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ABSTRACT

Two cleaning techniques were used for removal of co-deposits from the tested first mirrors exposed in JET: a) ultrasonic bath; b) a broad range of polishing conditions from manual buffing to machine polishing with the diamond grain size of up to 3μm. Reflectivity measurements were performed after each step in the cleaning procedure. Surfaces were also examined with electron microscopy and ion beam analysis methods. Ultrasonic cleaning leads to partial recovery of reflectivity due to enhanced detachment of deposits. Typically 30%-50% of the original reflectivity was recovered in the visible light and 50%-90% in the infrared region. One mirror was cleaned completely. Polishing with diamond paste may lead to successful removal of deposits but the damage to the surface in case of larger diamond grains was observed. Recovery of up to 100% of the initial reflectivity was achieved in some cases.

INTRODUCTION

Operation of optical diagnostic systems in ITER will rely on the performance of metallic first mirrors. As all plasma-facing components (PFCs), the first mirrors are a subject to degradation due to UV and γ radiation, neutron fluxes and impact of neutrals (e.g. charge exchange, hydrocarbons) which can lead to erosion and deposition processes [1-3]. The ongoing research at the major fusion experiments, i.e. TEXTOR [4], DIII-D [5], Tore Supra [6], HL-2A [7], JET [8] is to provide knowledge on the modification of mirrors and, to elaborate solutions for the prolongation of their lifetime.

The First Mirror Test (FMT) has been carried out at JET on the request of the ITER Design Team. The experiment in JET offers ITER-relevant combination of plasma configuration and placement of mirrors in critical locations. The overall aim of the project is to examine optical performance of tested specimens and to elucidate the cause of reflectivity losses. Two phases of the project in JET with carbon walls were completed: Phase I: 2004-2007, Phase II: 2008-2009 [9,10]. During this time a total of 61 metallic mirrors (stainless steel, molybdenum) were exposed at various locations in the JET divertor and on the outer wall providing a large database of exposure conditions. The set of mirrors for the second phase also included four Rh-coated mirrors which possess higher reflectivity and lower chemical activity compared to molybdenum mirrors [11]. Optical properties of all mirrors exposed in the divertor region were degraded by heavy deposition of the first wall materials such as carbon and beryllium while erosion by plasma impurity species (C, D, Be, Ni, etc) influenced mirrors on the main chamber wall. Implementation of the deposition mitigation techniques [12,13] may prolong the mirror lifetime but none of the methods discussed up to date is able to completely eliminate the growth of deposit. Even thin deposited layers (10nm) can reduce reflectivity of a mirror due to interference effects [3], while the thickness of deposits after exposure in JET often exceeds the micrometer range. Such effects may be expected in ITER, hence efforts are directed towards development and assessment of procedures for reflectivity recovery.

Some techniques for removal co-deposits are based on irradiation of PFC with a high-energy scanning laser beam. Photonic methods provide a possibility of remote operation for in-situ applications and demonstrate reliable removal of carbon layers under laboratory conditions [14,15]. However, when tested on the Be-containing deposits from JET, the laser cleaning did not give satisfactory results [16]. Despite multiple laser scans with the predefined laser parameters, it was not possible to remove all deposits while the damage to the mirror surface occurred: micro-cracking and local melting. The optimization of laser parameters would be challenging as each type of deposit has a different composition, thickness, density and adherence. This in turn, would require a specific set of parameters for each kind of co-deposit to ensure efficient removal. Hence the alternative cleaning techniques should be considered.

The goals of this work were: (i) to clarify whether mechanical cleaning would lead to efficient removal of deposits and recovery of high reflectivity; (ii) to gather more information on properties of mixed layers.

2. EXPERIMENTAL

Testing of the cleaning methods was performed with the test mirrors exposed in JET during 2008-2009 campaign. Surface studies after exposure revealed that co-deposited layers were formed on all mirrors exposed in the divertor and also on the mirrors located at the main chamber wall deep in the cassette channel, i.e. 3.0 and 4.5 cm from the channel mouth, as detailed in [10].

Two different cleaning methods were tested on these mirrors: a) ultrasound cleaning (US) in organic solvent; b) a broad range of polishing conditions from manual buffing to machine polishing. After each significant step in the cleaning procedure a visual inspection and total reflectivity measurements in the range 350-1700 nm were carried out using a GetSpec spectrophotometer suitable for work with beryllium and tritium contaminated samples. After cleaning specular and diffuse reflectivity of all mirrors was recorded in a wider range of wavelengths (250-2500 nm) by Varian Cary 5 system. The mirrors have also been examined prior to and after the cleaning with optical microscopy, Scanning Electron Microscopy (SEM) X-ray Photoelectron Spectroscopy (XPS), Ion Beam Analysis methods (IBA), e.g. Nuclear Reaction Analysis (NRA), Heavy Ion Elastic Recoil Detection Analysis (HIERDA) and secondary ion mass spectrometry (SIMS).

Ultrasonic cleaning was the first tested technique. Mirrors were placed in the ultrasonic bath (Sonomatic® 2800, effective HF-Power 450W, frequency 33kHz) which is routinely used for cleaning various components at JET. A set of mirrors was placed in isopropanol and treated by ultrasound for 55 minutes.

The second cleaning approach was based on mechanical polishing. In a preliminary test the manual buffing was tested for the most flaky and poorly attached deposits. Afterwards the cleaning would continue on the automatic polishing system (Struers®, 150 rotations per minute), which allowed for simultaneous treatment of up to 3 mirrors with a force of 30 N/cm^2 applied individually to each mirror. Polishing was done in steps lasting two minutes each until the initial (pre-exposure) reflectivity was reached. The measure of cleaning efficiency in this case would be the time required to reach the pre-exposure state. Another factor affecting the efficiency of cleaning was the type of a diamond paste grain size (1µm, 3µm).

In total 11 mirrors were treated by ultrasound and 13 mirrors were cleaned by polishing of which 7 mirrors underwent both cleaning procedures. The selection of mirror samples was representative for all the mirror locations: the main chamber wall, the inner and outer divertor legs, the divertor base.

3. RESULTS AND DISCUSSION

Many years of experience in storage and transportation the exposed mirrors with flaking carbon-based deposits demonstrated, that the exposure to air further enhances the brittleness of the layers. On several occasions the deposits partly peeled-off in the torus, during the dismantling of mirrors or during their transportation [9,10]. These observations suggested that even moderate cleaning of such mirrors in ultrasonic bath could be considered as an efficient cleaning method.

3.1. ULTRASOUND CLEANING

The results of the ultrasound cleaning varied in a broad range of effects from the minimal impact on reflectivity to the complete removal of deposit. Figure 1a shows the case when the US cleaning did not lead to any noticeable effect for the mirror exposed on the outer wall of the main chamber. The example in Figure 1b demonstrates a mirror from the inner divertor with the fully restored reflectivity. For most mirrors the performed treatment resulted in partial recovery of reflectivity due to enhanced detachment of deposits. Typically 30%-50% of the original reflectivity was recovered in the visible range on spectra and 50%-90% for the infrared region. This result was expected since the longer wavelengths are less sensitive to the surface imperfections and a similar dependence in recovery efficiency was observed during the earlier cleaning attempts by laser pulses [16].

The efficiency of US provides initial qualitative information on the adherence of co-deposits. Lack of any reflectivity improvement after the bath would signalise the need for harder polishing conditions.

3.2. POLISHING

Figure 2 demonstrates a successful cleaning process of a mirror exposed on the outer wall of JET. Restoring the initial level of reflectivity by polishing with 1μm diamond paste took about 30 minutes and gradual improvement in reflectivity was recorded after each 2 minutes of polishing (only a few reflectivity curves are included in the plot in Figure 2). Time required to clean a mirror varies from 2 to 40 minutes for different samples and depends on the exposure conditions such as location of a mirror in JET vessel or depth into the cassette channel. Prolonged polishing (over half an hour) was required to clean the mirrors which were located on the outer wall during JET operation and deep in the cassette channel. Contrariwise, the thickest deposits from the mirrors in the divertor at the channel mouth happened to be poorly attached and cleaning of such mirrors required relatively little effort. Irrespective of the exposure location of mirrors, manual buffing and mechanical polishing with a clean soft cloth without diamond paste proved to have minimal impact on reflectivity of mirrors even in the case of flaking and loose deposits. The best observed effect was not more than 10% increase in reflectivity in the infrared range.

The incremental approach to the cleaning process allowed observing qualitatively different layers of deposit on some mirrors. Images in Figure 3 demonstrate the same mirror before the cleaning (a), after 6 minutes of polishing with 3μm paste (b) and after 16 minutes of polishing with the same paste (c). The corresponding change in the reflectivity curve is shown in Figure 3b. This mirror was located in the inner divertor of JET and remained in the torus during two consecutive campaigns, i.e. it was exposed to ambient atmosphere during the shut-down period between the campaigns. As a result, the lower older deposit was exposed to air with subsequent oxidation. The second layer of deposit was built up during the repeated exposure to plasma.

While cleaning efficiency of polishing may virtually reach 100%, usage of the diamond paste with large grain size (3 μm) increases the probability of damaging the mirror surface via scratching. However for most of the studied mirrors this type of damage did not affect the specular reflectivity, as shown in Figure 5, and the recovered specular and diffusive reflectivity are on the pre-exposure level [8]. Additional improvement can be achieved by finishing polishing with a fine grain paste. The polishing process is very sensitive to the mirror flatness. In the presence of any manufacturing imperfections some small areas on the mirror surface may be unreachable for cleaning and the deposit would not be removed (Figure 4).

This fact has also been confirmed by HIERDA studies which proved that polishing completely removes the deposit from most of the surfaces. Only in some cases the minimal amounts of impurities (H, C, O, Si) were observed on the polished surfaces due to not uniform removal of the deposit. The most important was the absence of deuterium on the cleaned surfaces thus suggesting that the observed hydrogen attributed to the adsorption of water vapour from atmosphere. Traces of Be were detected on the mirrors from the outer divertor where a high concentration of Be was observed after exposure. As revealed by XPS, molybdenum in the surface layer is oxidized.

In summary, satisfactory recovery of reflectivity may be achieved but cleaning conditions must be individually set for each mirror. This general statement is especially valid when treating mirrors with thin films. The impact of the ultrasonic bath on the Rh coating was comparable to that on the pure molybdenum mirrors and the restored reflectivity was as high as 90% of the initial Rh reflectivity for the infrared light. However, this method alone was not enough to clean such mirrors, whereas the mechanical polishing with diamond paste was too rough for these surfaces and lead to disintegration of Rh coatings.

CONCLUDING REMARKS

Mirrors after the exposure in JET during the 2008-2009 campaign were cleaned by two different techniques: ultrasound bath and polishing. The selection of mirror samples was representative for all the mirror locations: the main chamber wall, the inner and outer divertor legs, the divertor base. Ultrasonic cleaning leads to partial recovery of reflectivity due to enhanced detachment of deposits. Typically 30%-50% of the original reflectivity was recovered in the visible light and 50%-90% for the infrared region. Full recovery of optical performance by the US methods was achieved in one case. Unlike the ultrasonic bath, polishing provides consistent results and appears to be very effective. Manual buffing and mechanical polishing with a soft polishing cloth had minimal impact on reflectivity of mirrors even in the case of flaking and loose deposits. Polishing with diamond paste may lead to successful removal of deposits but the duration of such treatment requires accurate estimation. The damage to the surface in case of larger diamond grains was observed, while smaller grain size would result in significant increase of the required time.

Results show that ultrasound alone is not sufficient for surfaces coated by deposited carbon-metal mixed material layer. In the case of mirrors modified by co-implantation of impurity species (on main chamber wall) the only way to recover the initial reflectivity of these mirrors is to remove the modified layer (50-200nm) by polishing. The results obtained until now indicate that the replacement - or repeated coating - of first mirrors may be needed in the case of their degradation in a reactor-class machine.

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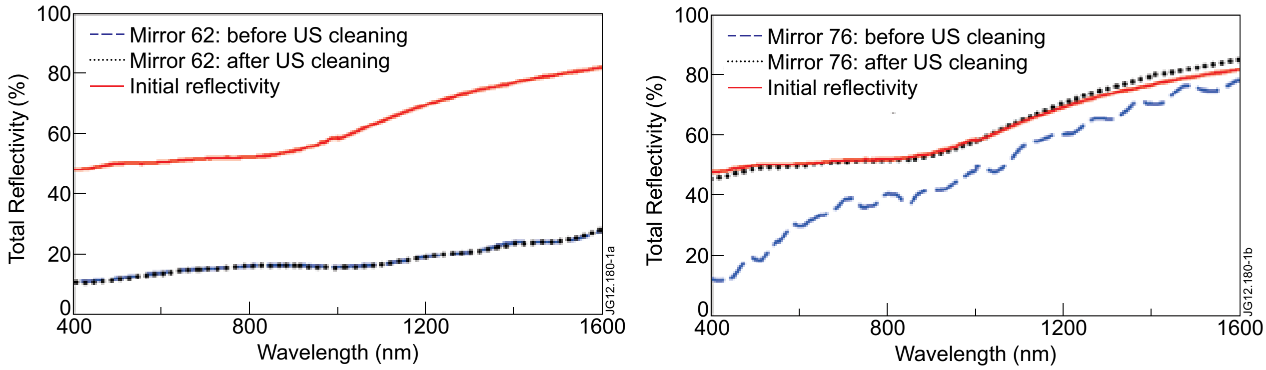

Figure 1: Efficiency of ultrasound cleaning performed on mirrors from the outer wall (#62) and the inner divertor (#76): a) no recovery of reflectivity, b) full recovery.

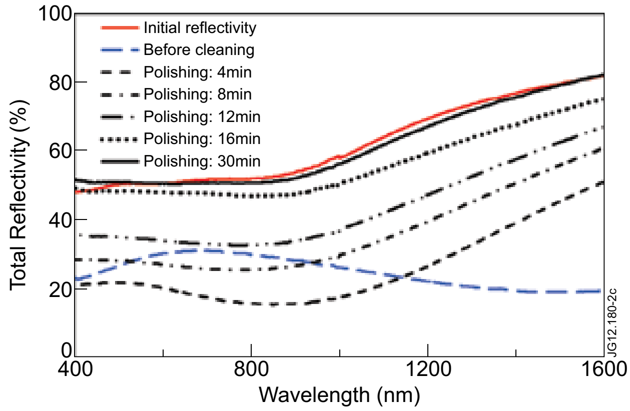

Figure 2: An iterative polishing process with 1μm diamond paste performed on a mirror exposed on the outer wall of JET.

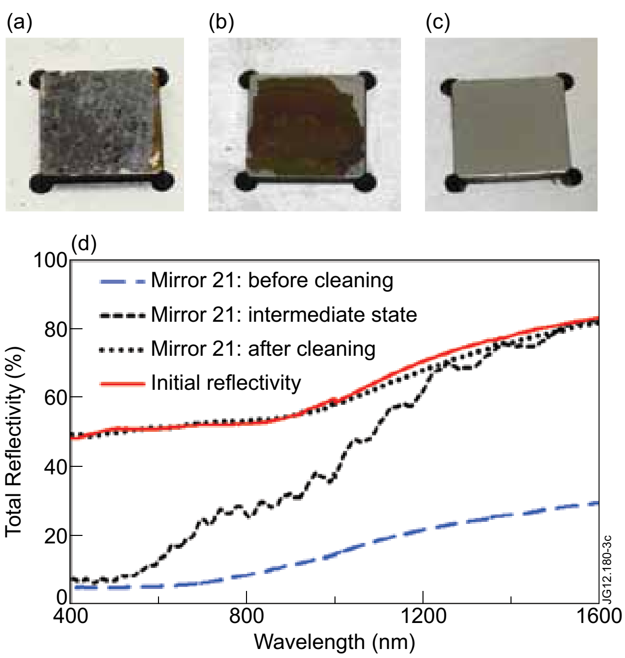

Figure 3: An example of the earlier deposited layer revealed during polishing (d). Photos depict the mirror before cleaning (a), after 6 minutes of polishing (b) and after 16 minutes of polishing (c).

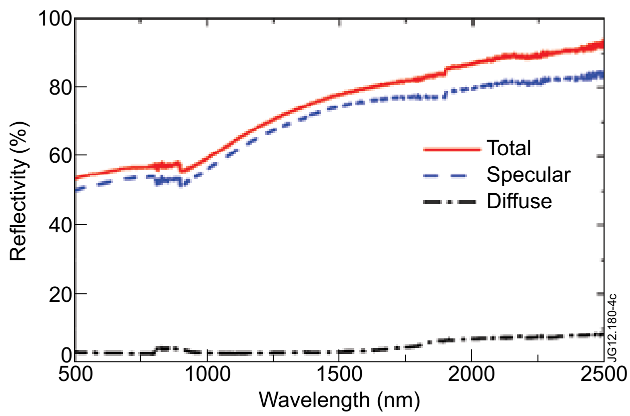

Figure 4: Specular, diffuse and total reflectivity of a mirror after cleaning.