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High efficiency Al/Sc-based multilayer coatings in the EUV wavelength range above 40 nanometers

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In this work, we have developed new and highly efficient periodic multilayer mirrors Al/Sc, Al/Sc/SiC and Mo/Al/Sc with optimized reflectance at wavelengths between 40 nm and 65 nm. We have reached record values in measured peak reflectance: 57.5% at 44.7 nm and 46.5% at 51 nm, with Al/Sc/SiC at near-normal incidence. Furthermore, we have achieved the largest reported bandwidth with Mo/Al/Sc at 57 nm and the narrowest bandwidth with Al/Sc at 60 nm wavelength. These new and promising results demonstrate that Al/Sc-based multilayer coatings are excellent candidates for future generations of EUV instruments for solar physics, EUV lasers and attosecond science, in a wavelength range that hasn't been fully explored.

realization of efficient multilayer coatings. Earlier, Sc/Si-based multilayers were developed to operate at wavelengths longer than 40 nm, to take advantage of the low absorption of Sc in the spectral region below its M_{2,3} edge [6]. Corrosion-resistant, MgAl/SiC multilayers were also developed for 25-80 nm, [4,7] as well as Tb/Si and Gd/Si multilayers for the 60 nm wavelength region [5,8]. Al/Sc-based multilayers were first proposed by our group [9]; we have also performed the first experimental demonstration of the improvement in reflectance with 3-material vs. 2-material multilayer systems [10]. In this paper, we are presenting the first EUV experimental demonstration of periodic multilayer coatings based on the Al/Sc material system. Samples were designed to peak near 44.7, 51.5 or 60 nm wavelengths and achieved record reflectance and bandwidth values. The results presented include grazing incidence X-Ray Reflectometry (XRR) at 8.05 keV and at-wavelength, normal incidence measurements in the EUV range at the Advanced Light Source (ALS) synchrotron.

Reflective multilayer interference optics have enabled the operation at normal incidence angles in the extreme ultraviolet (EUV) range for instrumentation (imaging and illumination mirrors, gratings, polarizers) for solar and planetary physics, plasma diagnostics, tabletop and free-electron lasers and high-harmonic generation sources. EUV spectral features of the Sun's corona and photosphere are currently scanned by several space-based telescopes equipped with such multilayer optics: SoHO, STEREO, SDO and soon Solar Orbiter (to be launched in February 2020). Most EUV imagers and spectrometers have been conceived to detect emission lines of Fe and He at wavelengths between 10 and 40 nanometers [1]. There are several interesting emission lines between 40 and 80 nm, for example Ne VII at 46.5 nm, He I at 58.4 nm and O V at 63 nm. However, few instruments have been developed to explore this portion of the EUV range due to technological limitations, most notably the absence of reliable refractive index values [2-5] that prevents the design and

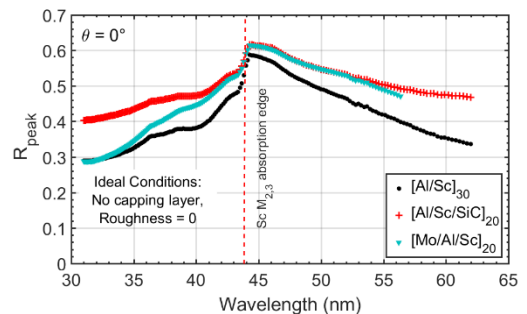


Fig. 1. Calculated ideal peak reflectance (R_{peak}) for Al/Sc, Al/Sc/SiC and Mo/Al/Sc multilayers from 30 to 60 nm wavelengths at normal incidence ($\theta = 0^\circ$). The number of periods in each multilayer has been chosen in order to saturate the peak reflectance in the entire wavelength range and is shown in the legend as a subscript.

Table 1. Fitted values of layer thicknesses (d_{Al} , d_{Sc} , $d_{SiC(Mo)}$, d_{Top}) and multilayer period thicknesses (D_{ML}) for Al/Sc, Al/Sc/SiC and Mo/Al/Sc multilayers deduced from X-Ray Reflectometry at 8.05 keV. The number of periods in each multilayer is shown under “Structure”, as a subscript.

#	Structure	d_{Al} (nm)	d_{Sc} (nm)	$d_{SiC(Mo)}$ (nm)	d_{Top} (nm)	D_{ML} (nm)
9055	[Al/Sc] ₂₅	14.3	9.7	--	5.23	23.9
9049	[Al/Sc/SiC] ₂₀	10.18	9.71	4.17	--	24.06
9051	[Mo/Al/Sc] ₂₀	7.92	11.40	4.56	5.05	23.87
9056	[Al/Sc] ₂₅	16	13.5	--	5	29.51
9060	[Al/Sc/SiC] ₂₀	11.97	12.11	5.68	--	30.02
8030	[Al/Sc] ₁₀	20.04	19.88	--	3.02	39.92
9052	[Mo/Al/Sc] ₇	11.32	16.72	8.13	9.73	36.17

The choice of materials and the specific multilayer designs were based on calculations using a homemade Matlab code and the best available optical constant (refractive index) values from the literature, in the 30-65 nm wavelength region [2,11-13]. It should be noted that the availability of reliable optical constants values in this wavelength range is sparse [2]. Fig. 1 shows the calculated “ideal” (zero interfacial roughness/diffusion) peak reflectance of the Al/Sc, Al/Sc/SiC and Mo/Al/Sc periodic multilayer systems at normal-incidence. The 3-material multilayers Al/Sc/SiC and Mo/Al/Sc were chosen because they have the potential for even higher peak reflectance than Al/Sc, with Al/Sc/SiC having the highest performance both above and below the Sc M_{2,3} edge (43.8 nm). In this study, we focus on wavelengths longer than the Sc M_{2,3}-edge. Close to the Sc-edge, where Sc absorption is the lowest, the difference in peak reflectance between Al/Sc, Al/Sc/SiC and Mo/Al/Sc is not so large. The advantage of using 3-material multilayers becomes more significant as the wavelength increases away from the absorption edge, where the material absorption increases as explored in our previous studies [10,14].

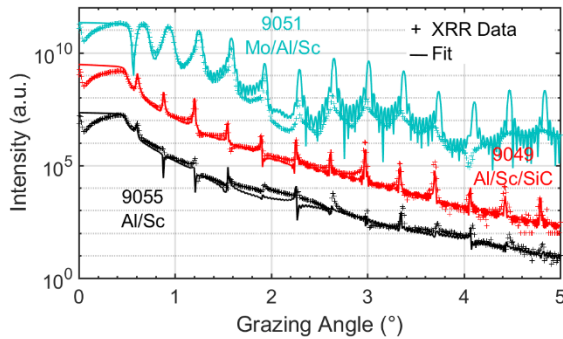


Fig. 2. Grazing incidence X-Ray Reflectometry measured and fitted curves at 8.05 keV for Al/Sc (9055), Al/Sc/SiC (9049) and Mo/Al/Sc (9051) multilayer samples.

All multilayers were prepared at *Laboratoire Charles Fabry* with a magnetron sputtering system, which can accommodate up to four targets [10] and can be used either in a RF or a DC mode according to the deposited material. The substrates were Si wafer pieces in (100) crystal orientation with surface microroughness in the 0.2 nm range. We used pure Mo, SiC and Sc targets (better than 99.95% purity) and a Si-doped (1.5 wt. %) Al target. During the deposition process, we used a 2 mTorr Ar pressure in the deposition chamber.

The plasma discharge was established with a DC current of 0.1 A for the Sc target, 0.06 A for the Mo target, and a RF power of 200 W and 150 W for the Al and SiC targets, respectively. We protected all multilayers with a SiC top layer thicker than 3 nm. We have previously demonstrated that SiC capping layers are able to assure a good temporal stability of Al-based multilayers [15]. For Al/Sc and Mo/Al/Sc multilayers, we optimized the SiC top layer thickness (shown in Table 1 as d_{Top}) in order to reach the highest reflectance.

All samples were measured by XRR at 8.05 keV (Cu K α line, $\lambda = 0.154$ nm) on a Bruker Discover D8 diffractometer. We fit the XRR data with a genetic algorithm by using the program Leptos®, and optical constant values from the “Henke tables” [16]. These fits allow to determine the multilayer period, layer thickness and average values of layer interface roughness/diffusion. The fitted layer thickness values for the samples discussed in this work are summarized in Table 1. The sensitivity is estimated to be 0.01 nm for the period thickness and close to 0.1 nm for the individual layer thicknesses according to the Bragg peaks positions. Fig. 2 shows the XRR results from 3 representative samples, indicating good layer quality and periodicity for all 3 samples. The quality of each fit is ensured by the good agreement between simulated and experimental intensities of the successive Bragg peaks. The broadening of the higher-order Bragg peaks for the Mo/Al/Sc multilayer may indicate a non-negligible Mo-related interface effect that is still under study. The absence of pronounced peaks in the XRR data from the Al/Sc sample is due to the low optical contrast between Al and Sc at this wavelength. The interfacial

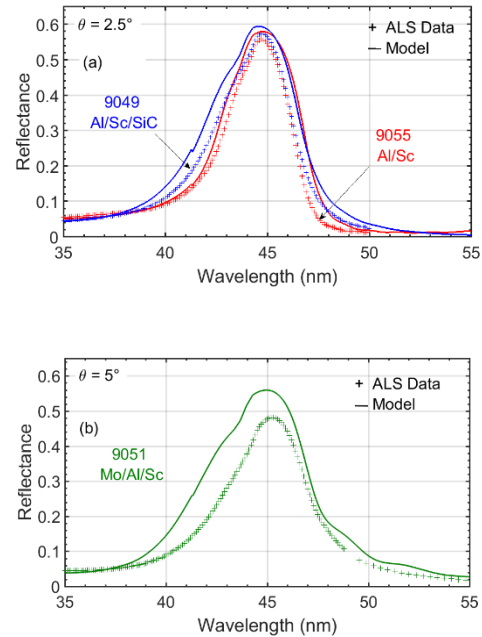


Fig. 3. ALS synchrotron reflectance measurements in the EUV range for (a) Al/Sc, Al/Sc/SiC and (b) Mo/Al/Sc multilayer samples at near-normal angles of incidence (indicated on the graph). The simulated (model) reflectance curves are also shown, using IMD [19].

roughness/diffusion values fitted from the XRR data were in the range 0.5-0.7 nm.

At-wavelength, absolute reflectance measurements of the samples were performed at the ALS synchrotron beamline 6.3.2 at

Lawrence Berkeley National Laboratory. This beamline is designed for high-precision EUV optical metrology including reflectivity measurements [17,18]. The beamline has a spectral resolving power ($\lambda/\Delta\lambda$) of up to 7000, a wavelength accuracy of 2×10^{-3} nm, and a reflectance accuracy of 0.1% (absolute). During the measurements, signal was collected with a Si photodiode detector. All samples were stored in a clean environment and measured 2-4 weeks after deposition.

Fig. 3 shows the measured near-normal-incidence reflectance of Al/Sc, Al/Sc/SiC and Mo/Al/Sc samples, optimized to peak near 44.7 nm. The simulated values for each curve are also shown, using the IMD software [19] with optical constants from [11] for Al, [12] for Sc, [11] for Mo and [13] for SiC. The layer model used in IMD to simulate EUV reflectance was based on the model derived from the XRR fits and included the layer thicknesses and the interface roughness/interdiffusion values determined from the XRR measurements (Fig.2 and Table 1).

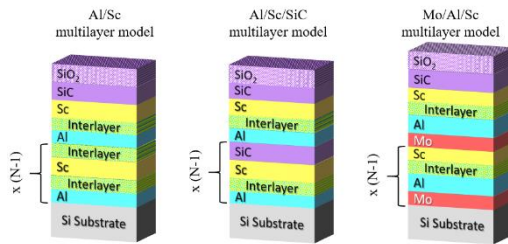


Fig. 4. Schematic layer models of Al/Sc, Al/Sc/SiC and Mo/Al/Sc multilayers used to fit the reflectance data in the EUV range. N represents the total number of periods in each multilayer.

Table 2. Simulated (R_{simu} , λ_{simu}) and measured (R_{exp} , λ_{exp}) peak EUV reflectance and peak wavelength for Al/Sc, Al/Sc/SiC and Mo/Al/Sc multilayers. The measured reflectance bandwidth (BW_{exp}), defined as the full width at half-maximum is also listed.

Angle	#	R_{simu} (%)	λ_{simu} (nm)	R_{exp} (%)	λ_{exp} (nm)	BW_{exp} (nm)
$\theta = 2.5^\circ$	9055	58.0	44.8	56.0	44.7	3.5
	9049	59.5	44.6	57.5	44.7	4.1
$\theta = 5^\circ$	9051	55.9	45	48.1	45.2	4.6
	9056	48.8	51.5	42.4	51.0	4.1
	9060	52.4	50.8	46.5	51.0	5.6
	8030	37.2	62.8	27.2	60.6	5.2
	9052	40.7	58.8	37.4	57.3	11.2

Some modifications to the XRR models were applied in order to better simulate the EUV data (Fig. 4). Based on earlier work [14], we have determined that a native oxide on top of the SiC capping layer modeled with an average 1.5 nm thick SiO₂ layer is a good approximation. Also, we chose to keep a simple surface model without carbon contamination because of the lack of optical constant values for such a compound in the 40 – 80 nm wavelength range. However, we added an interlayer between Al and Sc modeled by a “graded interface” function with the IMD software. The presence of such an interlayer of about 1-2 nm in thickness has been confirmed by Transmission Electron Microscopy analysis which will be discussed in an upcoming publication.

As shown in Fig. 3 and Table 2, Al/Sc and Al/Sc/SiC achieve measured peak reflectance (R_{exp}) close to their simulated peak reflectance value (R_{simu}), with Al/Sc/SiC producing $R_{\text{exp}} = 57.5\%$ and a spectral bandwidth BW_{exp} of 4.1 nm. Before this work, the highest reported R_{exp} at 44.7 nm at near-normal incidence was 56.6% (BW_{exp} of 4.8 nm), from Sc/Si with Cr barrier layers [20]. Our results produce higher peak reflectance and narrower bandwidth. The R_{exp} of Mo/Al/Sc is 48.1% - it is not as close to its simulated reflectance value, which is attributed to lack of accuracy in Mo optical constants values in the 44.7 nm wavelength region, and probable interfacial effects related to the Mo layer that are not taken into account in our present Mo/Al/Sc model.

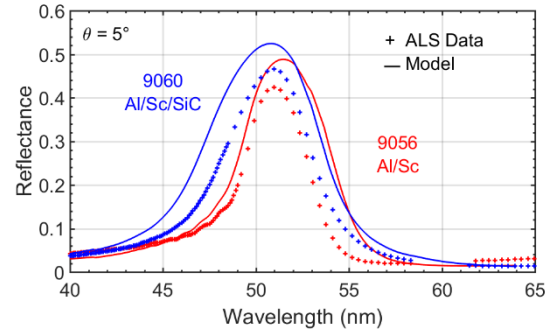


Fig. 5. ALS synchrotron reflectance measurements and simulations (model) [19] in the EUV range for Al/Sc and Al/Sc/SiC multilayer samples at 5° from normal incidence.

Fig. 5 shows the measured normal incidence reflectance of Al/Sc ($R_{\text{exp}} = 42.4\%$) and Al/Sc/SiC ($R_{\text{exp}} = 46.5\%$), optimized to peak near 51 nm. The simulated reflectance is also shown, using optical constants from [11-13]. The increase of reflectance and bandwidth with the addition of a 3rd multilayer material is evident (Table 2), as has been discussed in our previous studies [10] and in the theoretical calculations plotted in Fig. 1. To our knowledge, no other experimental multilayer reflectance value has been reported at 51 nm so far. The closest wavelength found in the literature is 49 nm, with $R_{\text{exp}} = 46.7\%$ ($BW_{\text{exp}} = 4.9$ nm), achieved with Sc/Si multilayers with Cr barrier layers [20]. The measured reflectance of our Al/Sc and Al/Sc/SiC samples, although it is amongst the highest reported in the literature, is appreciably lower than their simulated reflectance, indicating that the accuracy of available optical constants for Sc, Al and/or SiC is diminishing in the 51 nm region.

Fig. 6 shows the measured normal incidence reflectance of Al/Sc ($R_{\text{exp}} = 27.2\%$) and Mo/Al/Sc ($R_{\text{exp}} = 37.4\%$), as well as the simulated reflectance using optical constants from [11-13], optimized to peak near 60 nm. The effect of the 3rd multilayer material and the lack of accurate optical constants mentioned in the previous paragraph are also evident here. In earlier literature, the following measured values have been reported in the 60 nm wavelength region: $R_{\text{exp}} = 38.8\%$ with AlMg/SiC at 55.7 nm ($BW_{\text{exp}} = 5.6$ nm) [4], $R_{\text{exp}} = 26.2\%$ with Gd/Si at 62 nm ($BW_{\text{exp}} = 7.3$ nm) [5], $R_{\text{exp}} = 23\%$ with Si/Tb at 60 nm ($BW_{\text{exp}} = 10$ nm) [3], $R_{\text{exp}} = 23\%$ with Si/Tb at 60 nm ($BW_{\text{exp}} = 6.5$ nm) [8]. It should be noted that, in addition to very high reflectance, we have achieved the largest reported bandwidth with a periodic multilayer (Mo/Al/Sc) at 57 nm with $BW_{\text{exp}} = 11.2$ nm, as well as the narrowest bandwidth with Al/Sc at 60 nm wavelength with $BW_{\text{exp}} = 5.2$ nm. Therefore, the Al/Sc-based multilayer coatings

developed in this work may offer better out-of-band suppression at wavelengths near or longer than 60 nm, compared to the above-mentioned multilayers from the literature. This is of particular importance for applications requiring high spectral selectivity, such as solar imaging and high-order harmonic selection. Table 2 summarizes the experimental and simulated peak EUV reflectance values and bandwidths discussed in this manuscript, and Table 3 compares our most significant experimental results with the best values reported in the literature.

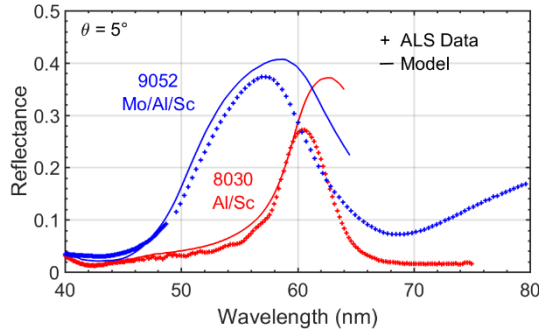


Fig. 6. ALS synchrotron reflectance measurements and simulations (model) in the EUV range for Al/Sc and Mo/Al/Sc multilayer samples at 5° from normal incidence. The simulations stop at 65 nm due to lack of optical constants values of Al and Mo in the 65-80 nm wavelength region.

Table 3. Main results and comparison with literature values near normal incidence

Al/Sc-based multilayer values				
R _{exp}	57.5%	46.5%	37.4%	27.2%
λ _{exp}	44.7 nm	51 nm	57.3 nm	60.6 nm
BW _{exp}	4.1 nm	5.6 nm	11.2 nm	5.2 nm
Structure	Al/Sc/SiC	Al/Sc/SiC	Mo/Al/Sc	Al/Sc
Literature values				
R _{exp}	56.6%	46.7%	38.8%	23%
λ _{exp}	44.7 nm	49 nm	55.7 nm	60 nm
BW _{exp}	4.8 nm	4.9 nm	5.6 nm	6.5 nm
Structure	Sc/Cr/Si	Sc/Cr/Si	AlMg/SiC	Si/Tb
Ref	[20]	[20]	[4]	[8]

In conclusion, despite being severely limited by uncertainties in the available optical constant values in the EUV range between 40 and 80 nm, we have reached record reflectance and selectivity performance with new Al/Sc-based multilayer coatings operating in the 40 – 65 nm wavelength region. Further studies will be developed to understand in detail the structure and test the evolution of these multilayers through time and under different environmental conditions. These promising results are establishing Al/Sc-based multilayer coatings as leading candidates for future EUV instrumentation. The findings in this work also highlight the need for dedicated, accurate measurements of the refractive index of thin film materials, in order to capture the full potential of scientific discovery in this important wavelength region.

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Disclosures. The authors declare no conflicts of interest.

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