Diagnostic mirrors with transparent protection layer for ITER


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A B S T R A C T

Fast degradation of in-vessel optics is one of the most serious problems for all optical diagnostics in ITER. To provide the resistance to mechanical and thermal stresses along with a high stability of optical characteristics under deposition-dominated conditions we suggest using high-reflective metallic (Ag or Al) film mirrors coated on silicon substrate and protected with thin oxide film in the divertor Thomson Scattering (TS) diagnostics. The mirrors coated with Al2O3 and ZrO2 films were tested under irradiation by deuterium ions. The experimental results on the oxide films sputtering are discussed in the context of their applicability for the first mirror protection in ITER.

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1. Introduction

The design of first mirrors is one of the most challenging issues in developing optical diagnostics for ITER. Fast degradation due to irradiation-induced defects, contamination, sputtering with fast particles, as well as high thermal and mechanical stresses poses a serious threat to optical elements located inside the ITER vacuum chamber. The requirements for the first mirrors may vary depending on their location and it is reasonable to use different design concepts for the mirrors to be placed in the areas featuring erosion-dominated and deposition-dominated conditions. The latter are typical of the divertor region, characterized by a high concentration of the products of plasma-induced erosion of first-wall components and divertor tiles and, as a result, by an intensive contamination of all optical surfaces. The deposition rate of amorphous hydrocarbon (a-C:H) films on the first mirror of the divertor TS diagnostics is estimated to be as high as 0.2 nm/min [1]. As shown in [2] the a-C:H films are transparent within wide spectral range. However, these films may cause crucial deterioration of the reflection spectra of the mirrors with moderate reflectivity like Rh or Mo (see Fig. 1a). The reflectivity of high-reflective mirrors is far less influenced by the transparent depositions in IR spectral region used for the divertor TS diagnostics (see Fig. 1b).

To minimize the negative effect of contamination we suggest using the high-reflective metallic mirrors (Ag, Al) coated with a transparent oxide layer (Al2O3, ZrO2) in the divertor TS system. The oxide film is designed to protect the mirrors from sputtering, and blistering on the one hand and to prevent the deposition-induced loss of reflectivity in the limited spectral region on another. The effect of Ag mirror coating with the transparent oxide is shown in the inclusion in Fig. 1c. The protection of the mirror with 210 nm of Al2O3 results in decreasing the influence of a-CH deposits on the reflectance in the IR range.

For the large-scale (tens of centimeters) mirrors to be used in the severe ITER conditions, the choice of substrate material resistant to mechanical and thermal stresses is a key factor. The mirror blank material should have:

- minimal density and linear expansion coefficient;
- maximal thermal conductivity and modulus of elasticity.

From this point of view, polycrystalline silicon (PCS) is considered to be a promising material for the mirror substrate [4]. The PCS combines significant specific rigidity E/ρ (E – modulus of elasticity and ρ – unit weight), with a low thermal distortion parameter α/λ, characterized by the ratio of the linear expansion coefficient α to thermal conductivity λ (see Table 1).

As is seen from Table 1 the specific rigidity of PCS is about twice as high as that of conventional substrate materials. In other words, PCS substrates of given stiffness are about twice as light as con-
Table 1
The parameters of PCS and some conventional substrate materials.

<table>
<thead>
<tr>
<th></th>
<th>PCS</th>
<th>Al</th>
<th>SiO₂</th>
<th>ULE</th>
<th>Sitall</th>
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<tr>
<td>Unit weight ρ, 10⁴ N/m³</td>
<td>2.3</td>
<td>2.7</td>
<td>2.2</td>
<td>2.205</td>
<td>2.5</td>
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<tr>
<td>Modulus of elasticity E, GPa</td>
<td>160</td>
<td>70</td>
<td>72</td>
<td>67</td>
<td>92</td>
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<tr>
<td>Specific rigidity E/ρ, 10⁸ m</td>
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<td>2.7</td>
<td>3.2</td>
<td>3.1</td>
<td>3.7</td>
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<tr>
<td>Linear expansion coefficient α, 10⁻⁶ 1/K</td>
<td>2.5</td>
<td>24</td>
<td>0.55</td>
<td>0.03</td>
<td>0.1</td>
</tr>
<tr>
<td>Thermal conductivity λ, W/mK</td>
<td>160</td>
<td>200</td>
<td>1.38</td>
<td>1.3</td>
<td>1.2</td>
</tr>
<tr>
<td>Thermal distortion parameter α/λ, 10⁻⁸ m/W</td>
<td>1.6</td>
<td>12</td>
<td>40</td>
<td>2.3</td>
<td>8.3</td>
</tr>
</tbody>
</table>

Fig. 1. Spectral reflectance of (a) Mo, (b) Ag and (c) Ag coated with 210 nm of Al₂O₃ without and with amorphous carbon deposits. The calculations were made for unpolarized light incident at 45° on the mirror surface [3].

Fig. 2. TEM image of the cross-section of (a) Si–Cu mirror with Al₂O₃ coating deposited at 20 °C and (b) Si–Al mirror with Al₂O₃ coating deposited at 200 °C.

Fig. 3. TEM image of the cross-section of (a) Si–Cu mirror with Al₂O₃ coating deposited at 20 °C and (b) Si–Al mirror with Al₂O₃ coating deposited at 200 °C.

As was shown earlier Al₂O₃ film deposited at 20 °C do not provide sufficient protection of the mirrors subjected to the...
deuterium ions bombardment. The exposition to a fluence of \( \sim 2 \times 10^{20} \text{ions/cm}^2 \) with the ion energy 40–50 eV results in blisters on the mirror surface covered with Al\(_2\)O\(_3\) [6]. From our recent experiments we found that Al\(_2\)O\(_3\) grown at 200 °C also reveals poor protective capability against blistering. When exposed to deuterium ion flux the mirror surface underwent fast degradation caused by blistering and flaking of the upper Al\(_2\)O\(_3\) layer. As a result we failed to measure the sputtering yield of Al\(_2\)O\(_3\) film and focused on testing ZrO\(_2\) films only.

In ZrO\(_2\) films, needle-like pores across the thickness of the film were observed for both grown at RT and at 200 °C films (see Fig. 3a). As shown in [6], ZrO\(_2\) grown at RT provides high protection against the deuterium ion bombardment. The mirrors with ZrO\(_2\) protective layer subjected to deuterium ion irradiation (ion energy was gradually increased from 40 to 100 eV, total fluence \( \sim 1 \times 10^{11} \text{ions/cm}^2 \)) underwent no noticeable changes in surface morphology. The observed good protective properties of the ZrO\(_2\) protective film are likely to be due to the porous structure of ZrO\(_2\) which facilitates the molecular hydrogen transport outside thus preventing the blistering caused by hydrogen accumulation at the oxide–metal interface.

The TEM study of ZrO\(_2\) coating revealed the presence of microcrystalline inclusions in the amorphous ZrO\(_2\) layer deposited at 200 °C (see fig. 3b). Some microcrystals were found to break away already during the deposition stage thus leaving the underlying metal layer unprotected. Although these unprotected spots eroded in deuterium flux, their concentration was too low to affect substantially the reflection spectrum of the mirror. At the same time, the morphology of the areas protected with ZrO\(_2\) did not undergo noticeable changes when exposed to a fluence as high as \( 1 \times 10^{21} \text{ions/cm}^2 \). The experiments on sputtering in the deuterium flux showed the sputtering yield to be approximately the same for ZrO\(_2\) films grown at RT and at 200 °C. All the results below are presented for the ZrO\(_2\) deposited at 200 °C on Si–Al mirror.

3. Zirconia sputtering by deuterium ion beam

3.1. Experimental

One of the key requirements to the transparent protective coating of mirrors – resistance to a sputtering by deuterium ion flux – is necessitated by the plasma cleaning of contaminated surfaces from CH deposits. For the first mirror cleaning we are going to use the capacitively coupled RF discharge with the first collecting mirror playing the role of one of the electrodes. The protective layer on the mirror surface in this case will undergo bombardment with deuterium ions for the entire period of ITER operation. The current density and ion energies in the deuterium flux, used for mirror cleaning are expected to be less than 0.1 mA/cm\(^2\) and 100 eV accordingly. More information on the application of plasma cleaning technique and the plasma source we are going to use can be found in [7]. To ensure the survivability of protective film and the mirror as a whole we should determine the upper nondestructive threshold energy of ion flows which can be used for in situ cleaning.

The treatment of the mirror samples with deuterium plasma was performed in the DSM-2 setup, described in [8]. The plasma was generated in a double-mirror magnetic configuration using the magnetron, operating at the electron cyclotron resonance frequency of 2.375 GHz. The working deuterium pressure was \( \sim 2 \times 10^{-2} \text{Pa} \). At the typical magnetron power of \( \sim 400 \text{W} \), the electron density and temperature were: \( n_e \sim 10^{19} \text{cm}^{-3} \), \( T_e \leq 5 \text{eV} \). The water-cooled mirror holder was located on the system axis outside the magnetic mirrors, and was biased negatively at the fixed potential in the range of 60–150 V. Current density through the sample mirrors coated with dielectric protective layer, 1.4–1.5 mA/cm\(^2\), was only slightly less than that for the bulk Cu samples under the same conditions. The anomalously low resistance of dielectric oxide films may have originated by the photo-induced ionization processes in oxide, or may have been caused by ion bombardment itself.

The reflection spectra of mirrors were recorded at the very beginning of the experiment and after each of the subsequent expositions to deuterium ion beam (see Fig. 4). The oxide film thickness was being determined by fitting the experimental reflection spectra with the calculated ones. The spectral dependences of refractive index \( (n) \) and extinction coefficient \( (k) \) used for the reflection spectra calculations were taken from [9]. The errors of the fitting procedure were estimated to be lower than ±2 nm.

![Fig. 3. TEM image of (a) needle-like open pores and (b) microcrystalline inclusions in amorphous ZrO\(_2\) deposited on Al sub-layer.](image-url)

![Fig. 4. Spectral shift of the interference maximums and minimums in the reflection spectrum of Si–Al–ZrO\(_2\) mirror caused by ZrO\(_2\) film sputtering with deuterium ion flux (1.4–1.5 mA/cm\(^2\), 60–150 eV).](image-url)
3.2. Results

Gradual thinning of ZrO₂ coating under deuterium ion bombardment and energy dependence of the sputtering yield are shown in Fig. 5a and b. Fast sputtering at the beginning of the exposition (see Fig. 5a) is due to the imperfection of upper interface of oxide film and should be neglected. According to [10], the current density as low as 0.1 mA/cm² on the mirror surface and the accelerating voltage of 50 V are sufficient to provide effective cleaning the first mirror of the divertor TS diagnostics system from a-C:H deposits. As follows from Fig. 5a, the sputtering rate of the ZrO₂ film subjected to deuterium ion bombardment with the ion current density of 0.1 mA/cm² and ion energy of 60 eV is expected to be ~0.004 nm/h. Therefore, ZrO₂ film on the surface of the first mirror exposed to cleaning discharge for the entire period of ITER operation (~5400 h) will lose no more than 20 nm in thickness, which is far less than the total thickness of the oxide film – 200 nm (180–200 nm is an optimal coating thickness for the first mirror in divertor TS [1,3]). It should be emphasized that sputtering efficiency of ZrO₂ appeared to be rather low (see Fig. 5b) – even lower than for the bulk Al₂O₃ [11].

4. Conclusion

High-reflective metallic (Ag or Al) mirrors on silicon substrate protected with thin oxide film were suggested to be used in the divertor TS diagnostic system. The mirrors of this type combine high stability of optical characteristics under deposition-dominated conditions and resistance to the mechanical and thermal stresses. The experimental results on sputtering of ZrO₂ protective film with deuterium ion flux appear to be promising: ZrO₂ film on the surface of the first mirror of the divertor TS system was shown to lose less than 10% of the total thickness during the entire period of ITER operation in the case it is subjected to cleaning discharge with the parameters given in [10]. As was shown earlier [6], the ZrO₂ also provides a good protection from deuterium in-diffusion, thus preventing blisters on the mirror surface.

The basic concepts of the first mirror design, discussed in this paper are developed with account taken of the specificity of deposition-dominated conditions in the ITER divertor. The first tests evidenced the validity of these concepts. However, testing of the mirrors in the ITER-relevant condition is still in progress.

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