Development of efficient and stable Al-based multilayer reflective coatings for EUV range

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Laboratoire Charles Fabry, Institut d'Optique Graduate School, Palaiseau, France

Since 1917
First director - Charles Fabry
Campus of Ecole Polytechnique (Palaiseau, France)

Laboratory Charles Fabry

- Biophotonics
- Non-linear materials and applications
- Atomic optics
- Imaging systems and physics
- Lasers
- Nanophotonics and electromagnetism
- Quantum optics
- XUV optics and optical surfaces

Outline

LCF: 40 years of research in XUV multilayer coatings

Deposition and characterization techniques

Applications, recent developments :

- beamline optics (Soleil, Elettra)
- x-ray diagnostics (LMJ)
- attosecond physics (pulse compression; selection of harmonics, large band mirrors)
- microscopy in water window
- space EUV telescopes

Laboratory Charles Fabry

XUV optics & optical surfaces group: 40 years of research in XUV optics

*In 70s & 80s : L. Névot, P. Croce, B. Pardo et al. Studies of optical surfaces, thin films, XUV interference mirrors, interfacial roughness model, etc. (**more than 1000 citations**)*

Optical workshop: precise fabrication of optical components to the limits of nowadays possibilities in terms of surface shape and roughness

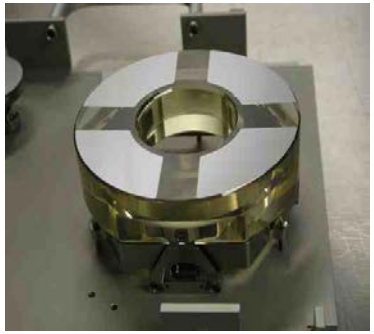
New cleanroom: 100 square meters, class 1000 (ISO 6)

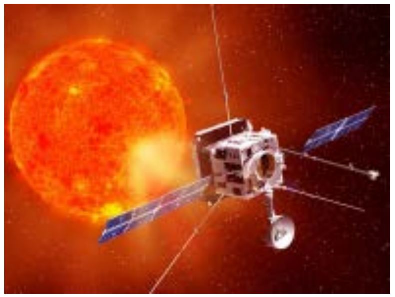
Optics for EUV telescopes

IAS Orsay

- First EUV multilayers on a satellite
  → SOHO mission (*J.-P. Chauvineau et al., 1995*)
- 3D imaging of the Sun corona
  → STEREO mission (M.-F. *Ravet et al., 2006*)

Primary mirror Ø105 mm for EUV Imager of STEREO
Mo/Si coatings (4 quadrants): λ = 17.1, 19.5, 28.4 and 30.4 nm





- Aluminum-based multilayer coatings
  → Solar Orbiter (to be launched in 2018)

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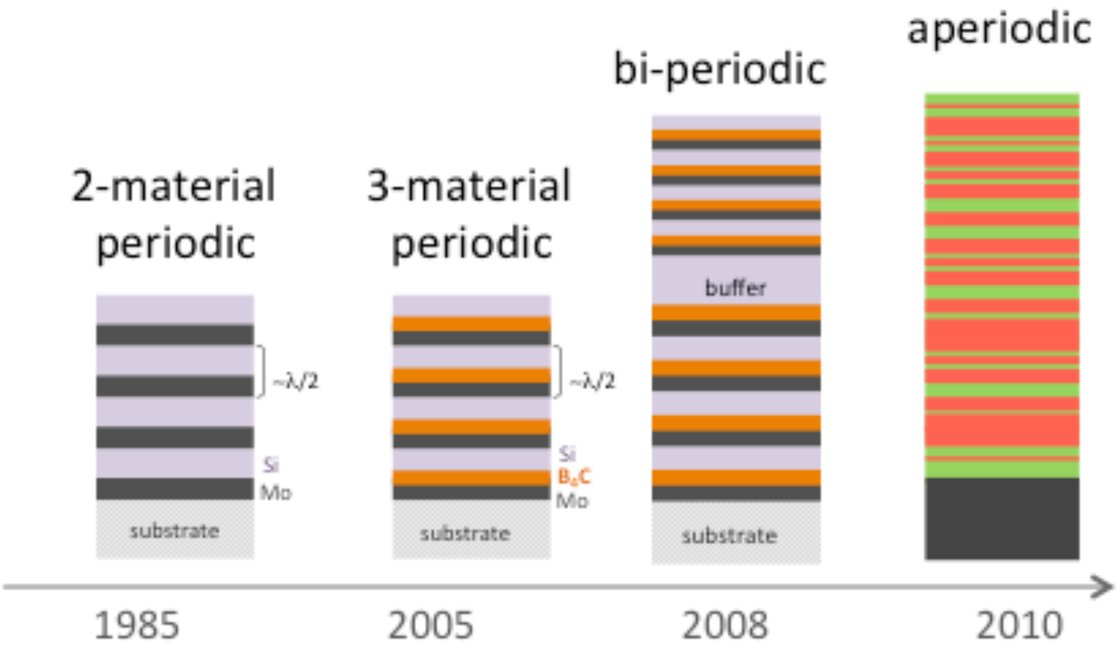
Evolution of multilayer structure design



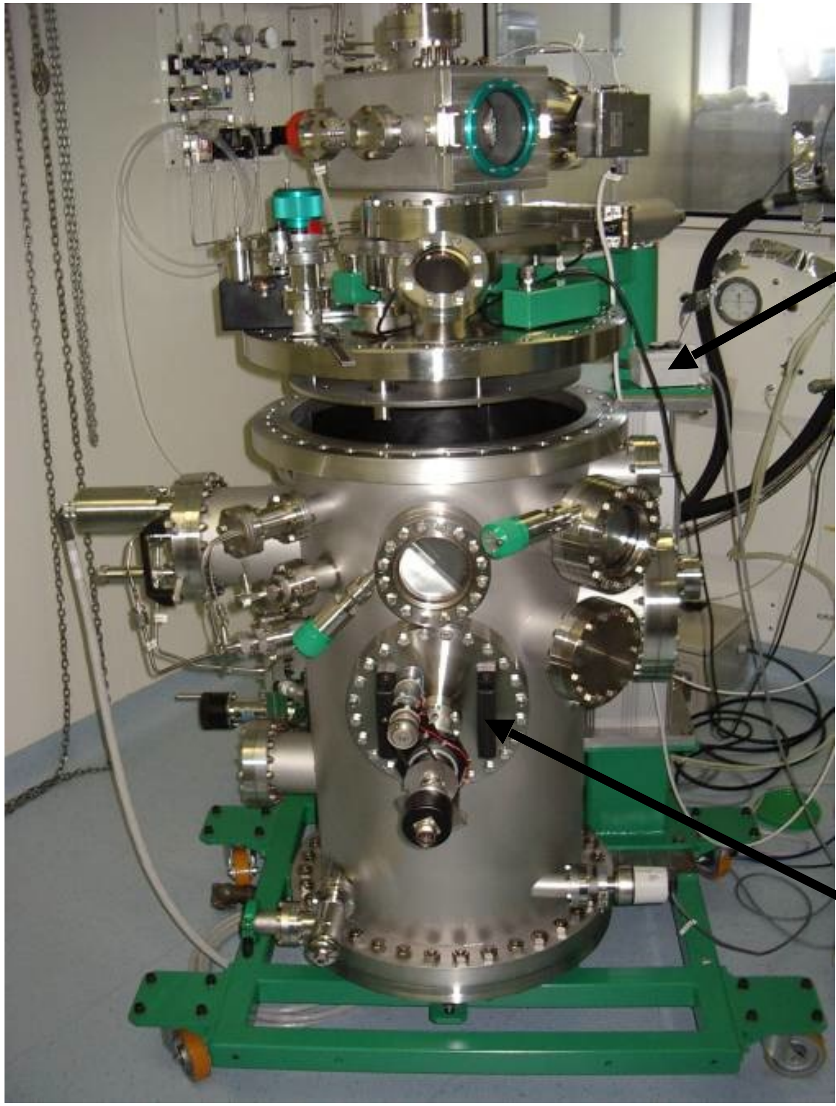
- Further progress in development of efficient reflective coatings requires a new approach to multilayer design
- Evolution of multilayer structures enables new solutions to be proposed to existing problems
- Multilayer coatings with new optical functions enlarge their application possibilities

Challenging issues

- ✓ Computer simulation (IMD, TFcalc, MatLab, home-made codes, etc...)
- ✓ Accurate knowledge of thin film material optical constants
- ✓ Physico-chemical properties of nanometric thin films and interfaces
- ✓ Optimization of deposition process for new material combinations

Multilayer deposition facilities (clean environment)

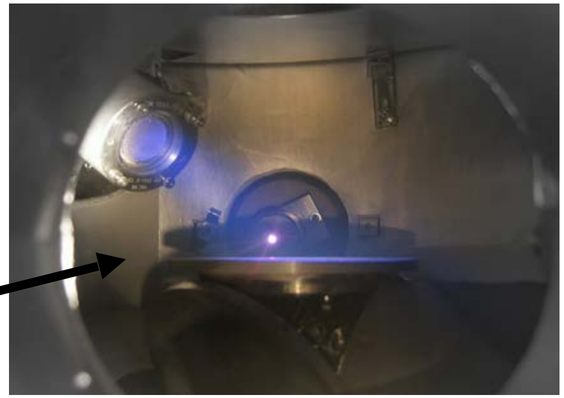
Ion beam sputtering - 4 targets



Lifting column

Transfer system

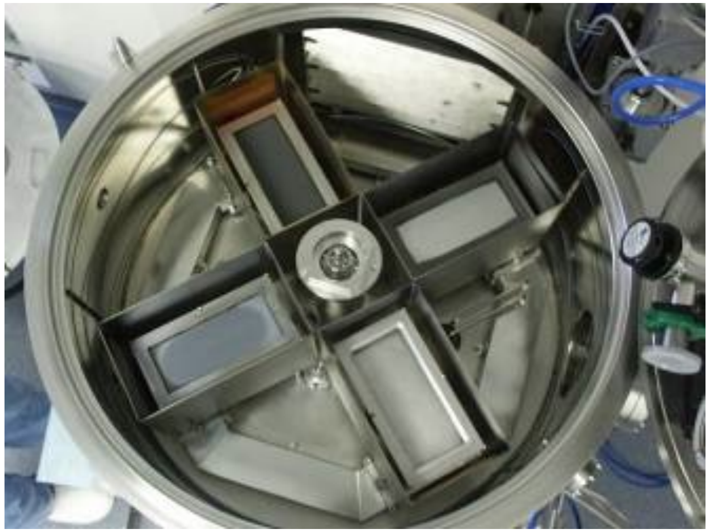
Ion gun



Rotating targets

Multilayer deposition facilities (clean environment)

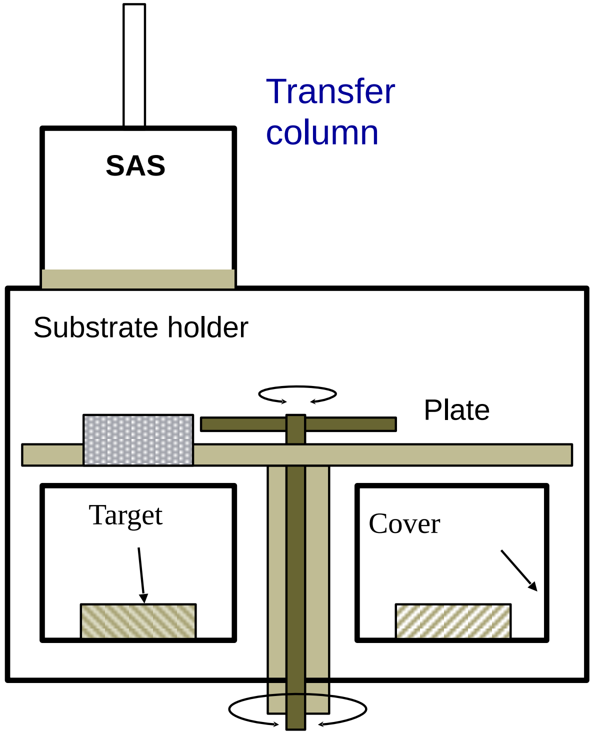
Magnetron sputtering Plassys MP800



4 targets
(2 *rf* + 2 *dc*)

Targets : 80 × 200 mm^2

Gas : Ar, Ar/O_2, Ar/N_2
P = 0.1 to 0.27 Pa



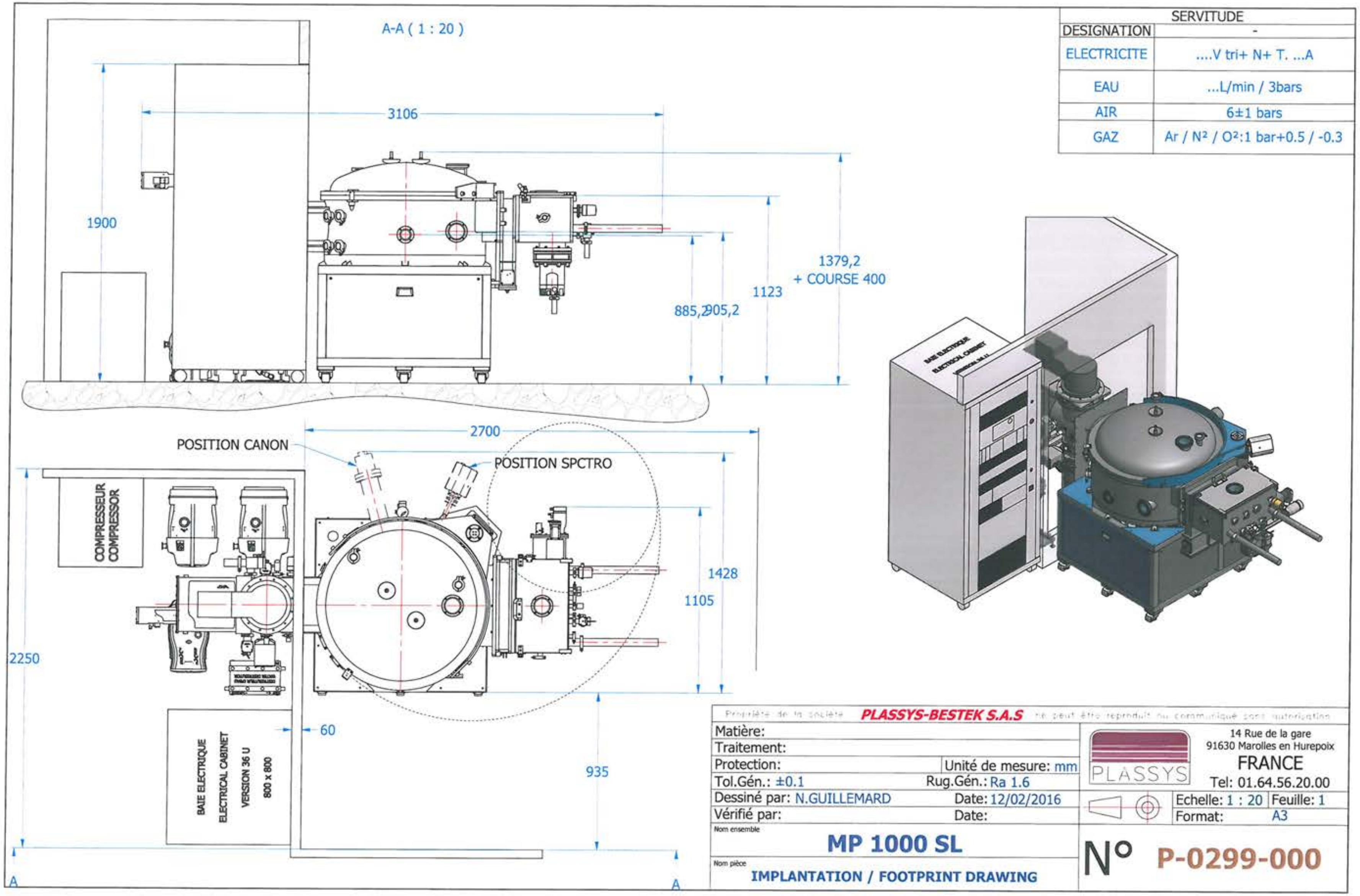
Precise thickness control: < 0.1 nm

High repeatability: in the order of 10^{-2} nm

Coating uniformity: ~ 0.5% @ 100 mm

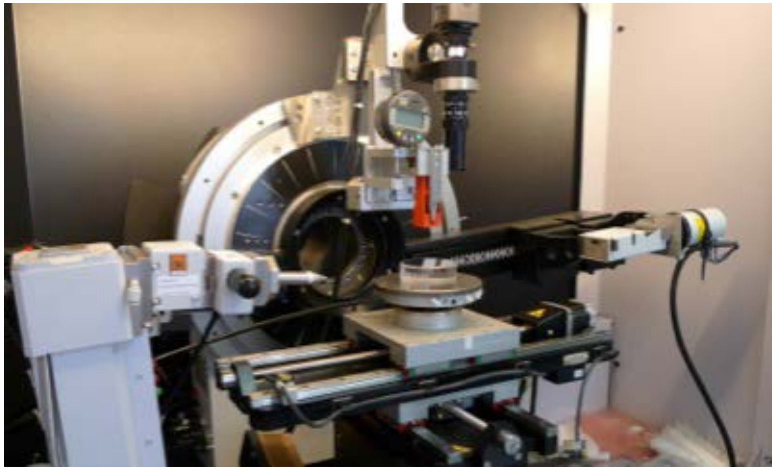
Multilayer deposition facilities (clean environment)

For the new cleanroom - a new magnetron sputtering machine (to be delivered in March 2017)



Characterization techniques

Grazing X-ray Reflectivity @ 0.154 nm



BRUKER D8 DISCOVER:

- *diffraction*
- *reflectometry*
- *scattering*
- *mapping*
- *heating stage Anton Paar - up to 1100°C*

At wavelength metrology

Reflectometer at ISMO (now moved to Soleil):

laser plasma, 532 nm, 400 mJ, 5ns, 1 Hz, spectral range from 4 to 50 nm

Calibration facility (CaliF) at IAS:

plasma discharge, 100 Hz, spectral range: from 12 to 120 nm

Synchrotron radiation facilities:

Elettra, Soleil, BESSY/PTB, ALS...

Techniques of physical-chemical analysis

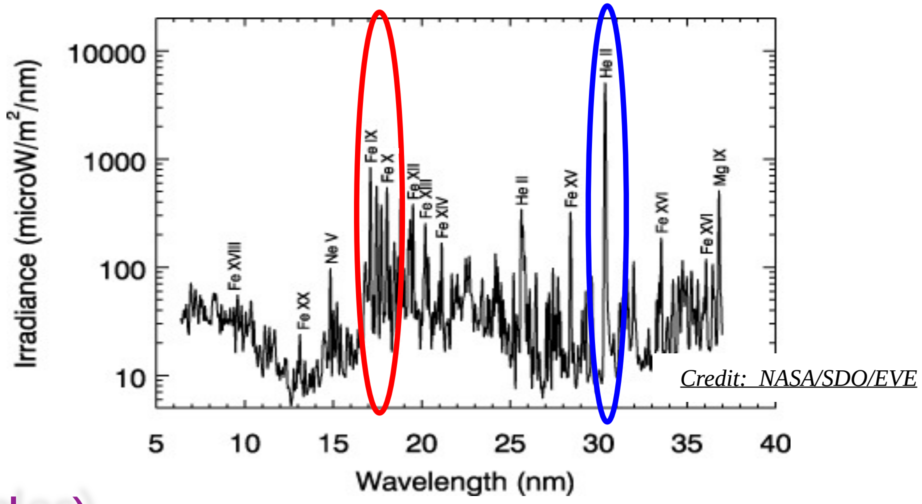
(TEM, AFM, XES, RBS, etc.) - accessible elsewhere

Solar imaging with EUV multilayer coated optics

EUV solar spectrum

17.1 - 17.4 nm (Fe IX-XI):
quiet corona, upper transition region
Log(T) = 5.9 – 6.1

30.4 nm (He II):
chromosphere, transition region
Log(T) = 4.9



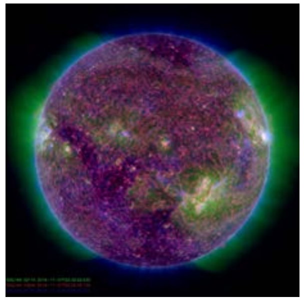
EUV onboard instruments (few examples)

1987: 1st high resolution solar images *A. Walker et al, Science 241, 781(1988)*
Sounding rockets (Stanford/MSFC)
Normal incidence telescope (resolution = 1.5 arcsec) :
λ =17.3 nm (Fe X); multilayer coating - Mo/Si (LLNL)

1995 : 1st EUV multilayers in space - SOHO mission (ESA/NASA)
Extreme UV Imager Telescope - EIT (resolution = 2.6 arcsec) :
λ =17.1/19.5/28.4/30.4 nm; multilayer coatings - Mo/Si (LCF)

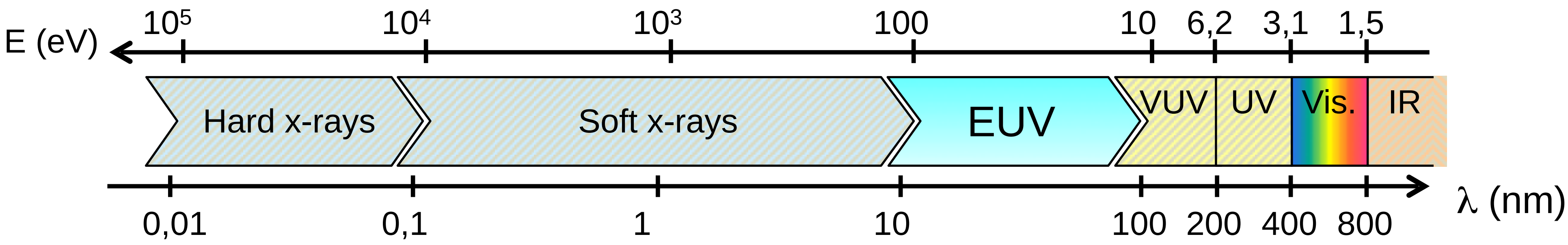
2006: 1st 3D images of the SUN - STEREO mission (NASA/ESA)
Extreme UV Imager - EUVI (resolution = 1.7 arcsec) :
λ =17.1/19.5/28.4/30.4 nm; multilayer coatings - Mo/Si (LCF)

2010: More EUV channels - SDO mission (NASA)
Atmospheric Imaging Assembly - AIA (resolution = 1.5 arcsec) :
λ = 9.4/13.1/17.1/19.3/21.1/30.4/33.5 nm;
multilayer coatings - Mo/Si, SiC/Si, Mo/Y (LLNL+RXO LLC)

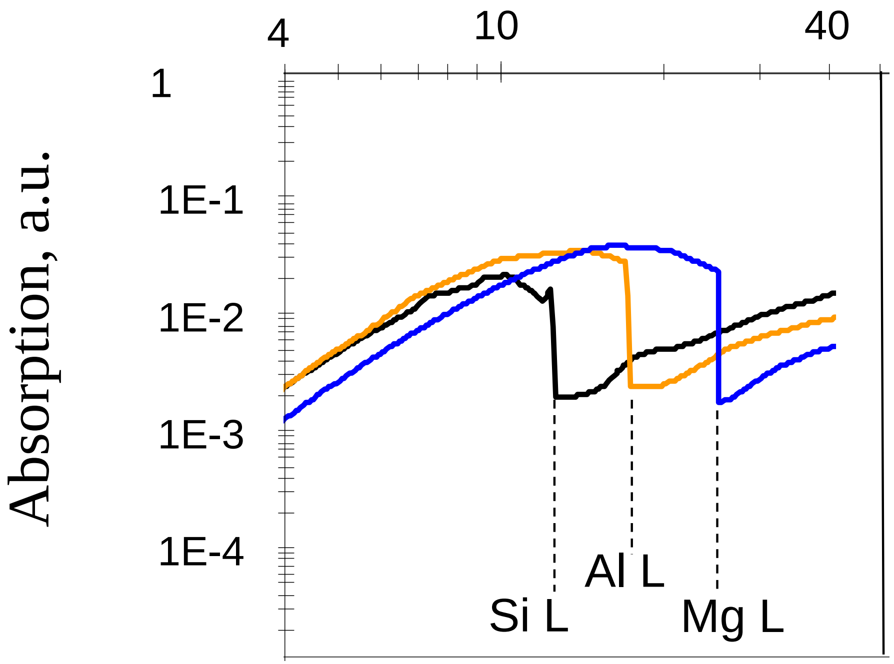


SDO image: 17.1+21.1+30.4 nm (from http://sdo.gsfc.nasa.gov - 2016/11/07)

Low absorbing materials in the EUV



✤ Limited number of low absorbing materials for a multilayer design



> 12.4 nm → Si **Mo/Si** *Barbee (1985)…*

> 17 nm → Al **Al/SiC** **Al/Zr** **Al/Y_2O_3** **Al/Be**

Windt (2009), Meltchakov (2010), Murakami (2011), Zhong (2012), Chkalo (2016)…

> 25 nm → Mg **Mg/SiC**

Kondo (2001), Yoshikawa (2005), Soufli (2005), Fernandez Perea (2012)…

Aluminum-based multilayers for the EUV

Problems of aluminum films growth:

- High chemical reactivity with oxygen and some other materials
- Inhomogeneous crystallization

  High interfacial roughness

  Low optical performance

  Uncertain long-term stability

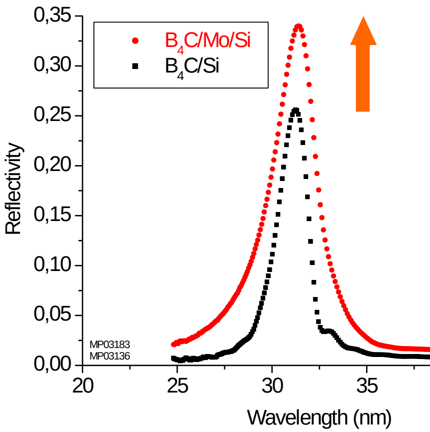
D. Windt and J. Belotti. Appl. Opt. (2009)
E. Meltchakov et al. Appl. Phys. A (2010)

Proposed solutions :

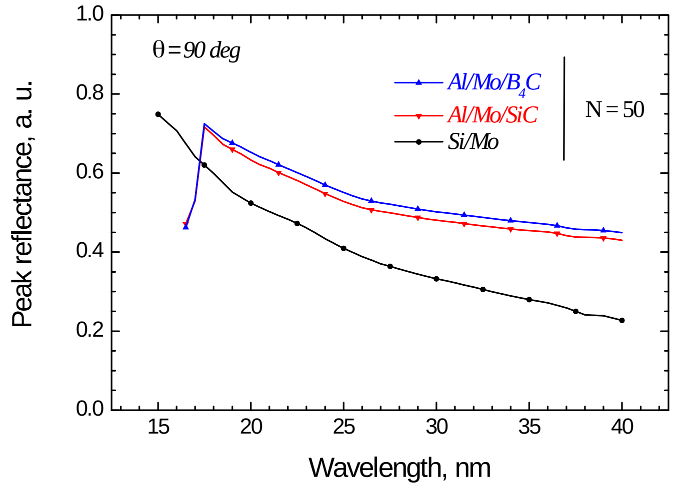
- *Optimization of deposition process*
- *Al target doped with Si, Cu, … to minimize crystallization*
- *Use of cap layers to protect from oxidation*
- *Use of more than two materials in multilayer structure*

Multilayer with more than two materials

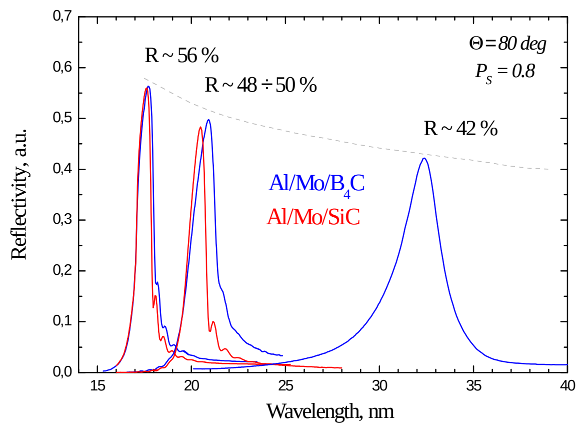
- Significant increase in theoretical peak reflectance *P. Boher et al., (1991), J. Larruquert (2002)*
- First experimental study of tri-component multilayers *J. Gautier et al., (2005)*



Computer optimization of tri-component Al-based multilayers for a maximum peak reflectance



Reflectivity measurements at Bear beamline (Sincrotrone Trieste)



Highly reflective multilayers (R > 50 %) at near normal incidence

1985	Mo/Si	13 nm	Barbee et al.
1995	Mo/Be	11 nm	Skulina et al.
1998	Sc/Si	46 nm	Uspenskii et al.
2009	Mg/Sc/SiC	28 nm	Aquila et al.
2010	Al/Mo/SiC	17 nm	Meltchakov et al.
2013	$La/B_4C/C$	6.8 nm	Chkhalo et al.

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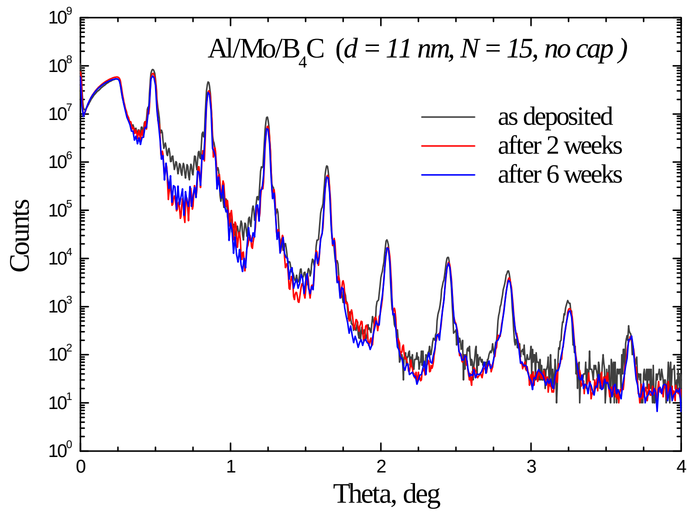
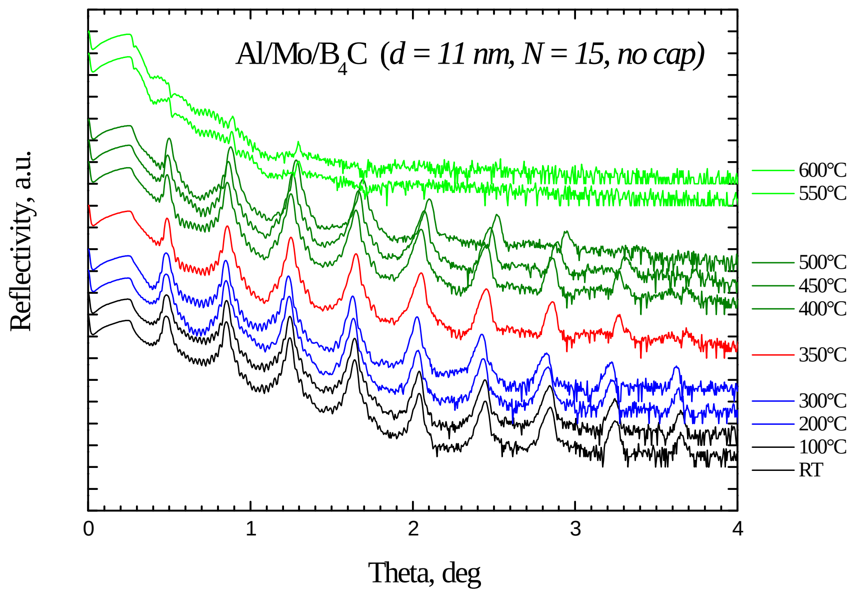
Stability of Al-based multilayers

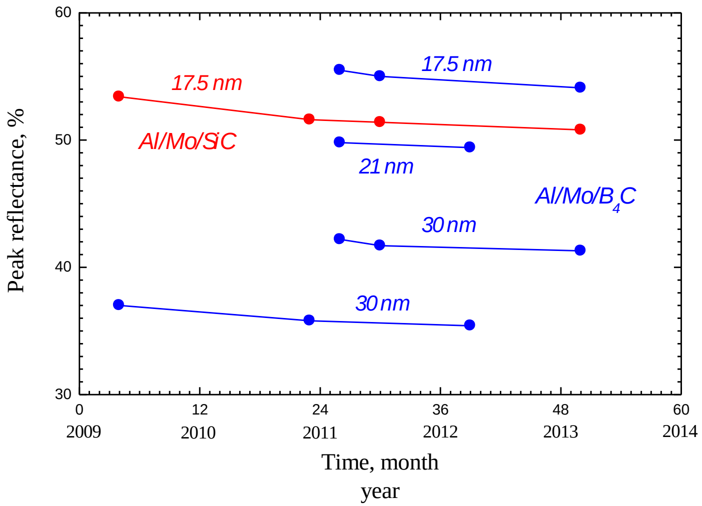
Stability of samples annealed at 100 °C in air



Changes induced by heating up to 600° in air



EUV peak reflectance of samples stored in air



- After a small initial reduce, the EUV peak reflectance (due to formation of a surface oxide layer) remains stable during all the period of observation (more than 6 years) even if stored in air
- No structural changes observed in multilayer annealed at 100°C or undergone a thermal cycling (-20° + 50°C)
- No significant changes occur upon a heating up to 300°C (just a slight increase of the multilayer period (0.07 nm) due to the thermal expansion)
- Multilayer is still generally stable between 300 and 500°C however the period is decreasing (structural phase transition started)
- Periodic structure is irreversibly lost above 500°C

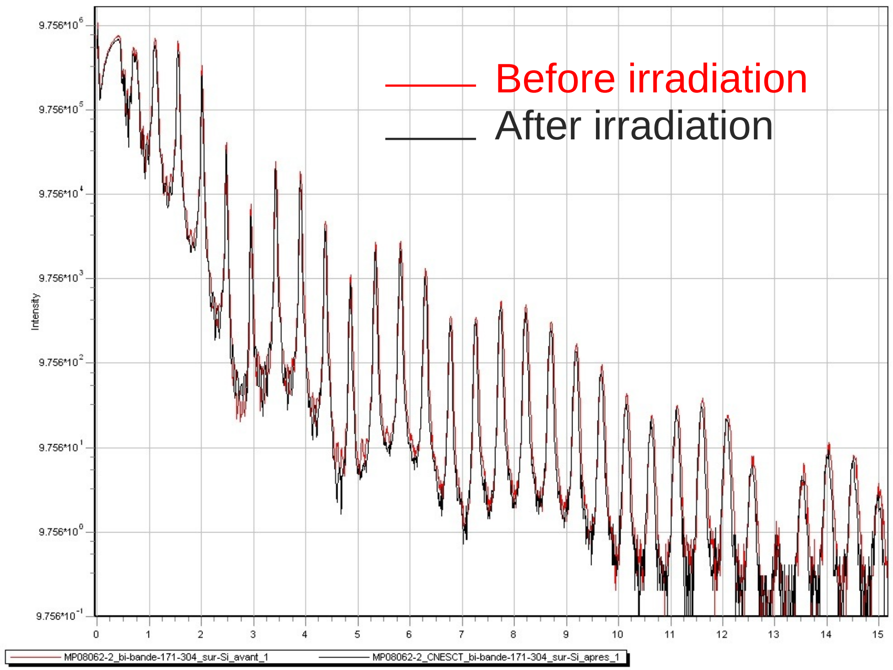
Stability of Al-based multilayers

Proton irradiation tests on Al/Mo/B_4C and Al/Mo/SiC

Energy (keV)	Dose (p/cm^2)
1	7.4×10^{12}
1	9×10^{15}
100	7.4×10^{12}

HZDR (Dresden, Germany)

Comparative tests - before / after irradiation



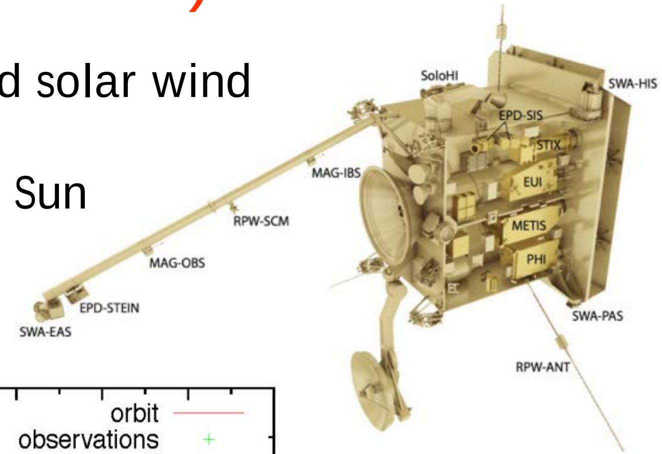
- Visual inspection
- Grazing x-ray reflectometry @ 0,154nm
- EUV reflectivity measurements (Elettra)

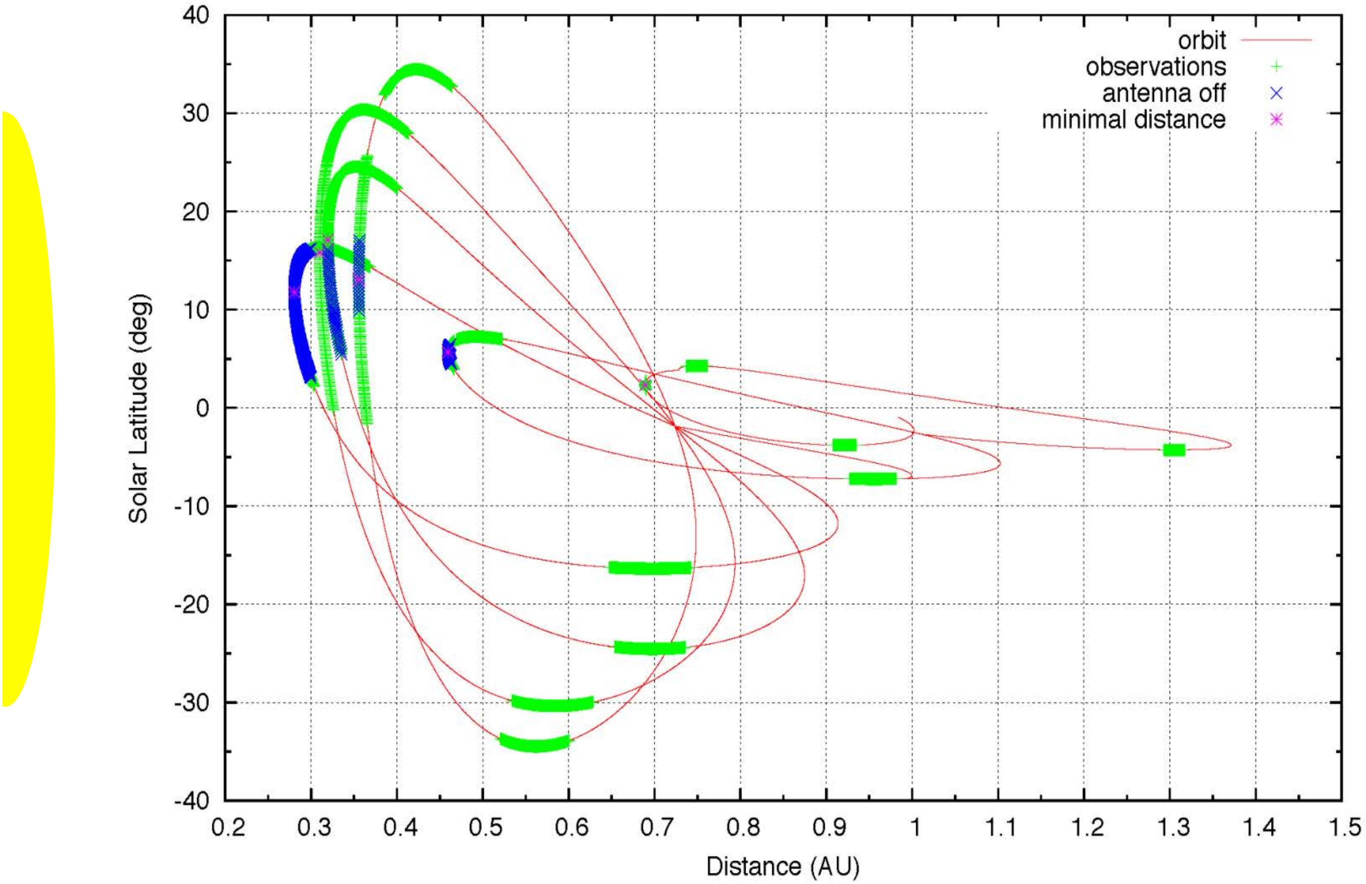
- ✦ No surface damage
- ✦ No significant structural changes
- ✦ No shift of EUV peak position (± 0.2 nm)
- ✦ No EUV peak reflectance loss (±0.5 %)

Imaging the Sun with multilayer optics

Solar Orbiter Mission (ESA/NASA)

- To provide a connection between inner heliosphere and solar wind
- Approaching as close as 0.2 solar radii
- Imaging with high spatial resolution : ~ 100 km on the Sun
- Polar regions observation : tilt up to 34°





Extreme Ultraviolet Imager of Solar Orbiter

PI: Pierre Rochus, Centre Spatial de Liège, Belgium

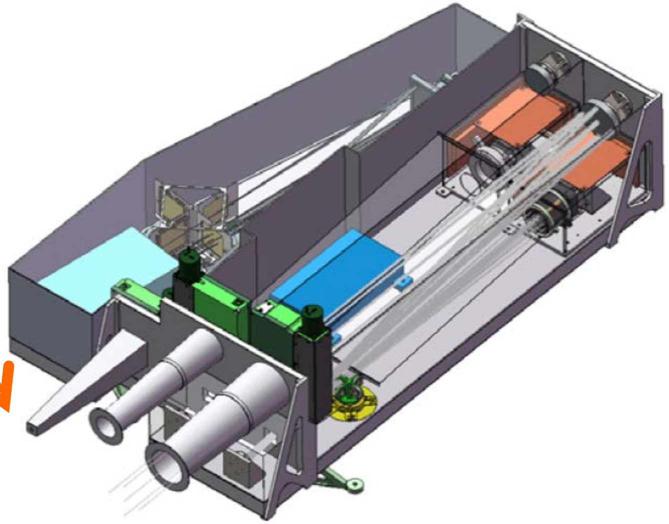
Collaborating countries (hardware): Belgium, UK, France, Germany, Switzerland

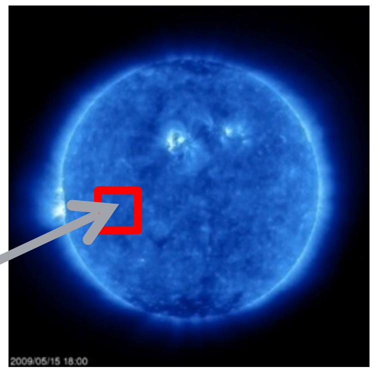
FSI: one mirror telescope
Dual-band coating
λ= 17.4 / 30.4 nm
High selectivity required

Single entrance aperture
2 channels in 1 telescope
1 mirror optical design
=> reduced thermal load, mass

HRI-EUV: two-mirror telescope
λ = 17,4 nm
High reflectivity required

High spatial resolution :
~100 km on the Sun (~ x4)



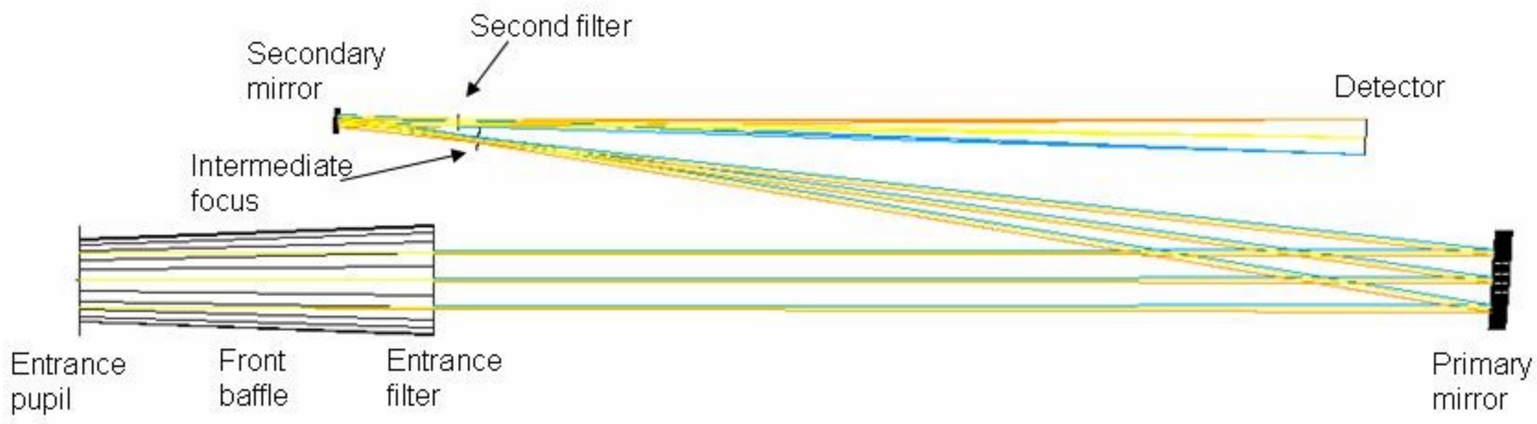


High Resolution Imager: Lyman α **Subsec-100s cadence** **Pixel=100km@ perihelion**
High Resolution Imager: Fe IX/X 17.4 1-100s cadence Pixel=100km@ perihelion
Full Sun Imager: **Fe IX/X 17.4 & He II:30.4** **10-600s cadence** **Pixel=860km@ perihelion**

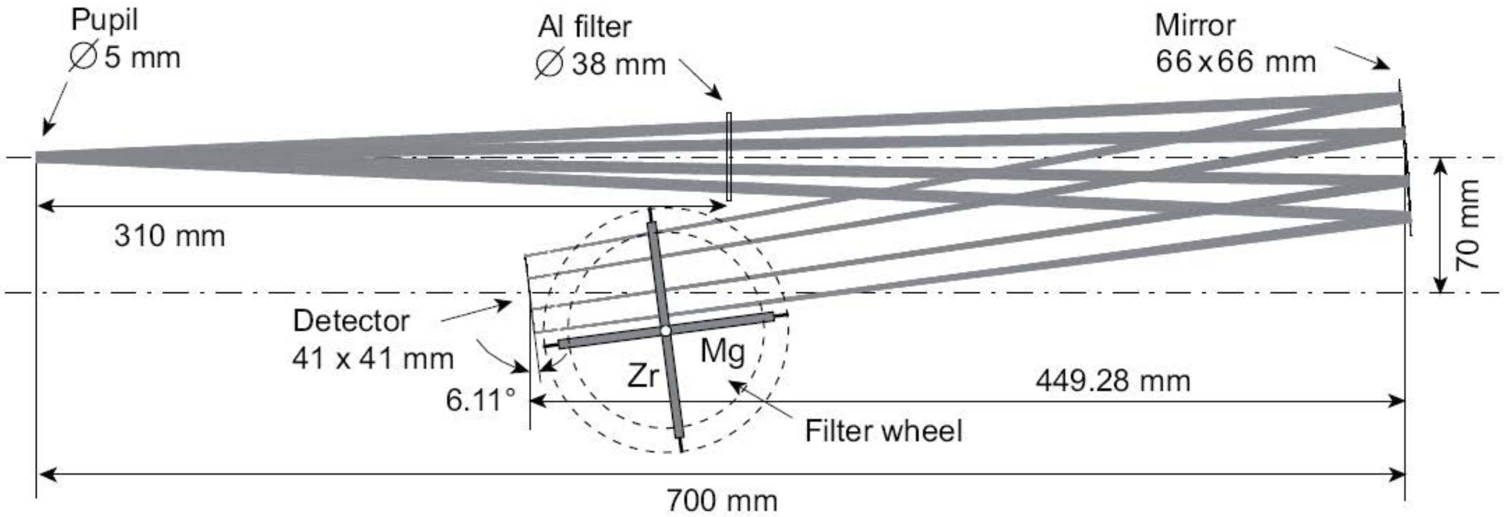
- Provide image sequences of the solar atmospheric layers from the chromosphere into the corona
- Provide images of the Sun from an out-of-ecliptic viewpoint (up to 34° of solar latitude during the extended mission phase)

Solar Orbiter : context

High Resolution Imager (HRI): 1 channel (17,4 nm) 2 mirrors

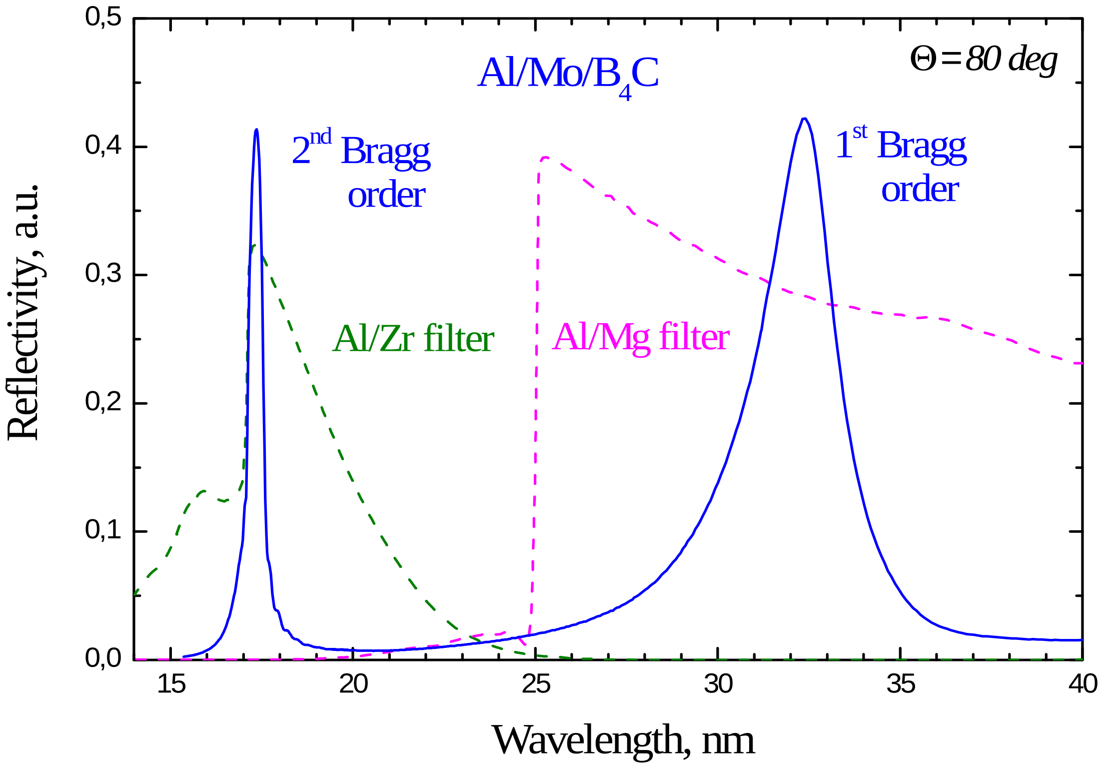


Full Sun Imager (FSI): 2 channels (17,4 nm & 30,4 nm) 1 mirror



Dual-band multilayer mirrors

Multiple order Bragg reflections from simple periodic multilayer ?

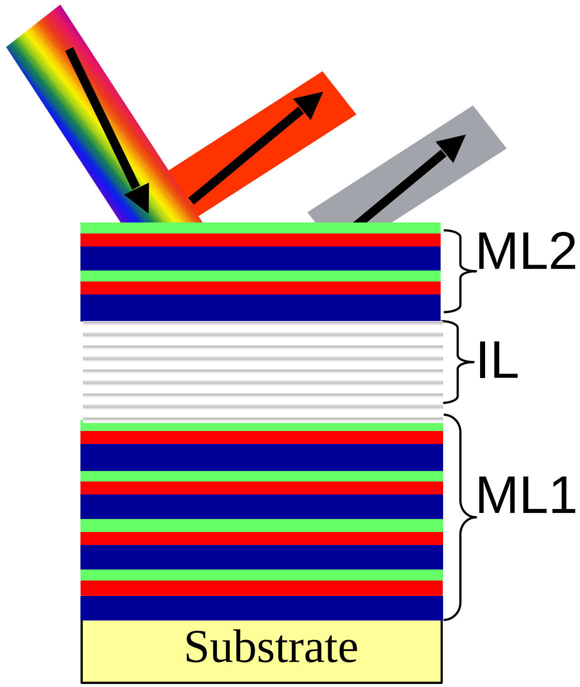


EUV reflectivity of simple periodic multilayer Al/Mo/B_4C (d = 18.3 nm, N = 25)

- Relatively narrow spectral region above an absorption edge
- No way to adjust positions of Bragg peaks to emission lines of interest

Aperiodic multilayers ?

Dual-band multilayer systems design



Principle:

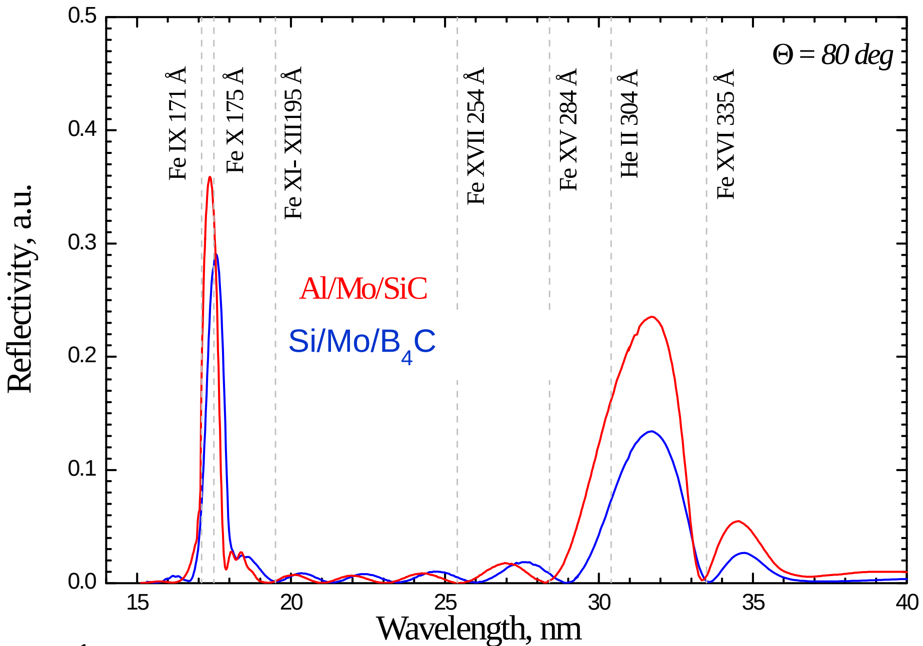
- Superposition of 2 periodic multilayers (ML1 and ML2) separated by an intermediate layer
- Use of first and/or second Bragg's orders of ML1 and ML2

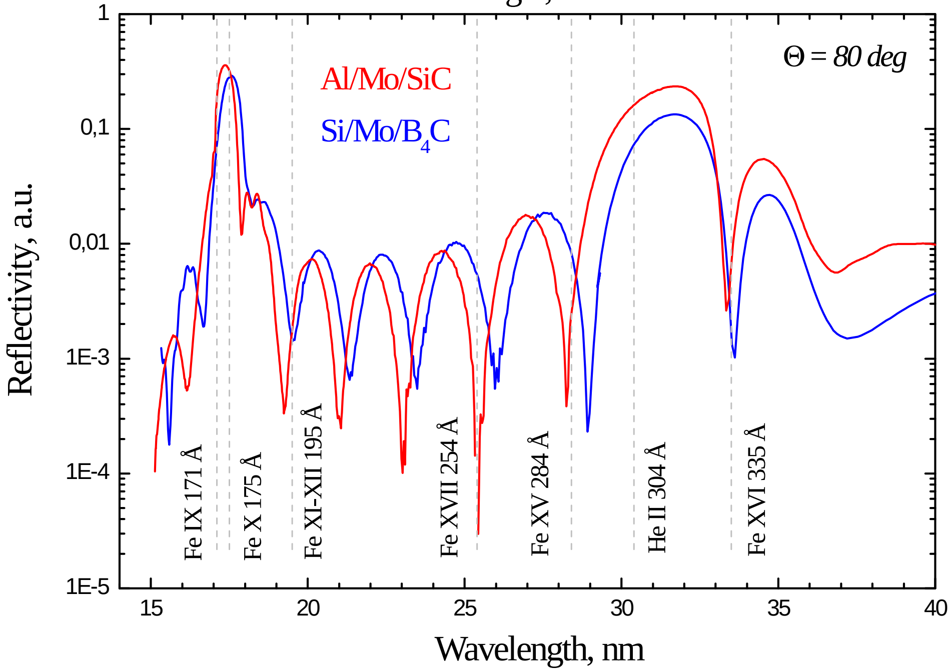
J. Gautier et al., Opt. Comm. 281, 30 (2008)
C. Hecquet et al., Appl. Phys. A 95, 401 (2009)
E. Meltchakov et al.,Proc. SPIE 8777, 47 (2011)

Computer optimization of structural parameters:

- Maximum peak reflectance in main channels
- Rejection of specific wavelengths_
- Adjustable band position
- Provide solutions considering the multilayer technology aspects (use of new multilayer materials, protection from oxidation, etc.)

Candidate systems for FSI telescope





Initially proposed materials – $Si/Mo/B_4C$

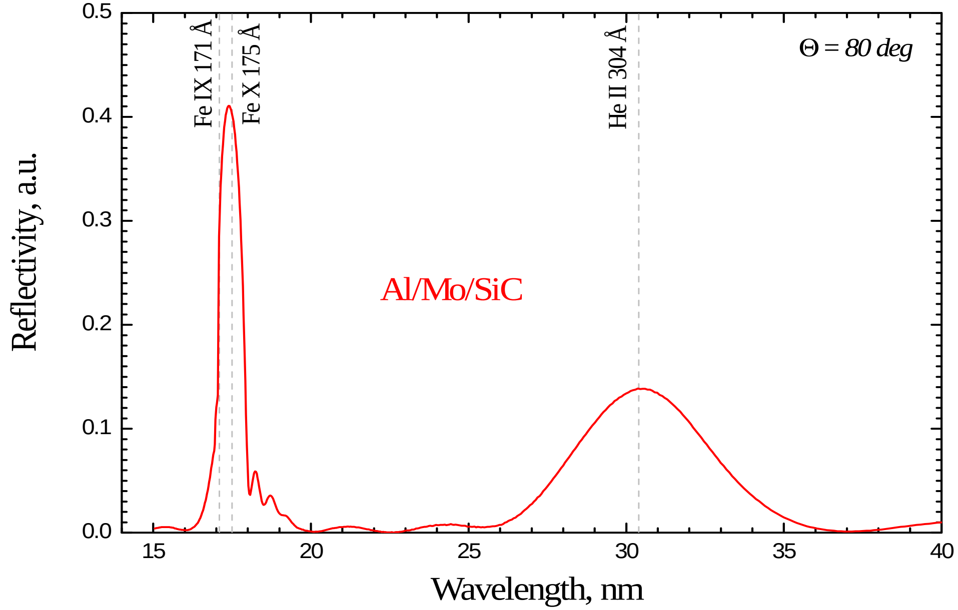
C. Hecquet et al., Appl. Phys. A 95, 401 (2009)

New materials – $Al/Mo/SiC$ (or B_4C)

E. Meltchakov et al., Proc. SPIE 8777, 47 (2013)

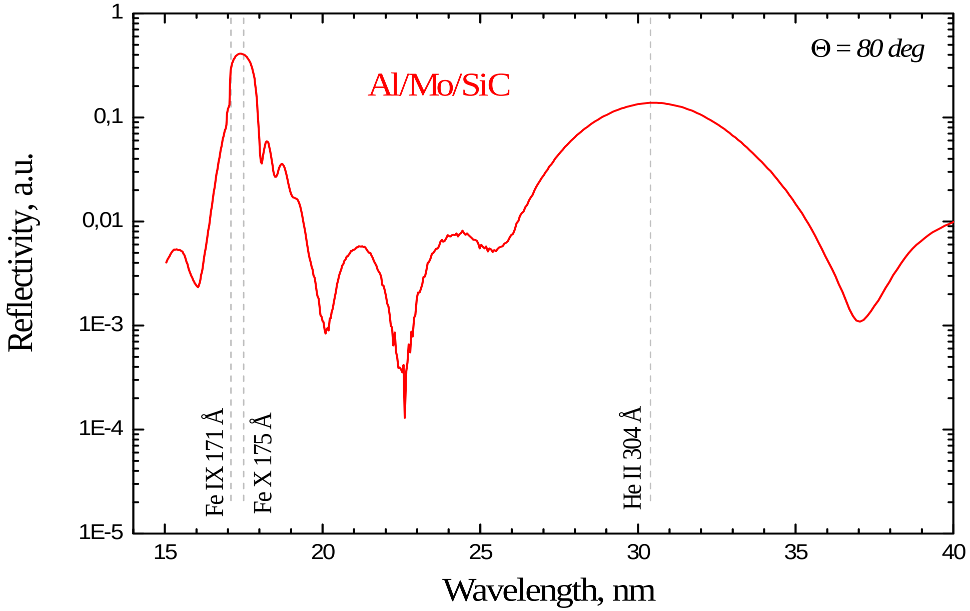
- Significant reflectance enhancement in main channels
- Better rejection of unwanted emission lines

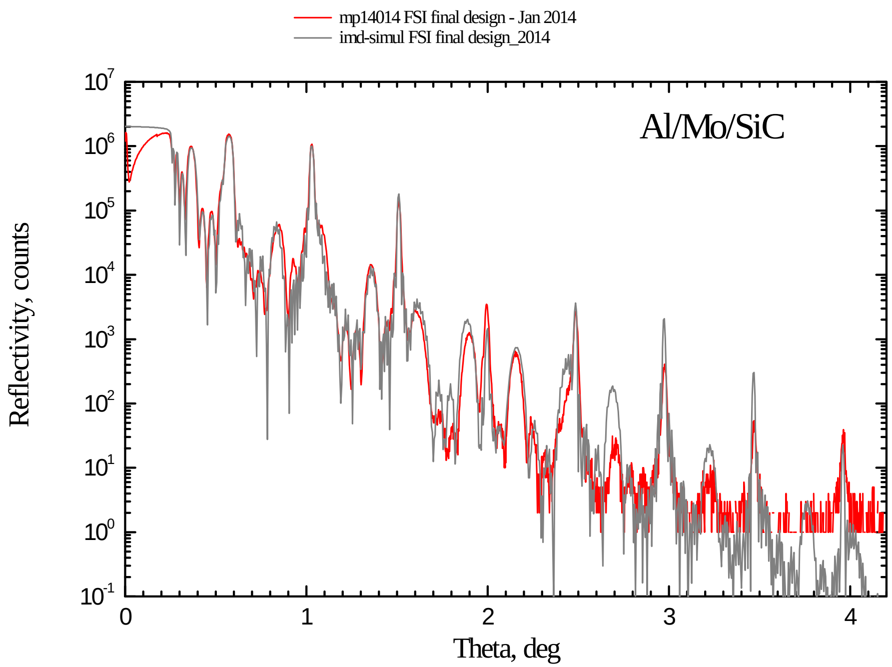
FSI mirror: final design



$[Al/Mo/SiC]_{30}//Al//[Al/Mo/SiC]_4$

ML1 – Al/Mo/SiC
(1st Bragg order)
Intermediate layer – Al
ML2 – Al/Mo/SiC
(1st and 2nd Bragg orders)





Fabrication of EUV optics for Solar Orbiter

Optical workshop LCF:

substrate surface shape

better than λ/14 RMS

with sub-nanometer roughness

(< 0.2 nm)

Thanks and congratulations to

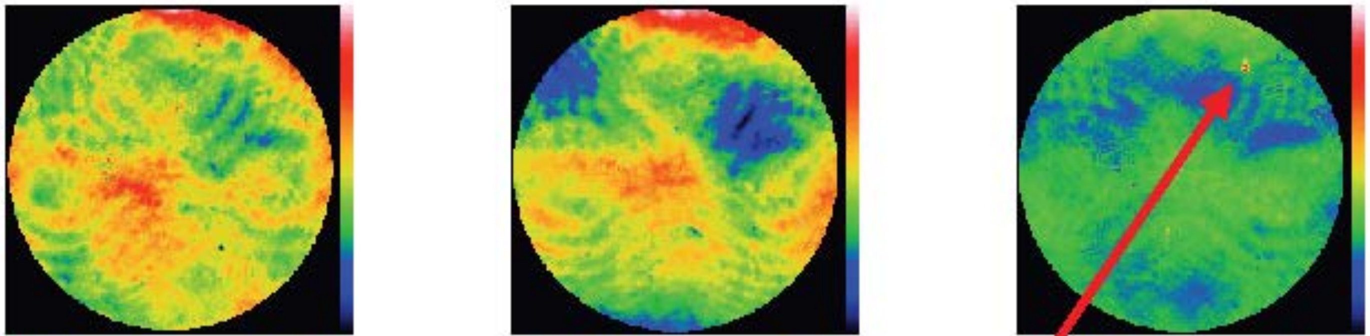
Christian Beurthe –

Crystal Medal CNRS (2015)

Fabrication of EUV optics for Solar Orbiter

An example of HRI-S mirrors

Second asphérisations:



N°1: 12.3 nm P-V,
1.19 nm RMS,
0.92 nm RMS estimated

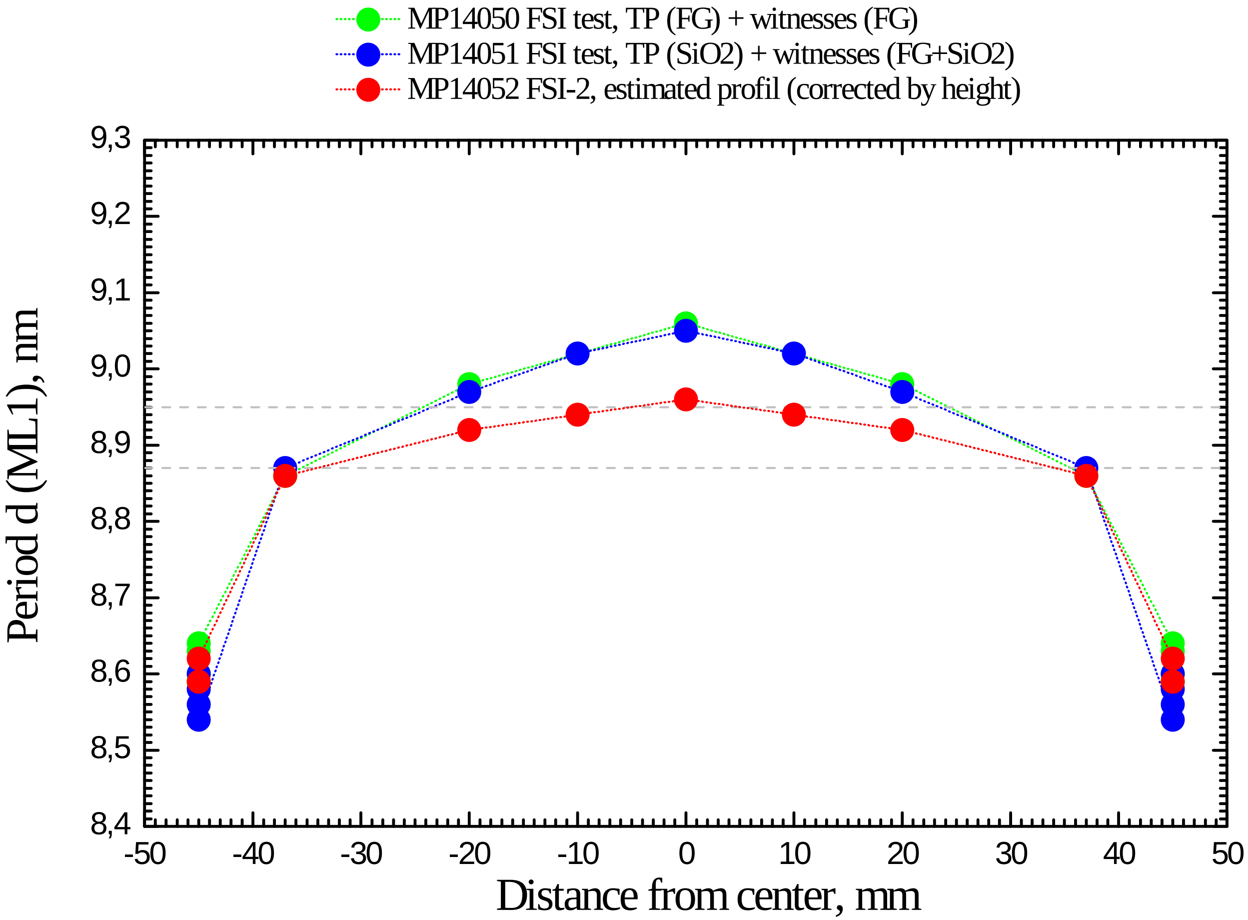
N°2: 12.2 nm P-V,
1.71 nm RMS,
1.55 nm RMS estimated

N°3: 33 nm P-V,
1,47 nm RMS,
1,27 nm RMS estimated

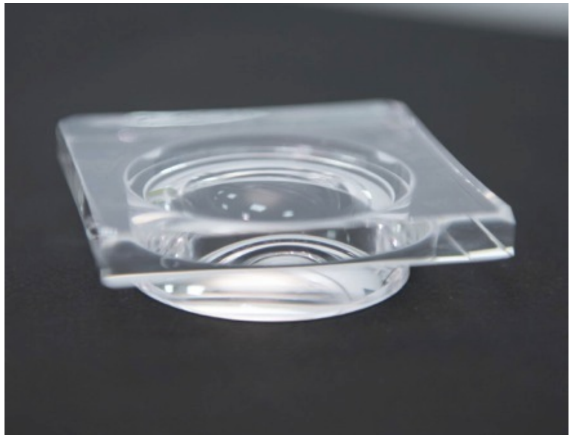
Aim: better than 2 nm RMS, so perfect …
or almost … small local defect on S3!

FSI tests and final deposition

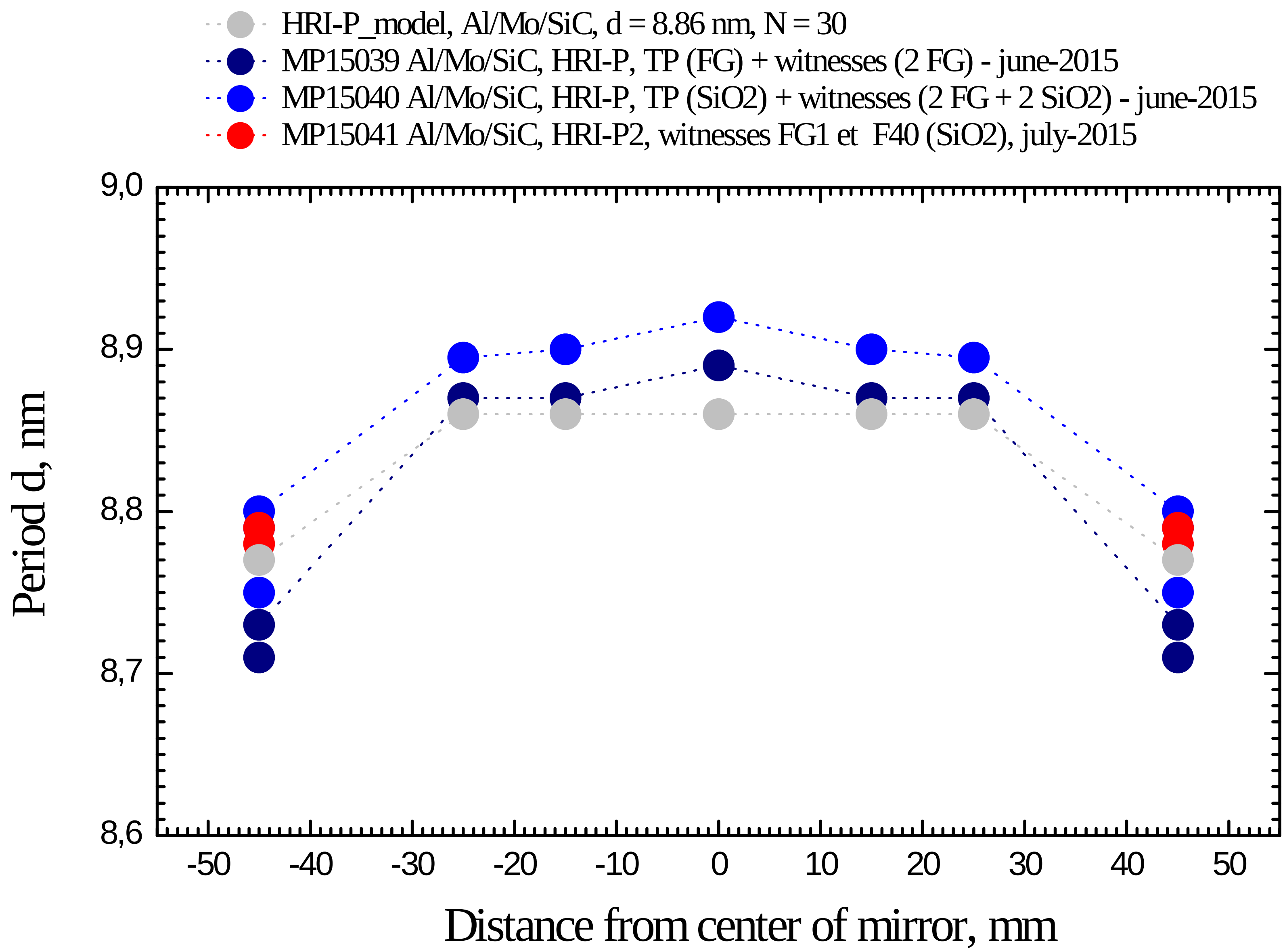
Test samples:
flat substrates 65 x 65 mm (SiO_2)
witness samples Ø20 mm (FG and/or SiO_2)



FSI mirror and witness samples before deposition

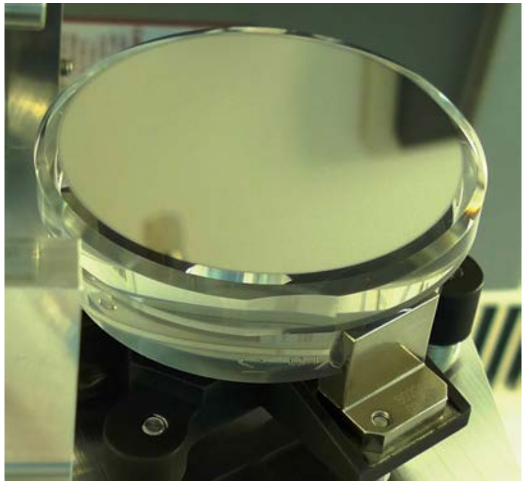


HRI-P tests and final depositions



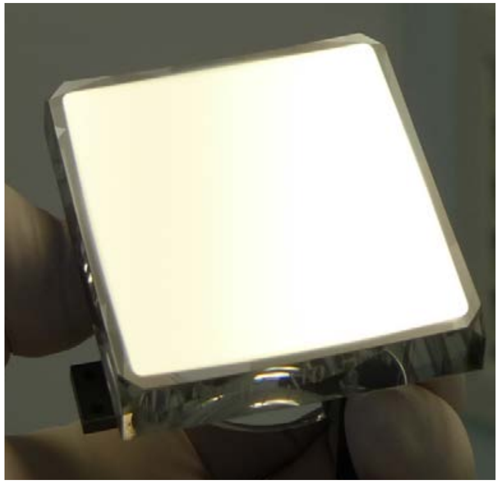
Summary

- Highly reflective Al-based multilayer coatings for EUV have been developed and characterized.
- New multilayers have good temporal, thermal and radiation stability
- FSI and HRI mirrors for EUV instrument of Solar Orbiter have been fabricated and characterized



HRI-P Flight Mirror

HRI-S Flight Mirror



FSI Flight Mirror

Thank you for your attention!