Extreme-ultraviolet and X- ray Training in Advanced Technologies for Interdisciplinary Cooperation

Optical And Structural Characterization Of Nb, Zr, Nb/Zr, Zr/Nb Thin Films On Si3N4 Membranes Windows

PhD Student

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Host University : **University of Aachen**
Supervisor : **Larissa Juschkin**

Overview

- Motivation
- Introduction
- Project development
- Activities during second year.
- Future activities

Motivation

- High brilliance sources such as synchrotron and Free Electron Laser (FEL) are very important nowadays due to their multiples application in the development of science and technology.

Applications such as:

- Environmental spectromicroscopy and Biomicroscopy.
- Spectromicroscopy of surfaces.
- Surface and material science.
- Chemical dynamics.
- Photoemission spectroscopy of strongly correlated systems.
- EUV interferometry and Coherent optics.
- Atomic and molecular physics studies.
- Magnetic materials and polarization studies.
- Protein crystallography.

- One strong requirement on the beam delivered by these sources besides brilliance, coherence and bandwidth is often related to the spectral purity; in fact the beam can be the superposition of various harmonics.

- The rejection of high harmonics or diffuse light in order to improve the quality of the beam can be achieved by suitable optical systems acting as band pass filters.

This project will be focused in the searching for potential materials, design, fabrication and characterization of self standing transmittance thin films filters between 4-20 nm and 20-100nm spectral range.

ELETTRA AND FERMI Light sources

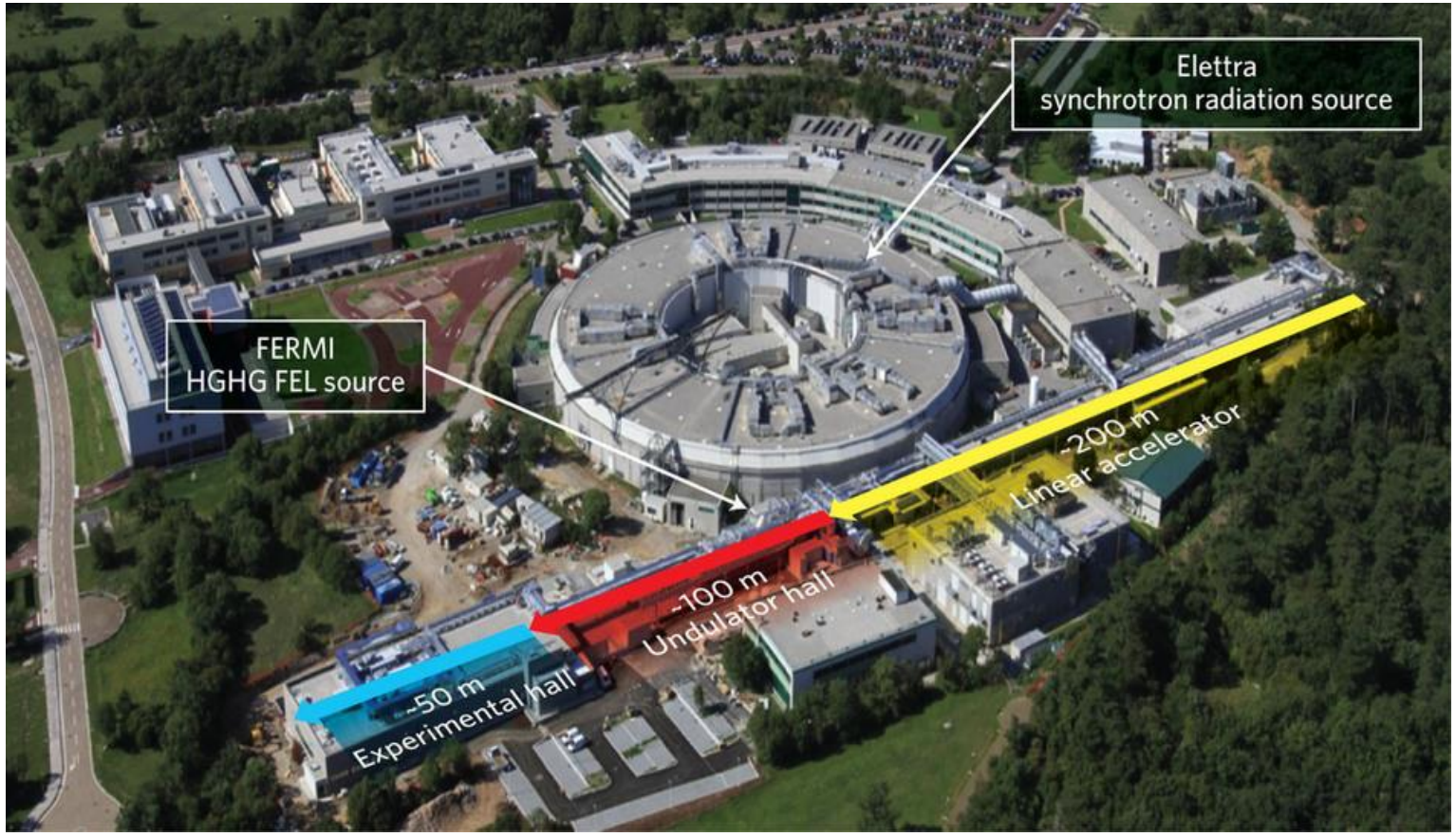
The FERMI FEL in Trieste is endowed with two power sources:

Seeded FELs

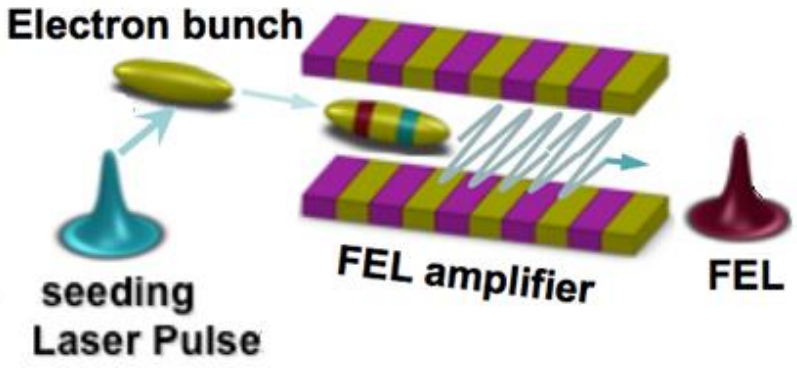
FEL-1 covers the wavelength range 100-20nm.

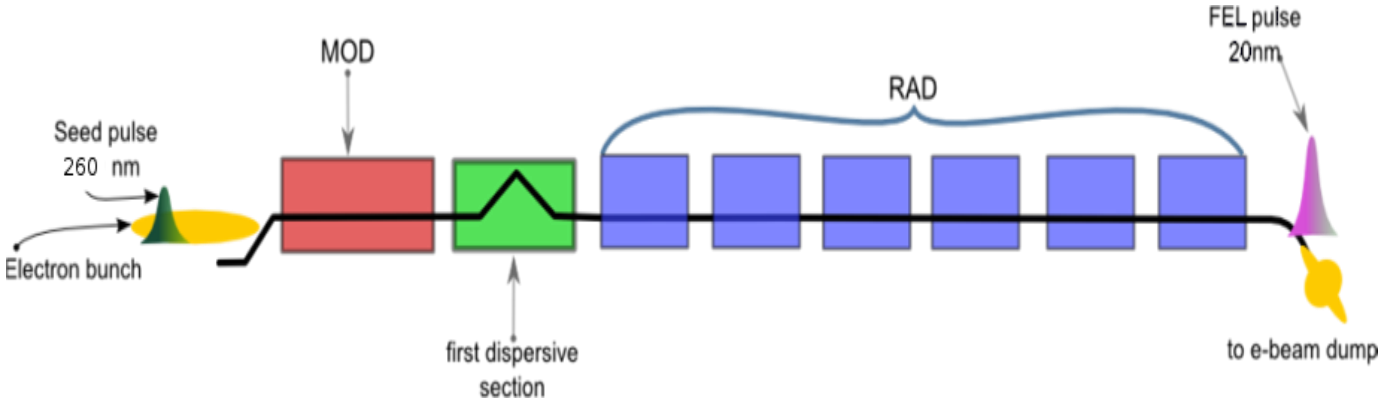
Double cascade for creating short wavelength photons

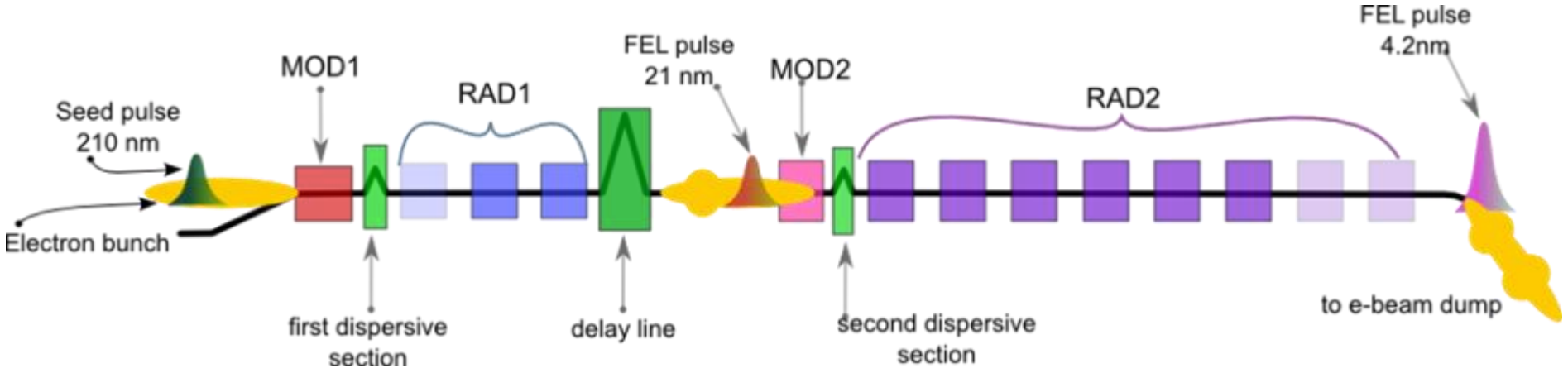
FEL-2 the wavelength range 20-4nm.



FERMI seeding Description







Transmittance Filters

Transmittance: is the ratio of transmitted energy to incident energy.

The transmittance T of a filter respect a particular wavelength is determined by the relationship

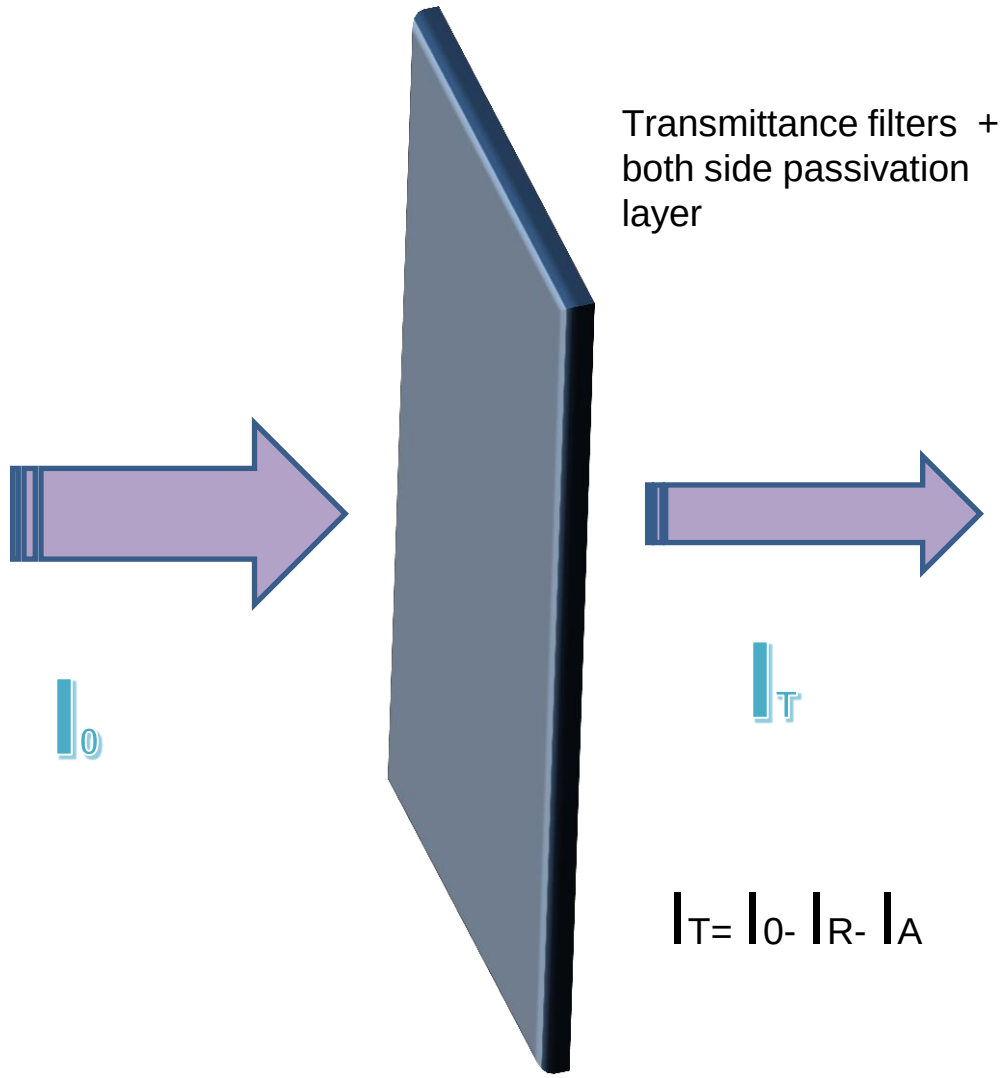
$$T = \exp(-\mu x)$$

μ= linear absorption coefficient at the chosen wavelength

X= thickness of the material.

For composite filters

$$T_{filters} = T_1 T_2 T_3 \ldots\ldots T_n$$



Project Development

Selection of materials

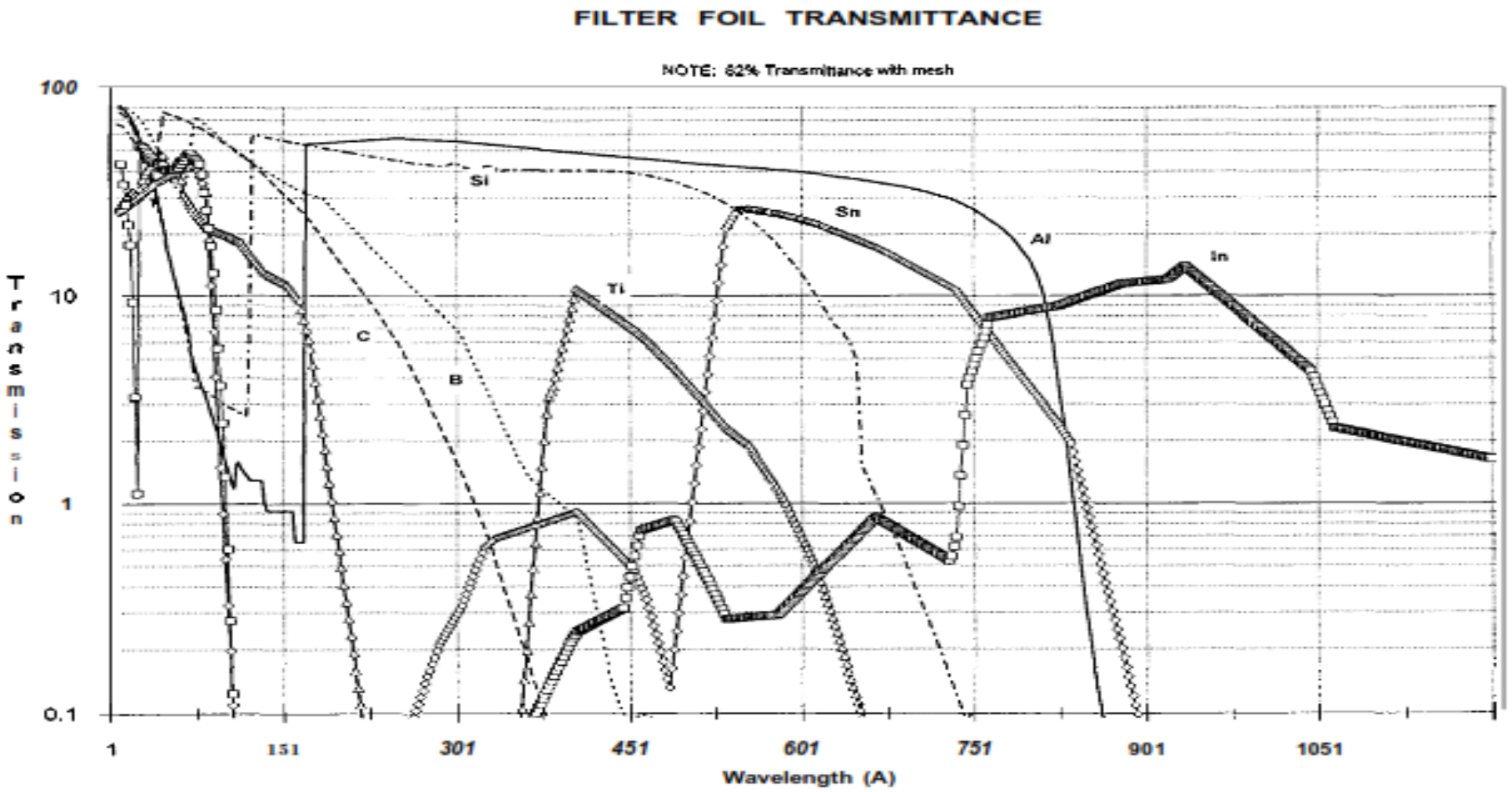


Figure 1. Transmission vs. wavelength for various materials

Niobium And Zirconium

In this stage *our core motivation is to develop thin film transmission filters in the EUV spectral range for applications at FERMI's FEL-2 and the Synchrotron sources.*

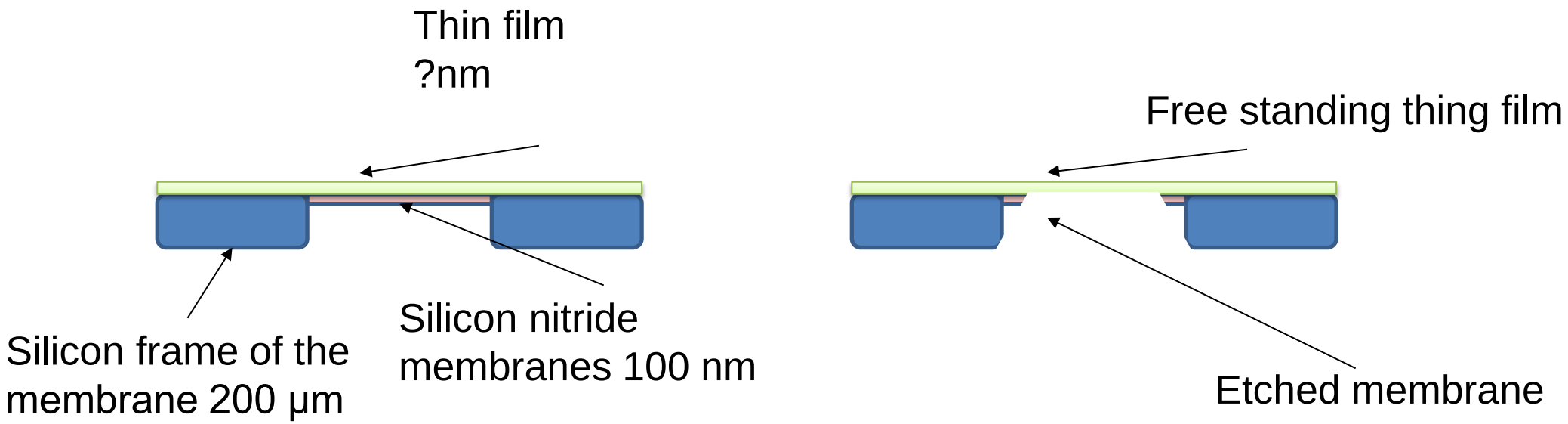
The simulated transmittances of Zr and Nb thin films show high transmittance in the 20 – 4 nm wavelength range that FEL-2 is designed to work,. Bilayers of Zr/Nb and Nb/Zr also show good transmittance as shown,

Transmittance of Nb, Zr, Nb/Zr and Zr/Nb thin film filters in the extreme ultraviolet

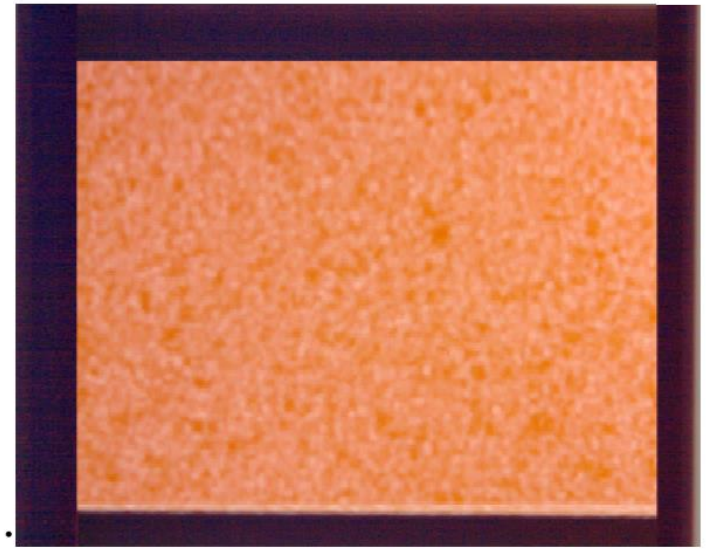
Filters Design

Deposition of materials. Zr, Nb, Zr/Nb and Nb/ zr on Si_3N_4

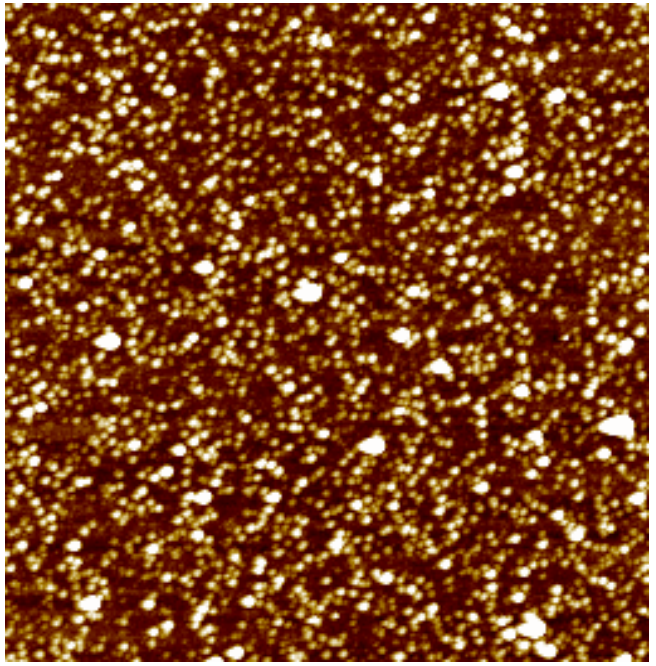
Etch the membrane after deposition to have free standing thin film



Silicon Nitride membrane used as a substrate



Area : 3mmx3mm
Thickness = 107 nm
RI @ 632 nm = 2.097
Roughness= 1.2-1.4 nm

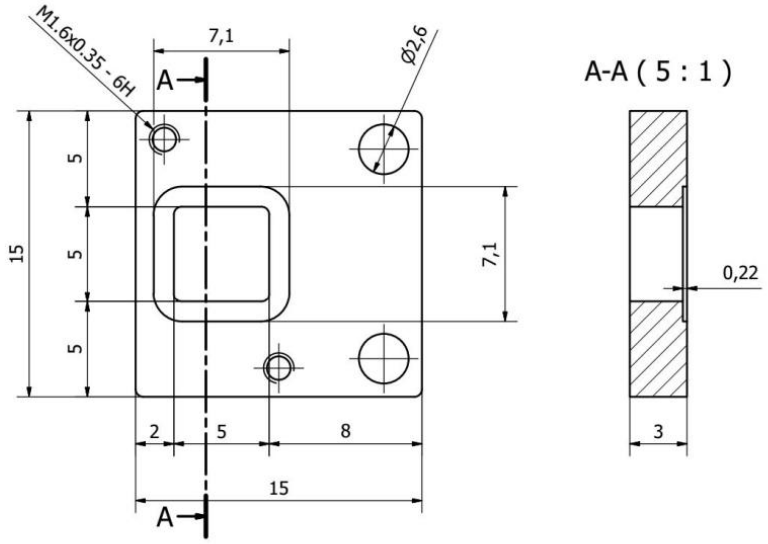


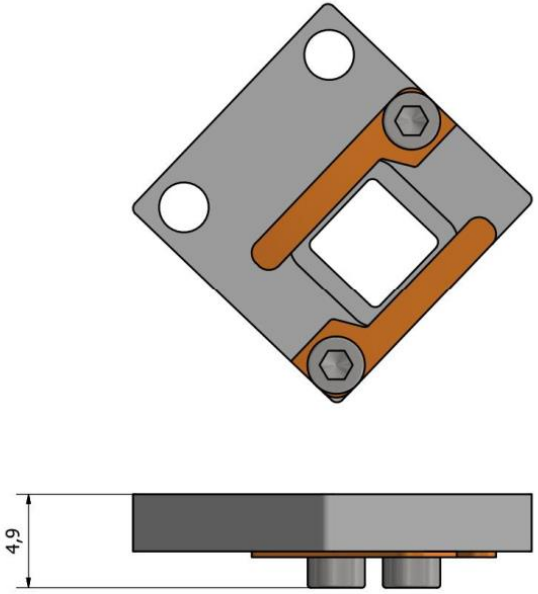
10um

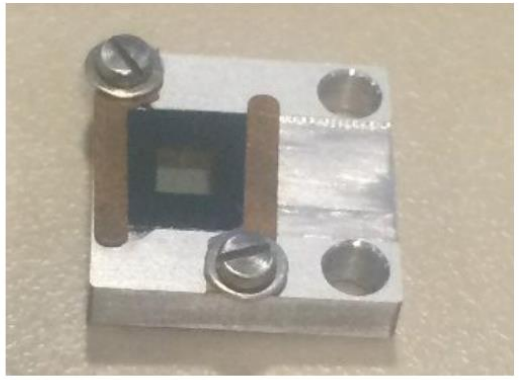
AFM Image

5. Optical Microscope image of Silicon Nitride (Si3N4) membrane.

Sample Holder for the fabrication and characterization of the sample







Sample's holder design and actual picture of a silicon nitride membrane mounted on the holder.

Samples

Table 1 type of Sample to be prepared and analyzed.

Sample Material	Substrate type	Nominal thickness
Zr	Si_3N_4 window	50nm, 75 nm, 100nm
Zr	Silicon wafer	50nm, 75 nm, 100nm
Nb	Si_3N_4 window	50nm, 75 nm, 100nm
Nb	Silicon wafer	50nm, 75 nm, 100nm
Zr/Nb	Si_3N_4 window	50/50 nm, 75/75nm
Zr/Nb	Silicon wafer	50/50 nm, 75/75nm
Nb/Zr	Si_3N_4 window	50/50 nm, 75/75nm
Nb/Zr	Silicon wafer	50/50 nm, 75/75nm

Problems Facing in actual Thin films Filters and the Proper selection of materials

- **Wavelength Target**: Since materials present high absorption in the EUV spectral range, the selection of devices acting as filters in order to select suitable spectral bandwidth or reject harmonics is quite challenging.
- **Fragility**: Transmittance increase with the decrease of the thickness of the materials, but with a small thickness the fragility of the material increase .
- **Oxidation and carbonation contamination.** Environmental exposure can reduce the performance of the filters and contribute to the production of higher harmonics photons.
- **Exposure time to high energy and aging**

Development Facilities

The design, fabrication and characterization of the thin films transmittance filters have been carried on the following facilities.

- Luxor Laboratories, Padova University , Italy.
- Department of Molecular Sciences and Nanosystems Università Ca' Foscari Venezia, Italy.
- CNR - IMM Istituto per la Microelettronica e Microsistemi e Instituto di Fotonica e Nanotecnologie. Sede Secondaria di Roma.
- Optoelectronic institute at Warsaw University of Technology. Warsaw, Poland.
- Bessy Syncrotron , Optics beamline. Berlin, Germany.

First Set of Samples

Table 2 AFM measurement results

Nominal thickness	AFM measurement, real thickness	Roughness
Zr 50nm	41nm	0.80nm
Zr 75nm	62nm	0.85nm
Zr 100nm	85nm	1.01nm
Si_3N_4 100nm	---	1.40 nm

Samples were deposited using E-beam deposition Technique at CNR - IMM Istituto per la Microelettronica e Microsistemi via del Fosso del Cavaliere 100 - 00133 Roma (ITALY)

Measurements performed:

- AFM Images.
- Thickness and roughness measurements.
- Reflectometry measurements.

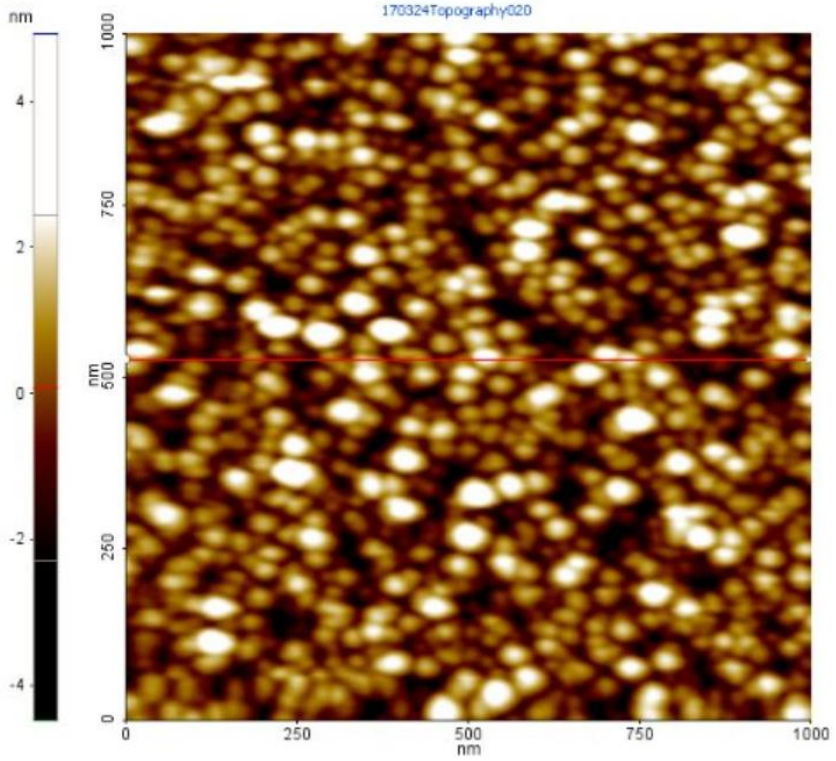
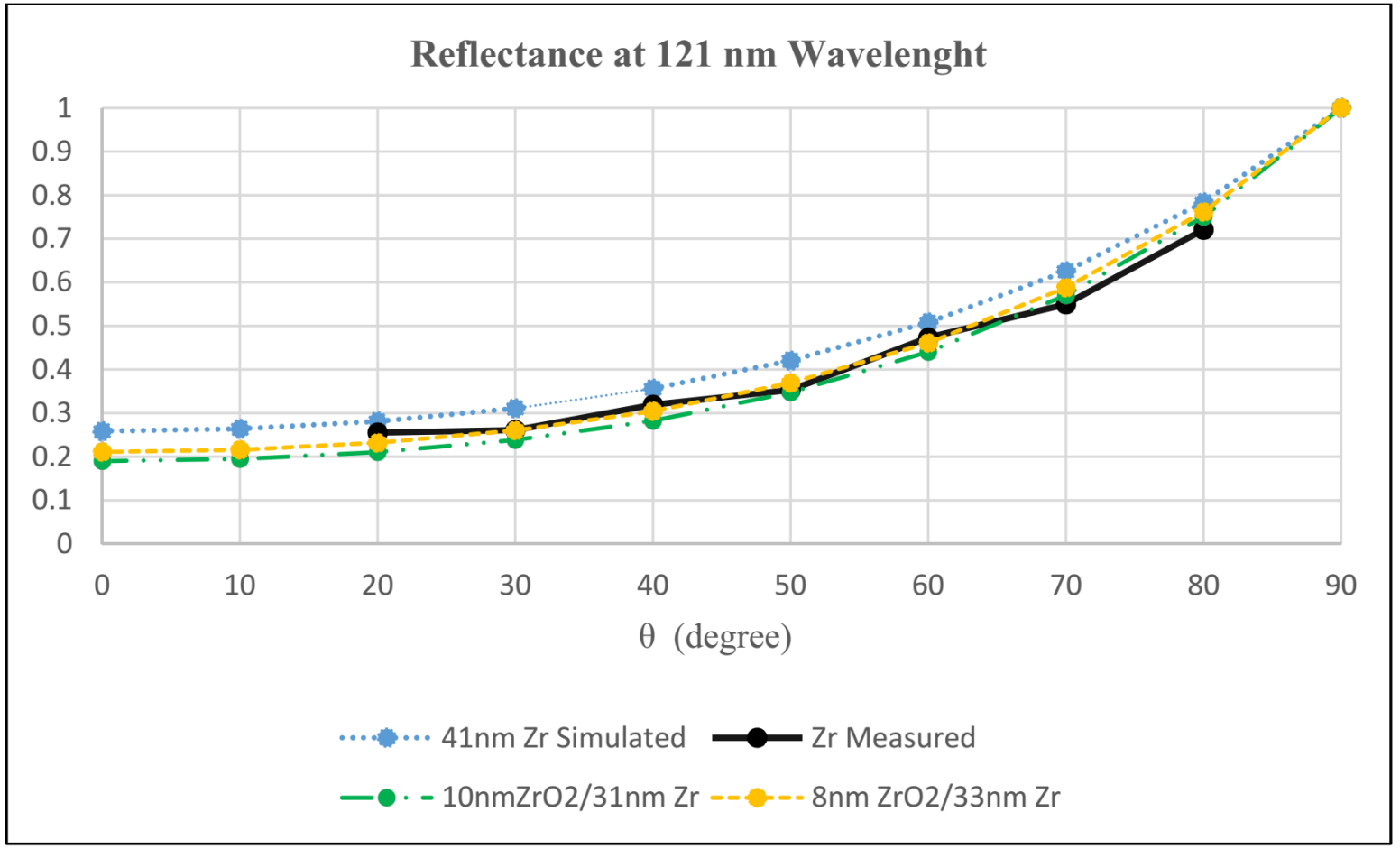


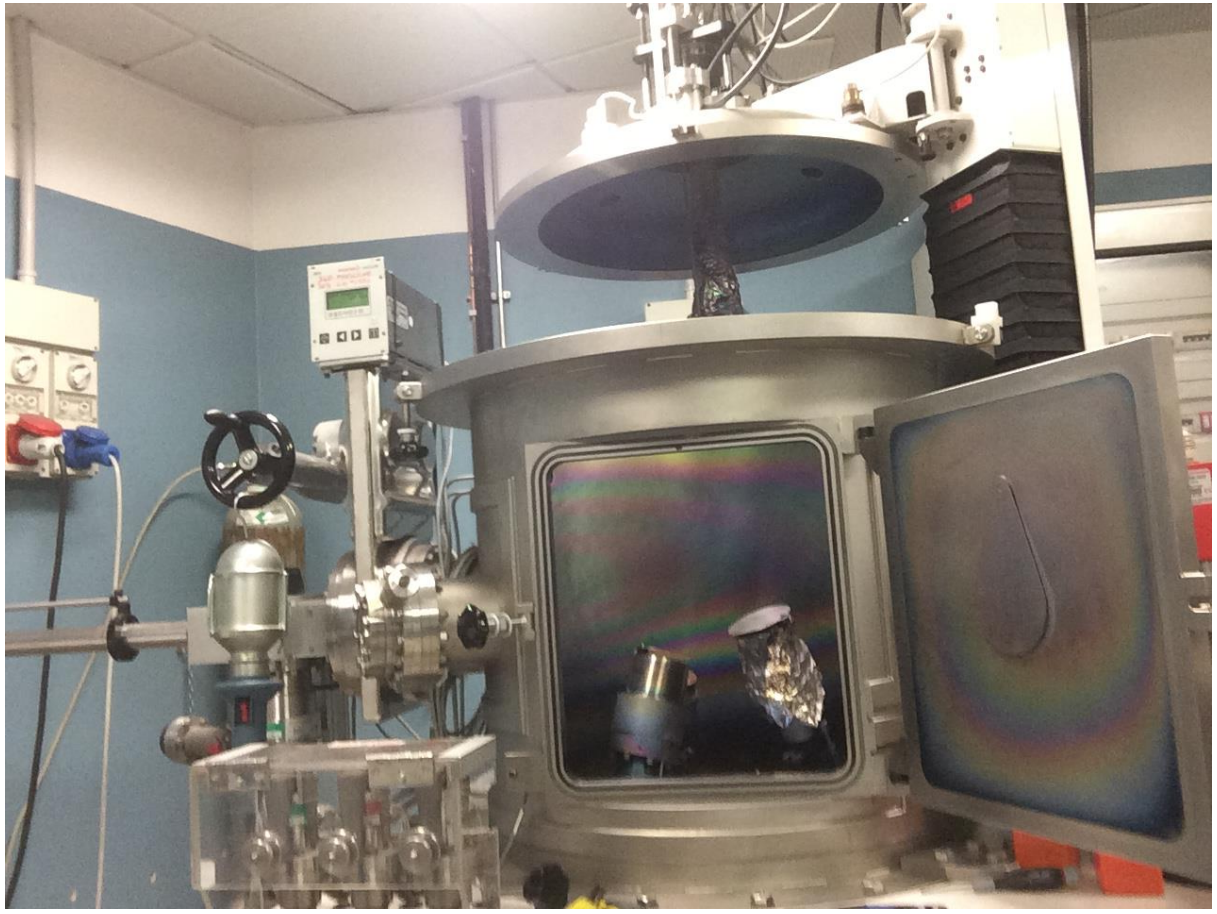
Figure 6. 2D topography AFM image of Zr/Si sample 100 nm Thickness

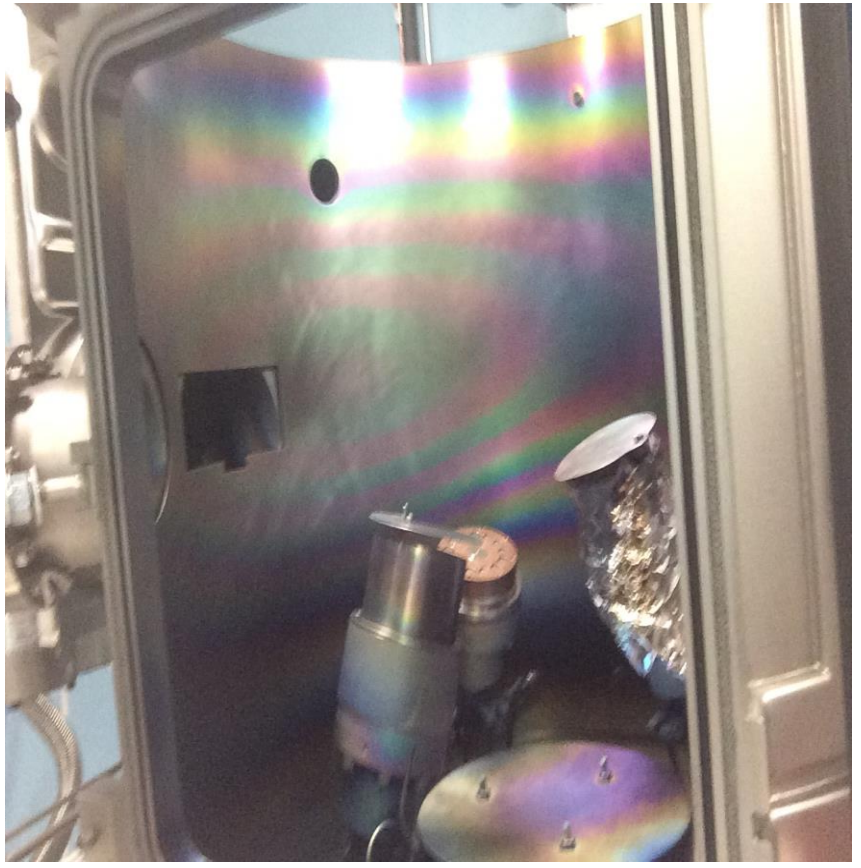
The FUV-EUV normal incidence reflectometer Facility located in LUXOR, Padova, Italy

Reflectometry of sample of Zr, 41nm of thickness



Second set of Samples

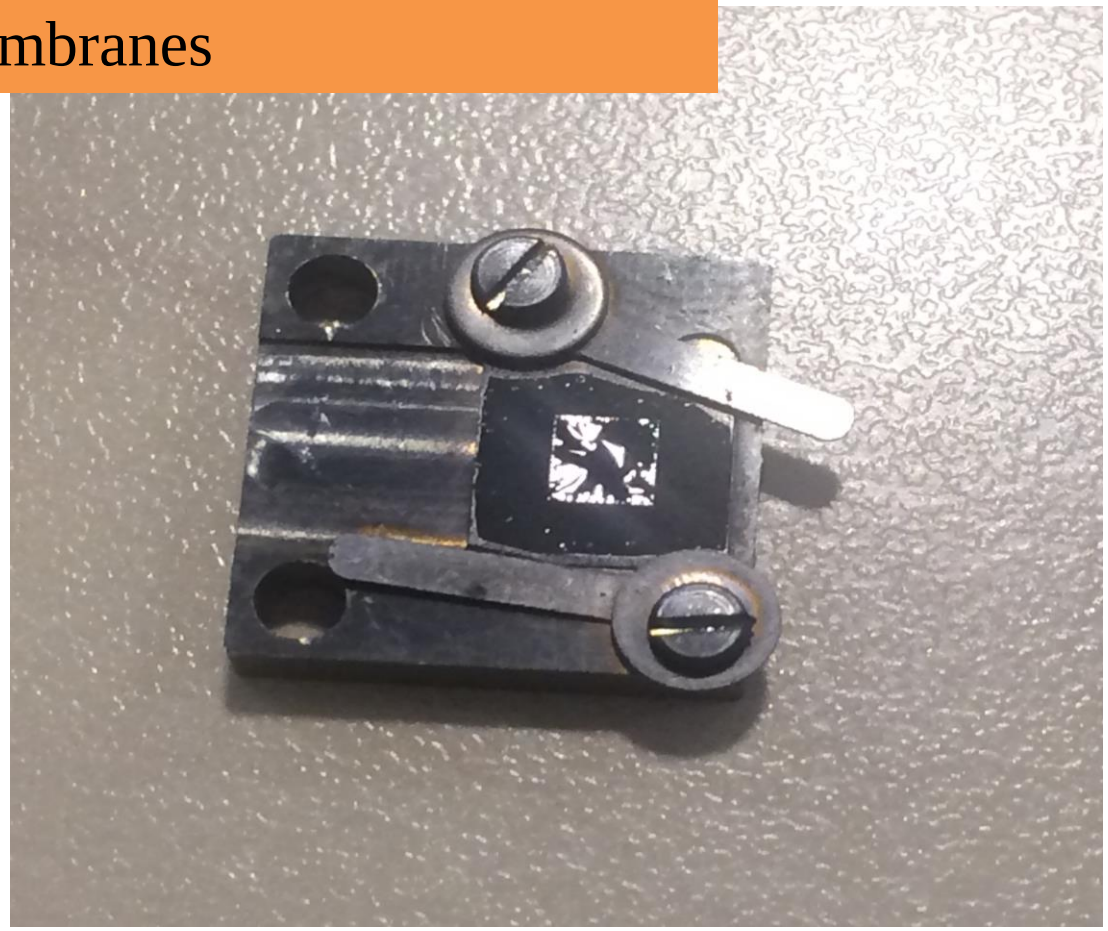




Magnetron Sputtering at the Department of Molecular Sciences and Nanosystems Università Ca' Foscari Venezia

Samples obtained

Structures of samples of Zr and Zr/Nb on silicon nitride membranes



Structures of samples of Nb and Nb/Zr on silicon nitride membranes

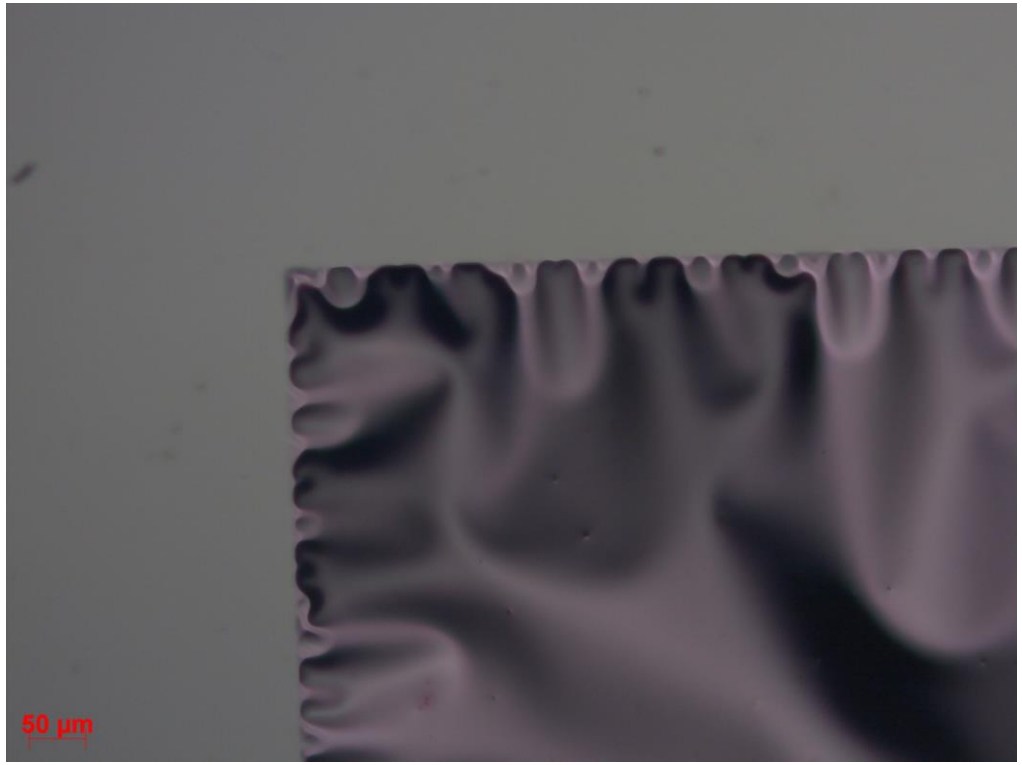
Rutherford backscattering spectrometry Results

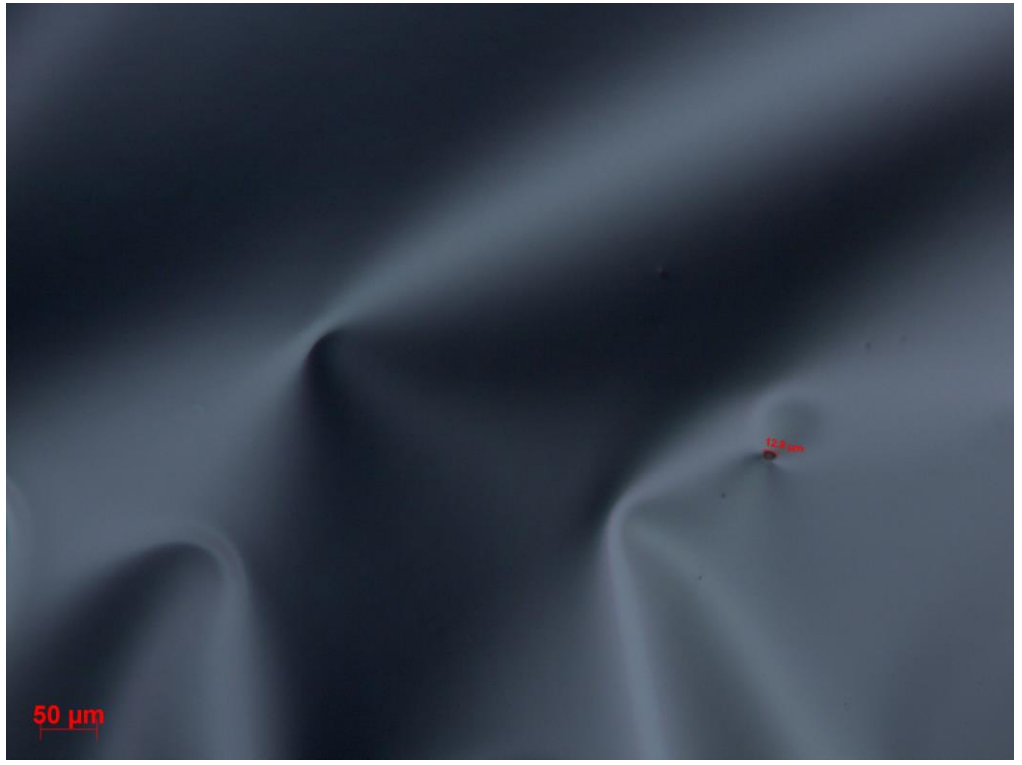
Sample	Nominal Thickness	RBS thickness Calculation	Mean *Stoichiometry*
#372 Zr	141 nm	108 nm	$ZrO_{1.1}$
#373 Nb	120 nm	112 nm	$NbO_{0.4}$
#374 Zr/Nb	50/50 nm	37/52 nm	$ZrO_{1.2}/Nb_{0.7}$
#375 Nb/Zr	75/75 nm	77/58 nm	$NbO_{0.2}/ZrO_{0.6}$
#376 Nb/Zr	59/50 nm	52/56 nm	$NbO_{0.05}/ZrO_{0.3}$
#377 Zr/Nb	75/75 nm	86/76 nm	$ZrO_{0.7}/NbO_{0.1}$

RBS conclusions

- Contamination of Hf in the Zr target of 1.1-1.2%.
- The presence of oxidation on Zr is more elevated than the presence of oxidation on Nb.
- All samples show a gradient oxygen concentration, increasing in the interface and it get reduce when is near the surface (contamination during the synthesis), increasing again in the surface contamination due exposition to the air), this behavior is higher when Zr is on the top.

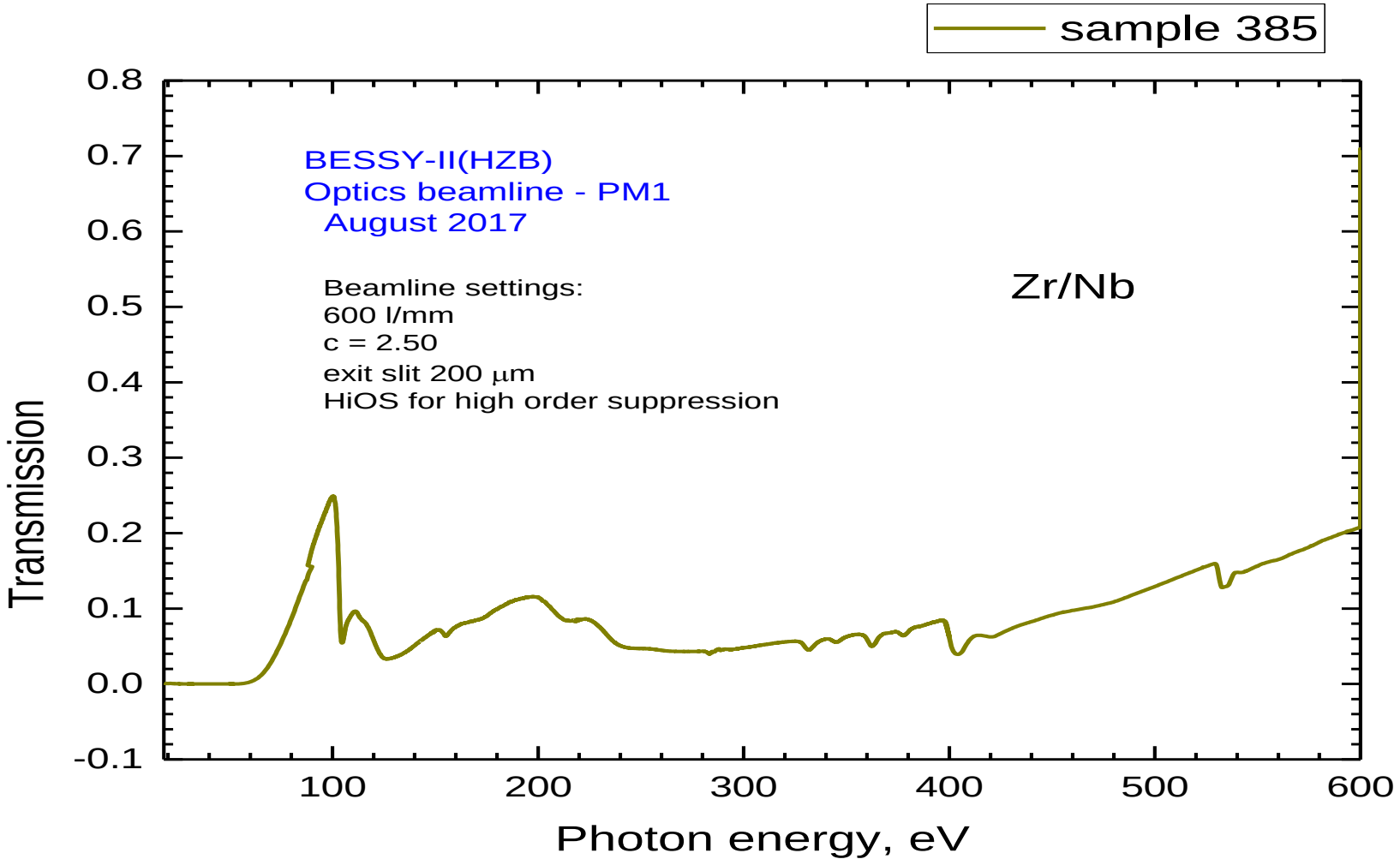
Structures of samples of Zr and Zr/Nb on silicon nitride membranes



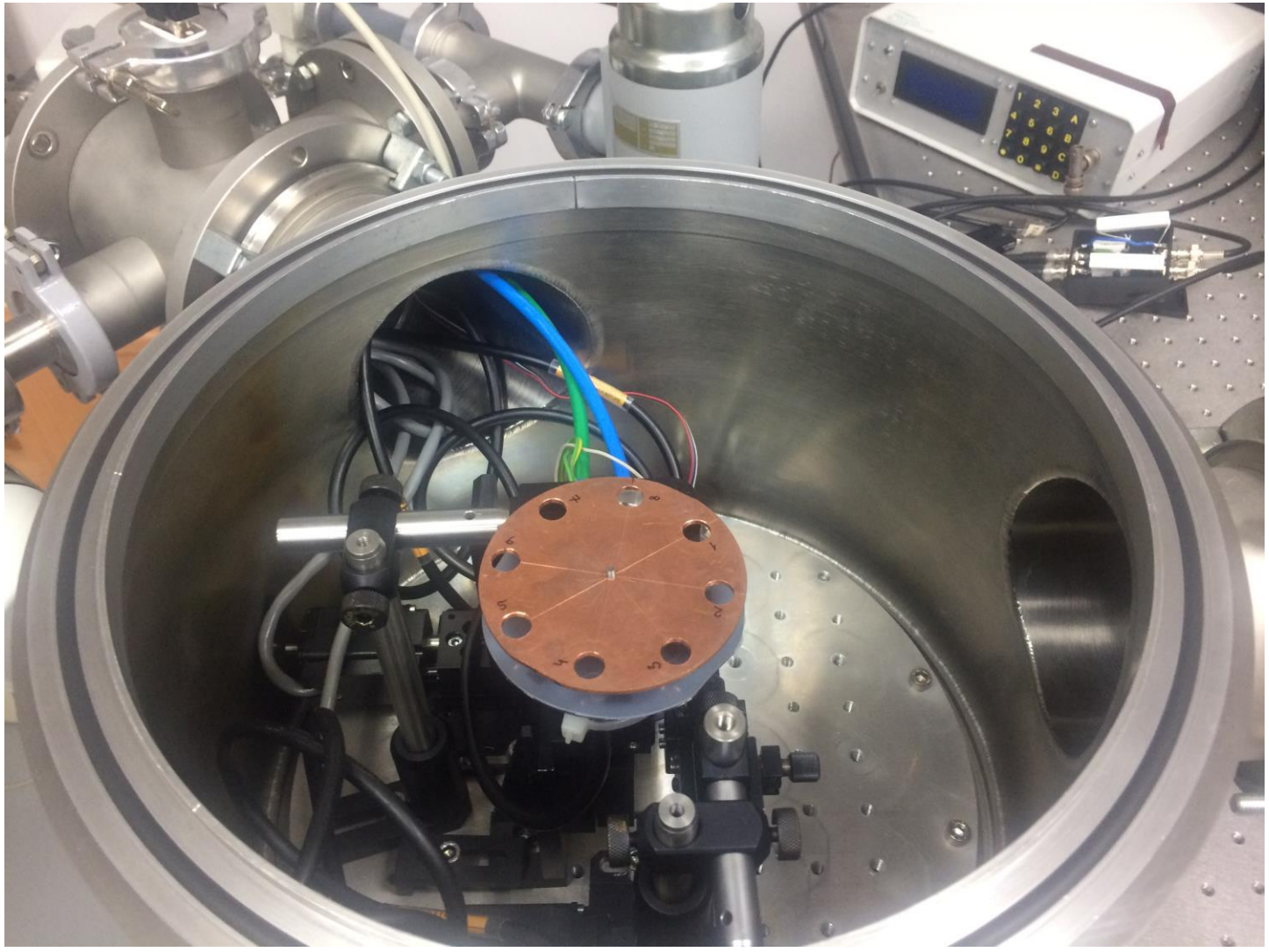


Structures of samples of Nb and Nb/Zr on silicon nitride membranes

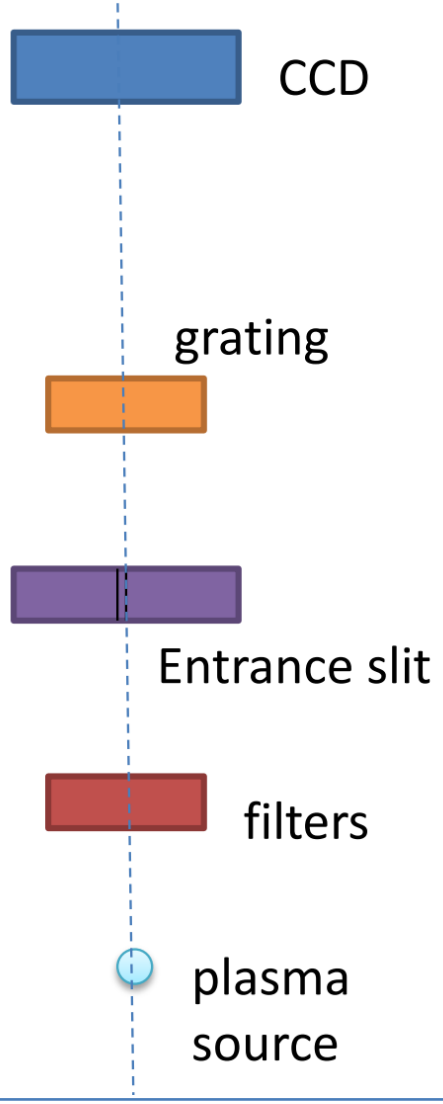
Transmission measurements at Bessy

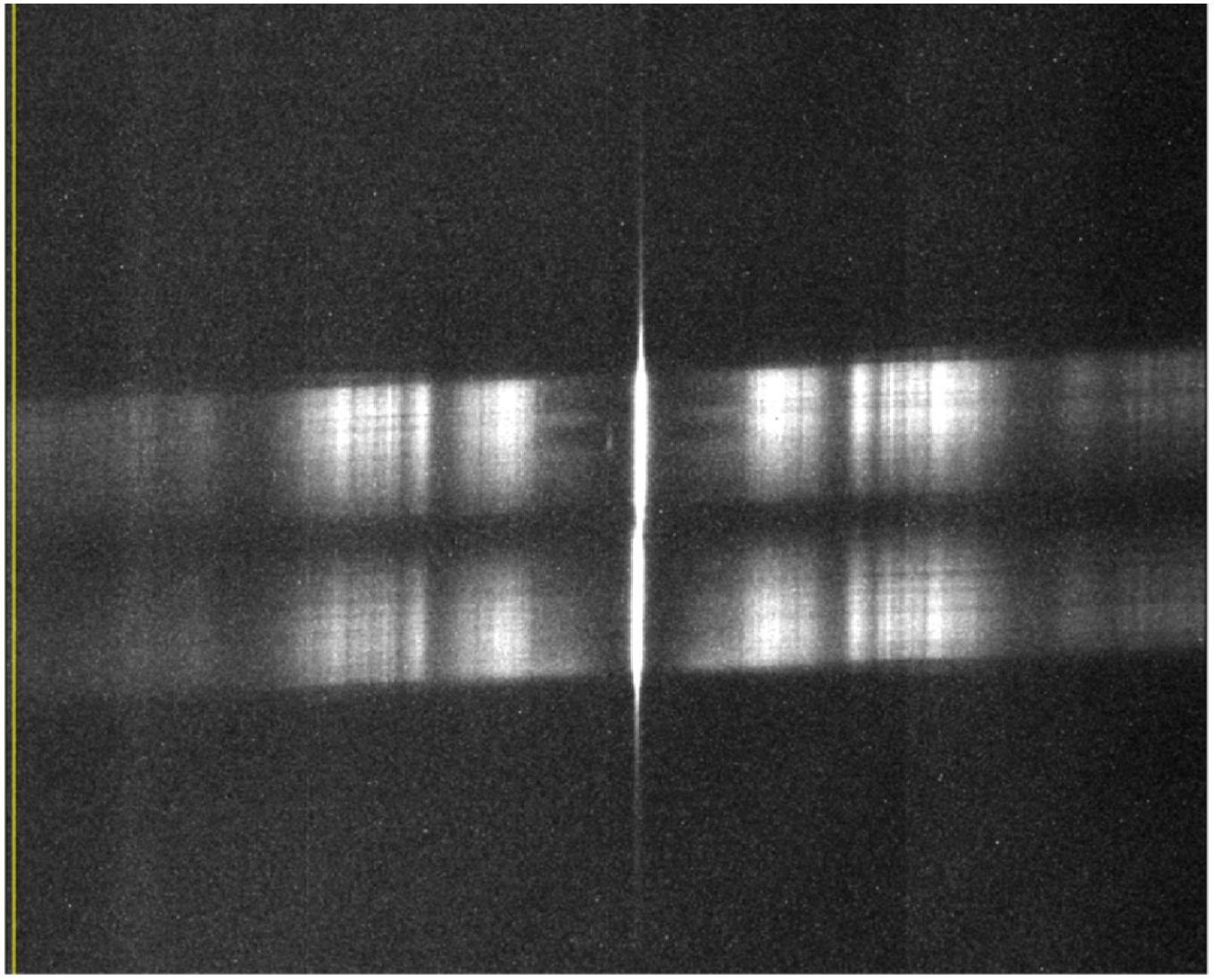


University of Warsaw optoelectronics facilities



Transmittance measurements Setup





Activities

PART 1 - COURSES, CONFERENCES

Seminars

- *"Functional materials for astronomical instrumentation" by Dr Andrea Bianco (INAF – Osservatorio Astronomico di Brera) on Thursday July 6th in room 201/DEI, 11am.*

Participation to International Conferences and Workshops

- ***Poster presentation:*** *Design, Development and characterization of thin film filters for high brilliance sources in the UV-X-ray Spectral range. PXRNMS 2016, Multilayer-workshop2016 (TNW), November 2016, Twente University, Twente, Holand.*
- ***Poster presentation***: *Optical and structural characterization of Nb, Zr, Nb/Zr, Zr/Nb thin films on Si3N4, SPIE Optics + Optoelectronics, 2017, Prague, Czech Republic.*

- ***Oral Presentation***. Extatic Welcome Week, 16-20 January 2017 at the International Centre for Theoretical Physics in Trieste, Italy.

2 Other learning activities

- Extatic Welcome Week, 16-20 January 2017 at the International Centre for Theoretical Physics in Trieste, Italy.
- Training at Bear Beamline, Elettra Syncrotron, Trieste; November 2016.
- Educational visit to Fermi Free Eletron Laser, Trieste; May 2017.

- Proposal submited at Bear Beamline*: EUV, Soft x-rays Characterization and Study of High energy EUV Radiation Damage on of Nb, Zr thin films on Si3N4 membranes windows for transmittance filters application.* The proposal was not allocated but classified as middle top, with final comment:
- *" Straightforward characterisation of Nb/Zr thin-films for use as EUV and soft x-ray filters subject to high-power irradiation. This will be useful support for XFEL experiments".*

PART 3 - PUBLICATIONS

List of publications on conference proceedings

- ***Optical and structural characterization of Nb, Zr, Nb/Zr, Zr/Nb thin films on Si3N4 membranes windows***

  K. Jimenez; A. E. H. Gaballah; Nadeem Ahmed; P. Zuppella; P. Nicolosi

Proceedings Volume 10236, Damage to VUV, EUV, and X-ray Optics VI; 102360K (2017); doi: 10.1117/12.2267348
Event: SPIE Optics + Optoelectronics, 2017, Prague, Czech Republic

Future Work

- Analysis of transmission measurements data obtained in the Optoelectronic facilities at Warsaw University.

- Study stress mechanism in the samples.

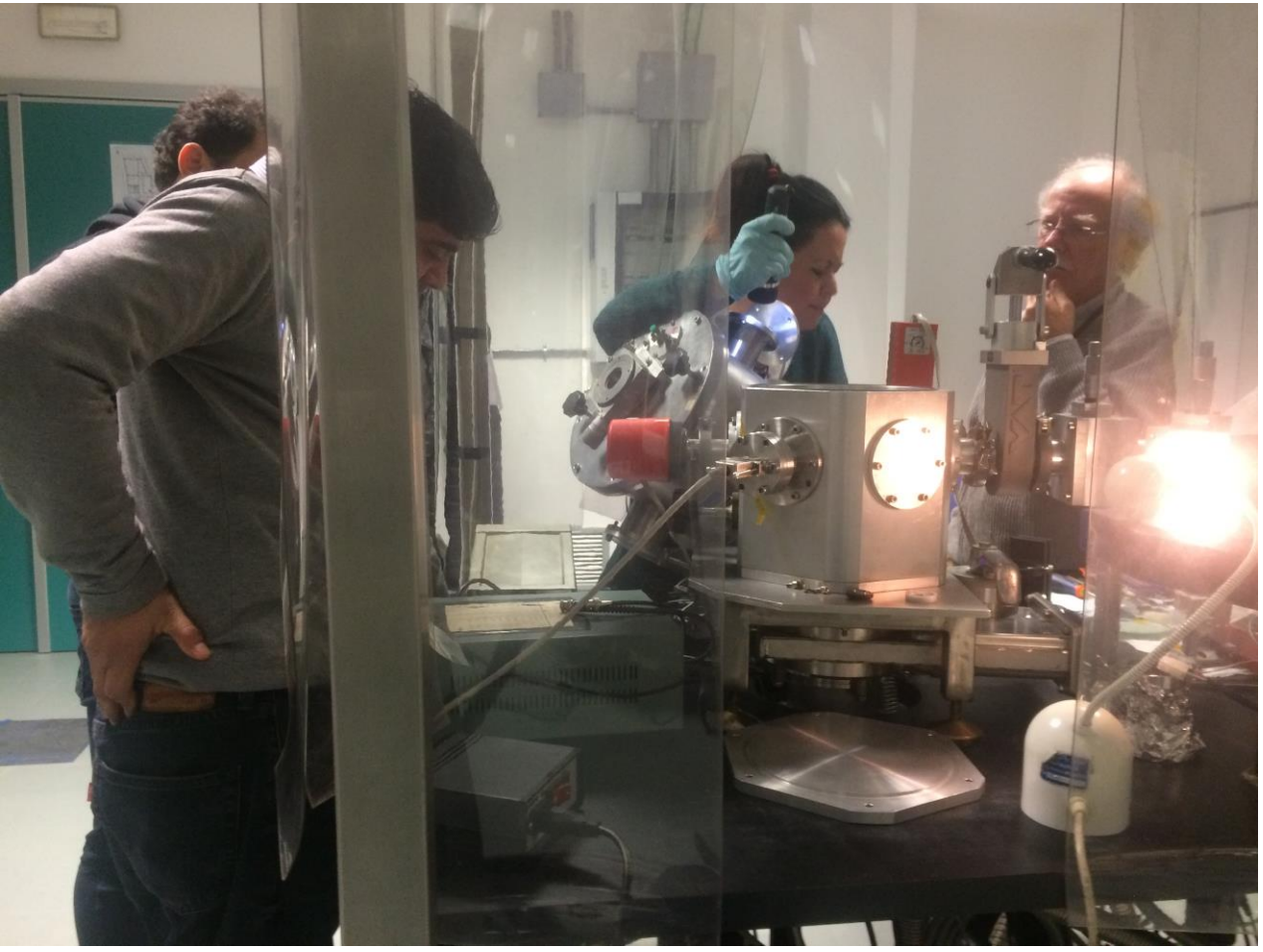
- Selection of new material for new transmittance filters and looking coating solution to avoid oxidation on the surface of the samples.

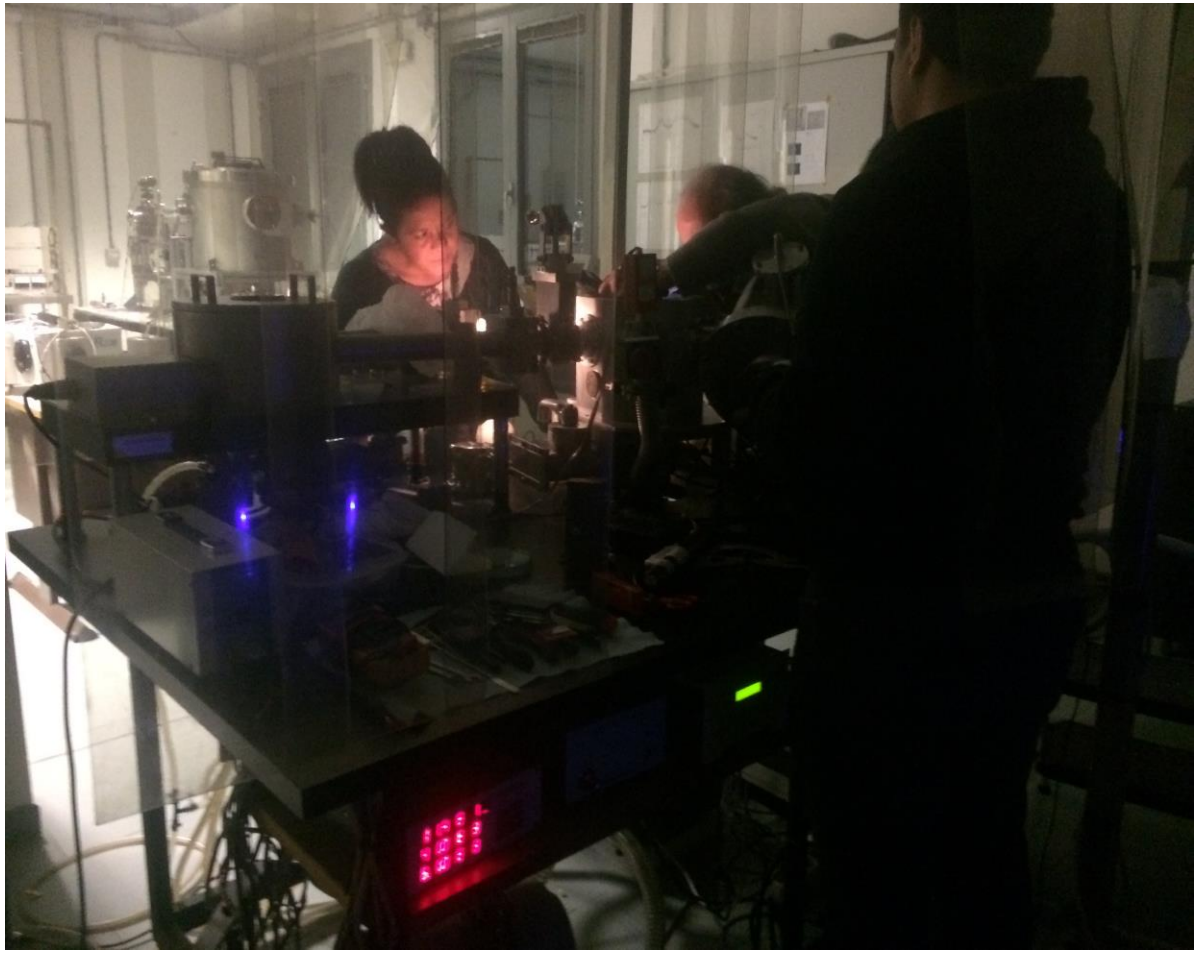
upcoming Activities

- Mobility to Aachen University, Confirmed to Starts in October 1, 2017.
- Poster Presentation accepted at the 3rd International Workshop on Frontiers of X&XUV Optics and its Applications to be held in the conference room of the Institute of Plasma Physics AS CR, Prague, Czech Republic, on October 4-6, 2017.
- Poster Presentation accepted at the PTB's 304. Seminar "VUV and EUV Metrology". October 19, 2017.

EXTATIC

Erasmus Mundus





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- Professor Henryk Fiedorowicz and professor Przemysław Wachulak; Optoelectronic institute at Warsaw University of Technology.

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