

# EUV multilayer coatings for solar imaging and spectroscopy

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## ABSTRACT

This paper describes recent progress in the development of new EUV multilayer coatings for solar physics. In particular, we present results obtained with Pd/B<sub>4</sub>C/Y, Al/Zr, and Al-Mg/SiC multilayers, designed for normal incidence operation in the 9 – 50 nm wavelength range. We describe the development of both periodic multilayer films designed for narrow-band imaging, and non-periodic multilayers designed to have a broad-spectral response for spectroscopy. The higher EUV reflectance provided by these new coatings, relative to older-generation coatings such as Si/Mo, Mo/Y, and others, will facilitate the development of future solar physics instruments for both imaging and spectroscopy having higher spatial and spectral resolution, while supporting the exposure times and cadences necessary to capture the evolution of flares, jets, CMEs and other dynamic processes in the solar atmosphere.

**Keywords:** Multilayers, EUV, Solar Physics

## 1. INTRODUCTION

Nanometer-scale multilayer coatings that reflect efficiently at normal incidence in the extreme ultraviolet (EUV) have proven extremely valuable to current space-based solar research targeting that spectral region. Multilayer-based instruments have been used in a number of major solar satellite instruments since the 1990's, including SoHO/EIT, TRACE, STEREO/EUVI, Hinode/EIS and SDO/AIA, and have flown in numerous sounding rocket experiments since the 1980's as well. They are by now an essential, mission-enabling technology that is widely used in both imaging and spectroscopy instruments, in rockets and satellites; currently almost all EUV observations of the Sun are made with multilayer-coated optics.

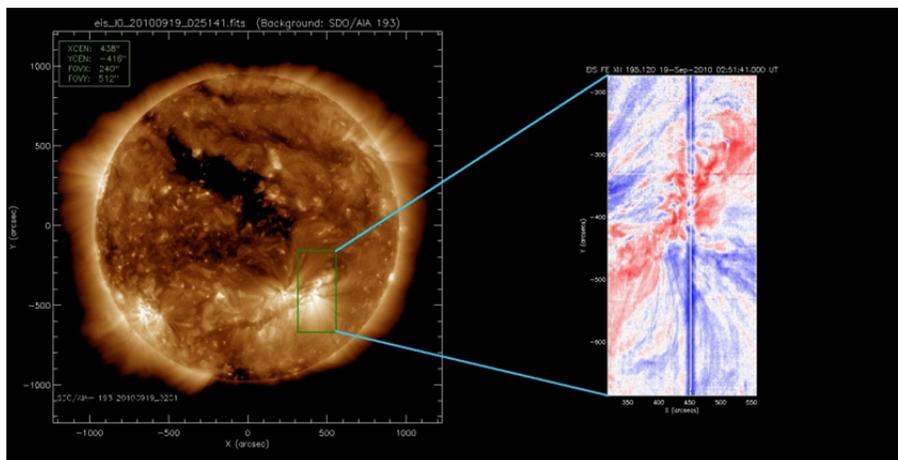


Figure 1. EUV imaging and spectroscopy results from the SDO/AIA (left) and Hinode/EIS (right) instruments. Both instruments use optics coated with EUV multilayers.

Multilayer coatings make possible the construction of mirrors and reflection gratings that operate efficiently at normal incidence in the EUV. Normal incidence optics can be fabricated with far greater precision, and at much lower cost, than grazing incidence optics (the only practical alternative until multilayers became readily available in the 1980's.) Furthermore, diffraction gratings working in reflection near normal incidence can achieve much higher spectral resolution than those operating at grazing incidence. Consequently, the use of normal-incidence EUV mirrors and

gratings over the past three decades has led to imaging and spectroscopy instruments for solar research having much higher spatial resolution, spectral resolution, and cadence than would have been possible otherwise.

In order to effectively address a variety of open questions in coronal dynamics, future solar missions targeting the EUV region that are now being formulated, such as FACTS, RAM, Solar-C, and others, will require even higher spatial resolution and better signal-to-noise than has been achieved thus far in any satellite instrument, while supporting the exposure times and cadences necessary to capture the evolution of flares, jets, CMEs and other dynamic processes in the solar atmosphere.<sup>1</sup> For EUV instruments using multilayer coatings, these performance requirements on instrumental resolution and signal-to-noise translate, in practice, into the need for better multilayer coatings. In particular, narrow-band multilayer coatings for imaging instruments having higher peak reflectance and better spectral selectivity are needed to avoid the problems of wavelength mixing and long exposure times, while efficient broad-band coatings are needed for sensitive, high-resolution spectroscopic instruments, particularly at EUV wavelengths below  $\sim 12$  nm, where efficient, broad-band coatings have been unavailable (until now.)

In this paper we describe some recent progress in the development of new EUV multilayer coatings for solar physics, including both narrow-band coatings designed for imaging, and broad-band coatings designed for spectroscopy. After a brief overview of the technology, we describe three new multilayer systems that are currently under development – Pd/B<sub>4</sub>C/Y for the 9–14 nm band, Al/Zr for 17–25 nm, and Al-Mg/SiC for 25–50 nm – and compare their measured performance to multilayer coatings developed previously. We conclude with a brief summary of our findings, and an outline for future research directions.

## 2. EUV MULTILAYERS: OVERVIEW

At EUV wavelengths shorter than about 50 nm, “single-layer” coatings – reflective coatings that comprise just one thin layer of material, such as Au, SiC, B<sub>4</sub>C, etc. – provide high reflectance only at grazing incidence; at normal incidence the reflectance of even the best single-layer coatings is too small for the construction of efficient solar instrumentation. Multilayer coatings, in contrast, use optical interference to achieve high reflectance at normal incidence in the EUV, and their high reflection efficiency has enabled remarkable advancements in a variety of scientific and technological disciplines over the past three decades since their development, particularly solar physics and microlithography, but also ultrafast science, plasma diagnostics, and many other areas as well.

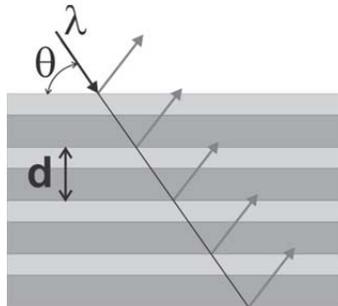


Figure 2. Schematic diagram of a periodic multilayer coating.

EUV multilayer coatings comprise a stack of nm-scale bilayers (typically) of optically dissimilar materials, as illustrated in Fig. 2; the layer thicknesses are designed so that the small reflections that occur at each interface in the stack add coherently, in phase. Just as Bragg's law describes the condition for constructive interference of X-rays in a crystal, the same law describes the condition for constructive interference in a multilayer film (albeit with small, yet important corrections for refraction within the layers, which we ignore for this simplified discussion): for a “periodic” multilayer, i.e., a film stack containing a number of repeating, identical bilayers of thickness  $d$ , that is meant to provide high reflectance over a narrow range of wavelengths, Bragg's law (in 1st order) is given by  $\lambda=2d\sin\theta$ , where  $\theta$  is the grazing incidence angle and  $\lambda$  is the photon wavelength. Near normal incidence ( $\theta\sim 90^\circ$ ), therefore, the bilayer thickness  $d$  is equal to half the photon wavelength (approximately – again, we have neglected the small corrections due to refraction), and thus the individual layers in each bilayer are only a few nanometers thick for operation at EUV wavelengths. By adjusting the bilayer thickness during deposition, typically with sub- $\text{\AA}$  control, it is possible to tune the peak of the multilayer spectral reflectance curve to precisely match the desired wavelength.

The narrow-band response of periodic multilayer coatings is ideally suited for quasi-monochromatic imaging, as in the SDO/AIA instrument,<sup>2</sup> for example. For spectroscopy, however, broad spectral response is required. At longer EUV wavelengths, this can be achieved by reducing the number of bilayers, in order to increase the spectral band-pass at the expense of peak reflectance. This approach was used, for example, on the Hinode/EIS instrument.<sup>3</sup> An alternative approach for creating broad-band reflective coatings is to use a non-periodic coating containing a range of layer thicknesses within the multilayer stack. Broad-band, depth-graded multilayer (DGML) coatings, in which the distribution of layer thicknesses in the film stack is specified analytically, have been successfully developed for the hard X-ray band, for example, and have been used in the construction of the telescopes for the NuSTAR astronomy mission,<sup>4</sup> and in other astronomy instruments as well. For coatings designed to operate over a broad-band in the EUV, however, the best performance at normal incidence can be realized using aperiodic multilayer (AML) coatings, which comprise a stack of layers whose thicknesses are specified numerically rather than analytically. An AML coating can be designed to provide flat response over a range of wavelengths. The layer thicknesses in such AML coatings can be determined using various numerical methods, including those available in the IMD software package.<sup>5</sup> AML coatings have been developed previously at certain EUV and X-ray wavelengths, for applications such as synchrotron instrumentation and ultrafast science.<sup>6,7,8</sup>

The achievable performance in a multilayer coating, be it a periodic, depth-graded or aperiodic design, is ultimately determined by the optical constants of the constituent materials that comprise the multilayer stack, and on the quality of the interfaces between the layers. That is, interface imperfections – interfacial roughness and diffuseness, which are typically quantified by an interface width parameter  $\sigma$  – have the effect of reducing the specular reflectance at that interface, either by scattering light into non-specular directions in the case of roughness, or by increasing the transmittance of the interface in the case of interfacial diffuseness.<sup>9</sup> In any case, the reduction in specular reflectance at the interfaces due to interface imperfections degrades the overall reflectance of the multilayer stack; for maximal performance, therefore, such imperfections must be minimized. In the case of ‘well-behaved’ material combinations (i.e., those that form smooth, continuous layers, and interfaces that are stable over time), interface imperfections can be minimized, in general, through control of the film deposition process parameters. While optical constants generally cannot be controlled once the constituent materials are selected, accurate values of the constituent material optical constants are nevertheless required in order to accurately design and model the performance of multilayer coatings, particularly AML designs.

There are additional considerations for the development of high-quality multilayers suitable for use in solar instrumentation. First, the net film stress in the coating must be sufficiently low so as to minimize the risk of stress-driven adhesion failures such as crazing, cracking or delamination. Such failures are generally catastrophic. Second, the coatings must have sufficiently high temporal and thermal stability, in order to avoid any degradation in coating performance over the lifetime of the mission. Third, the coatings must have high resistance to energetic particles in the case of instruments used in harsh environments (e.g., as in certain orbits subjected to a high particle flux.) Finally, in the specific case of aperiodic multilayer coatings, in order to achieve optimal broad-band EUV performance, the thickness of each layer in the AML stack must correspond to the design thickness with high accuracy; even small layer thickness errors can manifest as large errors in the resultant reflectance curve. It is thus necessary to calibrate the individual deposition rates of the constituent materials with high accuracy, so as to minimize such thickness errors during deposition.

Shown in Fig. 3 is a plot of the normal incidence reflectance of some of the best periodic multilayer coatings that were available for use in solar instrumentation approximately ten years ago. The coatings shown in Fig. 3 – Si/Mo, SiC/Si, and Mo/Y – were in fact used on the SDO/AIA telescope mirrors (among many other instruments). Si-based multilayers (i.e., Si/Mo, SiC/Si, etc.) provide high reflectance at wavelengths longer than the Si L-edge near 12.4 nm, due to the low optical absorption of Si in this range. Si/Mo in particular can provide nearly 70% peak reflectance close to the bright lines of Fe VIII, XX, and XXIII near  $\lambda=13$  nm, although the peak reflectance of Si/Mo falls steadily with increasing photon wavelength. At wavelengths shorter than  $\lambda=12.4$  nm, however, Si has much higher absorption, and so Si-based multilayer coatings work poorly in this range. Fortunately the Mo/Y multilayer system, developed in the 1990’s,<sup>10</sup> works reasonably well below 12.4 nm, providing over 30% peak reflectance near the Fe XVIII line at 9.4 nm wavelength.

As we describe in the following section, we are currently developing new multilayer coatings that have significantly better performance than the Mo/Y, Si/Mo and SiC/Si coatings used on SDO/AIA at many important solar wavelengths in the 9–35 nm wavelength range.

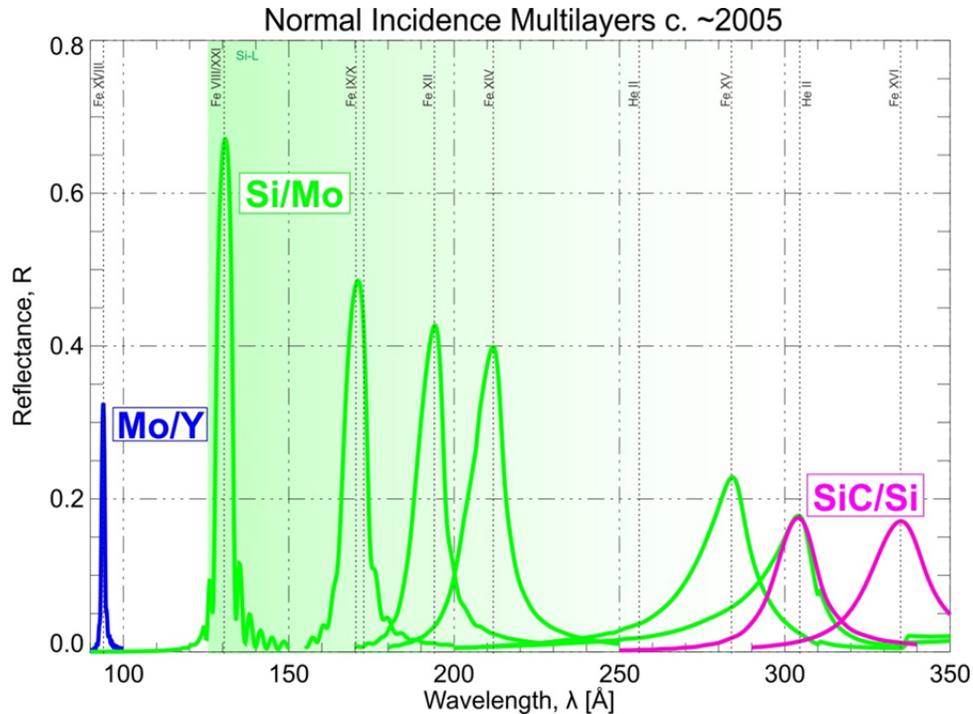


Figure 3. Some of the best normal incidence periodic multilayers that were available for use in solar instrumentation approximately ten years ago.

### 3. EUV MULTILAYERS: RECENT DEVELOPMENTS

#### Pd/B<sub>4</sub>C/Y Multilayers

Periodic, narrow-band Pd/B<sub>4</sub>C multilayers have been shown to have significantly higher peak reflectance than Mo/Y near 9 nm wavelength.<sup>11</sup> However, the Pd/B<sub>4</sub>C films developed thus far also have extremely high stress, thereby increasing the risk of coating failure. Motivated by the good EUV performance achieved with both Mo/Y and Pd/B<sub>4</sub>C, we have recently developed a new system comprising Pd and Y layers with thin (~0.6 nm) layers of B<sub>4</sub>C deposited at each interface;<sup>12</sup> the thin B<sub>4</sub>C layers are used to mitigate the diffusion of Pd and Y that would otherwise occur. Fig. 4 shows the normal-incidence reflectance, measured using synchrotron radiation at the ALS, of periodic Mo/Y, Pd/B<sub>4</sub>C, and Pd/B<sub>4</sub>C/Y multilayers tuned near 9.4 nm. Periodic Pd/B<sub>4</sub>C/Y multilayers provide 43% peak reflectance at 9.4 nm wavelength. The performance of Pd/B<sub>4</sub>C/Y exceeds that of both Pd/B<sub>4</sub>C and Mo/Y by significant margins. For a two-reflectance telescope, for example, the use of Pd/B<sub>4</sub>C/Y in place of Mo/Y would yield a 1.7x increase in effective area. Furthermore, this coating has acceptably low stress (-480 MPa), whereas the Pd/B<sub>4</sub>C film shown in Fig. 4 was found to have compressive stress of nearly -1.2 GPa. While calculated reflectance curves are not shown along with the measured curves for the three multilayers included in Fig. 4, from their analysis we find significant discrepancies between measurement and calculation for Pd/B<sub>4</sub>C/Y, suggesting that the optical constants of one or more of these materials may be inaccurate.

Cross-sectional transmission electron micrographs of Pd/B<sub>4</sub>C and Pd/B<sub>4</sub>C/Y films are shown in Fig. 5, where we compare the bottom six repetitions of each film at the highest magnification. Selected Area Electron Diffraction (SAED) patterns are included as inserts. Both films show relatively smooth, well-defined layers. Lattice fringes are visible in the Pd layers in the Pd/B<sub>4</sub>C multilayer, and the SAED image for this sample shows a diffraction pattern with 6-fold symmetry, indicating that the Pd layers in this film are polycrystalline. There is no evidence of crystallinity in either the Pd or Y layers in the Pd/B<sub>4</sub>C/Y film, however. The Pd/B<sub>4</sub>C/Y film also shows somewhat smoother, sharper interfaces, consistent with the higher EUV reflectance found in this type of structure.

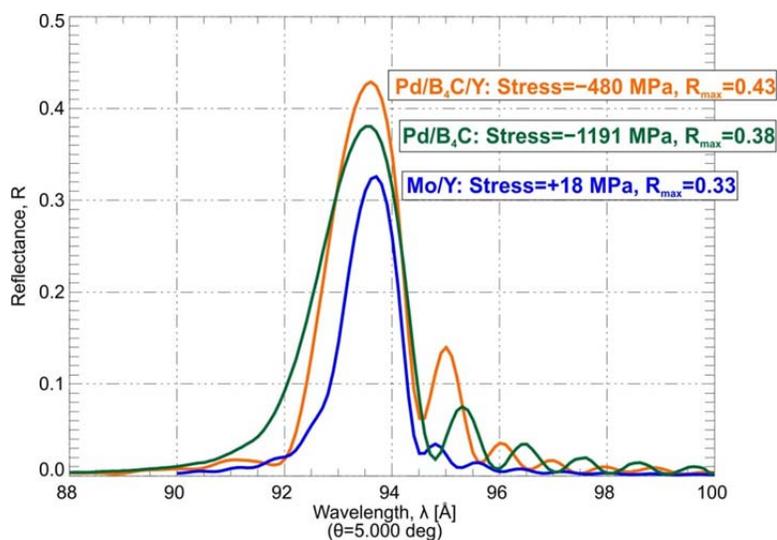


Figure 4. Normal-incidence reflectance of Pd/B<sub>4</sub>C/Y, Pd/B<sub>4</sub>C, and Mo/Y periodic multilayers tuned near 9.4 nm wavelength, as measured using synchrotron radiation at the ALS. The measured stress values for each film are labeled.

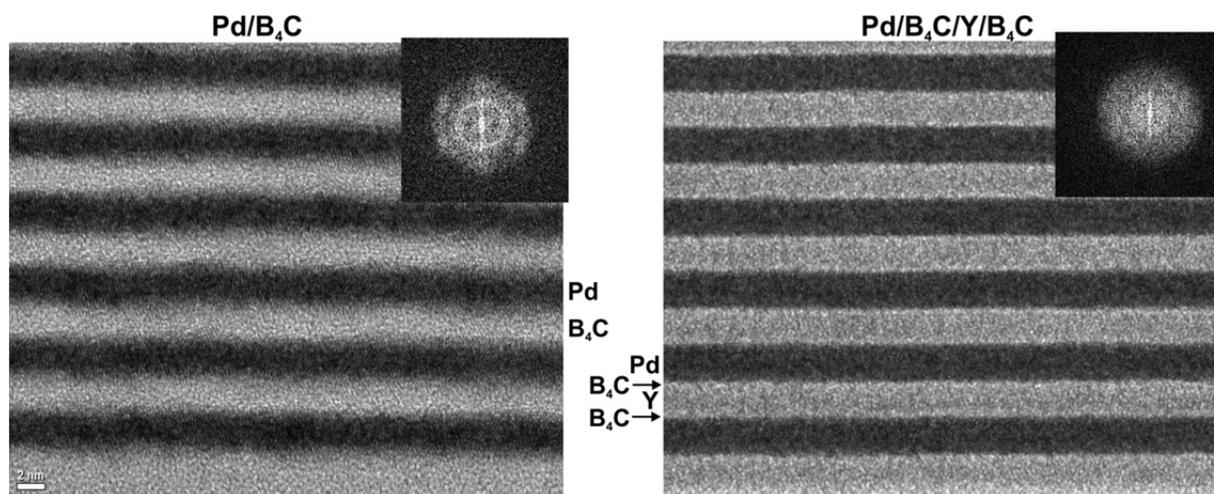


Figure 5. High-resolution transmission-electron-micrographs of multilayers containing repetitions of either Pd/B<sub>4</sub>C (left) or Pd/B<sub>4</sub>C/Y/B<sub>4</sub>C with  $d_{B_4C}=0.6$  nm (right), with a period of  $d \sim 5$  nm in both cases. SAED patterns from each film are shown as insets in the upper right corner in each case.

We have also begun to develop non-periodic Pd/B<sub>4</sub>C/Y multilayer films designed for broad-band response in the 9–14 nm region; periodic coatings having a reduced number of repetitions cannot provide sufficiently broad spectral response in this wavelength range, and up until now, non-periodic, broad-band coatings have not been demonstrated below ~12 nm wavelength.

Sample results of two non-periodic Pd/B<sub>4</sub>C/Y multilayer films are shown in Fig. 6. The measured and calculated normal-incidence reflectance curves for an analytically depth-graded structure containing  $N=120$  repetitions are shown in Fig. 6a. This film, which could be deposited onto a diffraction grating for spectroscopy from  $\lambda=8.9$  nm to  $\lambda=11.2$  nm, provides a relatively smooth response over this wavelength range, with an average reflectance of 5%. Shown in Fig. 6b is a fully aperiodic design containing the same number of repetitions and designed for a flatter response over the same wavelength band. Unfortunately layer thickness errors that occurred during deposition (and perhaps optical constants inaccuracies as well) have degraded the response significantly relative to the design, although the coating still provides an average reflectance of 5.2% over the designated wavelength band. Layer thickness errors can arise from imprecise deposition rate calibrations and/or coating system instability; both problems likely occurred during the deposition of the

films shown in Fig. 6, although the aperiodic design is far more sensitive to such errors. Future research will be directed at reducing layer thickness errors in order to produce broad-band films with better performance in this wavelength band.

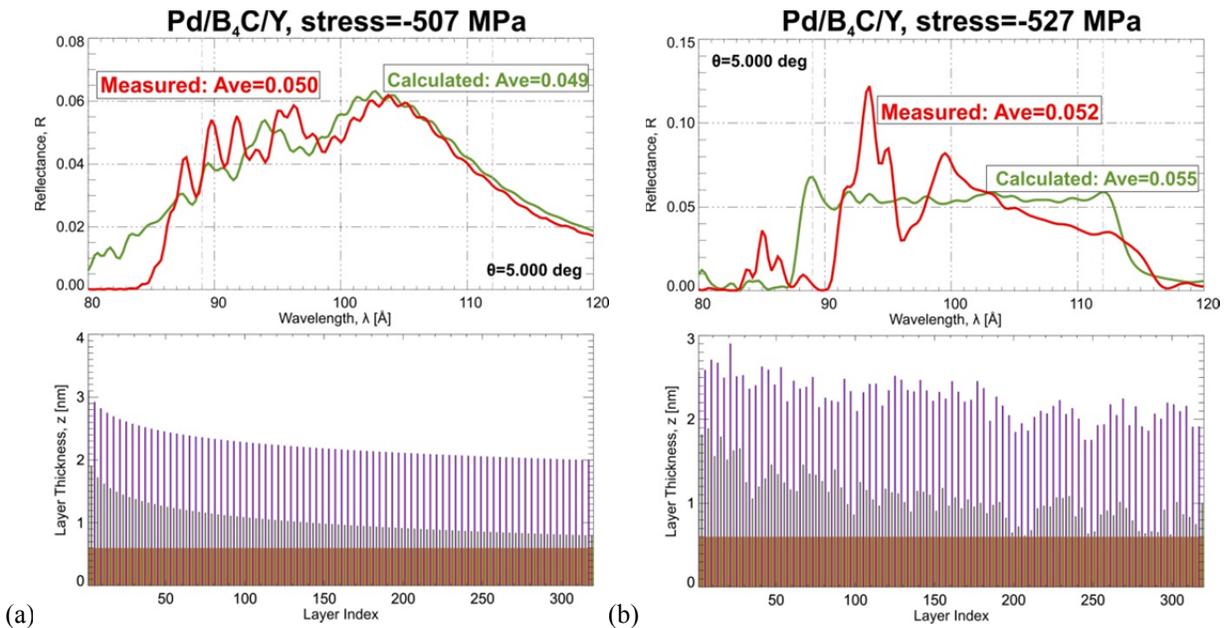


Figure 6. EUV reflectance measured at 5° incidence at the ALS, and modeled using IMD (top), and layer thickness distribution (bottom), for non-periodic multilayers: (a) analytically depth-graded design, and (b) aperiodic design.

We have begun to investigate the temporal and thermal stability of Pd/B<sub>4</sub>C/Y. The system appears relatively stable up to temperatures of 100C; at higher temperatures we observe significant changes in peak reflectance. By monitoring the reflectance of a prototype film stored in air at room temperature over a period of one year, we have so far observed only a small reduction (~10%) in peak reflectance. The observed reduction in peak reflectance might be due to surface contamination or oxidation, or it might be due to interface instability. Further research will be directed at more comprehensive characterization of the temporal stability of this new multilayer structure, in order to demonstrate its suitability for use in space-flight instrumentation.

### Al/Zr Multilayers

Two Al-based multilayer coatings, Al/SiC and Al/Zr, have been investigated in recent years for use at wavelengths longer than the Al L-edge near 17 nm.<sup>13,14,15,16</sup> The Al/Zr system in particular provides the highest reflectance thus far. For example, shown in Fig. 7 are the measured and calculated reflectance curves (assuming  $\sigma=1.2$  nm) for three periodic multilayers comprising layers of Zr and of an Al alloy containing 1% Si, with periods  $d=8.65$  nm, 8.85 nm and 9.05 nm as labeled. The Al/Zr coating having  $d=8.85$  nm provides nearly 60% peak reflectance near  $\lambda=17.3$  nm. As the multilayer period is decreased, the 1<sup>st</sup> order Bragg peak occurs closer to the Al L-edge. Of particular note is the large discrepancy between the measured and calculated reflectance near the edge, especially at wavelengths shorter than about 17.2 nm. This discrepancy is likely due to inaccurate (or insufficiently detailed) optical constants for Al in this range. Future research directed at improved Al optical constants should help resolve the discrepancy evident in Fig. 7. While not shown in Fig. 7, other Al/Zr coatings have been tested for somewhat longer wavelengths as well (e.g., for the Hi-C rocket instruments<sup>17</sup>); for example, normal-incidence peak reflectance in excess of 50% has been demonstrated near  $\lambda=19$  nm.

Broad-band, aperiodic Al/Zr coatings have been designed for spectroscopic applications. The theoretical reflectance of one such design, containing  $N=30$  repetitions of Al/Zr, is shown in Fig. 8. Also shown are the calculated reflectance curves of the periodic Si/Mo multilayer coatings used for the short-wavelength-band of the Hinode/EIS instrument, and for the TXI sounding rocket instrument. These periodic Si/Mo coatings were designed with only  $N=20$ , and  $N=5$ , repetitions, respectively, in order to provide a more broad spectral response while sacrificing the higher peak reflectance that would be possible with more repetitions. While the Si/Mo periodic coatings provide higher peak reflectance than the

aperiodic Al/Zr coating, the latter coating is expected to provide a much flatter response over the 17 – 21 nm wavelength band. Over the coming year, we plan to fabricate and test aperiodic Al/Zr multilayers like the one shown in Fig. 8.

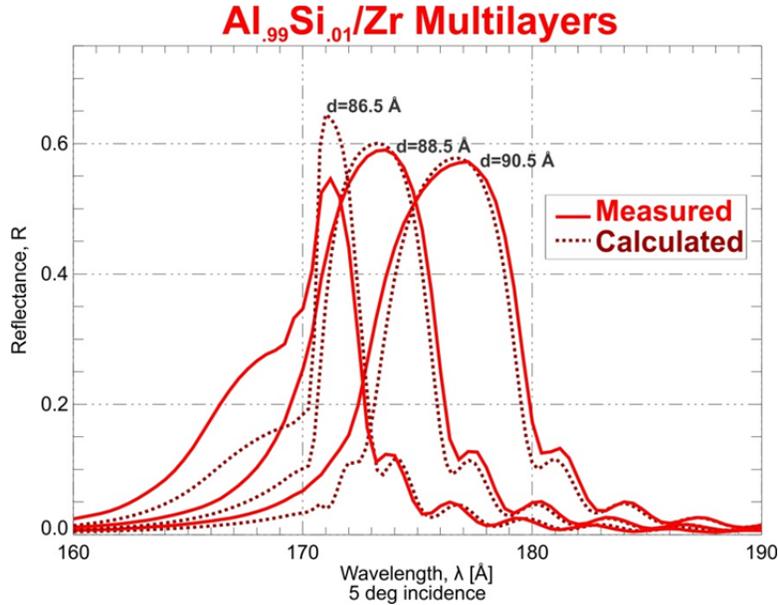


Figure 7. Normal incidence reflectance of periodic Al/Zr multilayers with periods  $d=86.5\text{\AA}$ ,  $88.5\text{\AA}$ , and  $90.5\text{\AA}$ , as indicated. The reflectance was measured at the ALS. Calculations (dotted lines) used optical constants from reference [18], and interface widths  $\sigma=1.35\text{ nm}$ .

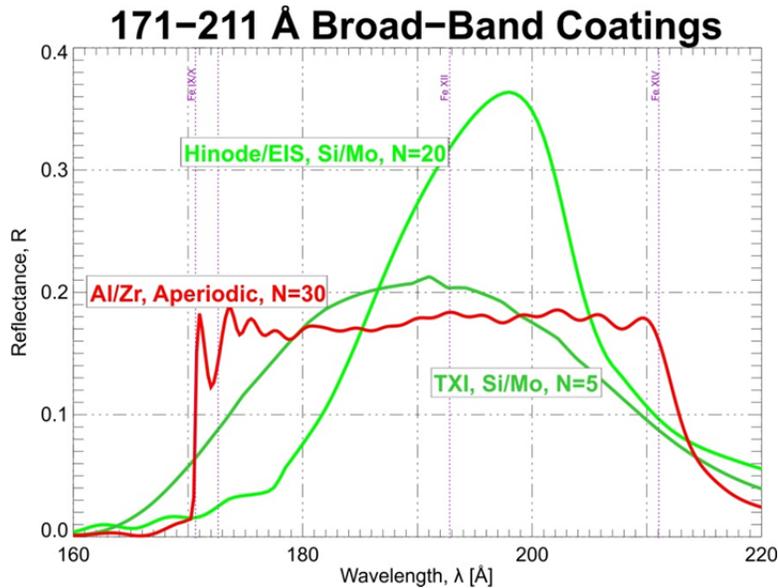


Figure 8. Calculated reflectance of an aperiodic Al/Zr multilayer designed for flat response from ~17 to 21 nm. Also shown are calculated reflectance curves for the periodic Si/Mo coatings used on the Hinode/EIS and TXI instruments.

Periodic Al/Zr multilayers have been found to have very low film stress and good temporal stability: a prototype film shows almost no change in reflectance after a period of several years. The thermal stability of Al/Zr remains to be fully investigated.

## Al-Mg/SiC Multilayers

SiC/Mg multilayers have already been shown to provide very high reflectance EUV wavelengths longer than the Mg L-edge near 25 nm.<sup>19,20</sup> However, the SiC/Mg system is also prone to catastrophic degradation, ostensibly due to corrosion of Mg. In recent years, Soufli et al have developed a new Mg-based multilayer that is apparently far more stable than SiC/Mg.<sup>21</sup> The new multilayer system contains Al layers deposited adjacent to the top and bottom Mg layers; over a short period of time (~weeks), the top and bottom Al-Mg layers react to form a stable Al-Mg alloy that is apparently far less susceptible to Mg corrosion. The addition of the top Al layer in Al-Mg/SiC multilayers degrades the peak reflectance of this system relative to the SiC/Mg system. As shown in Fig. 9, however, periodic Al-Mg/SiC multilayers nevertheless provide significantly higher reflectance than other available multilayer systems such as Si/Mo and SiC/Si in the 25–35 nm range, a spectral region that is rich with bright solar emission lines.

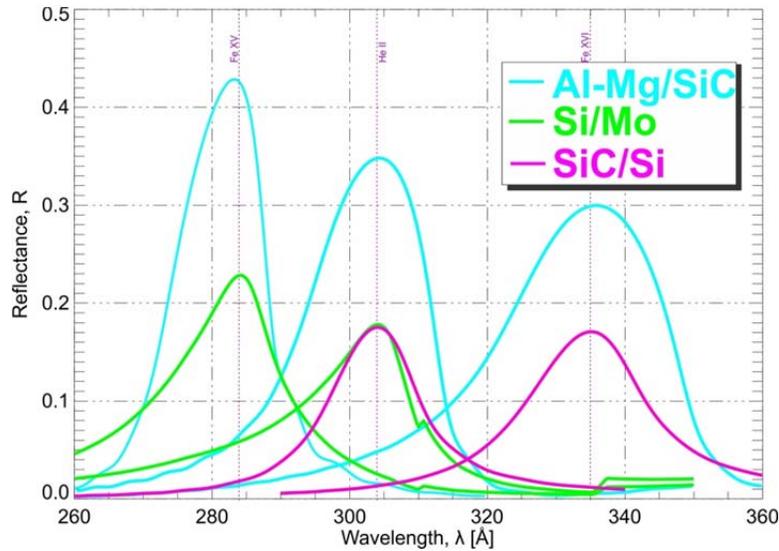


Figure 9. Calculated reflectance curves for periodic Al-Mg/SiC, Si/Mo, and SiC/Si coatings in the 25–35 nm range.

It should also be possible to fabricate aperiodic Al-Mg/SiC multilayers having broad-spectral response in this wavelength region. For example, shown in Fig. 10 are two Al-Mg/SiC AML reflectance curves, one designed for flat response from ~25 nm to ~34 nm, the other for flat response from ~25 to ~31 nm. Also shown in Fig. 10 is the response of the periodic Si/Mo coating containing N=20 repetitions that was used for the long-wavelength channel of the Hinode/EIS instrument. Again, while the periodic structure provides higher peak reflectance, the aperiodic coating designs are expected to provide flat response over a significantly wider spectral band-pass. We will fabricate and test aperiodic Al-Mg/SiC designs (along with aperiodic Al/Zr designs, as mentioned above) in the near future, and investigate in further detail their temporal and thermal stability. We also plan to investigate the use of various Mg alloys, such as Mg<sub>2</sub>Si, in place of pure Mg,<sup>22</sup> with the hope of developing a stable coating that has even better performance than Al-Mg/SiC.

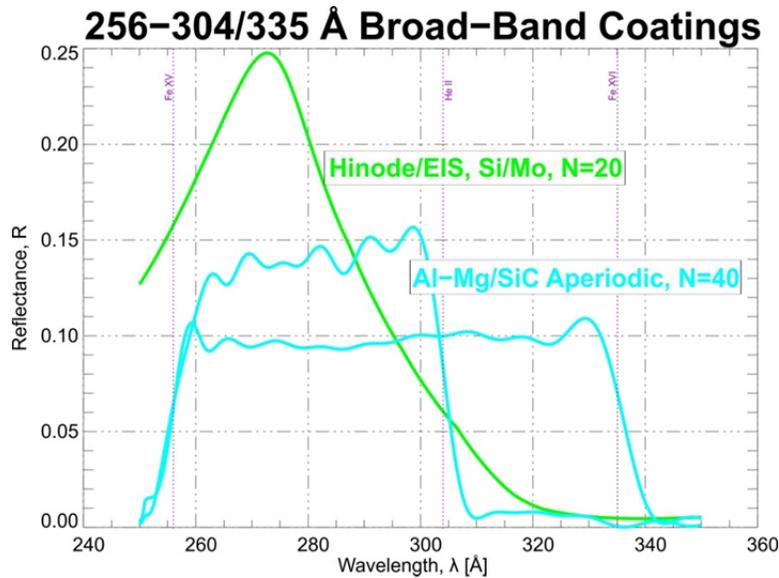


Figure 10. Calculated reflectance of a two aperiodic Al-Mg/SiC coatings designed for flat response from  $\lambda=25$  to 31 nm, and from  $\lambda=25$  to 34 nm. Also shown is the calculated reflectance of the periodic Si/Mo multilayer used for the long-wavelength band on the Hinode/EIS instrument.

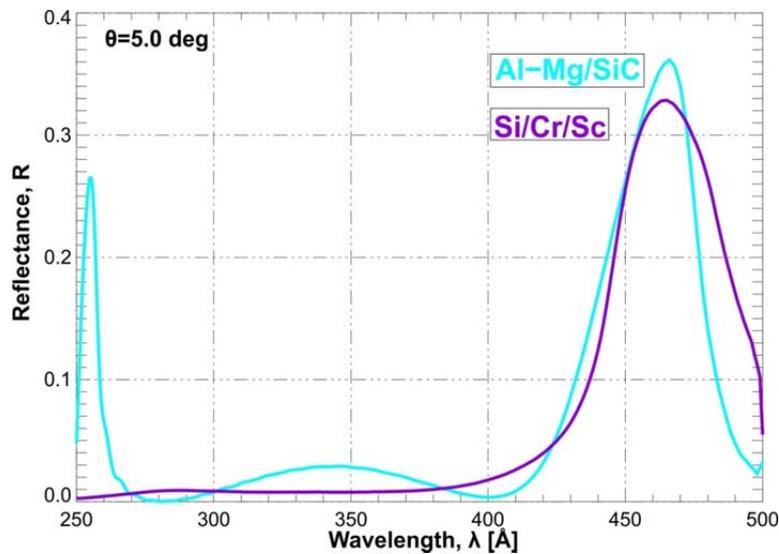


Figure 11. Measured normal incidence reflectance of periodic Al-Mg/SiC and Si/Cr/Sc multilayers tuned near 46.5 nm. The high reflectance in 2<sup>nd</sup> order of the Al-Mg/SiC coating precluded its use in the MOSES instrument.

The performance of periodic Al-Mg/SiC coatings were also investigated for use in the MOSES spectroscopic imaging sounding rocket instrument<sup>23</sup> targeting much longer EUV wavelengths, specifically in the vicinity of the Ne VII line at 46.5 nm. Shown in Fig. 11 is a plot of the measured normal-incidence reflectance, from 25 to 50 nm wavelength, of a periodic Al-Mg/SiC coating designed for maximum reflectance at 46.5 nm. Also shown in Fig. 11 is the reflectance of a periodic multilayer comprising Si and Sc layers with thin (0.6 nm) Cr barrier layers deposited at each interface, a multilayer system developed previously.<sup>24</sup> While the Al-Mg/SiC multilayer provides higher peak reflectance (36%) at the target wavelength, it also gives relatively high reflectance (~26%) in 2<sup>nd</sup> order near 25 nm; the shorter wavelength light is problematic for the MOSES instrument. As the Si/Cr/Sc coatings provide reflectance in first order that is also relatively high (33%), with 2<sup>nd</sup> order reflectance below 1%, those coatings were chosen for use in the MOSES instrument.



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