/Sc is a promising material combination for multilayer mirrors in the water window region. A possible X-ray source for laboratory use in this wavelength range is the N K-line at 3.16 nm. High reflectivities at this wavelength can be achieved with Cr/Sc multilayer mirrors if the interfaces between adjacent layers are extremely smooth. Hence, the deposition parameters of the magnetron sputtering process for these materials have to be optimized carefully.

For X-ray microscopy, the wavelength range between the absorption edges of oxygen (2.28 nm) and carbon (4.36 nm) is very important because it provides a good contrast between organic materials and water^[10]. Therefore this spectral region is called "water window". The development of X-ray microscopes for this region has been retarded for long time by the absence of

optical elements like mirrors or lenses. Modern X-ray microscopes are usually based on zone plates as diffractive elements, and the resolution of these microscopes is mainly determined by the smallest spacings between the concentric rings that form the zone plates^[11].

An improvement in future developments of X-ray microscopes may be possible by using multilayer mirrors as optical elements, e. g. in a Schwarzschild objective^[12]. The advantages of a Schwarzschild objective are a large aperture and no chromatic aberrations. In addition to imaging optics multilayer mirrors are best suited for condenser optics of plasma sources^[13] because of their possibility to act simultaneously as monochromator and focusing element.

It has been demonstrated previously that the combination Cr/Sc is suitable to realize multilayer mirrors for the water window^[14]. Model calculations show that for wavelengths close to the nitrogen K-line at 3.16 nm, the material combination Cr/Sc has a higher theoretical reflectivity than other material combinations^[15]. The N K-line can be used as an X-ray source in the water window, especially for the laboratory use where no synchrotron radiation is available.

3.2.1 Measured soft X-ray reflectivity

The soft X-ray reflectivity at near normal incidence was measured with synchrotron radiation at the undulator beamline U125/1-PGM at BESSY II, Berlin, Germany. Due to the absorption behavior of scandium, the highest reflectivity of $R = 17.3\%$ was achieved close to the Sc-2p absorption edge at a wavelength of $\lambda = 3.11$ nm. The reflectivity curve as well as the multilayer design parameters are shown in Fig. 8.

3.2.2 Structural characterization by TEM and AFM

The cross-sectional TEM image of Cr/Sc show that the multilayer is homogeneous without any perturbations (Fig. 9). The multilayer structure is continuous and smooth; there is no evidence of amplifying roughness or columnar

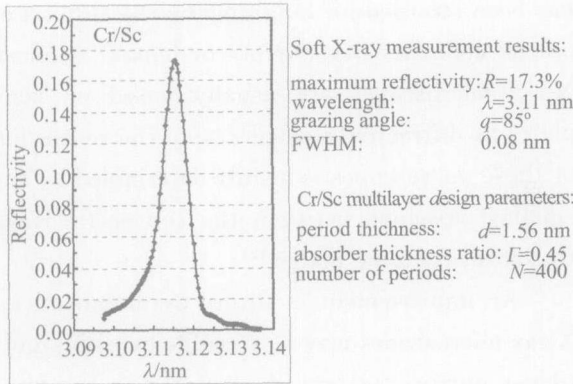


Fig. 8 Measured soft X-ray reflectivity of a Cr/ Sc multilayer and corresponding design parameters

growth through the multilayer stack. Since Cr has a higher atomic number, the Cr layer is dark and the Sc layer is bright. Fig. 10 shows the multilayer surface measured by atomic force microscopy over a scan area of $1\ \mu\text{m} \times 1\ \mu\text{m}$ with a resolution of 512×512 data points. A rms roughness of $\sigma = 0.14\ \text{nm}$ was calculated from the measurement. This fact indicates on the insignificant development of roughness in Cr/ Sc multilayer structure with increasing number of periods. Hence, in order to enhance the reflectivity behavior, the number of periods should be increased further to $N = 500 \dots 600$.

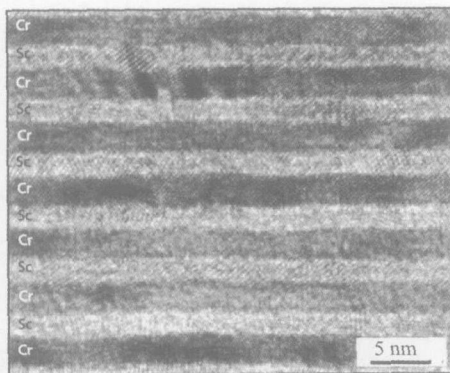


Fig. 9 High resolution cross section TEM image

3.3 Sc/ Si multilayers for $\lambda = 35 \sim 50\ \text{nm}$

The Sc/ Si material couple is a promising candidate for high-reflective normal incidence multilayer mirrors in the wavelength range between $35\ \text{nm}$ and $50\ \text{nm}$ ^[16-17]. The development

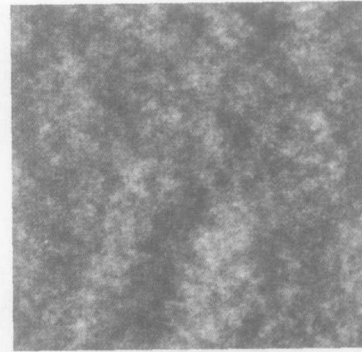


Fig. 10 Nanotopography, measured by AFM

of multilayer optics for this part of EUV range can give new impulses in the wide applications of discharge X-ray lasers^[18-19], study of generation of high-order spatially coherent harmonics from solid targets^[20] and construction of new astrophysical^[21] and spectroscopy^[22] instrumentations.

3.3.1 Measured EUV reflectivity

A series of Sc/ Si multilayer samples with a period $d = 18.7\ \text{nm} \dots 28.4\ \text{nm}$ was prepared on Si (100) substrates for reflectivity measurements with synchrotron radiation. The multilayer periods d were optimized to achieve maximum reflectivity at near normal incidence in the wavelength range from $35\ \text{nm}$ to $50\ \text{nm}$. The samples consist of 20 bilayers with silicon on top. All samples were measured at near-normal incidence ($\theta = 85^\circ$) and the energy of the incoming synchrotron beam was scanned over the region of maximum reflectivity (Fig. 11).

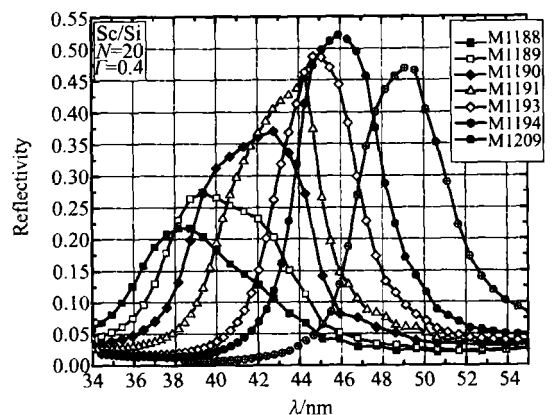


Fig. 11 Measured reflectivity of Sc/ Si multilayers at an grazing angle of $\theta = 85^\circ$

In the spectrum range from 38 nm to 46 nm the reflectivity R of Sc/Si multilayer mirrors have the tendency to growth from 23 % to 52 %, respectively. Maximum reflectivity of $R = 52 %$ at $\lambda = 45.9 \text{ nm}$ ($E = 27 \text{ eV}$) was measured for the sample M1194. The FWHM is $\Delta\lambda = 4.62 \text{ nm}$ and the selectivity of the mirror $S = \Delta\lambda / \lambda$ is comparable to the number of periods N . With increasing wavelength more above 46 nm a reflectivity decrease was found. According to these experimental results the strong transition of the 3p-core electrons to the practically unoccupied 3d in scandium occurs at the wavelength about 46 nm ($E = 27 \text{ eV}$), in the spectral range where maximum reflectivity was found.

The comparison of theoretical (using IMD software^[23] and Henke's optical constants^[24]) and experimental reflectivity behaviors vs. wavelength from different studies of Sc/Si multilayer mirrors is shown in Fig. 12. It has to be noted that our experimental results has a considerable disagreement with the reflectivity measurements from Ref. [16]. According to these previous studies the maximum reflectivity $R = 54 %$ at the wavelength of 36 nm Sc/Si material pair can be achieved and the reflectivity has the tendency to drop with increasing wavelength. This behavior agrees with theoretical simulations using optical constants from Henke^[21]. In con-

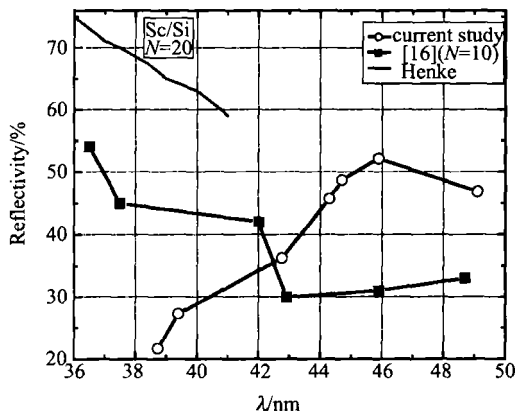


Fig. 12 Comparison of calculated and measured normal incidence reflectivities of Sc/Si multilayers

trast our measured reflectivity results in the wavelength range $41 \text{ nm} > \lambda > 36 \text{ nm}$ show an opposite behavior and cannot be describe by Henke's optical constants.

3.3.2 Structural characterization by TEM and AFM

It has to be noted that due to strong chemical activity of scandium a conventional preparation process of Sc/Si samples for TEM investigation can result in artifacts. Therefore, a low-temperature preparation process ($T < 30 \text{ K}$) was used. Crosssectional TEM image of Sc/Si multilayer coating is shown in Fig. 13. The light and dark (with light regions) bands correspond to the Si and Sc layers, respectively. The multilayer structure is continuous and smooth. The metal layers are a polycrystalline fcc scandium. The silicon layers appear to be amorphous. In growth direction the width of the scandium crystallites

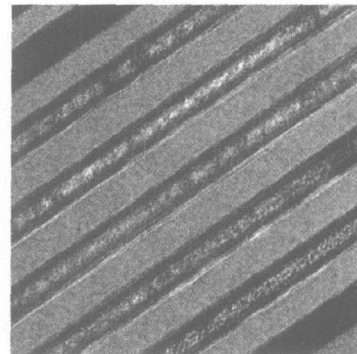


Fig. 13 Cross section TEM image of Sc/Si ($d = 24.5 \text{ nm}$ and $\sigma = 0.37$)

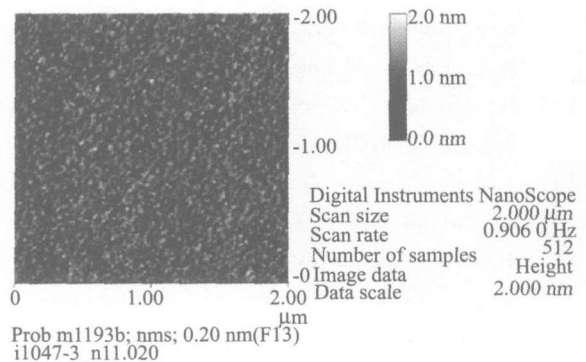


Fig. 14 Nanotopography, measured by AFM

and the pure amorphous silicon layers are 5.5 nm ± 0.2 nm and 14.9 nm ± 0.2 nm, respectively. The pure layers are separated by amorphous transition zones. The width of the transition zones is 2.5 nm ± 0.2 nm and 1.7 nm ± 0.2 nm on Sc-on-Si and Si-on-Sc interfaces, respectively. The interpretation of Sc/Si TEM images and especially separation of the real physical phenomenon and artifacts due to sample preparation for this material system is difficult and not obvious.

The quantitative analysis of the surface roughness has been performed by AFM investigation. It was found that the surface roughnesses are ± 0.2 nm and ± 0.21 nm for the Sc/Si multilayer mirrors with top silicon (Fig. 14) and top scandium layers, respectively. These values are close to the surface roughness of the Si-substrates.

4 Summary

High reflective Mo/Si, Cr/Sc and Sc/Si multilayer mirrors for different applications in the EUV and soft X-ray wavelength range were designed, deposited by dc magnetron sputtering

and characterized by different optical and non-optical methods. Maximum near normal incidence reflectivities of $R(\text{Mo/Si}) = 68.8\% @ \lambda = 13.45 \text{ nm}$, $R(\text{Cr/Sc}) = 17.3\% @ \lambda = 3.11 \text{ nm}$ and $R(\text{Sc/Si}) = 56.6\% @ \lambda = 44.6 \text{ nm}$ were measured at BESSY II, Berlin, Germany using the PTB reflectometer (Mo/Si) and the Bessy polarimeter (Cr/Sc and Sc/Si). To enhance the reflectivity, further work will concentrate on optimized diffusion barrier layers for Mo/Si and Sc/Si.

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Brief professional biography of the author :

Torsten Feigl received his Diplomphysiker in 1995 and his Ph. D. in 2000 from the University of Jena , Germany. He has held his MBA from the University of Bradford , England , U. K. since 2004. Torsten Feigl joined the Fraunhofer IOF in 1994 working on surface characterization by atomic force microscopy and related methods. In 1997 he started to work on design , manufacturing and characterization of optics for the extreme ultraviolet spectral range and X-rays. He is currently leading the VUV , EUV and X-ray optics working group at the Fraunhofer IOF. Torsten Feigl is author of more than 20 EUV and X-ray optics related publications and patents.