

Tests of rhodium -coated molybdenum first mirrors for ITER diagnostics*

G. Maddaluno¹, M. Di Fino¹, F. Orsitto¹, A. Rufoloni¹, M. Montecchi², E. Nichelatti², E. Serra², A. Litnovsky³, G. Sergienko³, G. De Temmerman⁴, L. Marot⁴

¹Associazione EURATOM-ENEA sulla fusione, Frascati, Italy; ²ENEA – C.R. Casaccia, S. Maria di Galeria, (Italy); ³Institut für Energieforschung - Plasmaphysik, Ass. EURATOM-FZJ, TEC, Forschungszentrum Jülich, Germany; ⁴Institute of Physics, University of Basel, Switzerland

*Work carried out under EFDA Task TW5-TPDS-DIADEV

Introduction

Metal first mirrors are foreseen for ITER optical diagnostics [1]. Mirror performance degradation due to erosion and impurity deposition are of concern. Rhodium is a promising material for the first mirrors because of its good optical reflectivity, high melting point and low sputtering yield [2]. In this paper the manufacturing of reflecting Rh coatings on polycrystalline molybdenum (Mo) substrates and their morphological and optical characterisation are reported, followed by the description of the exposure of one out of them in the TEXTOR scrape-off layer and the subsequent new characterisation.

Mirror manufacturing and characterization methods

The polycrystalline Mo substrates ($\varnothing = 18$ mm) was polished by using small-dimension diamond powder. Substrates were ultrasound cleaned with trichloroethylene and isopropyl alcohol and washed with deionised water. Rhodium was electro deposited without intermediate layer and successively polished. Several tests on Mo sample targets were carried out, to optimize the Rh concentration in the electrolyte as well as the current density. The thickness of Rh coatings was $\geq 1\mu\text{m}$, as confirmed by Energy Dispersive X-ray Spectroscopy (EDX) after finishing processing.

Planarity and roughness of the surface were measured with Fizeau-type Wyko 400 interferometer, and a phase-shift interferential microscope WykoTOPO-3D, respectively. 3D profilometry with a white light interferometric profilometer Zygo NewView 5000 was carried out too. The hemispherical reflectivity at near-normal incidence (8°) was measured in the wavelength range 230-800 nm by means of a Perkin Elmer Lambda 19 spectrophotometer equipped with an integrating sphere of 15 cm diameter. The reflectivity at 45 degrees for s and p polarisations was measured in the wavelength range of 400-800 nm by using an integrating sphere of 10 cm diameter and an Ocean Optics USB2000 spectrophotometer. The specular reflectivity was measured at 632.8 nm at quasi-normal incidence (5°). A consistency check between the rms roughness value as measured with the interferential microscope and the one inferred from hemispherical and specular reflectivity was done. If the roughness of the

mirror surface follows Gaussian statistics, the specular (R_{sp}) and hemispherical (R_{he}) reflectivities are related each other by:

$$R_{sp}(\vartheta) = R_{he}(\vartheta) \exp \left[- \left(\frac{4\pi \cos \vartheta}{\lambda} \sigma \right)^2 \right] \quad (1)$$

where λ , θ and σ are the wavelength and the incidence angle of the probing light and the rms roughness of the surface, respectively. From Eq. (1) the roughness is given by:

$$\sigma = \frac{\lambda}{4\pi \cos \vartheta} \sqrt{\ln \left(\frac{R_{he}(\vartheta)}{R_{sp}(\vartheta)} \right)} \quad (2)$$

Characterization results

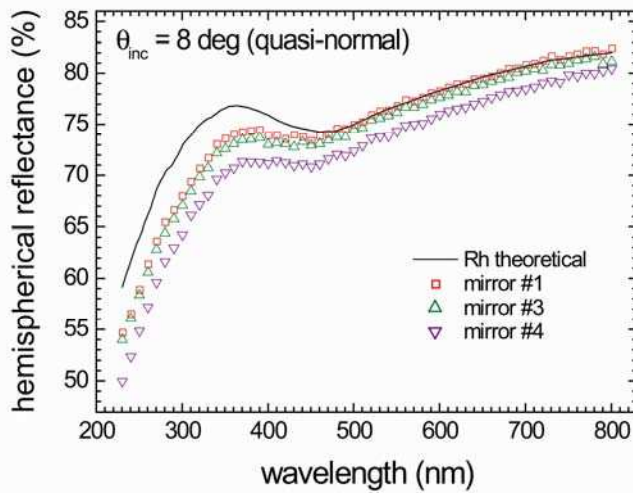


Fig.1 Hemispherical reflectance of mirrors and pure Rh

curves values are 3-5 % lower. The agreement is greatly improved when a 6 nm thick transition layer over the rhodium, modelling the coating porosity, is introduced. As measured with the interferential microscope, the rms roughness ranges from 13 to 40 nm; consequently a quite large diffuse component of the reflectivity is expected (about 20 % at $\lambda = 632.8$ nm for mirror #1). These results are in good agreement with the ones inferred from hemispherical and specular reflectance and Eq. (2) (see Tab I). The values of the average rms roughness as measured with the Zygo white light interferometric profilometer are also in satisfactory agreement, taking into account the different area which the data are averaged over (nearly all the mirror surface for Zygo, five $477 \mu\text{m} \times 477 \mu\text{m}$ squares for Wyko). The measurement of the reflectivity at 45° for s and p polarizations shows values larger than the one expected on the basis of Eq. (1) and the measured roughness, probably because the diffused component of the reflectivity is not totally rejected in the present experimental lay-out.

The hemispherical reflectivity of 3 mirrors is reported in Fig. 1 together with the curve calculated on the basis of the optical constants of pure Rh as reported in the literature [3]. At wavelengths longer than 450 nm the agreement with pure Rh is good for two out of three mirrors: at shorter wavelengths the experimental

Exposure of one Rh-coated mirror in the TEXTOR scrape-off layer

One of the Rh-coated mirrors was exposed in the TEXTOR scrape-off layer for 19 high



power NBI-heated discharges, together with an Rh-coated mirror from the University of Basel [4] and a SC Mo mirror. The mirrors, mounted on a test limiter, were introduced in the vacuum vessel with a limiter-lock system (see Fig. 2). Blue-colored deposit with the thickness of $> 70\text{-}80\text{ nm}$ was observed.

The bulk temperature of mirror holder

Fig. 2 Mirror holder for exposure in TEXTOR was $120\text{ }^{\circ}\text{C}$ at the beginning of exposure, as measured with thermocouples. During the exposure it rose up to 260°C . During the high-power discharges the average temperature increment was $\sim 24\text{ }^{\circ}\text{C}$. The corrected temperature of the mirror holder plasma-closest edge (top) was as high as 1100°C at the end of the high-power discharges. Our mirror was clearly in the erosion-dominated conditions. The temperature of the mirror was in the range $300\text{ }^{\circ}\text{C}$. Average electron density and temperature and ion energy were $5 \cdot 10^{18}\text{ m}^{-3}$, 30 eV and 250 eV , respectively. The flux averaged over the area of the mirror was $3.4 \cdot 10^{22}\text{ m}^{-2}\text{s}^{-1}$ corresponding to a total fluence of $3.4 \cdot 10^{24}\text{ m}^{-2}$, to be compared with a foreseen maximum fluence of particles with $E > 80\text{ eV}$ of about $4 \cdot 10^{21}\text{ m}^{-2}$ for a 1000 s ITER discharge [5].

Post –exposure morphological and optical analyses

After removal the ENEA mirror showed some halo in the lower part (the farthest from the plasma). After the exposure in TEXTOR, mirror n° 1 was characterized following the same procedures adopted before its exposure. SEM analysis did not prompt significant differences. Profilometry showed that the previous flat profile was modified in a slightly convex one. The same profile occurring along any diameter, erosion at the edge of the mirror seems to be ruled out as the cause of the observed convexity: this could be due to some strain, the constrained substrate was subjected to, during strong heating. A decrease (Fig.3) of the hemispherical reflectivity by $3\text{-}10\%$ a substantial invariance of the rms roughness was found after exposure (see Tab I). A very small decrease of both the s and p - polarization reflectivity under 45° incidence was measured too. After exposure, optical characterization was carried out also in the University of Basel. Hemispherical reflectivity results are in good agreement with ENEA

data, while a rather large discrepancy was found for the diffuse component. Presently comparative measurements on test mirrors are being carried out to clarify this point.

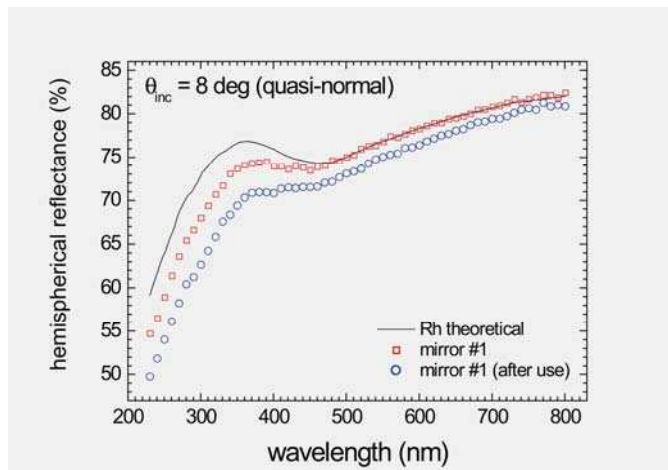


Fig. 3 Comparison between pre and post-exposure

Tab I – Rms roughness (nm)

Method	Pre-exposure	Post-exposure
Interferential microscope	21 ± 3	21 ± 3
Estimated from Eq.1	24.4	25.1
Interferometric profilometer	32.1	27.2

Conclusions

In the frame of Task TW5-TPDS-DIADEV and in collaboration with the Forschungszentrum Jülich and the University of Basel, Rh-coated molybdenum mirrors were manufactured by electroplating and optically characterized. Optimization of the manufacturing procedure (still ongoing) resulted in mirrors with hemispherical reflectivity close to that of pure rhodium, especially for $\lambda > 450$ nm. Diffuse reflectivity was in agreement with the measured roughness. One of these mirrors was exposed at 19 high power NBI-heated discharges in the TEXTOR scrape-off layer, withstanding an ion fluence of $\sim 3.4 \cdot 10^{24} \text{ m}^{-2}$. The main changes found after the exposure are the decrease of the hemispherical reflectivity by 3-10 % and the modification of the mirror surface shape from flat to convex, with consequent variation of optical characteristics like focal length. Manufacturing, as well as exposure in TEXTOR, of new Rh-coated mirrors are foreseen. In the frame of a programme devoted to the exploitation of ITER relevant diagnostics, the feasibility of testing first mirrors on FTU, by using the operating Sample Introduction System, is presently investigated.

References

- [1] V. Voitsenya et al., Rev. Sci. Instrum.72, (2001) 475
- [2] F. Orsitto et al., Rev. Sci. Instrum. 72, (2001) 540
- [3] Handbook of optical constants of solids, ed. E.D. Palik, Acad. Press, 1985 and 1991
- [4] L. Marot et al., “Characterization of magnetron sputtered rhodium films for reflective coatings”, Surf. Coat. Tech. In press.
- [5] A. Kukushkin, reported by T. Sugie – 11 ITPA of TG on Diagnostics- 4 – 8 September 2006, Tohoku Univ. Sendai Japan