Preliminary characterization of first multilayer mirrors for the soft X-ray water-window

**Gianfelice Cinque, Augusto Marcelli, Antonio Grilli and Agostino Raco**  
INFN, Laboratori Nazionali di Frascati, via E. Fermi 40, I-00044 Frascati, Italy  
**Valentino Rigato, Alessandro Patelli and Valentina Mattarello**  
INFN, Laboratori Nazionali di Legnaro, viale dell’Università 2, I-35020 Legnaro, Italy  
**Giannantonio Cibin**  
IMONT, Piazza dei Caprettari 70, I- 00186 Roma, Italy

Design and test of high reflectivity devices for soft X-rays at quasi-normal incidence: Synchrotron Radiation soft X-ray microscopy of organic systems and X-ray microprobe.

Multilayer mirrors for almost monochromatic X-rays in the low energy range, and quasi-normal incidence to fully exploit the angular aperture.

Design and deposition of multilayer at the INFN Legnaro Laboratories: first Ni/Ti and Ni/TiO₂ multilayers with $\Lambda > 1.3-1.4$ nm and number of layers from 150 to 300.

High Vacuum compatible reflectometer set-up at the INFN Frascati Laboratories: characterization of the multilayer reflectivity by SR beam below 1 keV at DAΦNE.
X-ray microscopy in reflection: an example

Spherical Reflecting optics

vertical divergence 5-3 mrad
horizontal divergence 5 mrad
angle of incidence

~10 mm
~20 mm

DAΦNE@LNF
SR source

X-ray fan

Object

Image

Normal incidence multilayer mirrors

\[ n_i = 1 - \delta_i \]

\[ m\lambda = 2\Lambda \sin \vartheta_m \sqrt{1 - \frac{2\delta}{\sin^2 \vartheta_m}} \]

\[ \delta = (d_1 \delta_1 + d_2 \delta_2) / (d_1 + d_2) \]

\[ \Lambda = d_{\text{absorber}} + d_{\text{spacer}} \]

\[ \delta \Lambda \leq \frac{\Lambda}{10} \quad \Gamma = \frac{d_{\text{abs}}}{d_{\text{tot}}} \]

\[ m = 1 \]

\[ \vartheta_1 \approx 90^\circ \]

\[ \delta_i << 1 \]

\[ 2\Lambda \approx \lambda \]
Multilayer material: selection for the X-ray water-window

\[ n(r) = \sqrt{\varepsilon(r)} = \sqrt{1 + \chi(r)} \approx 1 + \frac{\chi(r)}{2} = 1 - \frac{r_0 \lambda^2}{2\pi} \rho(r) \cdot (f_1 - if_2) = 1 - \delta + i\beta \]

Low absorption from spacer and absorber
Max contrast from the absorber
Spacer material sets the working range
Sharp interfaces

Mo/Si and Ni/Ti(O_x) deposition set-up at the LNL

Chamber
• 3 sputtering cathodes RF(13.56MHz)-driven
• Process gas: Ar, Xe, O_2
• DC biasable sample-holder (-100÷100V)
• Helmoltz coils (0÷60G)
• Quartz micro-balances
• quadrupole mass spectrometer
• Cylindrical Langmuir probe

Computer control
• Up to 500 Ni/TiOx bi-layers
• Shutters position
• Sample-holder position
• DC sample-holder bias
• cathodes applied power
• Helmoltz coils current
• O_2 gas admission

base pressure <1·10^{-7}mbar

operating Ar pressure 2.5÷3.3·10^{-3}mbar
First Ni/Ti and Ni/TiO\textsubscript{x} multilayers deposition at LNL

Multilayer Ni/Ti
4 multilayers deposited in the work on nickel titanium:
\textbf{NT012} project of 300 periods
Ni/Ti on Si(100) substrate

Multilayer Ni/TiO\textsubscript{x}
3 multilayers deposited in the Work on nickel titanium oxide:
\textbf{NT015} project of 150 periods
of Ni/TiO\textsubscript{2} on Si(100) substrate

Nominal values:
TiO thickness 8.6\AA
Ni thickness 6.0\AA
First Ni/Ti and Ni/TiO\textsubscript{x} multilayers: characterization by RBS

RBS: \(\alpha\)-beam a 2.2, 2.0 and 1.8 MeV scattering angle 160° - 170°

Spectrum: no sample contamination by Ar

values:

- **Ti thickness** 5.5 Å
- **Ni thickness** 7.6 Å
Bragg diffraction by a couple of KAP(100) crystals (2d=26.6 Å) is used for the X-ray energy selection down to 480 eV.

A double goniometer allows ω−scans and θ−2θ scans under vacuum with an angle precision/accuracy better than 1/2000 degrees in closed-loop.

The Wiggler source gives a continuous SR spectrum of softer X-rays with critical energy ~300 eV.
DAΦNE as Synchrotron Radiation source

The electron ring of the DAΦNE collider has unique characteristics as Synchrotron Radiation source: since the low energy, 0.51 GeV, and the high circulating current, routinely over 1 A, an intense photon flux spectrally peaked in the lower energy range is provided.

Wiggler SR at 1 A

Wiggler critical energy ~300 eV
X-ray fan 5 x 2 mrad$^2$ (h x v)
$\theta = 1/\gamma = 1$ mrad
**Boomerang monochromator and KAP crystals**

Double-crystal fixed-exit movement spanning Bragg angles from 15° to 75°

**KAP(100) crystals tested by a Cu source and a θ–2θ diffractometer**

*XRD by Claudio Veroli and Giorgio Cappuccio – CNR-ICSM*
HV experimental chamber and θ–2θ goniometer

2 Micos Precision Rotation Stage PRS-110
2 UHV VG magnetic rotary feedthrough
2-phase-micro-step endless motors
contactless limit-reference switches
optical rulers for absolute positioning

Rotation Range (°) 360 endless
Accuracy (°) +/- 0.006
Repeatability (°) +/- 0.0002
Reversal Error (°) 0.0004
Resolution: Open-Loop (°) 0.002
Closed-Loop (°) 0.0002

Micos CORVUS 2 axes Microstep Controller +
manual Joystick/Display
ADC Input 12 bit for signal 0-10V
LabVIEW code for ω scan and θ–2θ scans
Absolute photodiode AXUV100EUT
Area 10x10 mm² UHV preAmp 10V/nA +/-1%
Preliminary tests on a Si/Mo multilayer at LNF:

Characterization of the reflectometer set-up by an $\omega$-scan

Si/Mo $\Lambda = 64$ Å

40 bilayers optimized for 700-850eV@8deg

Si(1-1-1)@2,8keV

detector

whitebeam SR

$\omega$ scan@45deg

multilayer@550eV (50%)
+825eV (33%)
+965eV (16%)

upward scan

downward scan

average-background
Preliminary measurements on Ni/Ti multilayers at LNF:
characterization of the reflectivity by an $\omega$-scan

$\omega$-scan

$\omega$ [degrees]

$S [V]$

Ni/Ti 300 bilayers  SRwhitebeam@45deg

1$^{\text{st}}$ scan upward

2$^{\text{nd}}$ scan downward

3$^{\text{rd}}$ scan upward

4$^{\text{th}}$ scan downward

average-background

(x4)

670eV

SR

$\omega$ scan

46.0 46.5 47.0 47.5 48.0 48.5 49.0

0.0 0.5 1.0 1.5 2.0 2.5 3.0 3.5 4.0 4.5 5.0
Ni/Ti multilayer test by XRD and XRR:

XRD by Claudio Veroli and Giorgio Cappuccio – CNR-ICSM

Ni/Ti 300 layers

XRD by Cu $K\alpha_1$ line
Huber $\theta-2\theta$ goniometer
8048 eV @ 40 kV 30 mA

Ni/Ti 300 layers $\Lambda = 13.86 \pm 0.07 \text{Å}$

Ni/TiO$_x$ 150 layers $\Lambda = 12.78 \pm 0.18 \text{Å}$
Ni/Ti and Ni/TiO$_x$ now:

from XRR measurements:

$$\Lambda_{\text{Ni/Ti}} = 13.86 \pm 0.07 \text{Å}$$
$$\Lambda_{\text{Ni/TiO}_x} = 12.78 \pm 0.18 \text{Å}$$

from XRD intensity comparison:

$$R_{\text{Ni/Ti}} = \frac{1}{8} R_{\text{Si/Mo}}(72\%) \quad R_{\text{Ni/Ti}} \sim 9\%$$

from SR $\omega$-scan reflectivity comparison:

$$R_{\text{Ni/Ti}} = \frac{1}{15} R_{\text{Si/Mo}}(72\%) \quad R_{\text{Ni/Ti}} \sim 5\%$$

R$_{\text{Ni/Ti}}$ $\sim 7\%$

Future

Ideal multilayers tailored for the water-window: energy scan simulation of ideal Ni/Ti and Ni/TiO$_2$. 

<table>
<thead>
<tr>
<th>Multilayer Ni/TiO$_x$, N=500</th>
<th>$\Gamma=0.39$, $\Lambda=14.6$ Å</th>
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<tbody>
<tr>
<td>$R_{\text{max}} = 36.2%$ @ 425.8 eV</td>
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<th>Multilayer Ni/Ti, N=500</th>
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<tbody>
<tr>
<td>$R_{\text{max}} = 33.6%$ @ 426 eV</td>
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Conclusions:

The spectral region from 24 to 300 Å is strategical for fundamental research in astrophysics, for projection nano-lithography and for applications of future FEL.

In the so-called water-window between O and C $K$-edges, the X-ray microscopy on organic systems, cells and organelles, becomes important as well as the X-ray microprobe spectroscopy by Synchrotron Radiation.

INFN – Gr.V supports the experiment ARCHIMEDE (ARCHItects of Mirror Euv Devices) between LNL and LNF:

=> tayloring and deposition of multilayers at LNL
=> HV soft X-rays reflectometer set-up at LNF.

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Soft X-ray beamline at DAΦNE

ML mirrors in astrophysics

EIT SUN DISK IMAGE: EMISSION from Fe IX, X at 171 Å (SOHO Mission NASA Goddard Space Flight Center)

Solar disk and corona emission