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On the choice of materials for the first mirrors of plasma diagnostics in a fusion reactor

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Abstract

The reflectivity coefficient, *R*, of mirrors fabricated from different metals (Al, Cu, Mo, Ta, W) in the wavelength range 0.25–0.65 μm was investigated under long-term sputtering with ions of ECR plasma of hydrogen or deuterium, and *R* values versus thickness of sputtered layer were measured. For copper mirrors the influence of ion energy and ion energy spectrum on optical reflectivity was investigated in detail. Because a strong effect was found, the comparative test of mirrors made of different metals was carried out using ions with a wide energy distribution (0.1–1.5 keV) to have a more adequate simulation of fusion reactor environment conditions. The results obtained show that, with the exception of Al, the decrease of reflectivity coefficient with thickness of sputtered layer has approximately similar rate for metal tested. Thus, mirrors made of materials having lower sputtering coefficient, *Y*, will withstand a higher charge exchange atom fluence, in qualitative agreement with *R*/*Y* criterion suggested by the authors earlier. © 1998 Elsevier Science B.V. All rights reserved.

1. Introduction

According to the present state of the ITER Project [1], about half of methods of plasma diagnostics planned will need to use "first mirrors" (FM), which must be installed inside the reactor vacuum chamber in a straight vision of the core or divertor plasmas. Due to their location, FM will be subjected to bombardment with charge exchange (CX) atoms. Results of such an influence will be determined not only by properties of the FM material but also by the characteristics of CX atom flux: its value and atom energy spectrum. Up to now there is only one calculation of CX atom energy spectrum onto the ITER first wall [2]. This spectrum is very broad with total flux $\sim 2 \times 10^{15}/\text{cm}^2$ s, and the flux onto the mirror surface will depend on the mirror location, i.e., diameter and length of the mirror duct. The long-term sputtering of the mirror surface will result in increasing roughness and decreasing reflectivity (*R*). Thus, the optical stability under long-term CX atom bombardment should be a real criterion for the choice of a material for mirrors of a core plasma. In accordance with this criterion, the widely used mirror metals (Al, Cu, Ag, Au) are behind metals (Mo, Ta, W) having lower sputtering coefficients and reasonable values of reflectivity [3].

In this paper we present results of *R* measurements in visible spectral regions for mirrors made of several metals after their surfaces were long-term bombarded with ions of hydrogen or deuterium ECH plasmas. As distinct from other simulation experiments, the ion energy spectrum in our case was rather wide, (0.1–1.5 keV),

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in qualitative accordance with the predicted [2] and measured [4–6] CX atom energy spectra. Thus, better correspondence to the fusion reactor environment conditions was realised. Results for mirrors made of Al, Cu, Ta and W are given as dependencies of *R* versus sputtered layer thickness, *h*. From data obtained, Al and Cu (at small *h*) mirrors show much faster degradation than mirrors made of refractive metals. Data for *R* change are analysed along with a microstructure of mirror surface after ion bombardment for metals investigated.

2. Experimental details

As plasma source, a cw Electron Cyclotron Resonance (ECR) discharge (frequency 2.37 GHz) sustained in a conventional mirror-type magnetic trap was used. The stainless steel vacuum vessel was evacuated by a turbomolecular pump down to a base pressure $\sim 3 \times 10^{-4}$ Pa, and during exposure of samples the working gas (hydrogen or deuterium) was continuously fed in a pressure range $(3–5) \times 10^{-2}$ Pa. The typical plasma parameters with 200–400 W UHF power injected into the vacuum vessel were as follows: $n_e \leqslant 10^{10}/\text{cm}^3$ and $T_e \sim 5$ eV.

The mirror sample was inserted by a special holder into the plasma stream flowing out of the magnetic trap. In the case of exposure to fixed energy ions the mirror was negatively biased up to 1.5 kV. The variation of ion energy was realised by adding (to the fixed negative voltage) the positive half-wave voltage from a separate biphase rectifier. In Fig. 1 the time dependence of this part of voltage is shown as the curve *a*. Due to combination of two voltage sources, the effective energy spectrum of ions bombarding the mirror surface was spread between 0.1 and 1.5 keV. Curve *b* in Fig. 1 represents the ion energy spectrum obtained taking into account the sample current-voltage characteristic but not including the ion-electron secondary emission. The mean ion current density onto the sample was of the order of 1 mA/cm^2 and the average sample temperature was maintained near RT.

Al, Ta and W mirrors of 22 mm in diameter were prepared by mechanical polishing, and the diamond-turned Cu mirrors ($22 \times 22 \times 4$ mm^3) were manufactured of an oxygen-free material. The *R* value was measured at the normal incidence using a home-made attachment to a standard monochromator by means of the two-channel method [7] with reproducibility of ~1%. $R(\lambda)$ dependence for several points in the $\lambda = 253–650$ nm range, was measured as a function of the thickness of the near-surface layer sputtered by plasma ions accelerated by the sample biasing. The sputtered layer thickness, *h*, was determined by measuring the mass loss (Δm) of the tested sample. The $R(h)$ dependencies were obtained for many step by step exposures of mirrors to plasma measuring $R(\lambda)$ and Δm after every step. For these measurements the samples were taken out of the vacuum vessel, and after several number of repeated steps of sputtering the mirror surface was analysed by means of Scanning Electron Microscope (SEM).

3. Results

The important role of the projectile energy distribution for the proper simulation of environment effects on FM optical properties is evident from data of Fig. 2.

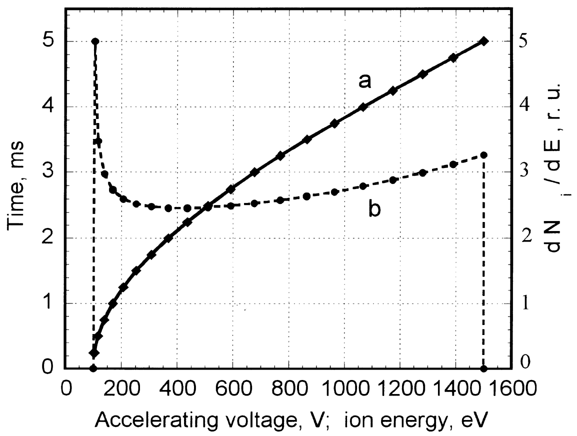

Fig. 1. (a) time variation of ion accelerating voltage, (b) equivalent ion energy spectrum found without taking into account the secondary electron emission from the biased mirror surface.

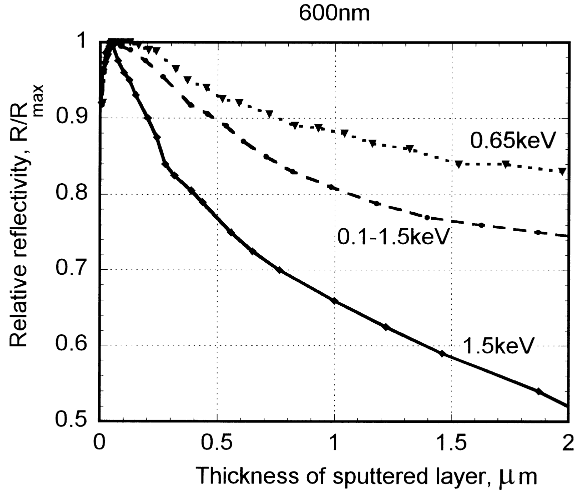

Fig. 2. Relative reflectivity of copper mirrors at $\lambda = 600$ nm versus thickness of a layer sputtered with ions of indicated energy.

The *R*(*h*) dependencies are presented here for three copper mirrors that have been long-term sputtered with ions of different energy. One mirror was exposed to the ion flux of a wide effective energy spectrum (0.1–1.5 keV), shown in Fig. 1. Another one was sputtered with fixed ion energy 1.5 keV that equals to the maximum energy of indicated range, and the third one – with the fixed energy 0.65 keV which was close to the mean energy of the ion spectrum used for the first Cu sample. The results shown in Fig. 2. were obtained for $\lambda = 600$ nm, but *R*(*h*) graphs for other wavelengths have a qualitatively similar behaviour.

The initial significant increase of *R*(*h*) is a result of a gradual cleaning of the mirror surface from oxidic film which has appeared due to the long time storage samples in an air atmosphere [8]. After reaching the maximum, *R*(*h*) degrades with different rates depending on the ion energy: the mirror bombarded with highest energy ions lost its reflectivity much faster. The ion energy spectrum widening led to the lower *R* degradation rate, and the lowest *R* decrement was observed for the mirror treated with ions of the mean energy (0.65 keV).

The differences in behaviour of *R*(*h*) for copper samples at different energies of projectiles was qualitatively confirmed by SEM photos: in the 1.5 keV ion energy case, the surface roughness increased faster (with increasing *h*), than in the case of an energy-distributed ions.

The comparison of *R*(*h*) dependencies for different metals bombarded with energy-distributed ions is shown in Fig. 3. It is evident from these data that the aluminium mirror has the lowest resistance when bombarded with ions of hydrogen plasma. This mirror lost a large portion of reflectivity after a layer of ~0.2 μm in thickness was sputtered, and it had practically fully degraded when $h \geqslant 0.4$ μm.

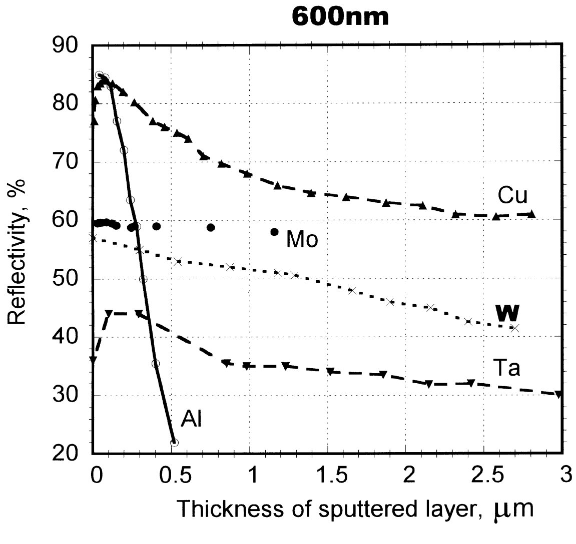

Fig. 3. Reflectivity versus sputtered layer thickness for different metals.

For the tantalum mirror, as for the copper one, the initial improvement of optical properties is probably related to cleaning the surface of oxides. After the maximum of the reflectivity, it decreases with various rates for both these mirrors. For a Cu sample, the initial decay of reflectivity (up to $h \approx 0.8$ μm) occurs much faster than in the last stage of sputtering. This difference correlates with different character of microrelief appearing as a result of the long-term bombardment of the mirror surface. The SEM photos made at the point where *R* begins to decrease (near $h \approx 0.5$ μm) show that the plane surface has started to turn into a step structure with flat parts limited by the each grain size. With increasing time of sputtering the step structure becomes more and more evident. Such a transformation can be explained by differences in the sputtering yield values, *Y*, for metallic microcrystals which have different orientation of crystallographic plane relatively to the projectile incidence [9].

The stepped surface morphology of the copper mirror surface results in some loss of phase information in the mirror image (i.e., decrease of optical resolution). Practically, the quality of the image becomes unclear in spite of a rather high energetic reflectivity measured at this state of experiment ($R \approx 70\%$ at $h = 0.8$ μm, Fig. 2.). With longer exposure of a (polycrystal) copper mirror to ion bombardment, the microrelief in the limit of grains develops with different rates, probably, again depending on the grain orientation. This local increase of roughness inside grains, even of a much smaller scale, is the reason for the monotonous decay of reflectivity (the range of $h > 0.8$ μm, Fig. 2).

The rate of *R*(*h*) dependence for tungsten and tantalum (after maximum) mirrors looks rather similar to *R*(*h*) dependence for the later stage of sputtering of a copper mirror. Thus, in terms of dependence of a reflectivity on sputtered layer thickness the W and Ta mirrors demonstrate no better resistance than the Cu mirror. In both cases the mirror degradation is a result of developing roughness that grows continuously with increasing time of exposure to ion flux, as is shown in Fig. 4. The character of microrelief is very different for these metals, and the step structure was not observed on both samples.

4. Discussion and conclusion

It was stated in Ref. [10] that for the adequate simulation of the FM behaviour in a fusion reactor, the long-term irradiation of the mirror surface has to be carried out with light ions (H^+, D^+, He^+) but not with

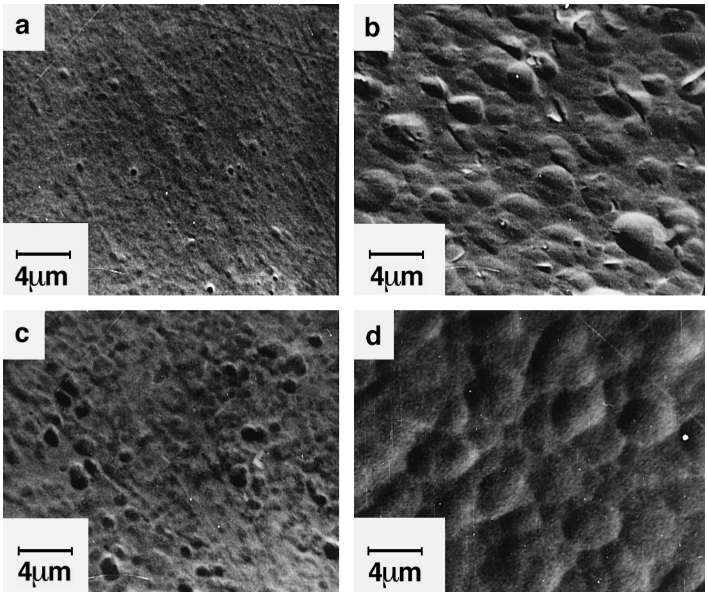

Fig. 4. Scanning electron micrographs of the surfaces of W (*a*, *b*) and Ta (*c*, *d*) mirrors after sputtering layers with thickness: (a) 870 nm; (b) 2710 nm; (c) 960 nm; (d) 2970 nm.

heavy ions (like Ar^+). The data of Fig. 2 show that the energy spectrum of light ions is also very important if one wants to predict correctly the influence of CX atoms with very wide energy spectrum on the FM properties. Such a strong effect of ion energy distribution on the R degradation is an indirect indication of the importance on the microrelief change rate of defects being created inside a mirror near-surface layer under ion bombardment. For the 1.5 keV case the ion energy was higher than the displacement-inducing threshold energy of hydrogen ions bombarding a copper sample ($E_{\mathrm{thr}} \approx 600$ eV). In the wide spectrum case, the energy of some portion of hydrogen ions was not enough to knock-on a copper atom, and only higher energy ions effectively create defects in a metal lattice by the displacement mechanism. In contrast, in the 0.65 keV ion energy case a great amount of ions had energy lower than E_{thr}. The reason for this is connected with peculiarities of ECH discharge in a magnetic trap: according to [11], the ion component of an ECR plasma is characterised by abundance of molecular ions (H_2^+, H_3^+). The disintegration of these ions with initial energy 650 eV on the mirror surface leads to the decrease of the projectile energy and, as a result, to a cancellation of their efficiency to create the displacement defects.

As far as we know, there is only one paper [12] devoted to the investigation of character of defects produced in the near surface layer depending on the projectile energy in keV range. It was found, using TEM, that the evolution of molybdenum microstructure under bombardment with hydrogen ions depends on the projectile energy. For an ion energy lower than E_{thr} ($\approx$860 eV for Mo) only platelet hydrogen clusters in the sample near-surface layer have been found, whereas for $E > E_{\mathrm{thr}}$ – dislocation loops were also registered. Amounts of different kinds of defects were observed to grow with increasing ion fluence in the range which corresponds to the decreasing stage of $R(h)$ curves in Fig. 2. However, special experiments should be carried out to determine the direct correlation between characteristics of defects produced in the near-surface layer, on the one hand, and the rate of change of the surface micromorphology, on the other hand.

In Fig. 3, it is seen that from the view point of R versus h dependence, the mirrors made of refractory metals do not differ significantly from a copper mirror. This means, taking into account the big difference in sputtering coefficients [13,14] that Ta, W and Mo mirrors will have a much longer life time as compared to Cu mirrors. Thus, data of Fig. 3 are in good qualitative agreement with the R/Y criterion suggested in [3] for ranging the priority of different metals to be a FM material. Note, that, in their capability to maintain a smooth surface for long-term sputtering, the refractory metals are a little worse than the stainless steel of O4Cr16Ni11Mo3T type. For mirror made of this material the sharp decrease of $R(h)$ has been observed only after sputtering ~3 μm layer in thickness [3]. The higher resistance of stainless steel to save the optically smooth surface for long-term bombarded with CX atoms is governed, possibly, by a fine grain structure of this steel.

In summary we conclude:

The energy spectrum of projectiles, as well as their mass, influences very strongly the rate of mirror degradation. Therefore, the correct simulation of CX atom effects on the long time survival of FM in a fusion reactor is possible only if fluxes of energy-distributed light ions are used for bombardment of mirror surface.

With the exception of the aluminium mirror, other material studied have approximately similar degradation rates as a function of thickness of a layer sputtered with energy-distributed ions. It means, that the service time of mirrors made of metals having the lowest sputtering yield (W, Ta, Mo) will last longer than mirrors made of metals with higher sputtering yield (Cu, Ag, Au). Thus, for those methods of plasma diagnostics where wavelengths longer than visible range are planned to be utilised (i.e., where $R \cong 1$ [15]) the refractive metals can be successfully used as the FM material with sufficient signal/noise ratio. Similar mirrors can be also suitable for spectroscopy in the visible part of spectrum, if the brightness of a plasma in this spectral range is high. As for FM of laser diagnostics, they have to be fabricated of metals with the highest reflectivity, and therefore should be located deep inside the long diagnostic channel where the CX atom flux will be strongly reduced.

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