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Overview of the Second Stage in the Comprehensive Mirrors Test in JET

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** See annex of F. Romanelli et al, "Overview of JET Results", (23rd IAEA Fusion Energy Conference, Daejon, Republic of Korea (2010)).*

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ABSTRACT

First Mirror Test for ITER in JET with carbon walls has been completed. 30 polycrystalline Mo mirrors including 4 coated with a 1μm Rh film were exposed to plasma in the divertor region and in the main chamber. The mirrors were installed in 8 cassettes of the pan-pipe shape. Reflectivity of all mirrors exposed in the divertor has been degraded by 80-90% because of formation of thick (> 20 μm) flaking co-deposits on surfaces. Only small reflectivity losses (5-10%) occurred on mirrors located at the channel mouth of the cassettes from the main chamber wall. This is due to the in-situ removal of deposited species by charge exchange neutrals. Deuterium, ^{12}C and ^{9}Be are the main isotopes detected on surfaces, but other isotopes (^{13}C) are also found in some locations thus indicating differences in the material migration. Rh-coatings with initial reflectivity 30% better than that of pure Mo survived the test without detachment but their resultant reflectivity was the same as of the exposed Mo surfaces.

1. INTRODUCTION

Metallic mirrors will be essential components of all optical systems for plasma diagnosis and imaging in a reactor-class device. Any degradation of the mirror reflectivity would influence the quality of signals that are crucial in many cases for the machine control. One has to recognize and assess the impact of the long-term exposure in a tokamak on the reflectivity change. Therefore, on the request of the ITER Design Team, the First Mirror Test (FMT) has been carried out at JET [1,2]. The entire research program comprises: (a) the selection of material for test mirror; (b) manufacture of mirrors and their carriers (cassettes of pan-pipe shape) for in-vessel installation; (c) optical pre-characterisation of mirrors; (d) exposure in the plasma boundary of JET during a complete operational campaign; (e) a broad range of post exposure analyses by means of optical and surface analysis methods; (f) analyses of deposition in the channels of the mirror carries [3] and (g) photonic cleaning of the exposed mirrors [4] followed by the analyses of cleaned surfaces and examination of products generated by the laser light impact. FMT is embedded in Tritium Retention Studies (TRS) [5] aiming at the detailed description of erosion-deposition pattern in JET measured by several types of diagnostic tools. They are located in the vicinity of the mirrors. Up to date it has been the most comprehensive test performed with a large number of specimens, ITER-relevant materials for mirrors and long-term exposures to exposures. Two campaigns in JET with carbon walls have been performed. The change of optical properties of mirrors retrieved from the vessel after the first campaign (2005-2007) has been discussed earlier [2]. The aim of this work is to provide an overview of results obtained during the second round of the test and, based on the outcome of the two campaigns, to discuss options for mirror maintenance and cleaning in a steady-state reactor.

2. EXPERIMENTAL: EXPOSURE AND ANALYSES

Until now two exposures have been performed in JET with carbon walls: Step 1 in 2005-2007 comprising 126600s (35h) of plasma operation including 96900 s of X-point operation [2] and recently accomplished Step 2 (2008-2009) with 161680s (45h) exposure with 117680s (32.7 h) of X-point plasma. These plasma exposure times, according to scaling by divertor fluxes, correspond to approximate 2/3 to 1 full ITER pulse [6]. One set of mirrors located in the inner divertor was facing discharges during both campaigns, i.e. 80 h (67.7 X point) of plasma in total. Mirrors were exposed to all operation scenarii realized in JET during several years. The test in 2008-2009 was performed with 32 mirrors made of polycrystalline molybdenum (Mo-poly) including 4 specimens coated with a 1 μm thick layer of rhodium (Rh). The coating was obtained by magnetron sputtering, as described in [7,8].

Mirrors were installed in carriers (8 cassettes with either 3 or 5 channels) placed on the outer wall and in the divertor: inner leg, outer leg and base plate under the load bearing tile. Mirrors were located in the cassettes at different distance from the channel mouth: from 0 to 4.5cm thus resulting in different aspect ration (depth in channel to mirror width). Exact information regarding the construction and location of the mirror carriers has been given earlier [1]. Only some figures of merit are invoked in the following: solid angle for particle bombardment 1×10^{-3} – 0.2sr which is in the range for the mirrors foreseen in ITER: 3×10^{-5} – 1.4sr. Rhcoated mirrors were placed in four cassettes: one in each of major locations, at a distance of 1.5cm from the channel mouth. Some mirrors which were placed on the main chamber wall near the beryllium evaporator where protected by a magnetic shutter which was open only in the presence of the magnetic field.

Before and after exposure mirrors underwent detailed surface analysis using optical methods, ion beam and microscopy techniques. Total reflectivity was measured in the in the range 350–1700nm using a photo-spectrometer (GetSpec) system complying with work procedures on materials retrieved from JET [1,2], i.e. contaminated with beryllium and tritium [9-11]. The integrating sphere was located in the glove-box and connected by fibre optics with externally located photo-spectrometers. The second, not-contaminated sphere, was used for precharacterisation of the mirrors. Full optical characteristic of some specimens was also performed using a Varian Cary 5 apparatus working in the range 250 -2500 nm [1].

Surface and sub-surface composition was studied using ion and electron beam methods. Nuclear Reaction Analysis (NRA) with a 2.5MeV $^{3}He^{+}$ beam was used to quantify deuterium, carbon (^{12}C and ^{13}C) and beryllium by detecting protons emerging from the following reactions: $^{3}He(d,p)^{4}He$, $^{3}He(^{12}C,p)^{14}N$, $^{3}He(^{13}C,p)^{15}N$, $^{3}He(^{9}Be,p)^{11}B$. Carbon and beryllium were also determined by Enhanced Proton Scattering (EPS) with a 2.5MeV H^{+} beam which allowed simultaneous studies of heavier elements using Proton-Induced X-ray Emission (PIXE). Secondary ion mass spectrometry (SIMS) with a VG IX70S double focusing magnetic sector equipment allowed depth profiling of hydrogen (^{1}H) deuterium (^{2}H), beryllium (^{9}Be), carbon (^{12}C), nickel (^{58}Ni), molybdenium (^{98}Mo) and rhodium (^{103}Rh). The analyses were performed using primary oxygen beam (O_2^{+}, 5keV, ion current 250nA). The sputtering at the rate of 0.35nm/s was done on areas $300\times220\mu m^2$. Topography was determined using Scanning Electron Microscopy (SEM) with a field emission gun (Zeiss Gemini), whereas composition was studied with energy dispersive X-ray spectroscopy using an

Oxford Instruments detector.

Sputter-assisted X-ray Photoelectron Spectroscopy (XPS) with an erosion rate of 0.5nm/s by an Ar^+ beam was used to study the material mixing on some mirrors from the main chamber wall. Chemical shift of the core levels: Rh3d, Mo3d, O1s, C1s was determined.

3. RESULTS AND DISCUSSION

3.1. TOPOGRAPHY OF THE MIRRORS

Series of photographs in Figure 1 show the appearance mirrors from the main chamber wall (a) and the outer divertor (b) after exposure in JET. The quality of images is somewhat obscured because of photographing through plastic of the glove box. The position of mirrors in cassettes is marked to inform about the distance in cm from the mouth of the channel. Bright areas on mirrors from the main chamber wall are spots eroded by sputtering during SIMS measurements. The images are representative for all specimens from the two major locations inside the tokamak. The visual inspection clearly indicates that: (i) on the main chamber wall, mirrors close to the channel mouth are free from deposition, whereas deposition increases with depth in the channel, as perceived on the surface located at 4.5cm; (ii) all divertor mirrors are covered by deposits which are rather thick and flaking and, the deposition decreases with the depth in the channel. These results are in full agreement with findings after the first step of the mirror test in 2005-2007, as reported in [2,3]. The results imply that the surface state of mirror in the divertor is governed by net deposition, whereas net erosion decides the state of mirrors located close to the channel mouth in cassettes on the main chamber wall. Micrographs in Figure 2 (a) and (b)show details of flaking co-deposits on surfaces exposed in the inner divertor (0 cm, 2005-2009) and in the base (0 cm, 2008-2009), respectively. In Fig. 2(a) one observes a stratified structure of the layer which peels-off thus forming terraces and areas of diversified deposit thickness. Flaking and poor adherence to the substrate, as can be judged from Fig. 2(b), indicates that during the entire period of exposure many consecutive layers could peel-off and contribute to the dust formation.

3.2 REFLECTIVITY OF THE EXPOSED MIRROR

Total reflectivity of all exposed mirrors was examined with spectro-photometry and compared it with the initial parameters. Plots in Figure 3 (a) and (b) show reflectivity of mirrors exposed in the inner divertor during the entire period of FMT (2005-2009) and during Step 2 (2008-2009), respectively. The initial reflectivity of Mo-poly and Rh-coated mirrors is also inserted in the graphs. For clarity of presentation it is illustrated by a single plot, as the initial values for mirrors from a given category (either Mo-poly or Rh-coated) were very similar. The main result is that reflectivity of nearly all mirrors in the divertor region has been degraded by 80- 90%. The results are fully consistent with those obtained after the exposure in 2005-2007 [2,3]. The strongest loss is measured on mirrors located close to the channel mouth. The exception for trace at 0 cm on Fig. 3 (b) is associated with peeling-off of deposits. There is no difference between Mo and Rh on the divertor samples because degradation of optical performance is connected with deposition, which is not surface specific once the first layer of a carbon deposit is formed. Reflectivity of mirrors from the main chamber wall is shown in Figure 4 (a) and (b), where results for units with and without the magnetic shutter are plotted, respectively. A clear general tendency is that the strongest reflectivity loss is on mirrors located deep in the channel (3 and 4.5cm), whereas only small changes are measured on mirrors located at the entrance (0cm) and close to channel mouth at 1.5cm. One may notice even slight improvement of reflectivity. This indicates that net erosion by impacting Charge eXchange (CX) neutrals resulted in further "polishing" of the surfaces. Such smoothening effects have been observed earlier in erosion zones of plasma-facing components from the TEXTOR tokamak [12]. The recent results for Mo-poly fully confirm the tendency measured after the first test on mirrors retrieved in 2007 [2]. Rh-coated specimens were used in JET for the first time. The big initial difference in reflectivity between molybdenum and rhodium has not been maintained after the exposure. The values measured on Rh-coated and not coated Mo mirrors are the same. This might be attributed to the loss of the coating or to material mixing occurring during the exposure. Strong emphasis in surface analyses was on the clarification of that issue.

3.3 SURFACE COMPOSITION OF THE EXPOSED MIRROR

NRA and EPS measurements combined with spectra modeling by means NDF Program [13] revealed the presence of carbon-12 and deuterium on all surfaces. The thickness of carbonrich deposits on several studied surfaces was greater than the information depth with IBA: approximately 10 μm with 2.5MeV $^{3}He^{+}$ and up to 18μm with a H^{+} beam. Graphs in Figure 5 (a) and (b) show carbon contents on mirrors from the divertor base and outer wall, respectively. They also demonstrate the general tendency for specimens from the two major locations: (i) decrease of deposition with the depth in channel in the divertor and (ii) low deposition at the channel mouth and then increase deeper in the channel on the wall. In the latter case, it is perceived that the deposition first increases and then decreases slightly. This recent result is fully consistent with previously obtained data [2] thus indicating that the CX flux reaching is reduced because of a small solid angle and attenuation by collisions in the gas phase and with the channel walls.

Other isotopes (^{9}Be and ^{13}C) are detected, but not on all surfaces. Beryllium, in measurable amounts, is not found on surfaces from the inner divertor because of the limited transport of non-volatile species to that region, as discussed in [10,11,14,15]. Carbon-13 used as a tracer for material migration studies (as described in [15-17]) was puffed into JET on the last operation day of the campaign in 2009 from the outer divertor ring of gas inlet modules. The isotope was detected only locally, i.e. on the set of mirrors from the outer divertor: around 5.5×10^{17} cm^{-2} and 1.5×10^{17} cm^{-2} on mirrors placed at the depth of 0 and 1.5cm, respectively. The result shows that there was no direct transport through the private region from the outer to the inner divertor. The amount of ^{13}C deposited during 30 discharges (round 460s X-point) is to be compared with the amount of ^{12}C detected on mirrors after the entire exposure period(161680 s): 1×10^{17} cm^{-2} and 3×10^{17} cm^{-2} on the above mentioned mirrors, thus giving the $^{12}C/^{13}C$ concentration ratio of about 2. This small value is no proportion to the exposure time. One may deduce that this could be attributed to quite frequent flaking and peeling-off of the carbon layers deposited on the mirrors in the divertor.

3.4 MODIFICATION OF RHODIUM COATING

The aim of the analysis was to verify whether the Rh coating remained on the mirrors, especially on the specimen from the outer wall which surface was strongly bombarded by charge exchange neutrals. SEM-EDX and PIXE measurements have shown that rhodium layer is present on all four coated mirrors. The signal intensity is clearly related to the amount of C and Be deposition surfaces. A relatively weak X-ray line intensity has been recorded for the inner divertor mirror and the strongest from the outer wall specimen. No noticeable molybdenum lines have been detected thus indicating: (i) no detachment or peeling-off of the Rh film; (ii) no significant erosion of the coating; (iii) no Rh-Mo intermixing or diffusion because of fairly low temperature of mirrors during the exposure: 220-280^{o}C for cassettes on the outer wall and 170-200^{o}C in the divertor. Plots in Figure 6 are SIMS depth profiles of several isotopes on the Rh-coated mirror from the outer wall, i.e. the surface free from visible deposition. These results are qualitative (no quantitative data can be extracted), but they bring crucial information on the relative changes regarding presence of respective species. The Rh coating extends to about 1μm. It is in agreement with the nominal pre-exposure value and clearly proves that the film was not detached from the substrate. However, one may perceive that the near surface region up to 50nm has a modified composition. In addition to carbon and beryllium, whose presence has been detected by NRA, there are also heavier species Ni and Mo. Their signals sharply decrease indicating that origin of metals is not related to the erosion of the mirror substrate. Both elements originate from the erosion of Inconel 625 alloy constituting the JET vacuum vessel. The alloy nominally contains up to 8-10 wt. % of Mo, and 58 wt % Ni, 20-23 wt % Cr as main constituents. Detailed analysis of profiles for the surface and sub-surface region of two mirrors, Rh-coated and Mo-poly, exposed at the same depth in channels (1.5cm) in two units on the main chamber wall clearly shows similarity in the overall surface composition: C, Be, Mo and Ni which were co-deposited and co-implanted as a result of the CX bombardment. This thin mixed material layer on the mirror surface is decisive for reflectivity. This may explain why the behavior of the Rh-coated and pure Mopoly mirrors exposed under erosion-dominated conditions is eventually on the same level.

CONCLUSIONS

First Mirror Test in JET with carbon wall has been completed. Results of two long-term campaigns (nearly 40h of plasma each) are fully consistent: significant loss of optical performance in the divertor and smaller changes for mirrors on the outer wall. An important and positive result is that Mo-poly mirrors located at the channel mouth during exposure on the main chamber wall retained good reflectivity despite surface modification by material mixing. This may indicate beneficial effects of net erosion of the Mo surface by charge exchange neutrals. However, the same type of mixing was determined by SIMS on the Rhcoated mirror (from the same location), but its reflectivity dropped to the level measured for Mo specimen. It still remains to be determined whether the surfaces contain just a mixture of co-implanted elements or a complex mixture of compounds. The fate of Rh coatings may be crucial in final selection and qualification of mirror materials for ITER. It has been clearly shown that the coatings survived the test. However, taking into account material mixing effects, these first results on long-term testing of the Rh layers do not provide convincing arguments for the application of coated mirrors in a reactor-class machine.

Optical performance of all mirrors in the divertor region has been fully degraded by deposition of carbon as the main constituent on the surfaces. It is not straightforward, and it is not intended here, to translate immediately these results to the ITER operation because of different densities and another wall composition. However, the results indicate that diagnostic mirrors in ITER may become coated with deposits in less than some tens of shots if carbon wall components are used. This result calls for intense efforts in development of mirror replacement [2] or protection systems [18] even if solutions constitute a serious engineering challenge. The other option is in elaboration of reliable and feasible cleaning techniques to remove deposits and regain reflectivity. Whichever cleaning method is pursued an issue of dust formation is to taken into account. The study has shown that deposits detach and peel-off rather easily, as proven by visual inspection, microscopy and with a 13C tracer, but this process occurs in an uncontrolled way. Dust mobilisation in a diagnostic channel would additionally obscure optical signals. Certain type of mechanical cleaning is planned at JET. However, the most important is to test mirrors and other diagnostic components in a tokamak with metal walls where carbon transport would be suppressed. Therefore, FMT will be continued during JET operation with the ITER-Like Wall [19-21]. For that purpose a new set of mirrors has been manufactured and installed.

ACKNOWLEDGEMENTS
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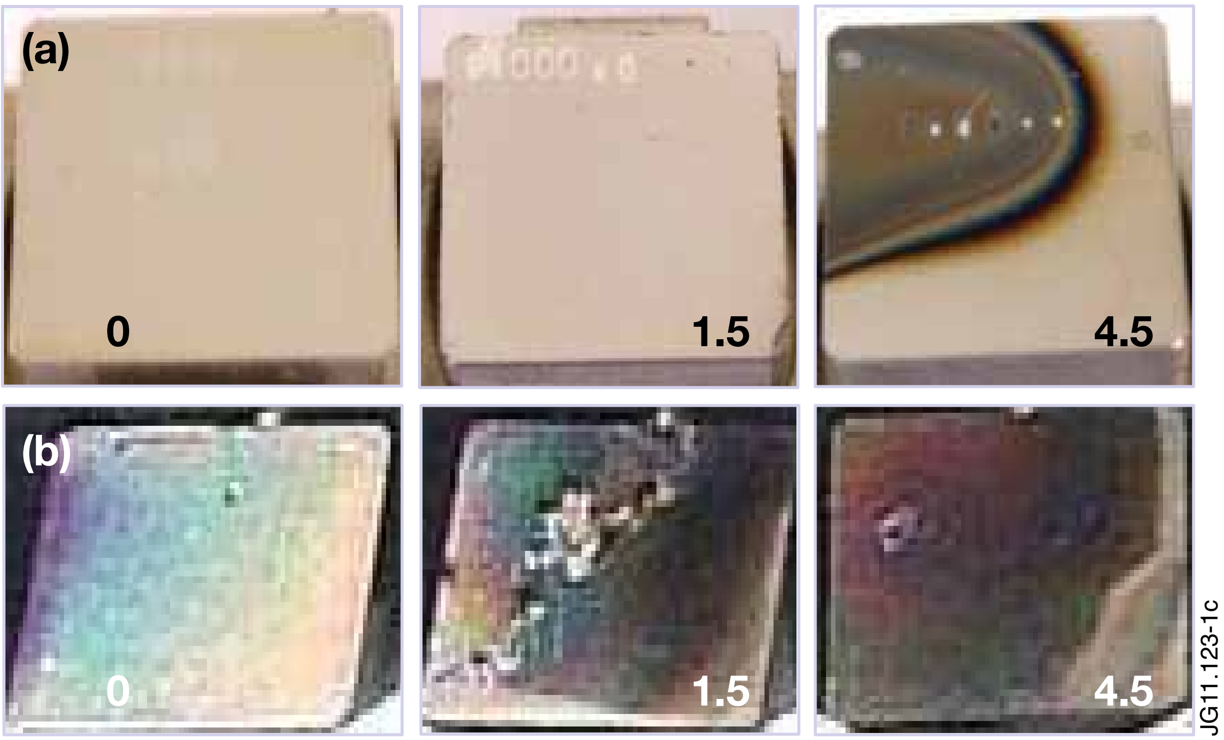


Figure 1: Mirrors retrieved after exposure in 2008-2009 from: (a) the main chamber wall; (b) the outer divertor. Numbers inform about the position in the channel, i.e. distance in cm from the channel mouth. Spots noticed on mirrors (a) are areas sputtered during SIMS measurements.

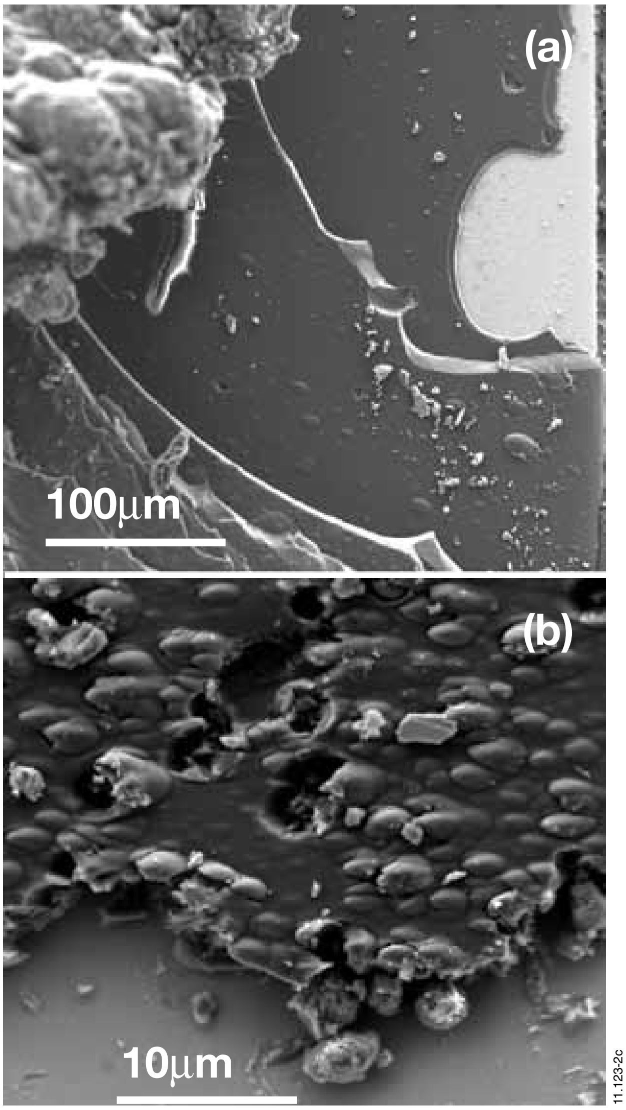


Figure 2: Surface topography of flaking co-deposits on mirrors from: (a) the inner divertor, exposure at 0cm during 2005-2009 operation; (b) the divertor base, exposure at 0cm during 2008-2009 campaign.

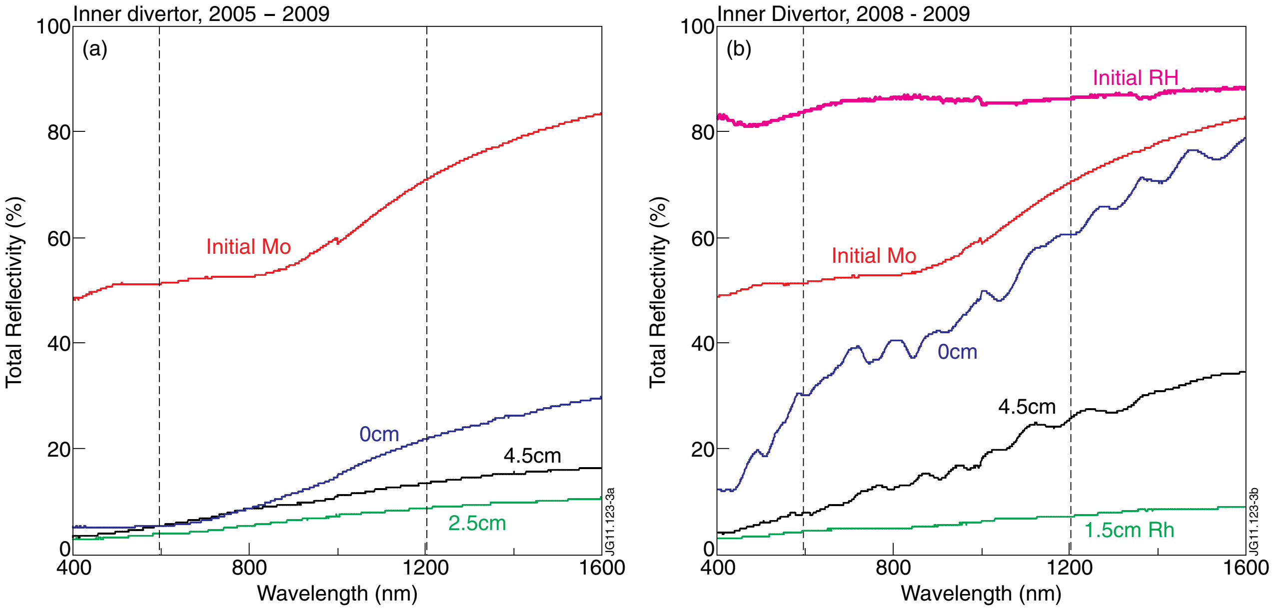
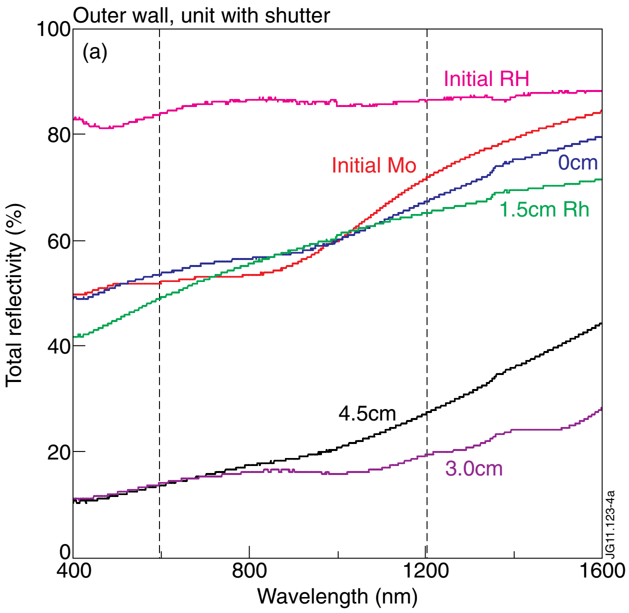


Figure 3: Total reflectivity of mirrors from the inner divertor after exposure: (a) 2005-2009 and (b) 2008-2009. An irregular trace for position 0 cm in (b) is related to the peeling-off layer.



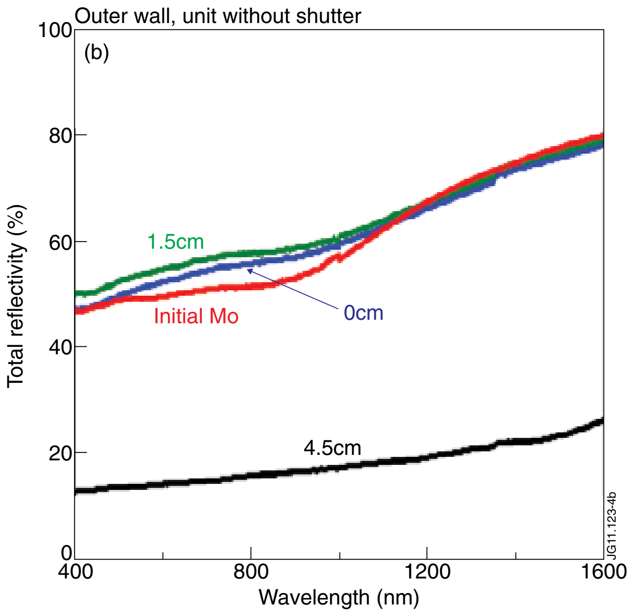
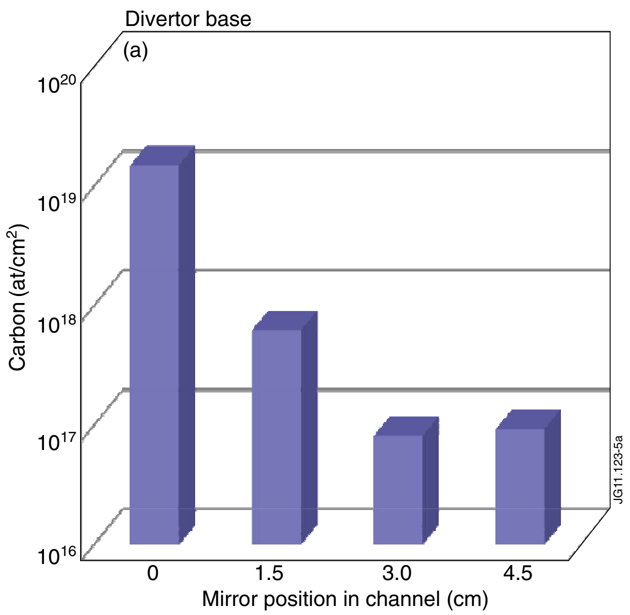


Figure 4: Total reflectivity of mirrors exposed in 2008-2009 on the outer wall in the main chamber: (a) unit with a magnetic shutter; (b) unit without the shutter.



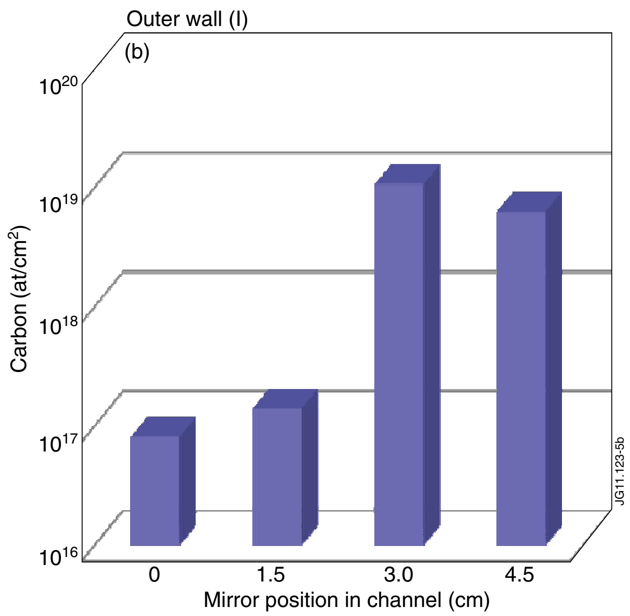


Figure 5: Carbon deposition on mirrors in: (a) divertor base; (b) main chamber wall.

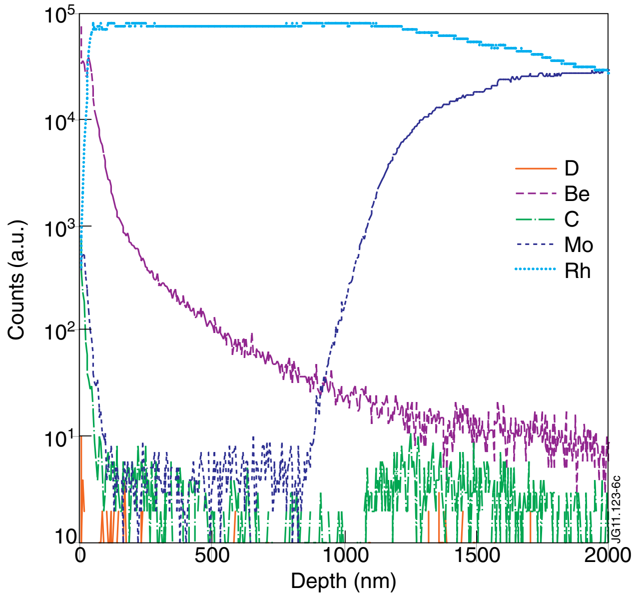


Figure 6: SIMS depth profiles of species in the surface region of a Rh-coated mirror exposed on the main chamber wall.