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# Some problems arising due to plasma–surface interaction for operation of the in-vessel mirrors in a fusion reactor

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#### Abstract

Effects of two contradictory processes on the survivability of the in-vessel mirrors for diagnosing plasma in a fusion reactor, namely, the sputtering by charge exchanged atoms and deposition of eroded material, are analyzed in detail using results of simulation experiments. The degradation rate of mirrors bombarded long-term by keV energy range ions of deuterium plasma was found to depend strongly not only on the mirror material but also on its initial structure and energy of projectiles. The highest resistance was demonstrated by mirrors fabricated of monocrystal W and Mo and the lowest by mirrors fabricated of hot-pressed Be. As for the effect of mirror contamination, an effective reflectance was measured and calculated for some metal mirrors coated by thin films typical for existing fusion devices or for a fusion reactor: boron, carbon, and beryllium. © 2001 Elsevier Science B.V. All rights reserved.

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#### 1. Introduction

The essential in-vessel components of some methods of plasma diagnostics in a fusion reactor will be the first mirrors (FM). Positioned in view of the hot plasma, these mirrors will be subjected to the same kind of radiations as the first wall. As simulation experiments demonstrated [1], the strongest effect on optical properties of metal mirrors will occur due to charge exchange atoms (CXA). The difference in CXA flux values to the first wall and to FM will depend on the mirror locations.

According to the ITER diagnostic scheme [2], the FMs of wide angle viewing systems will undergo the influence of the highest fluxes of radiation being those which are nearly flush-mounted with the first wall, and the Thomson scattering FM will experience the lowest fluxes. The spectroscopy mirrors will be placed not so deeply in diagnostic ducts as the latter one and will be irradiated by intermediate CXA fluxes.

The CXA bombardment can lead to the development of a microrelief on the mirror surface with corresponding degradation of reflectance, *R*. The rate of roughness growth depends strongly on the mirror material as well as on its structure, and one goal of this paper is to seek the correlation between reflectance degradation and the initial characteristics of material when mirrors are long-term bombarded by ions of deuterium plasma.

The second goal is studying the effect of an opposite process on R, which is characteristic for fusion devices,

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i.e., deposition of a contaminating film on the mirror surface. In most fusion devices under operation, the film growing on the inner surfaces of the vacuum chamber represents the carbon–boron film with a large amount of trapped hydrogen (deuterium). Till recently, only the effects of film growth on windows of laser scattering and spectroscopy were investigated [3–6]. Meanwhile, the deposited film can influence the mirror reflectance more strongly than the window transmissivity.

# 2. Long-term sputtering effect on evolution of microrelief with different initial structure

### 2.1. Experimental

We compared the behavior of mirrors fabricated of several metals of a polycrystalline structure (Be, Al, V, SS, Cu, Mo, Ta, W), mirrors made of single crystals (Mo and W) with different orientation of crystalline planes, and mirrors made of metal film on metal substrate (Be on Cu, Cu on Cu and Rh on Cu). As an ion source, the ECR discharge in deuterium was used with fixed or time-variable (negative) voltage accelerating ions to the mirror surface. In the latter case, the ion energy distribution was quite wide, in the range 0.1–1.5 keV [7]. Because of high difference in sputtering yields for these metals, the thickness of sputtered layer, h, instead of ion fluence was used as an independent variable. The temperature of water-cooled mirror holder did not differ noticeably from the room temperature. The reflectance under normal incidence in the range 253-650 nm and the mass loss of specimens (i.e., the h values) were measured ex situ after every exposure to plasma. Thus, for mirrors of the above mentioned metals, the R(h) dependences for several wavelengths were obtained. The surface morphology at different stages of mirror treatment was studied by SEM.

### 2.2. Effect of long-term sputtering on bulk metal mirrors

It was found that ion bombardment results in decrease of R with different decrement (as a function of h) for different metals [1]. As a rule, the reflectance decrease correlates with gradual increase of the mirror surface roughness. Under identical conditions, beryllium mirrors fabricated of a hot-pressed material showed the highest degradation rate among all metals tested, without direct connection with the sputtering yield, and this was attributed to high internal stresses appeared due to technology of mirror preparation. Very similar qualitative behavior was obtained with mirrors fabricated of Mo prepared by powder metallurgy (PM). Its reflectance versus h dropped much faster than for Mo mirror fabricated on different technology (Fig. 1).

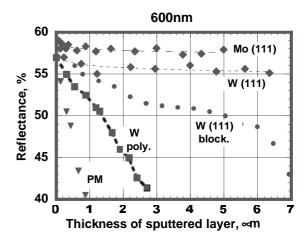


Fig. 1. Reflectivity at  $\lambda = 600$  nm for normal incidence versus thickness of sputtered layer of mono- and polycrystalline tungsten and molybdenum mirrors.

For the not-pressed mirrors made of polycrystalline metals, the characteristic feature of microrelief change under long-term ion bombardment was the appearance of a stepped structure (tens to hundreds um in size) due to different sputtering rates of grains with different orientation of crystalline planes. The stepped structure is most clearly seen on Cu and SS (analog of AISI 316 steel) surfaces even after a rather thin layer was sputtered [7]. The stepped structure transforms the mirror into a reflector with deterioration of an image transmission, but the absolute reflectance at normal incidence is affected in a considerably less degree. For these mirrors, the effect of ion energy variation on R degradation was studied in detail. It was discovered that R(h) decreases faster for higher ion energy, and for copper mirrors, the rate of degradation was much faster than for SS mirrors, Fig. 2. The main difference between the behavior of these metals consisted in the appearance of a chaotic microrelief of the etching pits inside almost every grain on the copper surface, practically in parallel with stepped structure growing and surface density and size of pits increased when ion fluence increased. In qualitative agreement with R(h) behavior, the in-grain microrelief did grow faster when mirror was sputtered by ions with higher energy [7] (note that bulk polycrystalline Ta and W mirrors behaved very similarly having passed a similar test [8]). This chaotic microrelief is probably a result of the crystallographic defects etching of the grain faces. In distinction from Cu, on SS surfaces, the grains turned into the very smooth plateaus, without developing the chaotic microrelief, even after sputtering layer with thickness  $\sim$ 5 µm. The SEM photos for Cu and SS mirrors are shown in Figs. 3(a) and (b).

In the case of W and Mo single crystal mirrors, there was not observed any reduction of reflectance even after

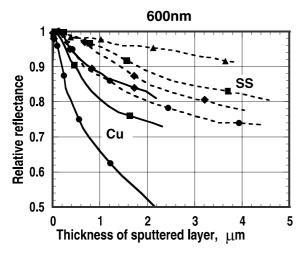


Fig. 2. Reflectivity at  $\lambda = 600$  nm versus thickness of sputtered layer of stainless steel and copper mirrors bombarded by ions of different energy. Solid lines and open markers for Cu and dashed lines and solid markers for SS. Triangles:  $E_i = 0.3$  keV; rhombus:  $E_i = 0.65$  keV; circles:  $E_i = 1.5$  keV; rectangles: wide energy distribution E = 0.1–1.5 keV.

sputtering layer of  $\sim$ 7 µm thick (Fig. 1) in agreement with the observation of no significant change in topography. At the same time, the rate of degradation of the block monocrystal tungsten was very significant, though much slower than for the polycrystal material.

# 2.3. Effect of long-term sputtering on single film mirrors

It is known that the size of grain in polycrystalline metal films (of 1–10  $\mu$ m in thickness) is much less than that in solid polycrystalline metals. Therefore, the stepped microrelief being developed under long-term ion bombardment of a film mirror also has to be of very small scale, not strongly affecting the initial smooth surface and the initial R value. The check of this hypothesis was carried out by testing some mirrors fabricated as a metal film on metal substrate: Be and Cu film on Cu substrate and Rh film of different thickness on some metal substrates. The results obtained will be shortly reviewed and discussed below.

The Be film deposited by magnetron sputtering on the polycrystalline Cu substrate in facility MAGRAS [9] has much lower reflectance than was measured for films deposited in a high vacuum [10]. The Be film occupied  $\sim$ 45% of the whole area of Cu substrate (22 × 22  $\mu$ m²), thus it was possible to compare R(h) for both surfaces in identical conditions. The thickness of Be film layer sputtered during every exposure to plasma ions ( $E_i = 0.65 \text{ keV}$ ) was estimated by measuring the mass loss of specimen taking into account the sputtering yield of copper measured earlier and exact relation between Be-coated and non-coated parts. In Fig. 4, the R(h) data

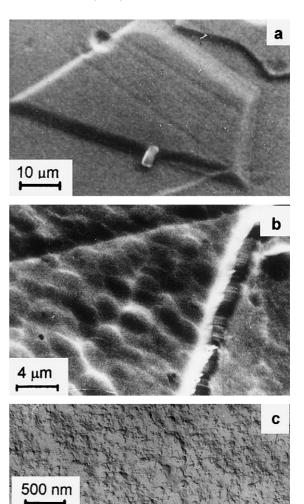


Fig. 3. Micrographs of surface: bulk polycrystal mirrors: (a) SS, (b) Cu after bombardment by ions of deuterium plasma with energy 1.43 keV,  $h \approx 4~\mu m$  and (c) Rh film mirror after bombardment D<sup>+</sup> ions with wide energy spectrum,  $h \approx 2.5~\mu m$ .

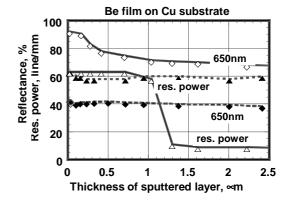


Fig. 4. Behavior on sputtered layer thickness of reflectivity and resolving power of Be film (dotted lines) and polycrystalline Cu (solid lines) mirrors.

for both surfaces are shown for  $\lambda=650$  nm together with a resolving power. The latter is the number of lines per mm seen after the test-object was imaged by mirror under the test, i.e., it characterizes the mirror quality. As seen, a Cu mirror practically fully lost the property to transmit an image already after sputtering layer of  $\sim 1$   $\mu$ m thick, though the power reflectance efficiency under normal incidence still was high enough. As for the Be film, it did keep the low-scale microrelief (similar to shown at Fig. 3(c) for Rh film) and maintained the optical properties during all sputtering time, limited by the film thickness.

Very similar was the behavior under long-term sputtering of the Cu film on a polycrystalline Cu substrate. The part of specimen initially coated by Cu film (of  $\sim 1.5 \, \mu m$  thick) kept the mirror-like quality of surface until the film was fully sputtered. At the same time, the non-coated part of specimen came to be similar to what was typically observed for a polycrystalline copper sample after sputtering layer of same thickness, as is shown in Fig. 3(b).

Rhodium is considered as a potential material for the film mirrors of plasma diagnostics in ITER [2]. However, the property of rhodium to maintain the optical properties after being ion-bombarded was not investigated until recent time. In our simulation experiment, the Rh films deposited on Cu, Ni and V were tested. The thinnest was a film on Ni ( $\sim$ 0.15 µm) and the thickest ones – on copper substrates (5, 10 and 14 µm). The latter mirrors were obtained by newly developed technology [11], without polishing after fabrication. In visible spectrum, their reflectance was ~8% below values of Rh mirrors fabricated by other technologies (Rh/Ni and Rh/ V). Some of the Rh/Cu mirrors maintained the reflectance after 7-8 micron layer sputtered. The Rh/V mirror behaved very differently. Soon after beginning bombarded, here and there the Rh surface became to show tarnished areas. Pretty similar patterns appeared on the surface of V mirror tested after mechanical polishing, so it looked as though the V substrate dictated the structure of the Rh film surface.

# 2.4. Discussion

It follows from data for long-term sputtering of metal mirrors that appearance of a stepped structure can be avoided by utilization of materials either with ordered structure (single crystals) or structure with small-scale size crystals (i.e., films). In both the cases, the maintenance of mirror reflectivity is possible even after sputtering a layer  $\sim$ 7 µm thick. Taking this quantity into account, one can estimate the total fluence of CXA ( $T^0$  and  $D^0$  in the case of a fusion reactor) which is necessary to sputter such thickness of a given material, if the energy distribution of atoms is known. Until now there is only one example of calculation of atom energy distri-

bution and flux on the first wall for ITER conditions [12]. Using data from that paper, i.e., atom flux  $\sim 3 \times 10^{19} \text{m}^{-2} \text{ s}^{-1}$ , atom mean energy not more than 250 eV, it is easy to see that monocrystal W and Mo mirrors can withstand during all the service time of ITER operation. The Rh film mirror of  $\sim 10~\mu \text{m}$  in thickness can withstand degradation during this time only if recessed in a duct which provides one order of magnitude weakening of atom flux to the Rh surface, and the film may be thinner for stronger flux weakening. Thus, in the case that sputtering is very dominant over deposition, the possible solution of the FM problem is monocrystal or metal film on metal substrate mirrors.

### 3. Effect of contaminating film on mirror reflectance

It is planned to use in ITER several materials for protection of the first wall and in the divertor region: tungsten, beryllium and carbon-based composite. The eroded beryllium and carbon will be transported in the vacuum vessel and accumulated on the deposition-dominated surfaces, i.e., on remote components of reactor construction, including diagnostic ducts with mirrors installed. As was shown (e.g., [13]), even in current fusion devices, the deposited layer can reach a few µm in thickness. Therefore, for much longer pulse duration in ITER, the deposit appearance on the FM surfaces cannot be excluded also.

Effects of boron-carbon (B-C) film on reflectance of the in-vessel mirrors is of interest in connection with the prevalence of the boronization procedure on a majority of modern fusion devices where some inner components are made of carbon-based materials. Data on structure and composition of B-C films in fusion devices were obtained by many authors [14], however, optical properties (i.e., n and k values) were obtained only once, for the carbon-based film which appeared on one of the diagnostic windows of JT-60U [15]. In contrast, in a special experiment [16], the properties of boron-based films were studied in detail for various experimental conditions, and these data can be used for prediction of contaminating film effect on metal mirror reflectance. Fig. 5 shows dependence of effective reflectance of SS mirrors coated by boron film in comparison with the result of a calculation provided for n and k data of the boron film and SS substrate. For the calculation of  $R_{\text{eff}}$ at normal incidence of the pair 'a semi-transparent film on metallic surface', the formula published in [17] was used. As seen, the boron film of only ~10 nm in thickness modifies the reflectance of SS mirror. A qualitatively similar effect occurs when metal mirror is coated by carbon film of similar thickness. Metals have a high extinction coefficient, thus the appearance of a Be film on the in-vessel mirror has to lead to much stronger change of mirror reflectance. In practice, the mirror of

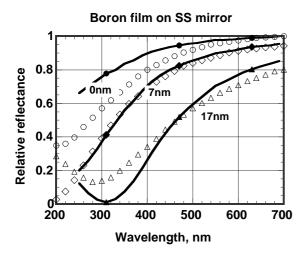


Fig. 5. Effect of boron film deposition on reflectance of stainless steel mirror.

any metal coated by Be film with  $\geq 20$  nm in thickness changes typically the optical characteristics for the bulk beryllium mirror.

#### 4. Discussion

Thus, the contaminating film of only 10-20 nm thick can drastically deteriorate the reflectance of metal mirror. So, the investigation of mechanisms of film growth on the FM surfaces is very important. Until now the only process potentially responsible for deposit growing on the diagnostic windows (and on the FMs in a fusion reactor) was discussed in [18]. It concerns the redeposition of material of the diagnostic duct sputtered by CXA. The use of material with lowest sputtering yield (W, Ta) for the duct wall protection could significantly decrease the role of this process in the contamination of FMs. However, such simple decision will have only minor effect in solving the deposit problem if the erosion products of components with high sputtering yield diffuse inside the duct due to the creation of volatile molecules (boron or carbon), i.e., when chemical erosion is predominant. In such a case, the effective methods of FM cleaning in situ from contaminants have to be developed.

# 5. Conclusion

It follows from results of simulation experiments that the hot plasma radiation can significantly modify the optical properties of the in-vessel mirrors either by directly (first of all, because of bombardment by CXA) or indirectly (redeposition of eroded material). In both cases, the effect will depend mainly on characteristics of CXA flux for any given mirror location and any combination of materials of the fusion reactor inner components. The long-term sputtering will result in mirror deterioration by two ways: increasing the surface roughness and growing a contaminating film. In the case the sputtering is predominant, there could be a two-fold solution of the FM problem: the use for mirror fabrication of the single crystal of metals with low sputtering yield or the single film on metal substrate with the film thickness high enough ( $\geqslant 10~\mu m$ ).

It can turn out, however, that deposition of contaminants will be the much more important reason for deterioration of mirror optical properties compared to CXA sputtering effects. Therefore, it is extremely important to develop the model of deposit evolution and to check it at operating fusion devices.

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