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** See annex of F. Romanelli et al, "Overview of JET Results", (24th IAEA Fusion Energy Conference, San Diego, USA (2012)).*

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ABSTRACT
A set of seven polycrystalline mirror samples retrieved from the JET tokamak have been cleaned in vacuum using a pulsed laser system. The samples were exposed between 2008- 2009 as part of the second phase of a comprehensive first mirror test and contain a mixture of carbon, beryllium and tritium. For this reason, the samples were treated in a vacuum chamber especially constructed for this purpose. Additionally, three samples coated in PISCES-B with a 110-120nm beryllium layer were cleaned as well. The results are quite positive, most mirrors showing a substantial increase of the specular reflectivity after cleaning. The ones that do not appear to have been damaged already prior to cleaning.

1. INTRODUCTION
In ITER, the most crucial element of each optical system is the first mirror in the optical path, which serves to guide light into the mirror labyrinth leading to the diagnostic located outside the neutron shielding. Though finding a suitable material is still an ongoing effort [1,2], the most promising candidates at present are molybdenum and rhodium, on account of their reflectivity and resilience to sputtering. Due to the prohibitive costs of manufacturing single crystalline mirrors of either of these materials, a common material at present is the cheaper polycrystalline form. However, its inferior erosion properties have motivated the investigation of nanocrystalline molybdenum and rhodium films on a metallic substrate for the preparation of mirror surfaces instead [3].

Two extensive first mirror tests have been carried out at JET, the first one between 2004 and 2007 [4] and the second one between 2008-2009 [5]. A total of 61 mirrors distributed over various locations in the machine were exposed and several materials were tested, including stainless steel, polycrystalline molybdenum and rhodium coatings. The results showed a severe degradation of the reflectivity of the mirrors located in the divertor region and a smaller degradation for mirrors near the outer wall, which was found to be due to the build-up of deposits of material eroded from the main chamber wall.

As all optical systems in ITER rely on the first mirrors for their operation, ways are being explored to prevent the build-up of deposits as well as to remove them in situ. One technique that is currently under investigation for the in situ removal of deposits is laser cleaning, which would offer the advantage of not being susceptible to the magnetic field.

So far, few experiments on the laser cleaning of surfaces contaminated with beryllium have been performed. The first ablation experiments on substrates containing beryllium were performed by Widdowson et al. [6], who investigated the cleaning of stainless steel and molybdenum mirror samples from the initial first mirror test between 2005-2007. The experiments were performed using 1064 nm, 120 ns pulses at 20 kHz. Unfortunately the mirrors were damaged in the experiment, though a substantial recovery of the reflectivity was achieved.

Equally unsuccessful experiments on beryllium surfaces were reported in [7], which describes experiments in an argon atmosphere on a set of single crystalline molybdenum mirrors coated with a 150nm beryllium film in the PISCES-B facility, using 1064nm, 220ns pulses at 8-10kHz.

Following our previous paper reporting on damage threshold tests of ITER first mirror materials and laser cleaning of stainless steel mirrors in vacuum [8], the present work concerns the cleaning of seven polycrystalline molybdenum mirror samples from the second mirror test (2008-2009), including five mirrors from the divertor base, one from the outer divertor and one from the outer wall.

As these samples still contain a considerable amount of carbon, which is most likely to be absent in ITER, as well as a lower beryllium concentration than is to be expected in ITER, three additional polycrystalline molybdenum samples, including one with a partial rhodium coating, were prepared with a beryllium coating in the PISCES-B facility to supplement the sample set from JET.

2. SAMPLE SET

2.1 SAMPLES FROM JET

The samples consisted of 10x10x10 mm cubes of polycrystalline molybdenum, each with one polished face. One of these was coated with a one micron rhodium layer at the University of Basel, using magnetron sputtering. In order to see the effect of plasma exposure on a rhodium coating. More details on the coating process may be found in [9]. The total reflectivity of each mirror between 350 and 1750nm was measured at JET before installation in the JET vessel, using a spectrophotometer. The concentrations of C, D and Be as determined by nuclear reaction analysis (NRA) using a 2.5MeV ^{3}He+ beam are listed in Table 1.

2.2 SAMPLES FROM PISCES-B

Three 15×25×4mm polycrystalline molybdenum mirror samples with one polished face were prepared at the University of Basel. One half of one mirror was coated with a one micron layer of rhodium. The mirrors were then coated with a layer of beryllium at the University of California in San Diego using the witness plate manipulator [10]. A beryllium coating was obtained on the mirrors during exposure of beryllium targets being eroded by deuterium plasma. Beryllium codeposits created in this way have been shown to consist almost entirely of beryllium and deuterium [11]. The codeposits were collected at room temperature by cooling the witness plate during the codeposition process. The film thickness was determined before and after laser cleaning by Secondary Ion Mass Spectrometry (SIMS) depth profiling and Rutherford Backscattering Spectrometry (RBS) measurements [5,12]. Examples of SIMS depth profiles of the codeposit on mirror P3 are plotted Figure 1. A complete listing can be found in table 2.

Figure 2 shows a picture of all mirrors mounted in a sample holder used for this experiment a well as a schematic showing the mirror identification numbers. J1 is a pristine polycrystalline molybdenum reference mirror installed to enable in situ reflectivity measurements on the samples.

3. EXPERIMENTAL SETUP

Due to health and safety issues associated with the JET samples, a vacuum system was constructed to contain the samples for the entire duration of the experiment. This system, shown in Figure 3, consists of a small vacuum chamber with a quartz window to provide optical access down to 200nm. The chamber was evacuated through a particle filter (Luwa JM Sterile Air Filter) using a conventional pumping system, after which the chamber was sealed and pumping was taken over by an ion getter pump, reaching a pressure of 3×10^{-8} mbar at room temperature. A resistive heater combined with a thermocouple allowed the samples to be heated during the experiment, as a 150°C mirror temperature is expected in ITER [9, 10]. In order to scan the laser beam across the sample surface, the chamber was mounted on two computer controlled translation stages, a counterweight being used to alleviate the strain on the stages. The laser system used for cleaning is described in detail in [8]. It consists of an optical parametric oscillator delivering tuneable radiation in the range of 220-2300nm, with a pulse length of 5ns and a repetition rate of 20Hz, with an output energy varying between 2 and 25mJ depending on wavelength.

4. IN SITU REFLECTIVITY MEASUREMENTS

The setup used at JET to measure the reflectivity can only be used to measure the total reflectivity of the whole sample surface, whereas it was desirable to divide some of the sample surfaces into smaller quadrants in order to make maximum use of the limited area available for the experiment. Therefore, a simple setup was constructed that allowed the specular reflectivity to be measured in situ, within the range of 400-800 nm, see Figure 4. The setup consisted of a 150 W tungsten light source coupled to a 400 micron optical fibre connected to 50 mm lens. The beam was shone through the quartz window, reflected from the sample surface and collected using an integrating sphere connected to an Avantes USB spectrometer (Avaspec 2048-USB2-UA). In order to determine the absolute specular reflectivity, the intensity of the reflected light was compared to that collected from a reference mirror that was installed in the chamber for this purpose, which is the top left mirror in Figure 3 (J1). The results were confirmed by ex-situ measurements at JET after cleaning.

5. CLEANING EXPERIMENT

The damage threshold of polycrystalline molybdenum as a function of wavelength in vacuum has been determined previously [8], providing an indication of the energy density desired for the current experiments. For example, the damage threshold at 532nm has been determined to be around $400mJ/cm^2$ (for a 5ns pulse). A similar test on the reference mirror, however, showed a threshold of almost $1000mJ/cm^2$. As a result, the energies used for the cleaning tests described here are higher than might be anticipated from the damage thresholds reported in [8]. It is unclear why this should be the case. Though the properties of polycrystalline molybdenum do vary from batch to batch, this is hardly likely to account for a factor of 2.5. The pressure obtained in this experiment is lower by a factor of ten compared to that reported in [8], which would also have the effect of raising the damage threshold, though again not by a factor of 2.5. It was assumed that the same would apply to rhodium as well, though this has not been measured.

In most cases, the samples were heated to 150 °C during the experiment (see the results section for details). As the experiments were carried out over several days and the heating was switched off overnight, the samples were heated a total of seven times, at a rate of 5°C per minute.

Three wavelengths have been used: 230, 532 and 1064 nm. The beam profile was recorded and used in combination with energy meter readings to deduce the spatial energy distribution incident on the sample. The samples were scanned at a speed so as to achieve a 90% overlap between consecutive pulses both in the x- and y-direction, where the spot size in these directions is defined as the central region covering 50% of the total beam energy. The spot sizes are given in Table 2, which also includes the damage thresholds measured in [8], with and without multiplication by a factor of 2.5 to account for the difference in the damage threshold of molybdenum observed in the current experiment. Note that although the higher value is stated here for rhodium as well, this value was not measured, but is an estimate based on the assumption that the increased threshold of molybdenum under the current conditions applies to rhodium as well.

6. RESULTS

The most interesting results are summarized below. The dashed line in all plots below corresponds to the reflectivity of one of the mirrors before exposure in JET and serves as an indication of the reflectivity that should be obtained when the deposits have been successfully removed.

6.1 JET MIRRORS

J2 had been coated with a 1 micron layer of rhodium before installation in JET. After heating to 150°C the surface appeared to be buckling, indicating delamination. It is the first time that this type of film is seen to delaminate upon heating. It is particularly surprising since the layer has survived two years of JET operation. After exposure to 532nm/760mJ/cm^2 most of the layer has delaminated, as is evident from the picture. During exposure, the pressure rose to 10^{-6} mbar, probably due to the release of deuterium from the ablated material. The pressure dropped again as soon as the exposure finished. The reflectivity in the top right corner was found to correspond to molybdenum, indicating the complete removal of the rhodium layer. Note that the delamination cannot be attributed to the laser energy being above the damage threshold, even when the assumption of the increased threshold is false; the damage threshold applies to the removal of material from the top surface, not the delamination of a layer of material, which is an entirely different process that may occur either at a lower or higher energy and results in a very different damage pattern. It is possible that the delamination was enhanced by mechanical stress building up during the ablation of the deposit on top, either by the removal of the deposited material or through the heating of gas trapped in the surface layer.

J3 and J4 showed mainly erosion and cleaning did not change their reflectivity. J5, J6 and J7 were cleaned at 150°C, using the same recipe as before: 2 cycles of 532nm / 760mJ/cm^2, with a substantial improvement of the reflectivity. The structure remaining on J5 is probably due to erosion.

6.2 PISCES MIRRORS

6.2.1 Mirror P1 and P3

Mirror P1 had been prepared with a one micron layer of rhodium covering its left half prior to coating in PISCES-B. A vertical line may be seen in the picture below, indicating the boundary. Cleaning tests at 150 °C were performed on three horizontal strips covering the width of the sample. See Figure 7 for a schematic showing the wavelength, energy density and number of exposures for each section. The second cleaning cycle on the middle strip accidentally started halfway the section covered with rhodium, which can be made out in the picture below. Blistering occurred both on the top and middle sections, indicating that the laser energy was too high, which suggests that the assumption that the damage threshold of rhodium was also a factor of 2.5 higher under the current conditions was too optimistic. The bottom strip does not show any blistering, but the reflectivity has not improved either (see Figure 9).

Mirror P3 was subjected to a similar procedure. The sample was heated to 150°C and six patches were cleaned, see the schematic in Figure 8 for details. The reflectivity of the bottom patches was accidentally recorded in the middle, so only one measurement is available. Although there is little difference between the various sections, a few observations can be made. The top two sections were cleaned at a moderate energy and show a very similar reflectivity after cleaning. The middle sections were cleaned at a higher energy, resulting in a higher reflectivity. Contrary to the bottom section of P1, which was also cleaned at 1064nm, the bottom section of P3 does show an improved reflectivity, be it a moderate one.

6.2.2 Mirror P2

The coating of this sample in PISCES-B had gone wrong, resulting in a nonuniform deposit with a very low reflectivity instead of the highly reflecting film that was present on P1 and P3. However, this could be advantageous, as it could very well be that this is much closer to the sort of deposit that might form in ITER. The sample was heated to 150°C. The deposit was found to ablate at 230, 532 as well as 1064nm, with no apparent visual difference. The illumination was different in the two pictures below, which somewhat impedes a direct comparison. However, reflectivity measurements clearly demonstrate a much enhanced reflectivity after cleaning.

Table 3 summarizes all the experimental conditions for the laser cleaning of each mirror, as well as the recovered reflectivity and the measured thickness of some of the films by SIMS and RBS 7.

CONCLUSIONS

Seven JET mirrors contaminated with carbon, beryllium, tritium and deuterium have been cleaned in vacuum, either at room temperature or at 150°C. All but one mirror show a substantial increase of the specular reflectivity. As no damage was incurred by the reference mirror at any of the power densities used during cleaning, it may be assumed that any damage observed after cleaning, in particular on the mirror whose reflectivity did not increase, was sustained during plasma exposure in JET.

The rhodium coating present on one mirror delaminated completely, which is the first time this is observed. The delamination process appeared to start already during heating and was completed after exposure, exposing the original molybdenum substrate underneath. It is possible that the delamination was enhanced by mechanical stress building up during the ablation of the deposit on top, either by the removal of the deposited material or through the heating of gas trapped in the surface layer. Another rhodium coating showed signs of blistering after laser exposure, though this may be attributed to the laser energy being too high.

Two of three mirrors containing only beryllium and deuterium were cleaned patch-wise, using 230, 532 or 1064nm. The reflectivity was already quite high before cleaning, and cleaning increased the reflectivity from about 40 to 50%. There is no clear dependence of the resulting reflectivity on the wavelength used for cleaning, though a smaller increase and even a decrease of the reflectivity have been observed when using 1064nm.

One mirror had accidentally received a non-uniform, badly reflecting coating covering two thirds of the mirror surface, which was found to ablate at 230, 532 as well as 1064nm, leaving behind a surface with a much enhanced reflectivity.

In conclusion, a clear cleaning effect has been demonstrated on all JET mirrors accept one that was most probably damaged beforehand. Removing highly reflecting beryllium coatings proved to be less successful, but a badly reflecting beryllium layer was successfully removed.

ACKNOWLEDGEMENTS

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Mirror (JET ID)	*C 10^{18} cm^{-2}*	*Be 10^{18} cm^{-2}*	*D 10^{18} cm^{-2}*
J2 (20)[1)]	*1 - 3.4*	*0.7 - 1.8*	*0.7 - 2.2*
J3 (75)[2)]	*0.45 - 0.6*	*0.02 – 0.9*	*0.17 – 1.25*
J4 (73)[3)]	*0.17 – 0.31*	*0.275 – 0.43*	*0.07*
J5 (62)[4)]	*0.1 – 5.9*	*0.2*	*0.08 – 4.3*
J6 (74)[5)]	*0.03 – 0.8*	*0.01 – 0.21*	*0.004 – 0.06*
J7 (72)[6)]	*0.05*	*0.04*	*0.04-0.01*
J8 (06)[7)]	*0.2 – 1.43*	*0.3 – 0.8*	*0.06 - 1*

Original location of each mirror inside the JET vessel:

1) Rh coated Mo. Outer divertor corner (Module 2 outer narrow carrier) recessed 1.5cm

2) Mo under LBSRP of divertor (Module 2 narrow LBSRP carrier) recessed 0cm

3) Mo under LBSRP of divertor (Module 2 narrow LBSRP carrier) recessed 1.5cm

4) Mo on outer wall (Octant 3 sector E) recessed 3.0cm

5) Mo under LBSRP of divertor (Module 2 narrow LBSRP carr ier) recessed 3.0cm

6) Mo under LBSRP of divertor (Module 2 narrow LBSRP carrier) recessed 4.5cm

7) Mo 45 deg under LBSRP of divertor (Module 2 narrow LBSRP carrier) recessed 1.5cm

Table 1: Concentration of C, Be and D on each mirror surface as determined with NRA using a 2.5MeV $^{3}He+$ beam [5]. The spread in values reflects the spatial spread in concentrations found on the surface.

Wavelength	*Spot width (micron)*	*Spot height (micron)*	*E_{th} (pcMo) (mJ/cm^2)*	*E_{th} (pcMo) * (mJ/cm^2)*	*E_{th} (Rh) (mJ/cm^2)*	*E_{th} (Rh) * (mJ/cm^2)*
230	*316*	*132*	*850*	*2125*	*700*	*1750*
532	*420*	*308*	*200*	*500*	*400*	*1000*
1064	*497*	*401*	*200*	*500*	*500*	*1250*

*Table 2: Spots sizes and damage thresholds for the three wavelengths used. *) Multiplied by 2.5 (see text)*

Mirrors	*Be thickness SIMS (nm)*	*Be thickness RBS (nm)*	*Reflectivity after laser treatment (%)*	*Experimental conditions: Temperature, laser wavelength, power density*
J2 (1 mm Rh)			*Delamination*	*150°C; 532 nm; 760 mJ/cm^2*
J3			*~15%*	*RT; 532 nm;760 mJ/cm^2*
J4			*~0%*	*RT; 532 nm ;760 mJ/cm^2 (2x)*
J5			*~85%*	*150°C; 532 nm; 760 mJ/cm^2 (2x)*
J6			*~100%*	*150°C; 532 nm; 760 mJ/cm^2 (2x)*
J7			*~100%*	*150°C; 532 nm; 760 mJ/cm^2 (2x)*
J8			*~50%*	*150°C; 532 nm; 640 mJ/cm^2 and 230 nm; 2100 mJ/cm^2*
P1 (600nm Rh)	*105*	*121*		*Codeposited film*
	100	*107*	*~65%*	*150°C; 230 nm; 2177 mJ/cm^2*
			~60%	*150°C; 532 nm; 760 mJ/cm^2 (2x)*
		115	*~55%*	*150°C; 1064 nm; 500 mJ/cm^2 (2x)*
P1 (Mo)	*105*	*121*		*Codeposited film*
	100	*126*	*~80%*	*150°C; 230 nm; 2177 mJ/cm^2*
	81	*124*	*~80%*	*150°C; 532 nm; 760 mJ/cm^2 (2x)*
			~75%	*150°C; 1064 nm; 500 mJ/cm^2 (2x)*
P2 (Mo)	*112*	*130*		*Codeposited film*
		15	*~100%*	*150°C; 532 nm; 760 mJ/cm^2 (2x) & 230 nm; 2050 mJ/cm^2*
	2	*8*	*~100%*	*150°C; 1064 nm; 500 mJ/cm^2 (2x) & 1064 nm; 800 mJ/cm^2(2x)*
			~100%	*150°C; 532 nm; 760 mJ/cm^2*
P3 (Mo)	*102*	*131*		*Codeposited film*
			~85%	*150°C; 532 nm; 460 mJ/cm^2*
			~90%	*150°C; 532 nm; 760 mJ/cm^2*
			~80%	*150°C; 1064 nm; 500 mJ/cm^2*
	136	*126*	*~85%*	*150°C; 532 nm; 280 mJ/cm^2*
	110	*138*	*~90%*	*150°C; 532 nm; 760 mJ/cm^2 (2x)*
	96	*130*	*~80%*	*150°C; 1064 nm; 500 mJ/cm^2(2x)*

Table 2: Summary of the experimental conditions used for laser cleaning: temperature, wavelength, power density, number of cycles, final reflectivity in the visible range. Some films thicknesses measured with SIMS and RBS are also listed.

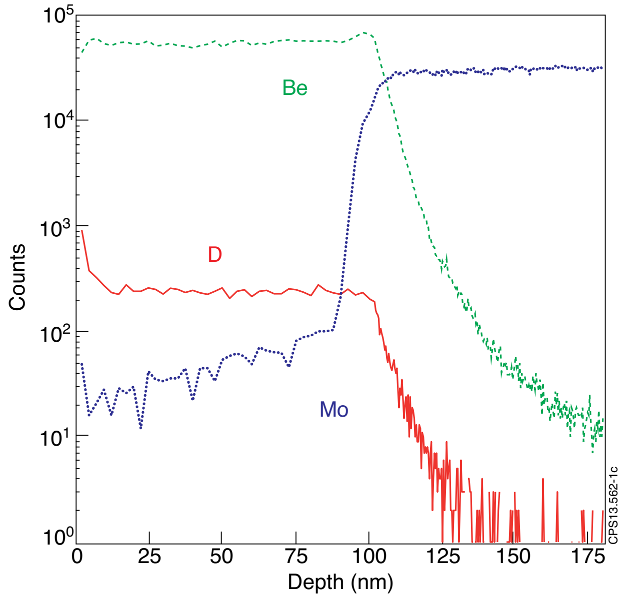


Figure 1: SIMS depth profiles of several species measured on the codeposit on mirror P3.

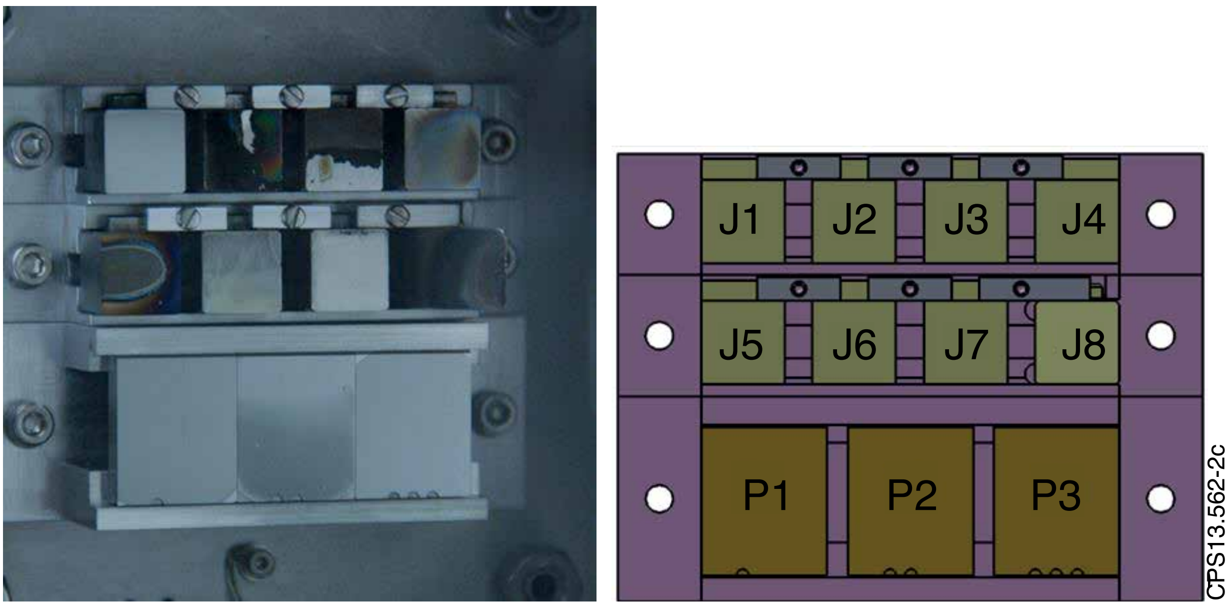


Figure 2: (left) Sample holder containing: a clean molybdenum reference mirror (top left), seven mirrors from JET (top and middle row) and three samples from PISCES-B (bottom row). (right) Schematic showing the mirror identification numbers.

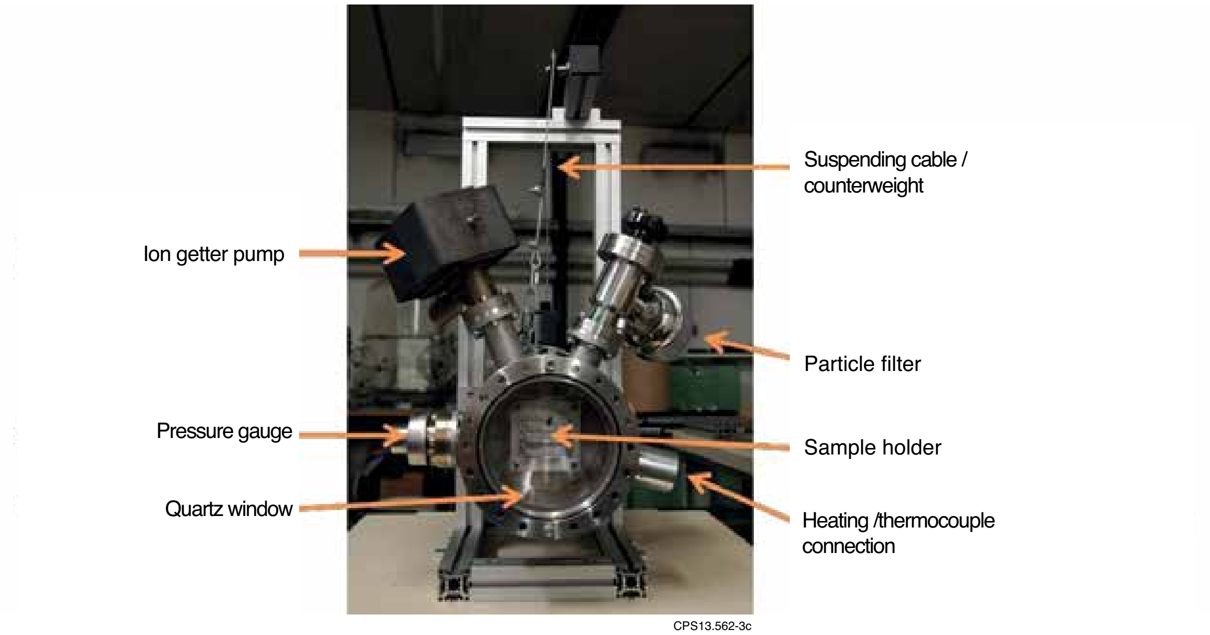


Figure 3: Vacuum chamber before installation of the samples.. The sample holder can be seen through the window. The chamber is suspended from a cable using a counterweight, and attached to motorized translation stages to allow computer controlled xy-motion.

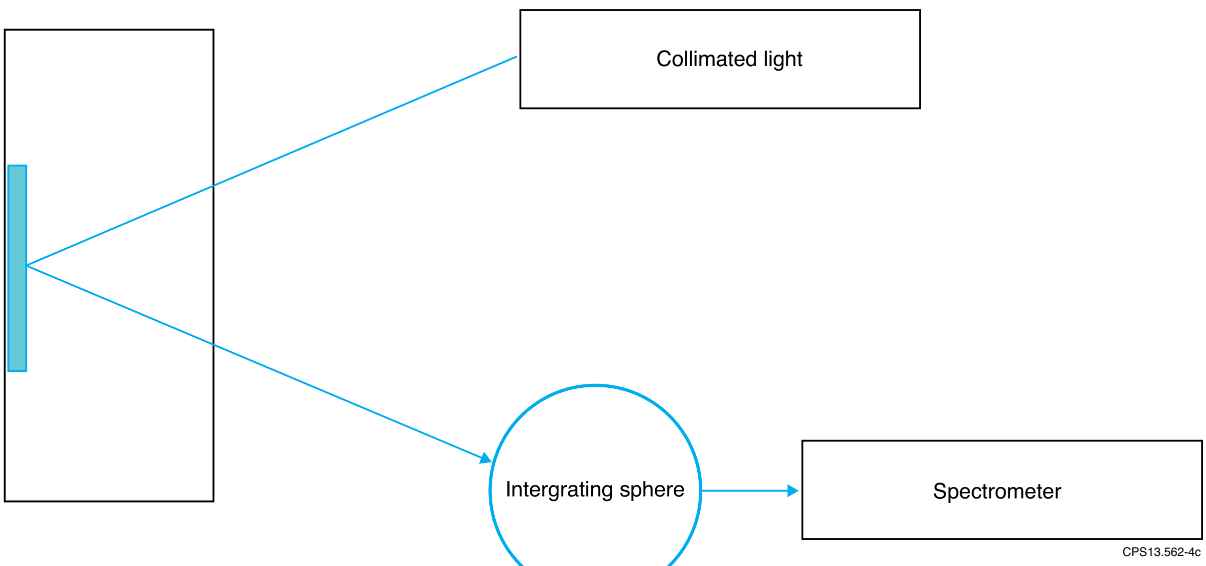
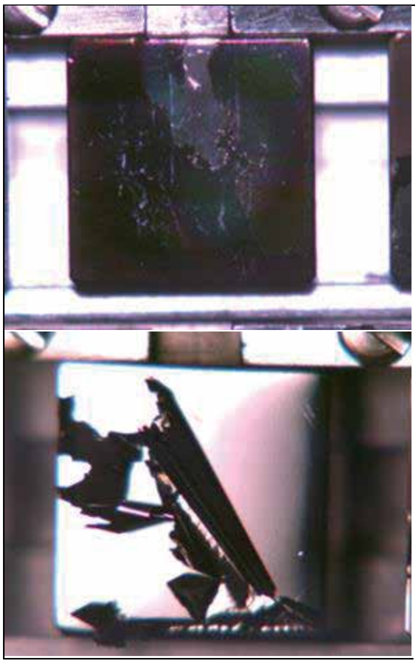


Figure 4: Schematic of the setup used to measure the specular reflectivity of the mirror samples before and after cleaning.



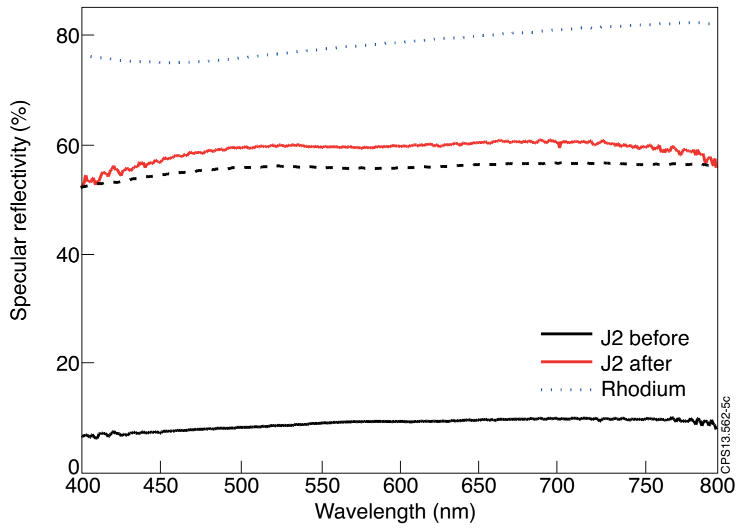


Figure 5: Mirrors J5-J7 before (top) and after (bottom) cleaning. The structure remaining on J5 most likely originates from erosion during exposure in JET.

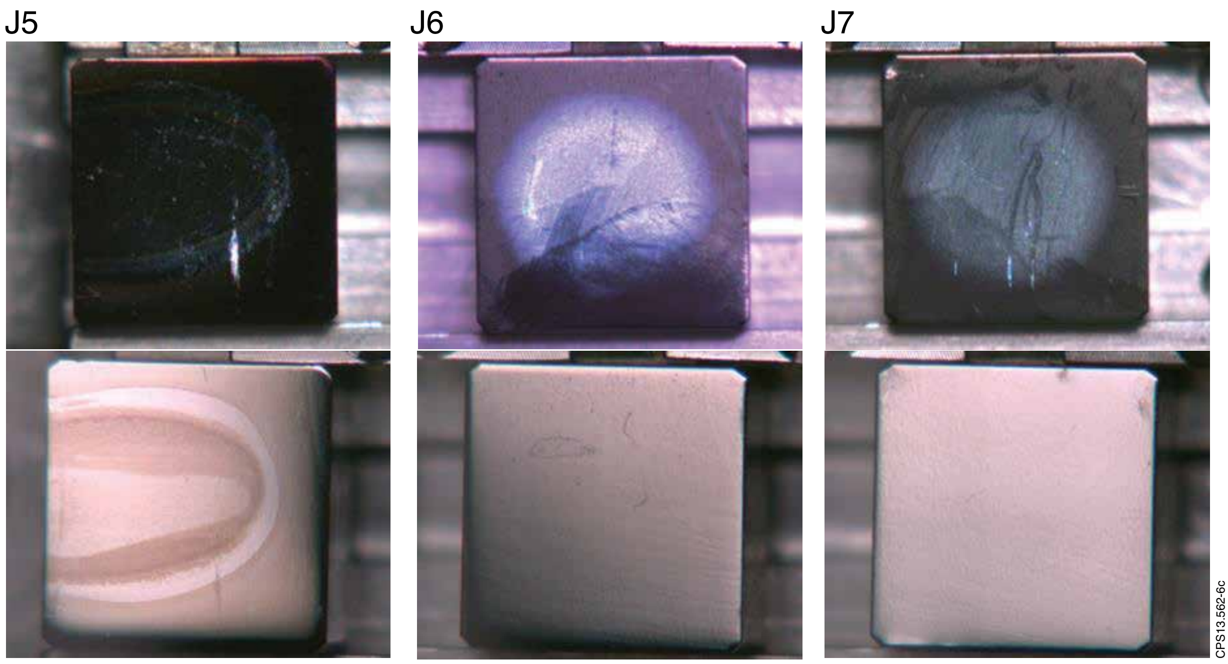


Figure 6: Specular reflectivity of J5-J7 before exposure in JET (dashed), after coating (solid black) and after cleaning (red).

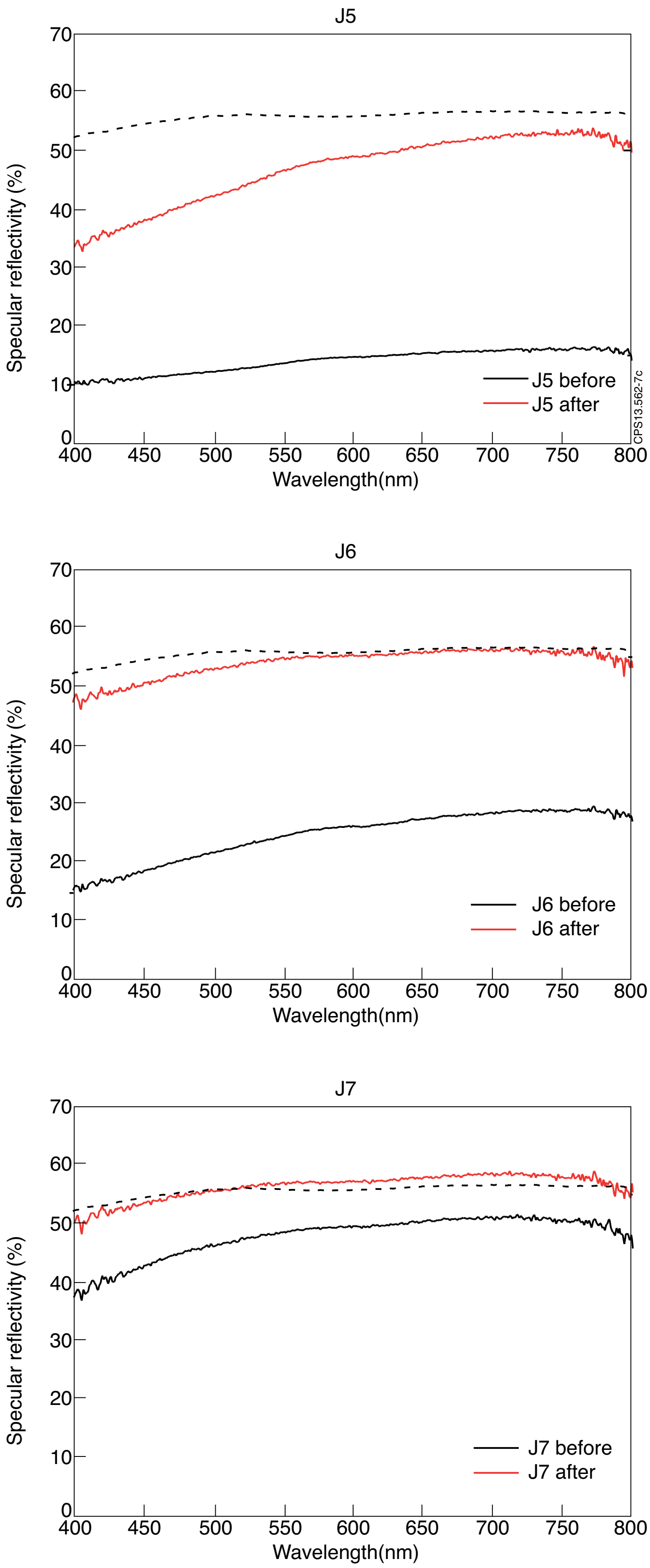


Figure 7: (left) Mirror P1 before cleaning. The left half of the mirror had been coated with Rh before exposure in JET. (middle) P1 after cleaning. Blistering of the Rh layer is apparent in the top lelft in particular. (right) Schematic showing the wavelength and energy used on each of the cleaned patches.

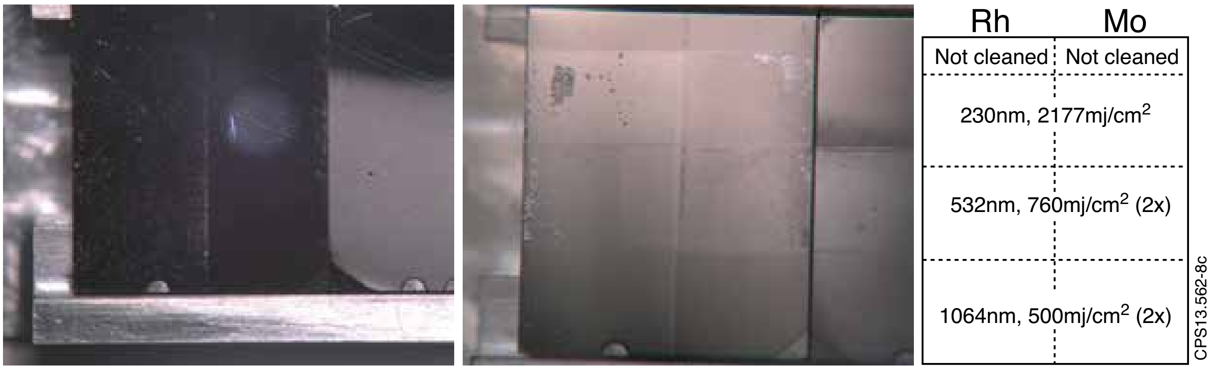


Figure 8: Mirror P3 before (left) and after (middle) cleaning. (right) Schematic showing the wavelength and energy used on each of the cleaned patches.

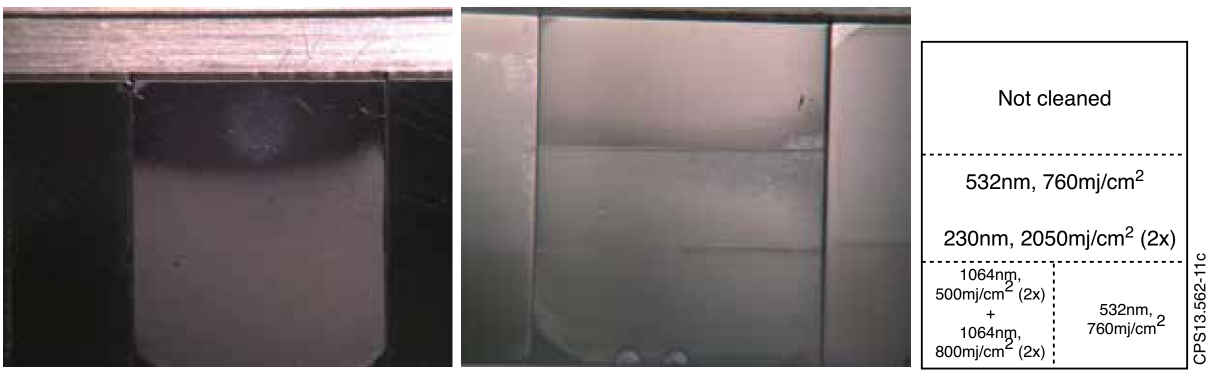
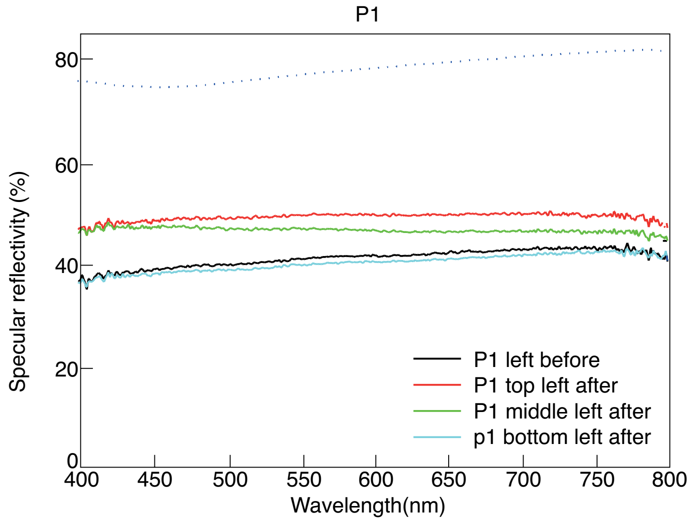
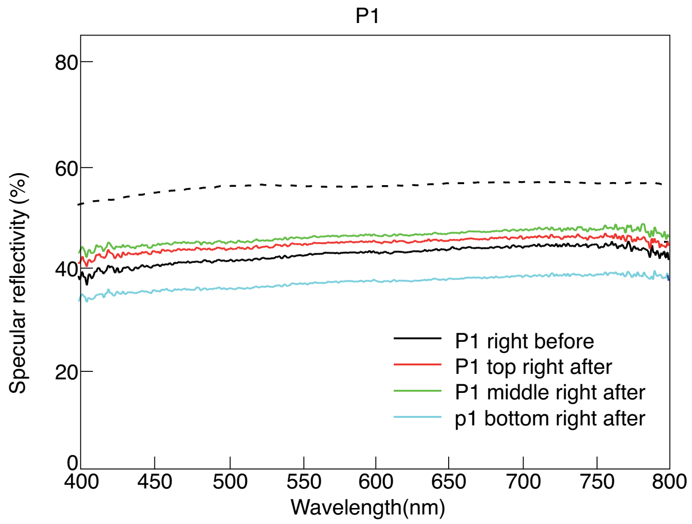
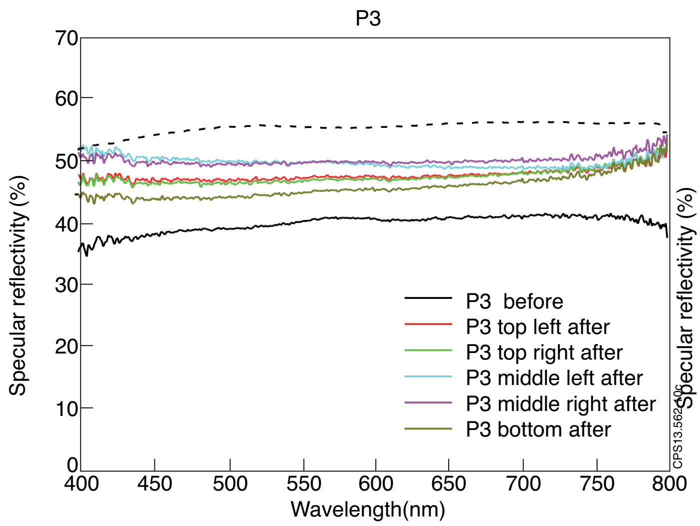


Figure 9: The dashed lines indicate the specular reflectivity of the Rh layer prior to exposure in JET (left) and the specular reflectivity of Mo (middle and right) respectively. The spatial designations correspond to those indicated in Figure 7 and Figure 8.







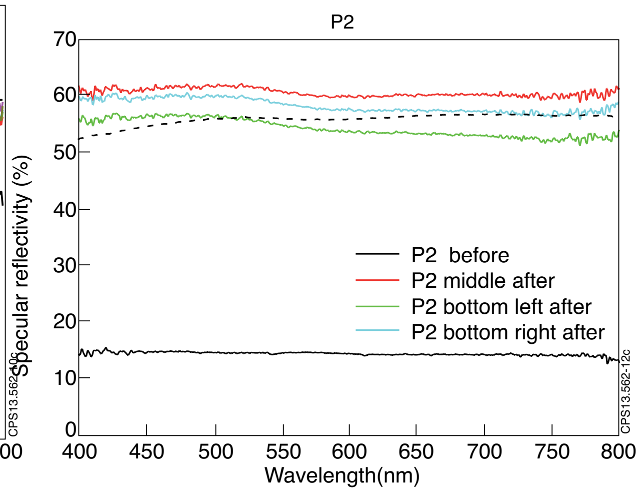


Figure 10: P2 before (left) and after (middle) cleaning. The Be coating was very nonuniform, covering only the lower two thirds of the sampe, and had a very low reflectivity of about 15 % throughout the visible region. (right) Schematic showing the wavelength and energy used on each of the cleaned patches.

Figure 11: Specular reflectivity of mirror P2 before (solid black) and after (solid coloured) cleaning. The black dashed line indicates the reflectivity of pristine Mo.