Corrosion-resistant Mg-based multilayer coatings for sources > 25 nm

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EUVL Workshop, LBNL, June 13-14, 2018

LLNL-PROC-754006

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Technical contributors

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- Luis Rodríguez-De Marcos, Jose A. Méndez, Manuela Vidal-Dasilva, Juan I. Larruquert (*Instituto de Óptica, Consejo Superior de Investigaciones Científicas (CSIC), Madrid, Spain*)

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Overview

- Motivation: applications at λ = 25 – 80 nm that benefit from Mg/SiC multilayers
- Design and experimental performance of Mg/SiC multilayers
- Origins, propagation mechanisms and impact of Mg/SiC corrosion
- Al-Mg corrosion barriers for Mg/SiC
  - Al-Mg spontaneous intermixing and amorphization
  - Lifetime properties and performance optimization of Mg/SiC with Al-Mg corrosion barriers

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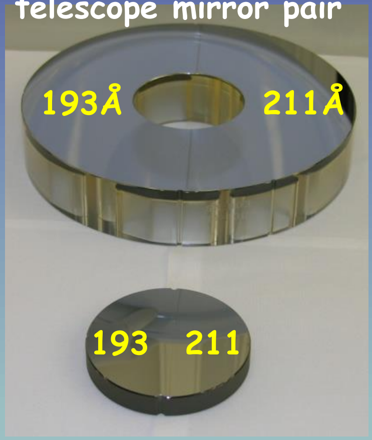
Multilayer coatings with stability > 10 years are needed for space-borne EUV solar physics telescopes

NASA's Solar Dynamics Observatory (SDO). Launched: February 11, 2010.

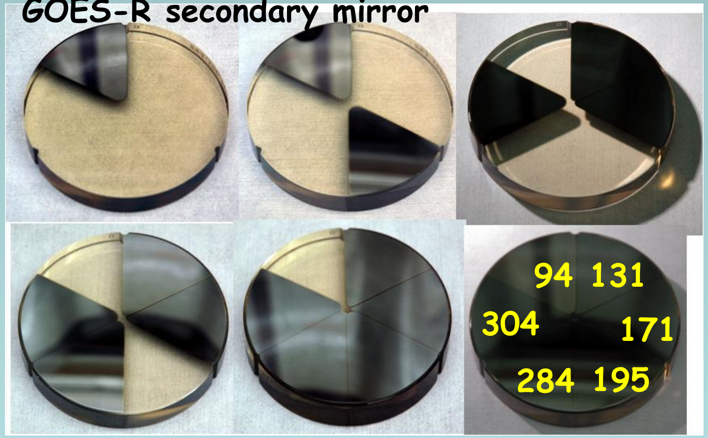
Multilayer-coated SDO telescope mirror pair



4 telescopes
2 wavelengths per telescope
7 EUV wavelengths, 94 - 335 Å.
1 arcsec resolution
41x41 arcmin FOV
12 sec cadence
2 Tbytes / day

NASA/NOAA's GOES-R,-S,-T,-U space weather satellites. Launch: 2016 - 2020

GOES-R secondary mirror



6 EUV wavelengths (94 - 304 Å) on a single telescope.

Soufli, Proc. SPIE 59010M (2005)
Soufli, Appl. Opt. 46, 3156-3163 (2007)
Boerner, Solar Physics 275, 41-66 (2012)
Lemen, Solar Physics 275, 17-40 (2012)
Soufli, Proc. SPIE 8443R (2012)
Martinez-Galarce, Opt. Eng. 52, 095102 (2013).

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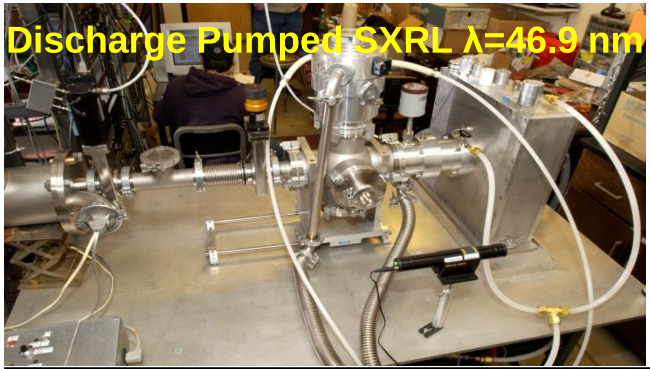
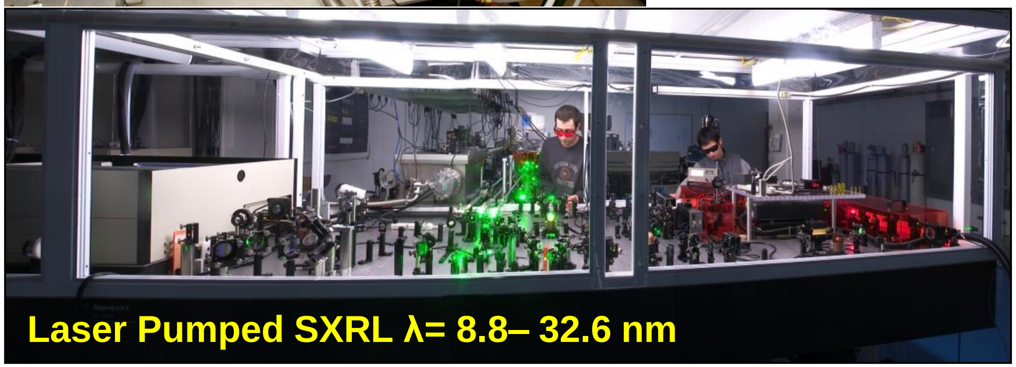
Reflective X-ray Optics

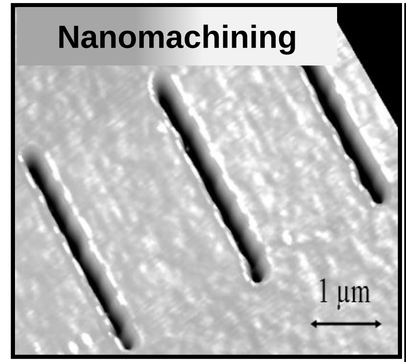
BERKELEY LAB

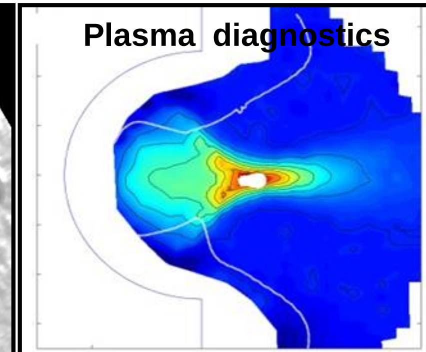
Compact plasma-based EUV laser sources and applications need efficient multilayer coatings

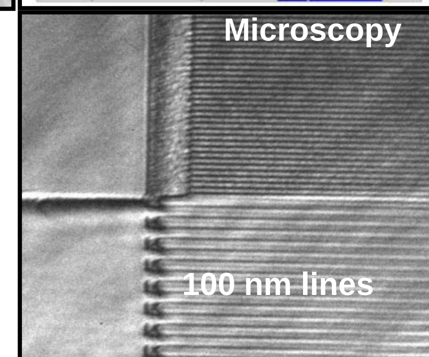
Courtesy: Profs. Jorge Rocca and Carmen Menoni, Colorado State University



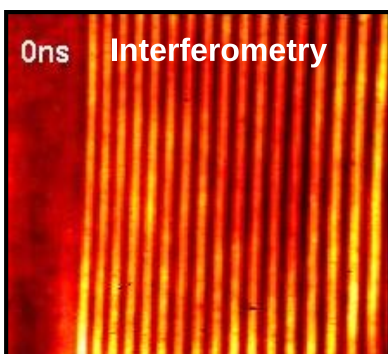


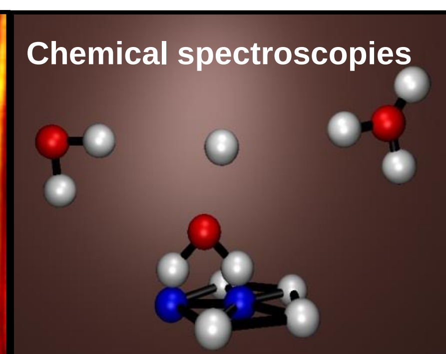


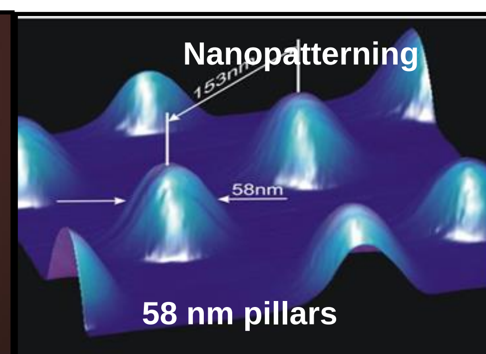


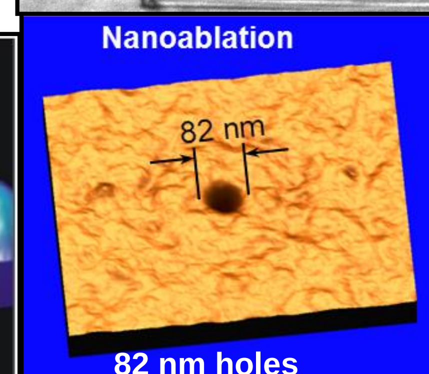


- High pulse energy (μJ-mJ)
- High monochromaticity ($\lambda/\Delta\lambda < 10^{-4}$)
- High peak spectral brightness



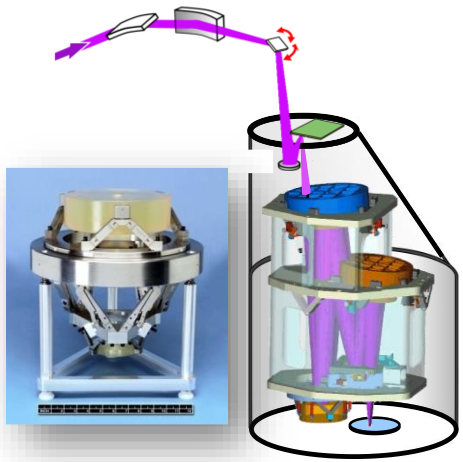




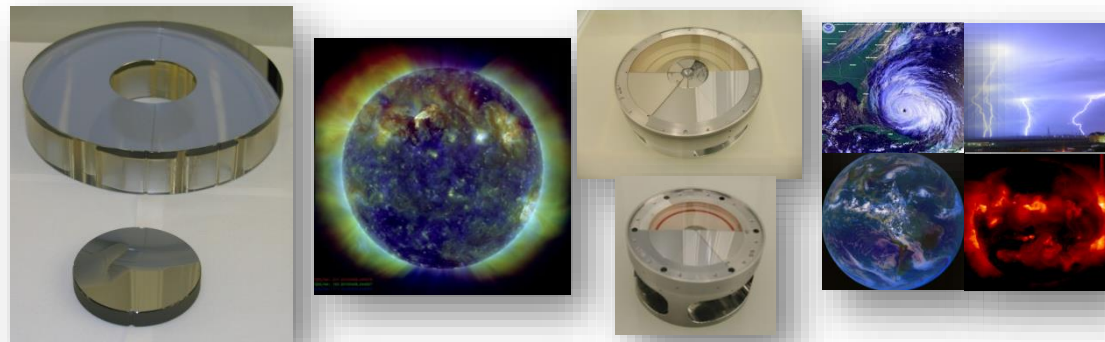


Our group at LLNL has participated in the development of short-wavelength optics for various applications

EUV Lithography:
ETS1, ETS2, MET2, MET5



EUV solar missions and laser sources



X-ray optics for the LCLS free-electron laser

Hard x-ray /gamma-ray astrophysics, radiation detection, target diagnostics

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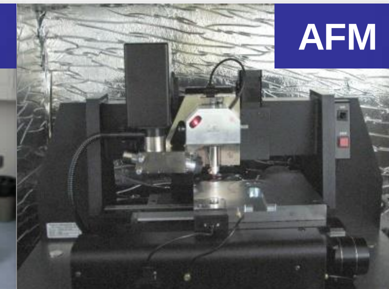
LLNL facilities for thin film deposition and characterization

DC- and RF-magnetron sputtering deposition systems

Precision surface metrology



Also (not pictured):
- Contact profilometers
- Thin film stress measurement apparatus
- Full-aperture interferometers

Custom cleaning facility for optical substrates

X-Ray Diffractometer

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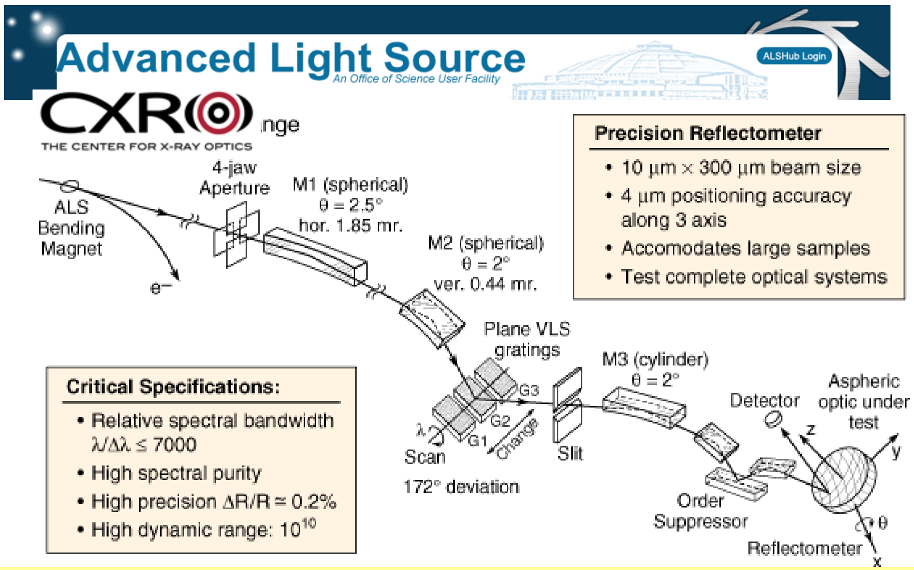
Light sources employed for at-wavelength testing of multilayer optics in this presentation



Calibration and standards ALS beamline 6.3.2
λ = 1-90 nm

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GOLD facility, λ = 50-200 nm

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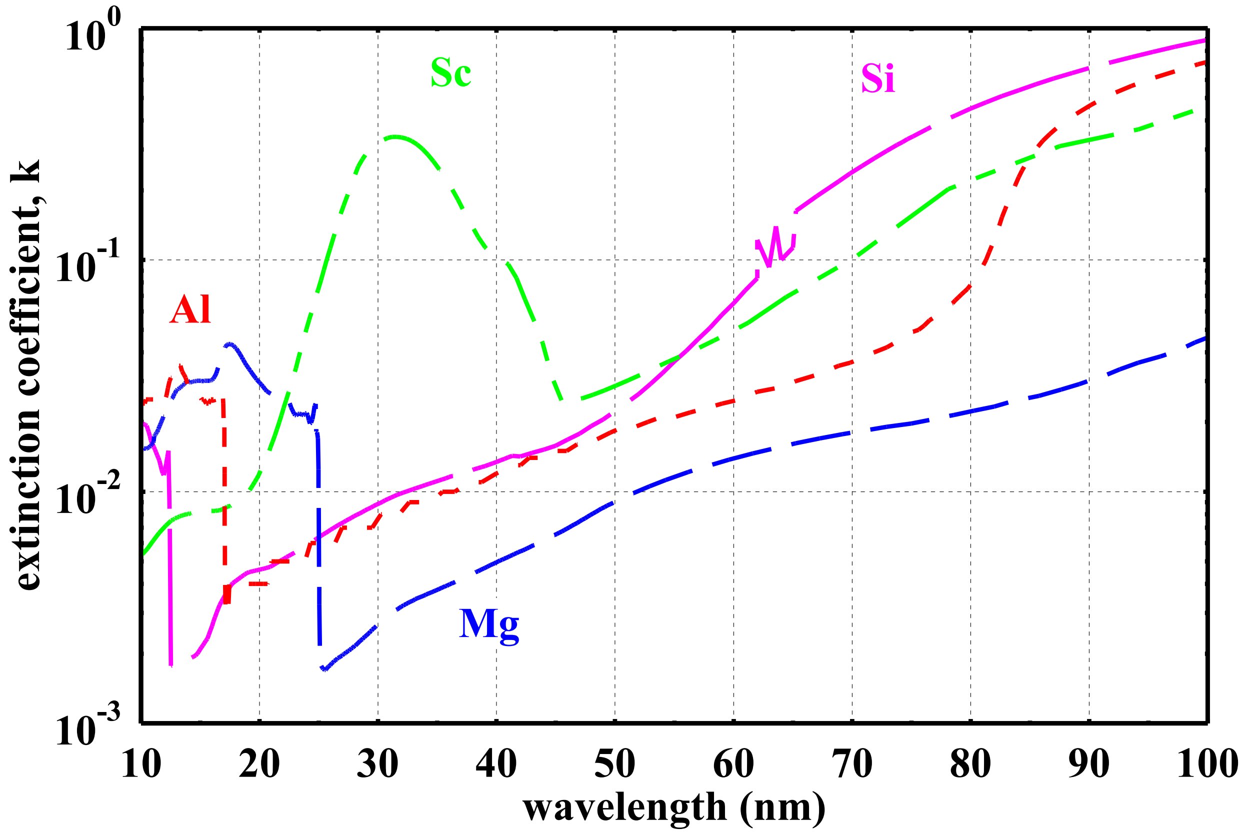
Mg exhibits low absorption in an extended wavelength range for λ > 25 nm (Mg $L_{2,3}$ edge)



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

Extinction coefficient *k* values obtained from:

1. <u>Mg</u>: M. Vidal-Dasilva, *et al*, J. Appl. Phys. 108, 063517 (2010).

2. <u>Al</u>: E. Shiles, *et al,* Phys. Rev. B 22, 1612-1628 (1980), as compiled by E. D. Palik, *Handbook of optical constants of solids* (Academic Press, 1985).

3. <u>Si</u>: R. Soufli and E. M. Gullikson, Appl. Opt. 36, 5499-5507 (1997).
H. R. Philipp, J. Appl. Phys. 43, 2835-2839 (1972), compiled by E. D. Palik, *Handbook of optical constants of solids* (Academic Press, 1985).

4. <u>Sc</u>: M. Fernández-Perea, *et al,* J. Opt. Soc. Am. A 23, 2880-2887 (2006).

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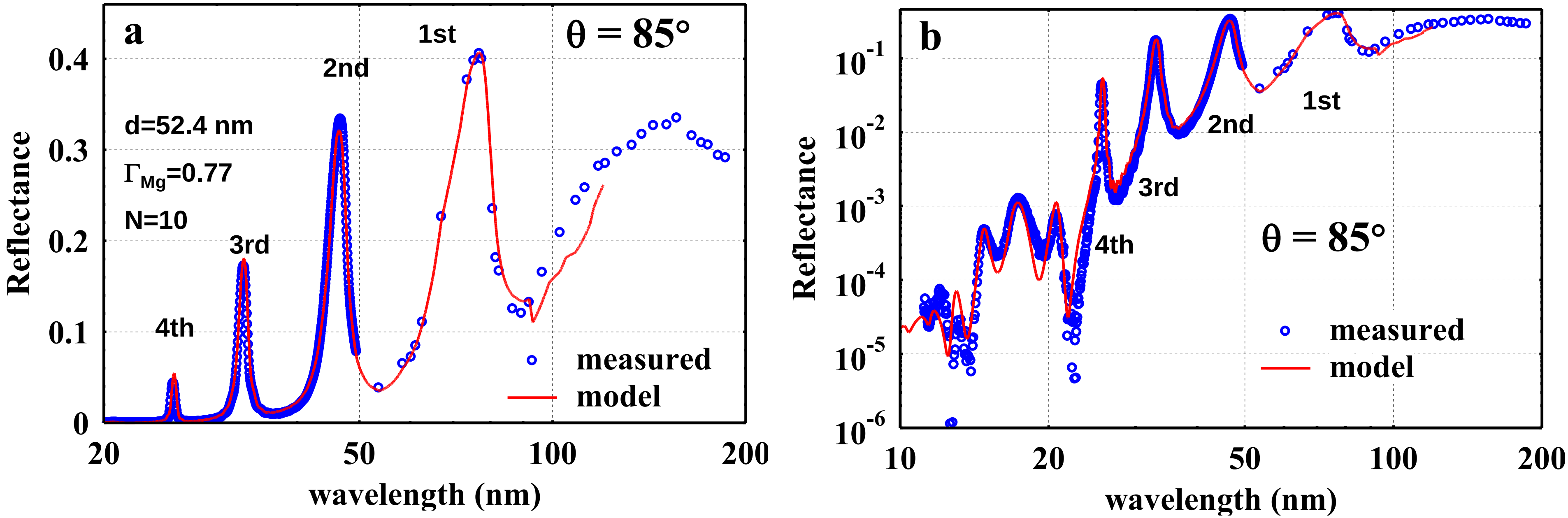
Mg/SiC achieves the highest narrowband peak reflectance at λ = 76.9 nm: R = 40.6% at near-normal incidence

Standard Mg/SiC (no corrosion barriers)



- ❑ Mesurements performed at:
- • Beamline 6.3.2., Advanced Light Source, LBNL, for λ< 50 nm
- • GOLD facility, Instituto de Óptica, Madrid, Spain, for λ = 50-200 nm
- ❑ Model = IMD software by D. L. Windt, Computers in Physics 12, 360–370 (1998)

M. Fernández-Perea, R. Soufli, J. C. Robinson, L. Rodríguez-de Marcos, J. A. Mendez, J. I. Larruquert and E. M. Gullikson, "Triple-wavelength, narrowband Mg/SiC multilayers with corrosion barriers and high peak reflectance in the 25-80 nm wavelength region", Optics Express **20,** 24018-24029 (2012).

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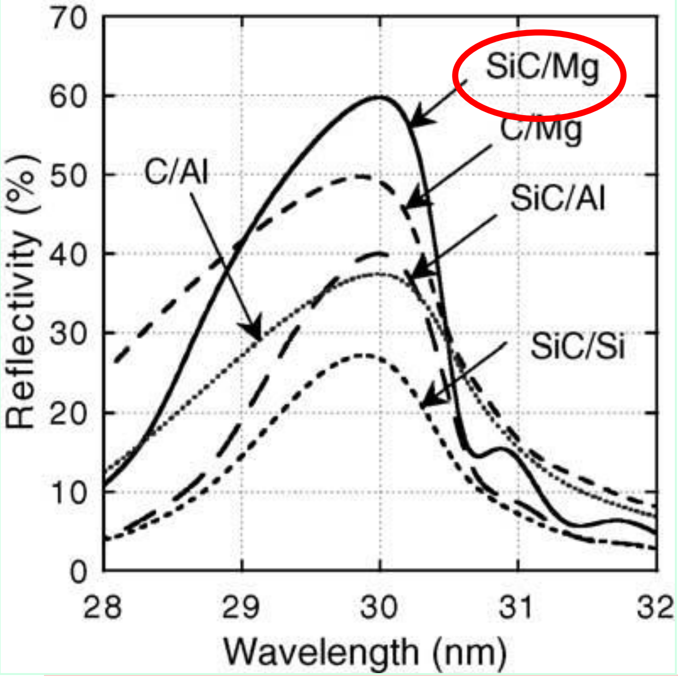
Mg/SiC is the best multilayer for the 25-80 nm wavelength region, except ….

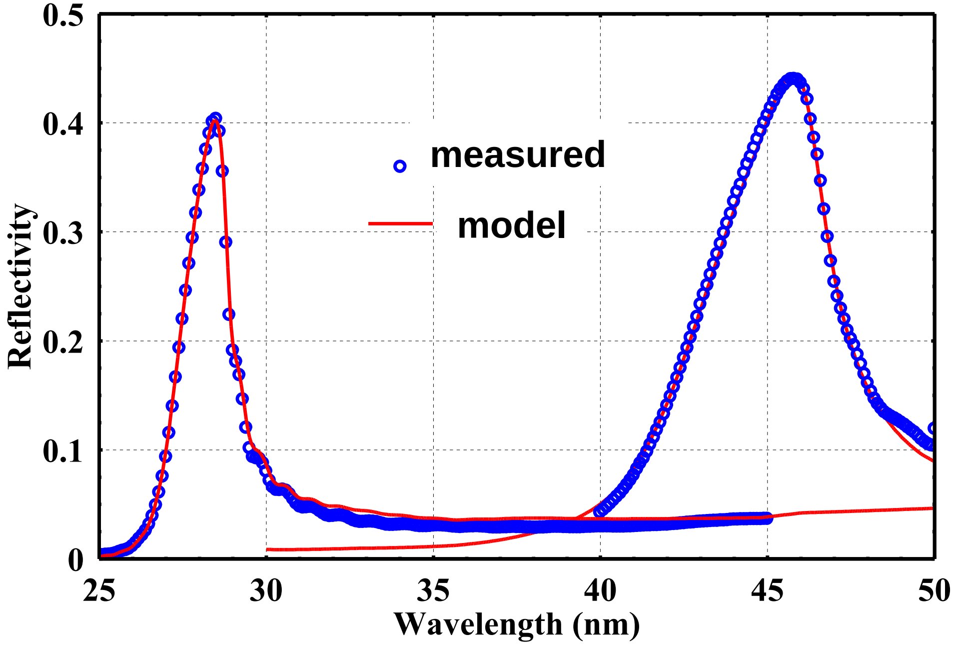
Mg/SiC exhibits a unique combination of high reflectivity, near-zero stress, thermal stability to ~350° C and good spectral selectivity compared to other candidate multilayer pairs in the 25-80 nm region

Calculated ideal reflectivity (roughness=0) for different multilayers



H. Takenaka *et al.*, J.of El. Spectr. and Rel. Phen. **144-147**, 1047 (2005).

Mg/SiC deposited at LLNL, measured at ALS beamline 6.3.2



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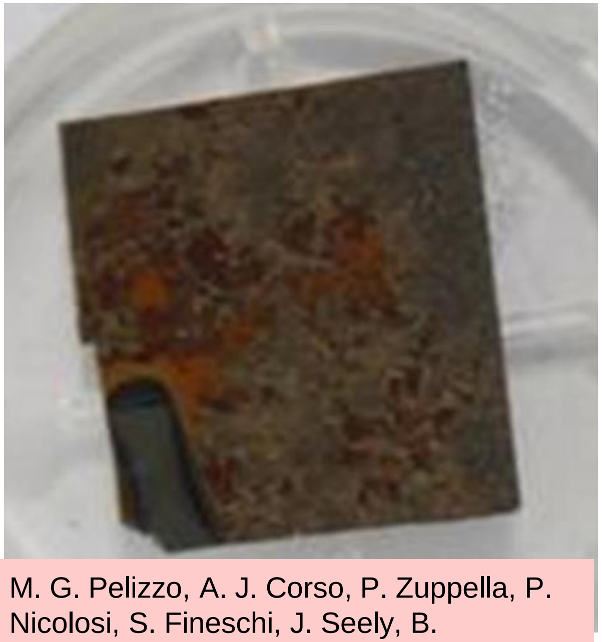
…atmospheric corrosion prevents Mg/SiC from being used in applications requiring long lifetime stability

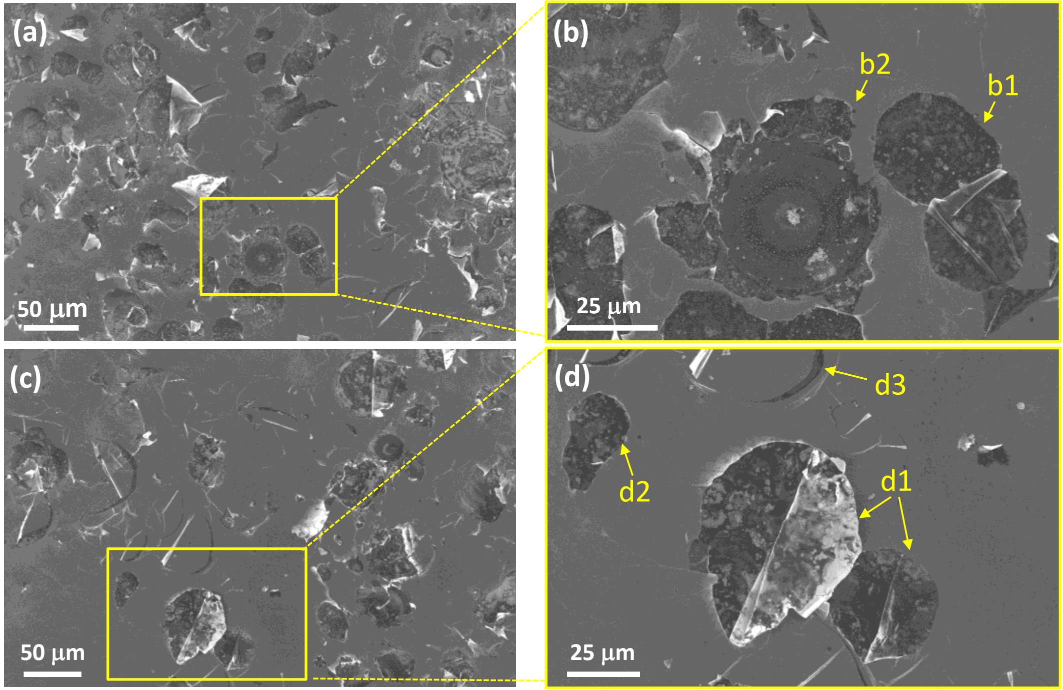
Corrosion has prevented Mg/SiC use in EUV solar telescopes, free-electron lasers, and HHG sources

Mg/SiC with advanced corrosion aged for 4 years



M. G. Pelizzo, A. J. Corso, P. Zuppella, P. Nicolosi, S. Fineschi, J. Seely, B. Kjornrattanawanich, and D. L. Windt, *Opt. Eng.* **51**, 023801 (2012).

Top-surface SEM of Mg/SiC with advanced corrosion aged for 2.7 years



R. Soufli, M. Fernández-Perea, S. L. Baker, J. C. Robinson, J. Alameda, C. C. Walton, App. Phys. Lett. **101**, 043111 (2012).

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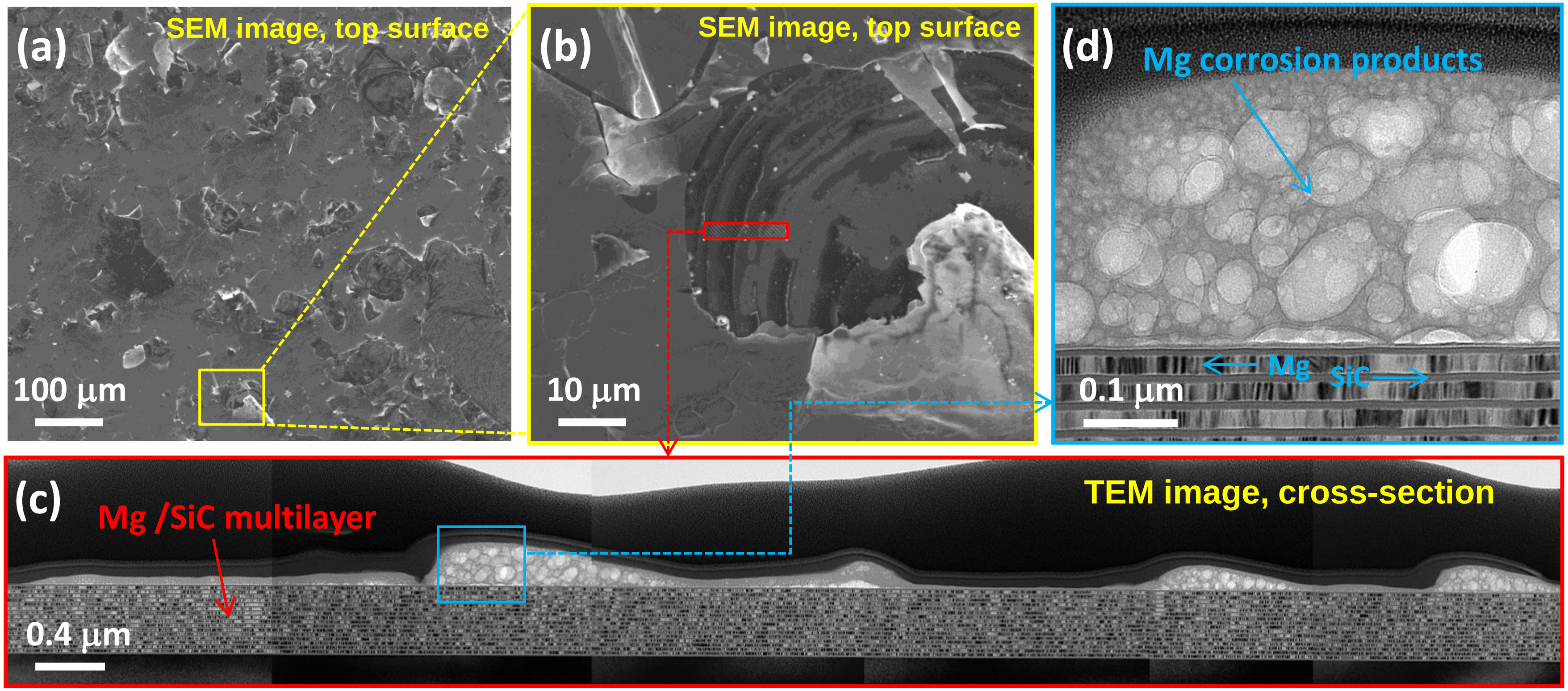
Advanced corrosion exhibits eruptive effects due to formation and volume expansion of corrosion products

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



Binding energy (eV)	Orbital	Species
50.8	Mg 2p	MgO, $Mg(OH)_2$, $Mg(CO)_3$
532.8	O 1s	OH^-, $Mg(OH)_2$

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

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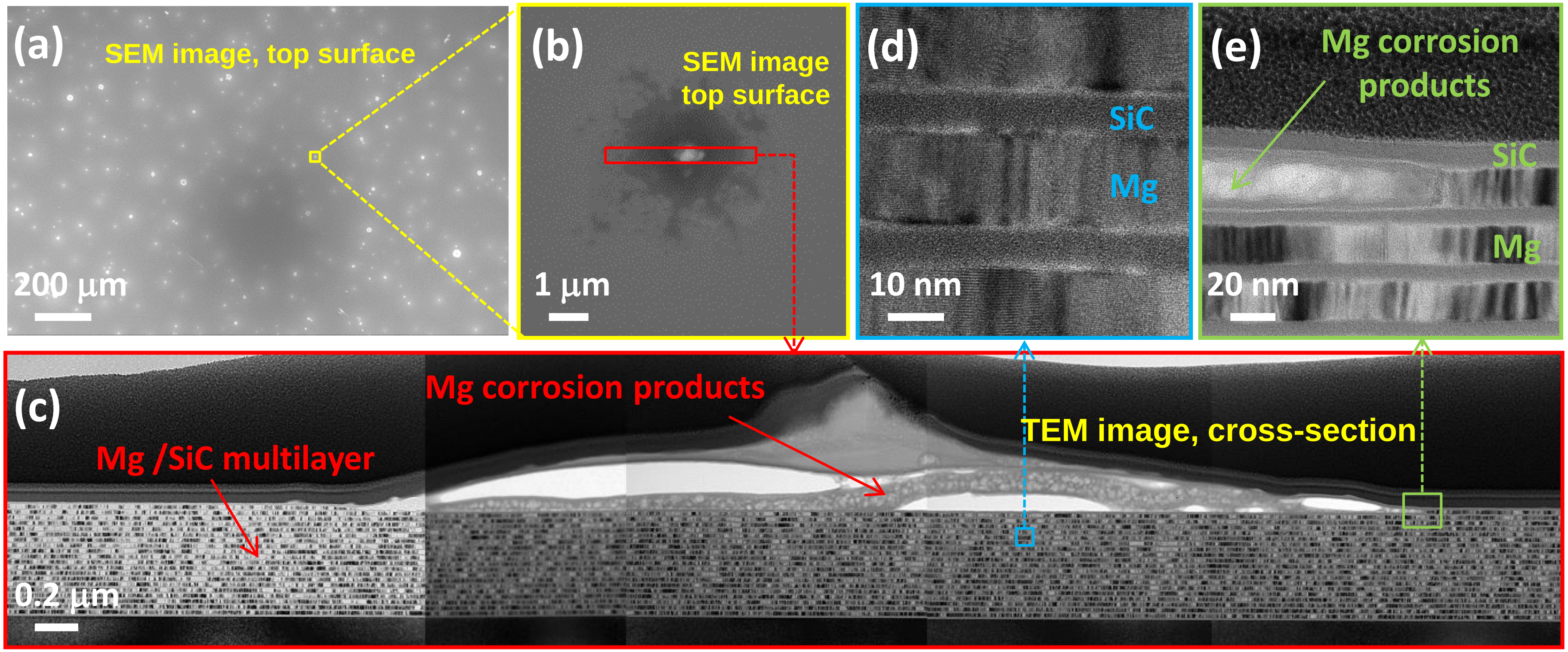
We have elucidated the origins and propagation mechanisms of corrosion in Mg/SiC multilayers

Atmospheric corrosion attacks Mg/SiC from the top surface via localized entry points such as pinholes and defects, inherent in sputtered thin films



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

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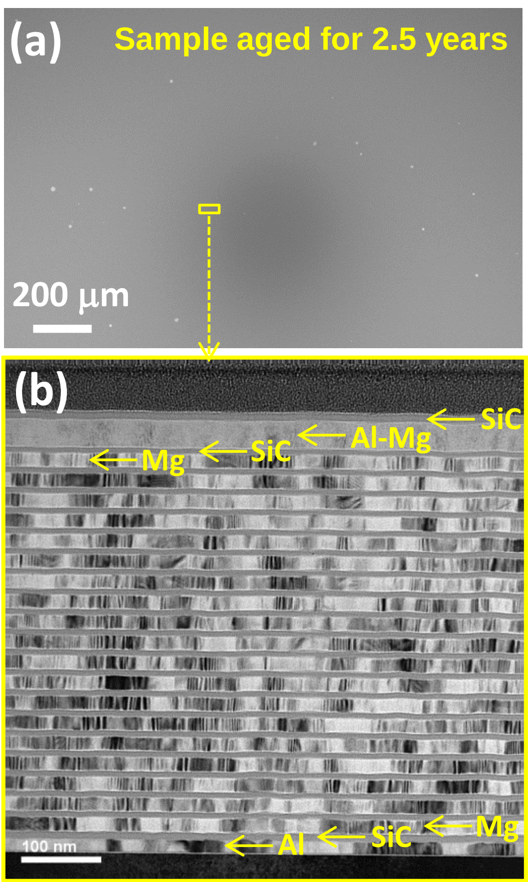
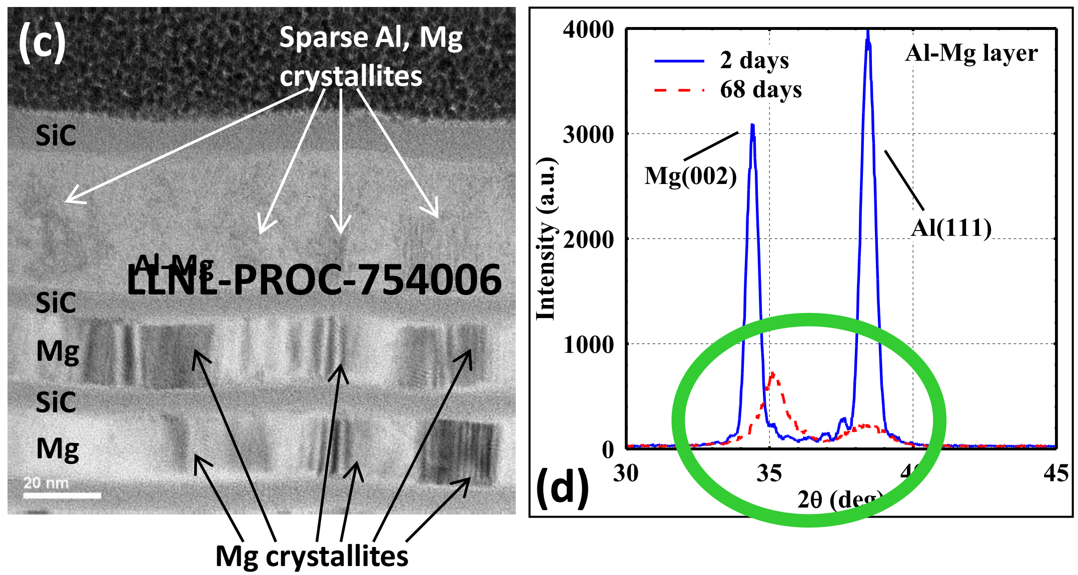
Al-Mg corrosion barrier structures for Mg/SiC multilayers

Polycrystalline Al (20 nm) and Mg (19 nm) layers spontaneously intermix to produce partially amorphous Al-Mg layer.

R. Soufli, M. Fernández-Perea, S. L. Baker, J. C. Robinson, J. Alameda, C. C. Walton, App. Phys. Lett. **101**, 043111 (2012).





(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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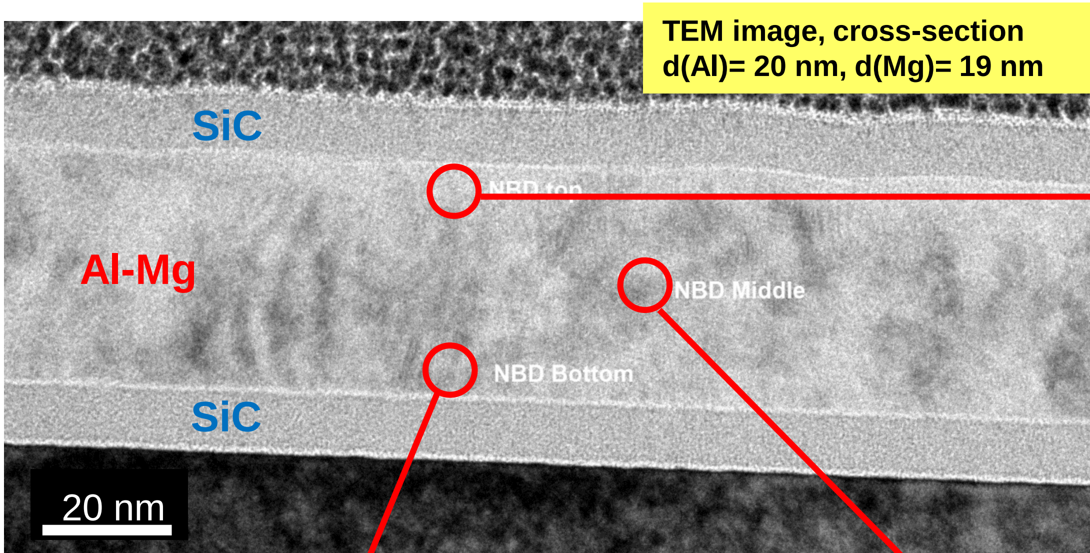
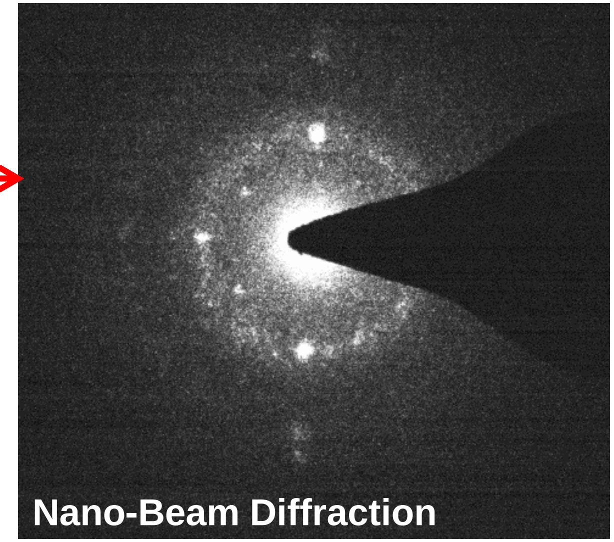
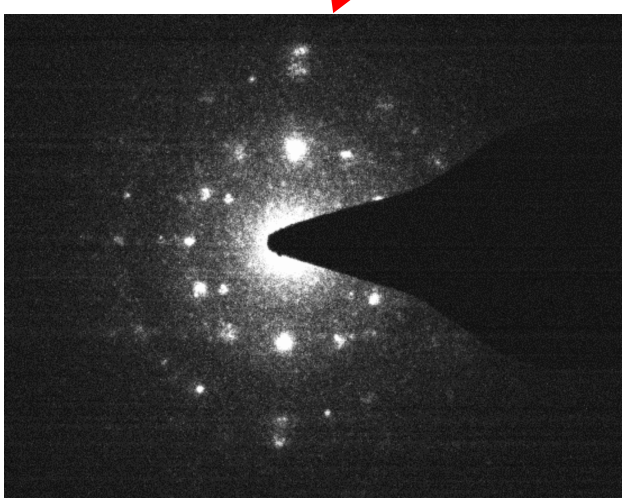
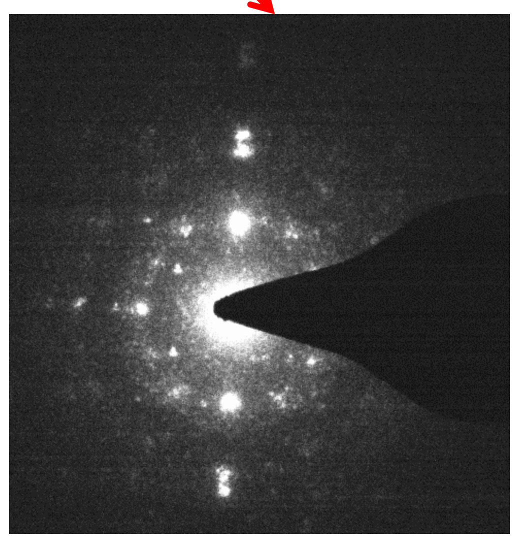
We are investigating in detail the physics of the Al-Mg intermixing and amorphization process









- Intermixed region of Mg layer (bottom) exhibits higher degree of “crystallinity” than Al layer (top)

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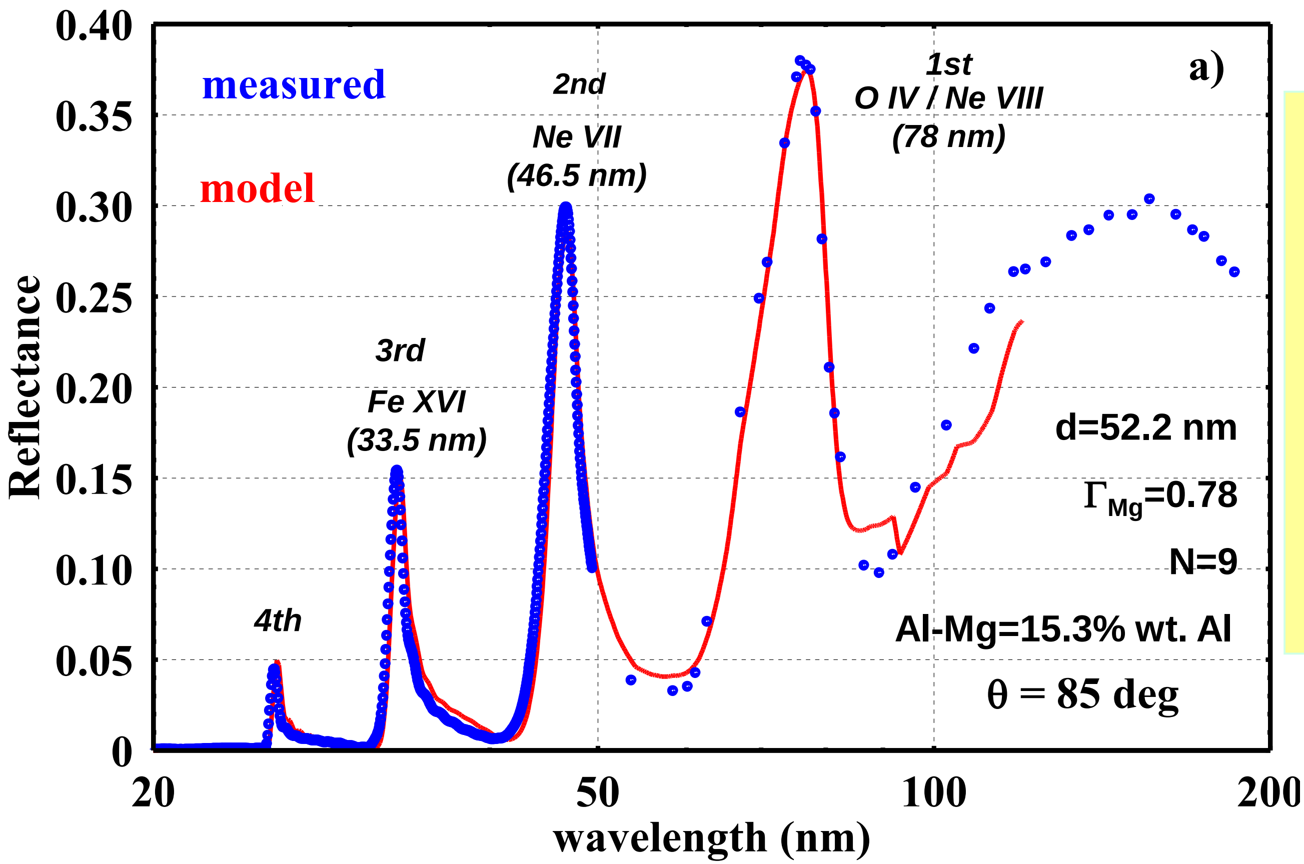
Triple-wavelength Mg/SiC multilayers with corrosion barriers can be employed in EUV imaging or spectrometer instruments



Refractive index values obtained from:

1. Mg: M. Vidal-Dasilva, *et al*, J. Appl. Phys. 108, 063517 (2010).

2. SiC: J. B. Kortright and D. L. Windt, Appl. Opt. 27, 2841-2846 (1988).

3. Al: E. Shiles, *et al,* Phys. Rev. B 22, 1612-1628 (1980), as compiled by E. D. Palik, *Handbook of optical constants of solids* (Academic Press, 1985).

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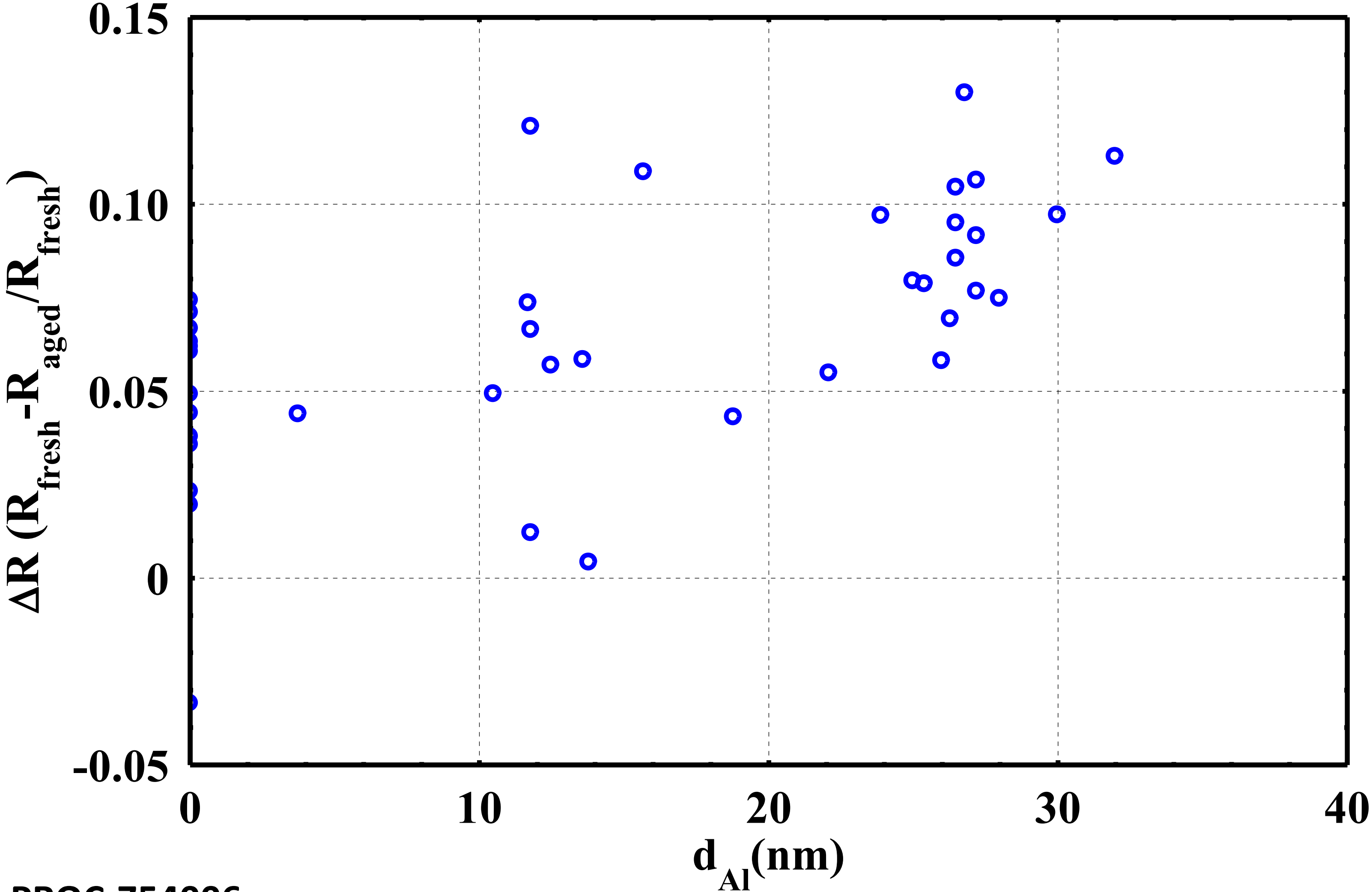
Reflectance loss after 3 years of aging appears to increase with Al thickness

Mg/SiC samples reflecting at 28 and 46 nm, with and without Al-Mg corrosion barrier layers



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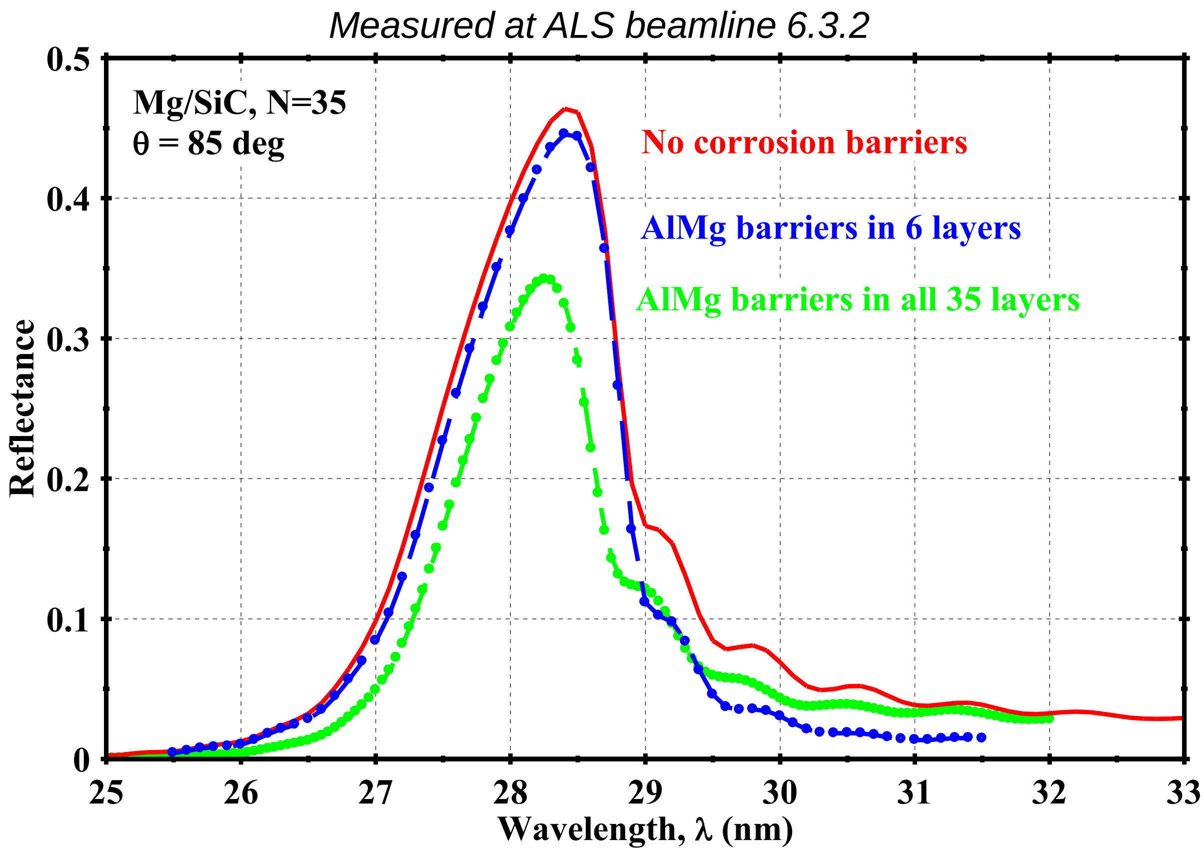
Performance optimization of Mg/SiC coatings with and without AlMg corrosion barriers



- Insertion of 6 barrier layers in a Mg/SiC stack of 35 layers has minimal impact in reflectance
- Significant reflectance can be achieved even with corrosion barriers in all 35 layers
- In addition to corrosion protection, AlMg barriers improve out-of-band suppression

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Summary

- Atmospheric corrosion has prevented the use of Mg/SiC multilayers in applications requiring good lifetime stability, such as EUV laser sources and solar physics

- We have developed Al-based barrier layers that dramatically reduce corrosion in Mg/SiC multilayers, while preserving high reflectance

- Corrosion barrier layers can be customized specifically for each multilayer design and environmental conditions

- Mg/SiC with Al-based corrosion barriers has been implemented in upcoming EUV solar physics missions

- Investigation of the physics of spontaneous intermixing and amorphization of sputtered Al and Mg layers is ongoing

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- GOLD acknowledges financial support from the National Program for Space Research, Subdirección General de Proyectos de Investigación, Ministerio de Ciencia y Tecnología, project number AYA2010-22032
- Additional funding was provided by Lockheed Martin Corporation Internal Research and Development

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