Nanostructured Rhodium Films for Advanced Mirrors Produced by Pulsed Laser Deposition

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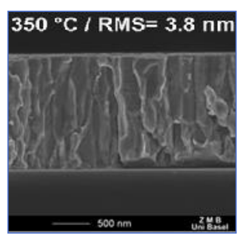
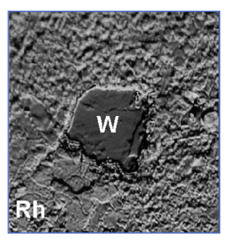
Abstract and motivations

Thanks to its high reflectivity and low sputtering yield, Rhodium is one of the best candidates for the development of metallic coatings to be used as *first mirrors*, required in many diagnostic systems of thermonuclear magnetic fusion machines. Since those devices work in a harsh environment, high priority issues about mirrors production are the optimization of thermo-mechanical and resistance properties to ensure good behaviour under operative conditions.

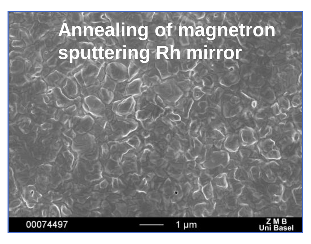
Deposition techniques exploited so far, like electrodeposition (Orsitto et al., Rev Sci Inst 2001), magnetron sputtering (Marot et al., Surf &Coat Tech 2008) and evaporation, were successful in depositing films with the desired thickness and extension, but showed problems regarding roughness, recrystallization and high temperature behavior.



Marot et al., Surf &Coat Tech 2008



Matveeva et al., 37[th] EPS Conf. 2010

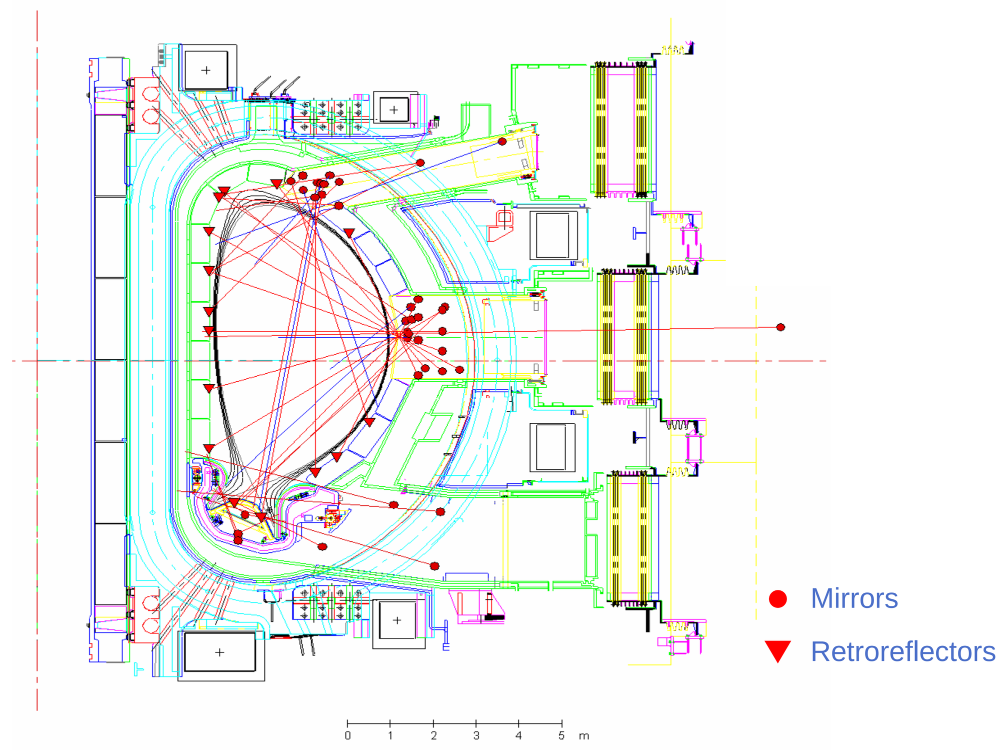


Marot et al., ITPA14 2008

In this work we use Pulsed Laser Deposition (PLD), to deposit Rh films with suitable properties. By varying the process parameters (laser fluence, background pressure, substrate temperature) it is possible to tailor the nanocrystalline domain size of the deposited films, down to less than 5 nm and separately control the other relevant features.

The films have been characterized by XRD, AFM, SEM and Ellipsometry. Adhesion has been tested with scratch tests.

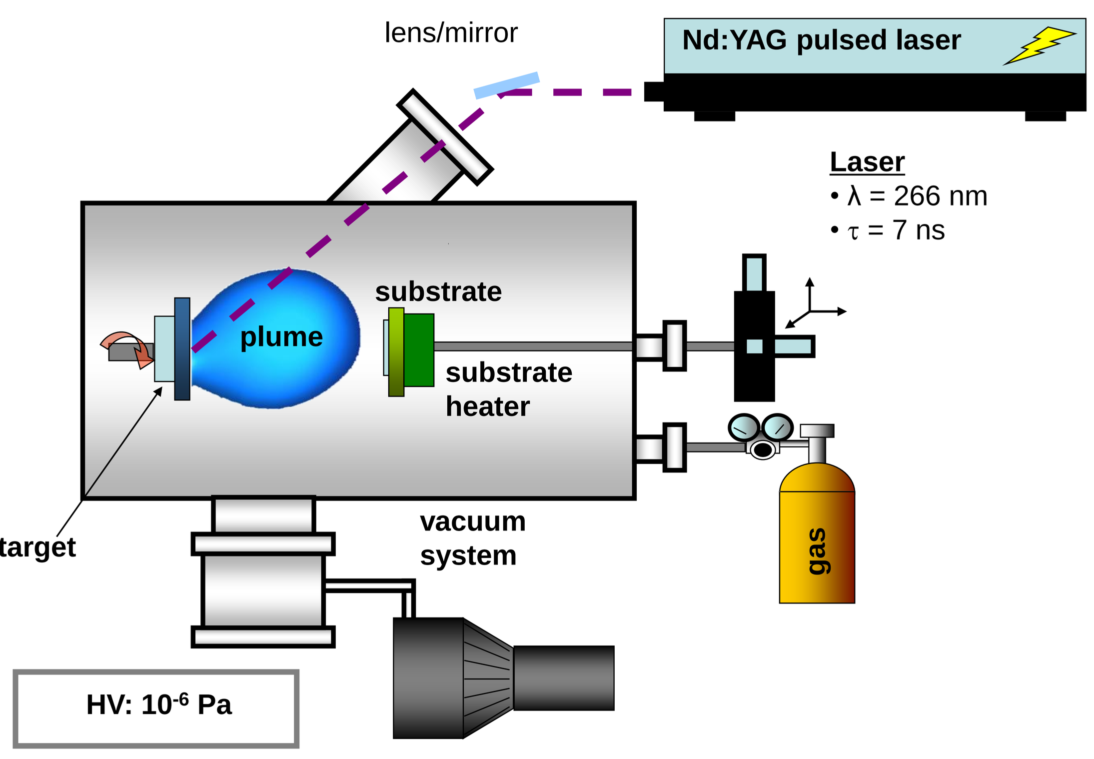
Mirrors in ITER



More than 50 mirrors planned for ITER diagnostics

T. Sugie et al. 12th Meeting of the ITPA TG on Diagnostics 2007, Princeton USA

Pulsed Laser Deposition



Deposition parameters

- Substrate Temp. T: 30°- 200°C
- He pressure P: 0-30 Pa
- Laser fluence F: 1-3 J/cm^2
- Target-sub. dist. d_{t-s}: 5-6 cm

Target

- Metallic Rhodium

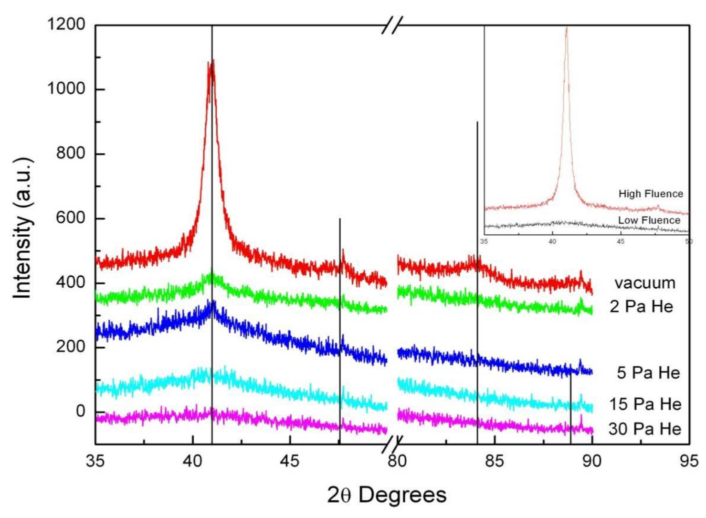
Substrates

- Si(100)
- Molybdenum

Nanocrystalline Rh films

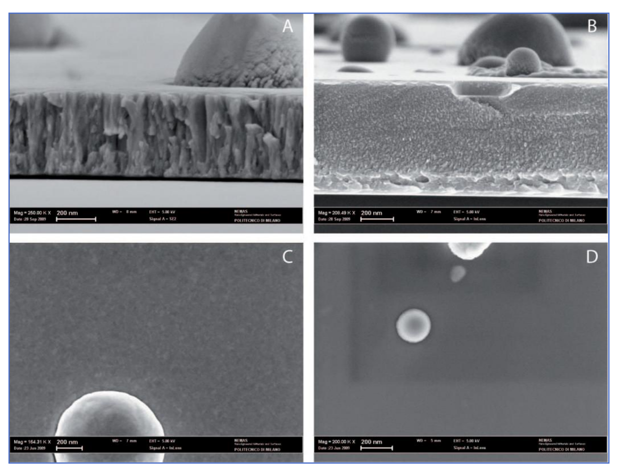
M. Passoni et al., J. Nucl. Mater. (2010), in press doi:10.1016/j.jnucmat.2010.06.015

XRD analysis



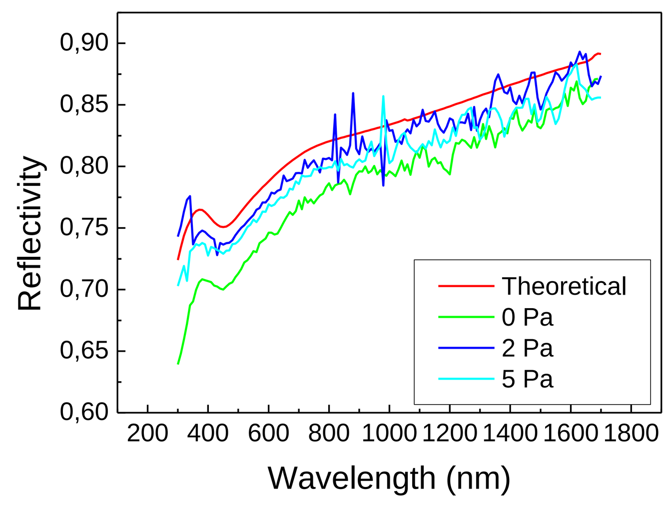
Role of deposition pressure and fluence

SEM analysis

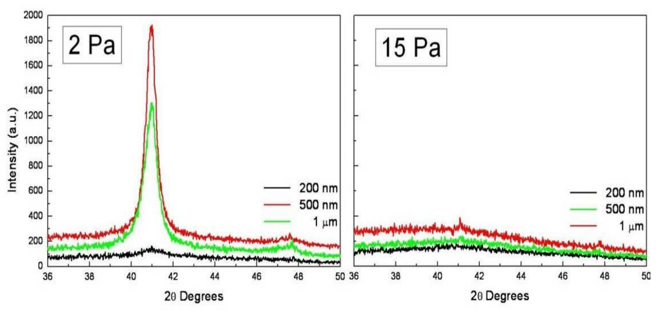


Films deposited in vacuum (a-c) and at 15 Pa He (b-d)

Specular Reflectivity

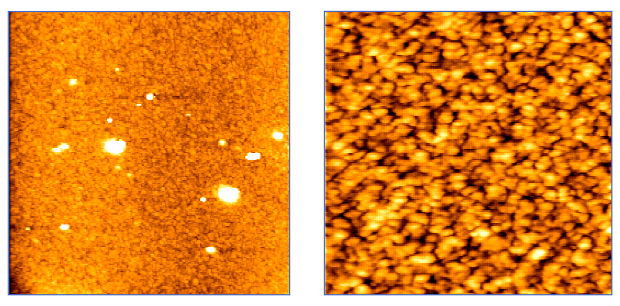


Reflectivity spectra collected using ellipsometry



Dependence on thickness at different pressure

AFM analysis



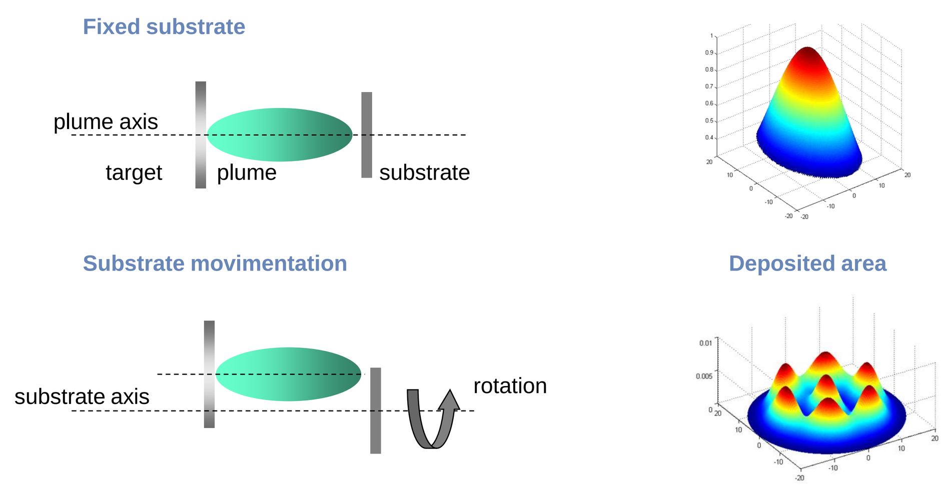
Films (200 nm) on Si with scan size: a) 3 µm, b) 10 µm

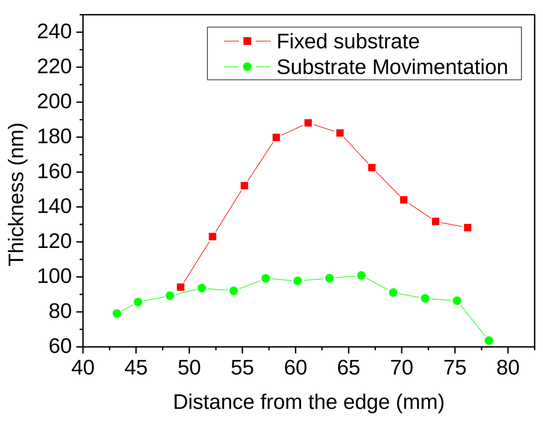
Deposition pressure (Pa)	Roughness RMS (nm)
0	0.3881
2	0.2176
5	0.4691
15	0.8867

Rh film roughness calculated using AFM analysis

Towards a realistic Rh mirror

Uniform Flat films of wide area





Effects of substrate movimentation

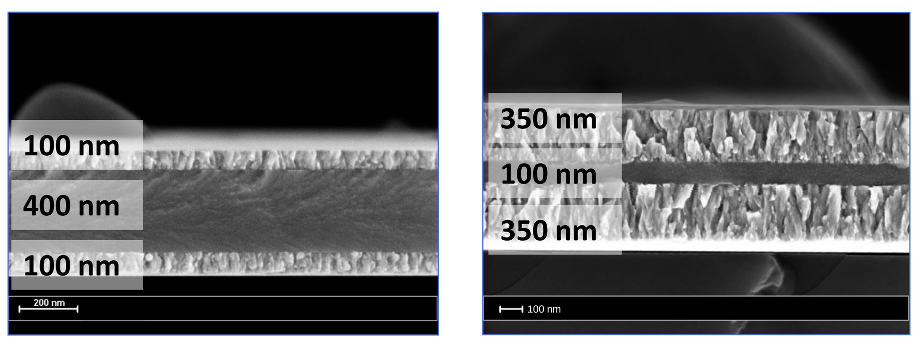
- large area (Ø up to several cm) with improved film uniformity (differences within 20 nm)
- effective deposition rate: 4,5 nm/min
- uniform thickness on suitable areas for a mirror device becomes possible

Thick films

Strategies:

Realization of multilayer films

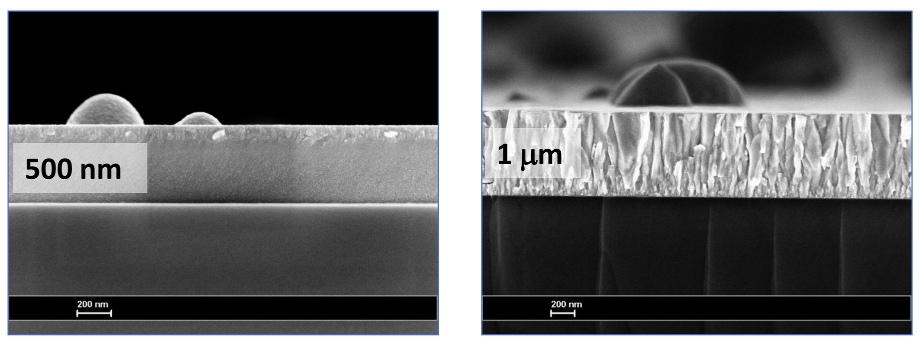
Insertion of amorphous-like layers between columnar ones to reduce stress (no delamination and buckling phenomena) → thickness ~ µm



Optimization of laser parameters

Increase of repetition rate (20 Hz)
- deposition rate: 10 nm/min

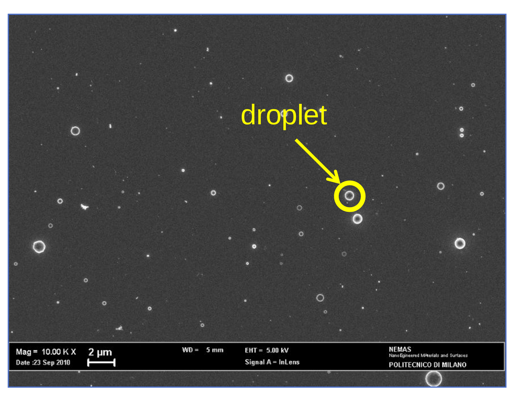
Increase of pulse energy (700 mJ)
- deposition rate: 25 nm/min

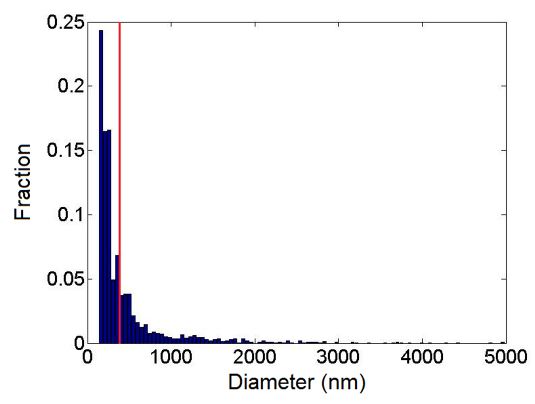


Film quality: reflectivity and mechanical behavior

Droplet properties and effects on mirror reflectivity

- mean coverage of mirror surface: <1%
- 70% of droplets has dimension < 400 nm (histogram area below red line)
- minor effects expected on mirror reflectivity
- optimization possible and foreseen working on the deposition parameters





Droplet size distribution

Mechanical behavior (Scratch tests)

Critical load on Mo substrate:

- columnar structure corresponds to better adhesion
- optimization using multilayer structure

Film morphology	Critical Load (N)
Columnar (0 Pa)	12,27
Amorphous-like (15 Pa)	8,9
Amorphous-like (30 Pa)	2,48

Behavior in operative conditions

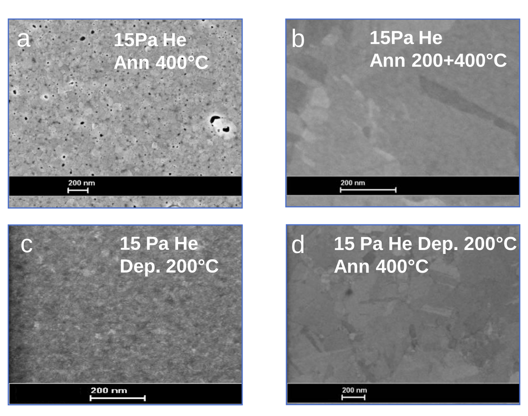
Temperature effects

a) Annealing @ 400°C
- surface modifications due to partial recrystallisation
- non negligible reflectivity degradation (presence of nano-voids)

b) Combined annealing @ 200°C+400°C
- no evidence of nano-voids
- significant growth of crystalline domains

c) Hot substrate depositions (@ 200°C)
- surface structural changes can be partially overcome with depositions on a heated substrate (no presence of nano-voids)

d) Hot substrate deposition + annealing @ 400°C
- Less recrystallisation than annealed sample deposited @ RT



Erosion/redeposition effects on mirror reflectivity

- PLD production of Rh mirrors
- PLD production of C, W coatings/dusts/clusters
- Laser cleaning investigation

Irradiation tests

- exposure to ion gun (fluence: ~10^{20} ions/cm^2)
- SOL of FTU TOKAMAK
- X-ray irradiation @ DAΦNE-INFN

Mo substrate for tokamak testing on FTU

DAΦNE-INFN

Conclusions and perspectives

PLD deposition of Rh films

- Fine tailoring of structure and morphology obtained achievement of nanocrystalline structure (domain size < 5nm)
- Very smooth surfaces (roughness < 1 nm)
- Uniform thickness on suitable areas (up to 12 cm^2) obtained with different strategies (in particular multilayer films)
- Characterization of adhesion properties
- Temperature modifications investigated
- Various irradiation tests are foreseen