Corrosion-resistant, triple-wavelength Mg/SiC multilayer coatings for the 25-80 nm wavelength region

Regina Soufli
Lawrence Livermore National Laboratory
Contributors

- **Lawrence Livermore National Lab:** Mónica Fernández-Perea, Jeff C. Robinson, Sherry L. Baker, Jennifer Alameda, Chris Walton

- **Instituto de Óptica, Consejo Superior de Investigaciones Científicas:** Luis Rodríguez-De Marcos, Jose A. Méndez, Juan I. Larruquert

- **Lawrence Berkeley National Lab:** Eric M. Gullikson
Overview

- Motivation: applications that could benefit from Mg/SiC multilayers with high performance and long lifetime
- Design and experimental performance of triple-wavelength Mg/SiC multilayers
- Origins, propagation mechanisms and impact of Mg/SiC corrosion
- Corrosion barriers for Mg/SiC multilayers
- Design and experimental performance of triple-wavelength Mg/SiC multilayers with corrosion barriers
- Summary and future work
Compact plasma-based EUV/soft x-ray laser applications need multilayer coatings as collector and imaging elements.

Laser Pumped SXRL $\lambda = 8.8 \text{–} 32.6$ nm

Discharge Pumped SXRL $\lambda = 46.9$ nm

Courtesy: Jorge Rocca and Carmen Menoni, Colorado State University

- High pulse energy ($\mu$J-mJ)
- High monochromaticity ($\lambda/\Delta \lambda < 10^{-4}$)
- High peak spectral brightness

Interferometry

Chemical spectroscopies

Nanomachining

Plasma diagnostics

Microscopy

Nanoablation
Multilayers with high performance and long lifetime are needed for EUV solar physics and space weather missions.


Multilayer-coated test mirrors for NASA/NOAA’s GOES-R space weather satellite. 6 EUV wavelengths, 9.4 nm to 30.4 nm (Mo/Y, Mo/Si). Launch date: 2014

7 EUV wavelengths, 9.4 nm to 33.5 nm. Mo/Y, Mo/Si, SiC/Si.


Multilayer coatings are needed for future solar missions designed to operate at 46.5 nm

HiLiTE: a 300-mm aperture Cassegrain telescope, aiming to study the Sun’s transition region at the 46.5 nm Ne VII emission line. Designed to be made entirely of SiC, including optical substrates and metering structure.

LLNL facilities used for multilayer deposition and characterization of Mg/SiC multilayers discussed in this presentation

- DC- magnetron sputtering multilayer deposition system
- Precision surface metrology
  - Zygo
  - AFM
  - SEM
- Custom cleaning facility for optical substrates
- X-Ray Diffractometer

Also (not pictured):
- Contact profilometers
- Thin film stress measurement apparatus
Our group at LLNL has participated in the development of optics for a wide range of applications for EUV, soft and hard x-ray wavelengths.

**EUV Lithography**


**EUV solar missions (NASA's SDO and NASA/NOAA's GOES-R)**


**X-ray optics for the LCLS free-electron laser**

- A. Barty, R. Soufli et al., Optics Express 17, 15508-15519 (2009)

**Hard x-ray space missions (NASA's NuSTAR)**


Regina Soufli
regina.soufli@llnl.gov
Mg exhibits consistently low absorption in an extended wavelength range, longer than the Mg L$_{2,3}$ edge (25 nm)

**Refractive index = $n + i*k$**

Extinction coefficient $k$ values obtained from:

We have achieved the highest reported narrowband peak reflectance 40.6% at 76.9 nm and near-normal incidence

Standard Mg/SiC (no corrosion barriers)

Mesurements performed at:
• Beamline 6.3.2., Advanced Light Source, LBNL, for < 50 nm wavelengths
• GOLD facility, Instituto de Óptica, Madrid, Spain, for 50-200 nm wavelengths


Mg/SiC is the best performing multilayer in the 25-80 nm wavelength region, except ....

Mg/SiC exhibits a unique combination of high reflectivity, near-zero stress, thermal stability to ~350° C and good spectral selectivity compared to other candidate multilayer pairs in the 25-80 nm region.
...atmospheric corrosion prevents Mg/SiC from being used in applications requiring long lifetime stability


Advanced corrosion exhibits eruptive effects due to formation and volume expansion of corrosion products

Mg/SiC with advanced, eruptive stages of corrosion aged for 3 years. 4 top bilayers (out of 20) have been consumed and are missing.

TEM and XPS analysis performed at EAG Labs, Sunnyvale, California

<table>
<thead>
<tr>
<th>Binding energy (eV)</th>
<th>Orbital</th>
<th>Species</th>
</tr>
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<tbody>
<tr>
<td>50.8</td>
<td>Mg 2p</td>
<td>MgO, Mg(OH)$_2$, Mg(CO)$_3$</td>
</tr>
<tr>
<td>532.8</td>
<td>O 1s</td>
<td>OH$^-$, Mg(OH)$_2$</td>
</tr>
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</table>
We have elucidated the origins and propagation mechanisms of corrosion in Mg/SiC multilayers.

Mg/SiC aged for 3 years, with early, pre-eruptive stages of corrosion.

Atmospheric corrosion attacks Mg/SiC from the top surface via localized entry points such as pinholes and defects, inherent in sputtered thin films.
We have demonstrated Al-Mg corrosion barrier structures for Mg/SiC multilayers

Polycrystalline Al (20 nm) and Mg (19 nm) layers spontaneously intermix to produce partially amorphous Al-Mg layer. Amorphization process takes several weeks to complete.

(a) Sample aged for 2.5 years

Polycrystalline Al (20 nm) and Mg (19 nm) layers spontaneously intermix to produce partially amorphous Al-Mg layer. Amorphization process takes several weeks to complete.

(b)

(c) Sparse Al, Mg crystallites

(d) Mg crystallites

(e) Days after deposition | Bragg peak, 2θ (deg) | Lattice spacing (nm) | Crystallite size (nm)
--- | --- | --- | ---
Al(111) | 2 | 38.44 | 0.23 | 15.13
" | 68 | 38.41 | 0.23 | 4.57
Mg(002) | 2 | 34.41 | 0.26 | 16.7
" | 68 | 35.14 | 0.25 | 9.15

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Regina Soufli
regina.soufli@llnl.gov
Triple-wavelength Mg/SiC multilayers with corrosion barriers can be employed in EUV imaging instruments or spectrometers.

Shown below is a periodic (constant d) multilayer. Aperiodic or “stacked” multilayer designs can also be implemented to achieve customized reflective performance. Corrosion barriers can be inserted in specific Mg layers or in each Mg layer.

Refractive index values obtained from:

Summary and future work

- Corrosion has prevented the use of Mg/SiC multilayers in applications requiring good lifetime stability.
- We have developed triple-wavelength Mg/SiC multilayers with Al-based barrier layers that dramatically reduce corrosion, while preserving high reflectance and low stress.
- We are currently studying in detail the physics of spontaneous intermixing and amorphization of Al and Mg layers.
- Corrosion barrier layers should be tested and optimized individually for each multilayer design and environmental conditions.
- The aforementioned advances may enable the implementation of corrosion-resistant, high-performance Mg/SiC coatings in applications such as EUV/soft x-ray laser sources and solar physics.
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