EFFECTS OF WATER IMPACT ON OPTICAL PROPERTIES OF METALLIC MIRROR SAMPLES

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The effects of exposure in water or water steam on optical properties of mirror samples fabricated of different metals were studied. Materials of samples were: tungsten, beryllium, molybdenum, stainless steel, nickel, copper, rhodium, and amorphous alloys of two compositions. The material of the mirror was found to play a decisive role in the degree of impact of water or water vapor on mirror degradation. The most strongly degraded Mo and Cu mirror samples, and the lowest degradation was observed for SS and Rh/SS mirror samples. The reflectance can be restored to practically initial level when after water impact procedure the mirrors are exposed to ions of Ar plasma with energy ≥100 eV up to ion fluence $>10^{19}$ ion/cm^2.

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INTRODUCTION

One of the problems of the reliability of optical diagnostics in ITER is the possibility of sudden release of refrigerant inside the vacuum chamber. Recently a quite detail experiments were provided with an aim to clear up the consequences of in-vessel coolant leak on optical characteristics of in-vessel component of optical diagnostics in ITER [1, 2]. The authors of [1] studied the behavior of mirror samples made of different materials from several suppliers under impact of water steam and high temperature (≤250°C), as imitation of the impact of accidental in-vessel coolant leaks on components of optical diagnostics in ITER. In [2] the degradation of optical characteristics of Mo mirrors under impact of hot water or water vapor was thoroughly investigated.

In our modeling experiments, we limited ourselves by studying the effects of exposure in a distilled water of two temperatures (100°C and RT) or in water vapor (~100°C) on the optical properties of mirrors made of different metals (W, Be, Mo, Rh, SS, Cu) that are prospect to be used as constructive elements in ITER. Such data can be useful to predict their behavior in ITER if ingress of water or water steam occurs.

The detail characterization of tested mirror samples and conditions of their exposing are given in the first part, the main results are presented in the part two, the discussion – in the third part, and the concluding remarks – in the last section.

1. EXPERIMENTAL

The experiments were provided with mirror samples prepared from: recrystallized tungsten and ITER grade W, beryllium, single crystal molybdenum, rhodium, copper, stainless steel (SS), nickel, and amorphous alloys.

All samples were polished to a mirror-like quality and before the water test each of them was cleaned by exposure to ions of Ar or D plasma produced in conditions of electron cyclotron resonance (ECR) in a simple mirror-like magnetic device DSM-2 [3]. The reflectance $R(\lambda)$ in the wavelength 220…650 nm at normal incidence was measured just after cleaning, after water (or water vapor) impact, and (rarely) at the stage of reflectance restoration by successive exposing in ECR plasma. The change of the surface stage of all samples was controlled with optical microscope.

Most samples were (22x22) mm^2 with thickness 2…3 mm, or round-shape with diameter 22 mm and thickness 2 mm. Several W samples have size [10×10×(1…2)] mm^3.

Characteristics of samples:

– W samples were recrystallized with chaotic grains orientation [4] and the ITER-grade ones with grains predominantly oriented normally to the surface [5];

– polycrystalline Be from two suppliers;

– Mo samples were single crystal: Mo(100), Mo(110), and Mo(111);

– stainless steel (analog of 316 steel) [6];

– amorphous samples were made of two different alloys: $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ (one sample, AMA – in amorphous state, the other, AMK – crystallized) and $Zr_{48}Cu_{36}Al_8Ag_8$ sample (*AMA*) that was preliminary exposed in D plasma;

– polycrystalline Cu sample;

– monocrystalline Ni(111) sample;

– Rh film deposited on stainless steel substrate.

After mechanical polishing and cleaning of samples by low temperature plasma in DSM-2, their reflectance was measured and the photos in optical microscope were made. These data were used as the initial ones.

During the test procedure the samples were exposed for 1.5…2 hours in boiling water or in water steam. In the latter case the sample was fixed to the holder located just under the surface of boiling water and inclined to 45° to provide continuous flowing off the condensed water. The room-temperature test lasted 20 hours continuously. After exposure in water or in water steam the reflectance $R(\lambda)$ was again measured, and photos of different parts of the sample surface in an optical microscope was provided.

2. RESULTS

2.1. OPTICAL MEASUREMENTS

Fig. 1 shows the results on reflectance degradation, i.e., the difference ΔR=R_{init}-R_{steam} for all samples after they were exposed in water steam during 2 hours.

It is seen that the strongest decrease of the reflectance was observed for the Cu sample (note, the ΔR values for Cu is shown with the scale that is factor two less than for other samples). Among those mostly degraded are also both Mo and W ITER-grade samples. The reflectivity drop for other samples does not exceed 10 % in the considered wavelength interval. The highest resistance to water steam impact showed the Rh film deposited on SS substrate, beryllium (not shown), and single crystal Ni(111) mirror sample.

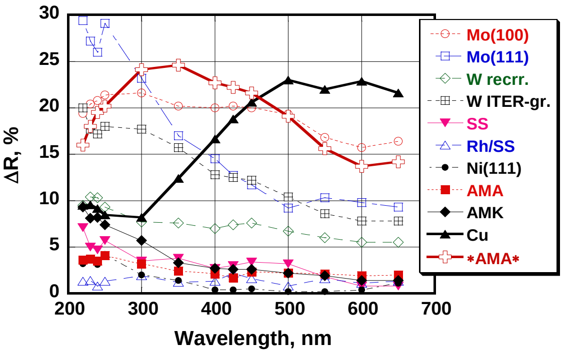


Fig. 1. Decrement of reflectance due to exposure of mirror samples in water steam during two hours. For the Cu sample ½·ΔR is shown for convenience

The data on reflectance decrease due to exposure of samples in boiling water, ΔR=R_{init}-$R_{boil.water}$, are presented in Fig. 2. They are in a qualitative agreement with those obtained with samples exposed in water vapor.

The results of ΔR drop after samples were exposed at room temperature in distilled water during 20 hours are presented in Fig. 3. Again the strongest effect is for the Mo samples including the mirror made of Mo-Ni(4 %) alloy. Among tested, the Cu sample showed the lowest R decrease among all samples tested: ΔR is practically within the measurement error.

2.2. SURFACE MODIFICATION

The reason of reflectance degradation is the change of sample surface. As an example, in Fig. 4,a,b the photos made in optical microscope are shown for W samples after they were exposed in water or in water vapors. The surface of samples after water tests looks like the surface of the grains with some orientations is modified much stronger than of the grains with other orientations. To some extent this reminds the data obtained in [4-6], where significant difference was found for sputtering rate of differently oriented grains of recrystallized W, ITER-grade W, and SS samples, respectively.

After exposure to water vapor, the surface of the samples becomes reflecting the effects of droplets (see Fig. 4,c), probably due to their weak runoff at an angle selected for the sample tilting (45°).

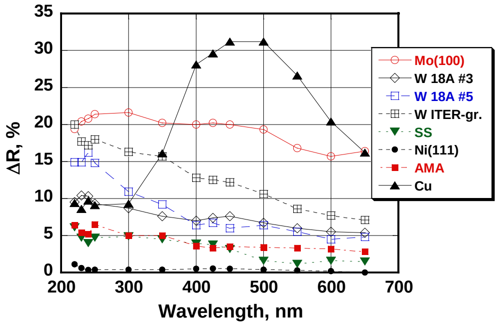


Fig. 2. Drop of reflectance after samples were exposed in boiling water during two hours

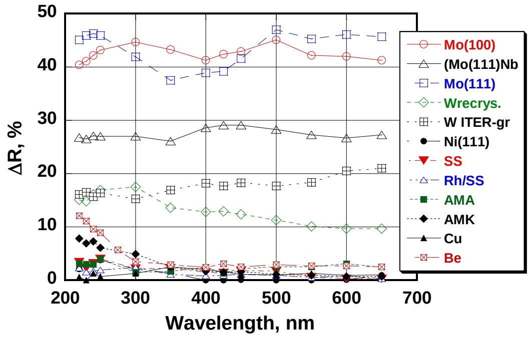


Fig. 3. Decrease of reflectance for indicated samples exposed in distilled water at room temperature during 20 hrs

2.3. RESTORATION OF REFLECTANCE

Almost in all cases, after water impact the reflectance of samples was restored rather easily by exposing them to flux of argon plasma ions in the DSM-2 stand. As an example, Fig. 5 shows the reflectance of the Mo(100) sample at different stages of its cleaning. The reflectance was restored to the initial value after 100 min exposing to Ar ions with energy 90 eV followed by 10 min exposure to Ar ions with energy 300 eV (total Ar^+ ion fluence ≈$4\cdot10^{19}$ ion/cm^2). The use of deuterium plasma ions of same energy was found not so effective for restoration of reflectance after water impact in comparison with Ar plasma ions.

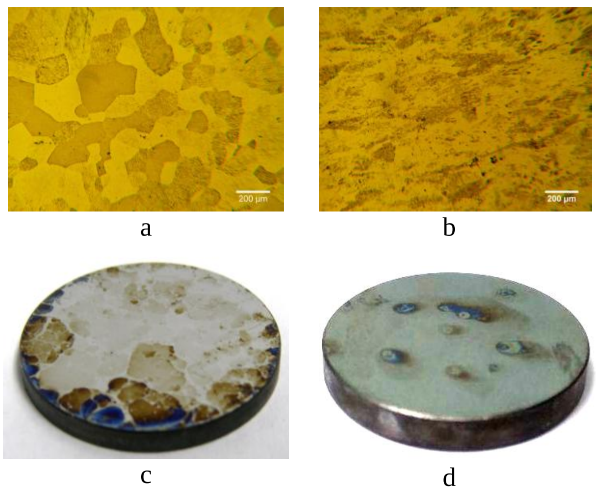


Fig. 4. W recrystallized, 1 hour in boiling water (a); W– ITER-grade, 2 hours in water vapor (b); Mo*(111) 2 hours in water vapor (c); Be 20 hours in water of room temperature(d)*

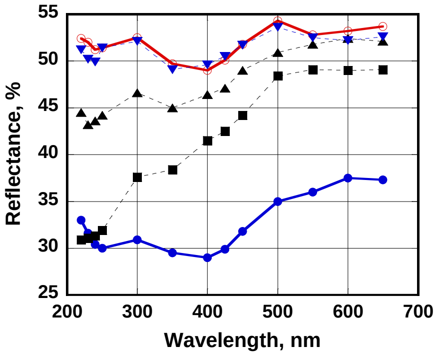


Fig. 5. Reflectance restoration of the Mo*(100) sample by exposing in* Ar *plasma: ○ – initial; • – 90' exposure in boiling water; ▲ – 20 min cleaning by Ar ions with energy 90 eV; ▼ – 60 min by Ar ions with energy 90 eV; ♦ – 100 min with* Ar *ion energy 90 min and 10 min with* Ar *ion energy 300 eV*

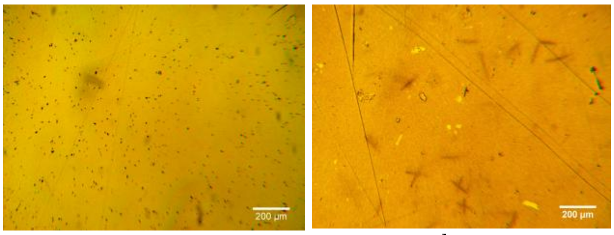
It should be mentioned that initial reflectance of the tested Mo(100) sample was somewhat below the values recommended in the reference book [7], what is explained by an imperfection of the surface of this particular sample.

3. DISCUSSION

When analyzing the experimental data, we can conclude that our results are in qualitative agreement with results of papers [1] and [2], namely: exposure of metal mirror samples in water or water vapor leads to degradation of their optical characteristics. The rate of degradation depends on the mirror material and exposure duration. Even bathing of samples in room temperature water leads to decrease of reflectance. The strongest effect (i.e., R decrease) of hot water or vapors was observed for Cu and Mo samples. But at the same time, after 20 hrs exposure in cold water of Cu sample the degradation of its reflectance R was practically in the limit of measurement accuracy (±1.5 %). The latter fact agrees with published data, e.g., in [8] the authors registered the onset of Cu corrosion process after exposure time exceeded ~200 hrs. In Conclusion they wrote: "we note a distinctly low rate of copper corrosion in pure anoxic water".

The samples made of $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ alloy showed rather high resistance to water impact both in amorphous (AMA) and crystallized (AMK) states for all three tests. At the same time, many small-size dark points have appeared on the surface Fig. 6,a, which, however, did not lead to strong reflectance degradation (see Fig. 3). Appearance of chaotic inhomogeneities on the surface of single crystal Mo samples (i.e., Fig. 6,b), that became the main reason of reflectance degradation, is quite striking, taking into account a perfect (single crystal) structure of Mo samples.

Strong inhomogeneous on Be mirror sample surface after RT bathing (Fig. 4,d) is also difficult to explain, as initially there was no any indication on surface inhomogeneities.



a b

Fig. 6. $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$, 20 hours in RT water (a); b – Mo(100) 1.5 hour in boiling water(b)

The samples made of Ni(111), SS, and Rh film on SS substrate showed low degradation in all tests with $\Delta R \approx 5$ % in UV wavelength range and $\Delta R \leq 2$ % in visible range.

CONCLUSIONS

Among all tested samples, enumerated in Part 1, Mo samples turned out themselves among the weakest ones in all three tests. In this connection it should be noted that just molybdenum is one of the materials recommended for the first mirrors because of its quite high resistance to sputtering and high enough reflective properties. The Cu samples strongly degraded at 100°C but saved their reflectance after 20 hour bathing in cold water. The next step in resistibility occupy W samples with noticeable difference among recrystallized and ITER-grade tungsten. At about equal positions among them are SS and AMA samples with perfect structure, but the sample with not perfect surface structure (*AMA*) degraded very strongly. The lowest degradation demonstrated SS and Rh/SS mirror samples. Beryllium weakly degraded during 2-hour exposures at 100°C, but much stronger when bathing in RT water for 20 hours. The contaminated layer appeared during water impact procedure can be successfully taken off by bombardment with Ar ions of moderate energy and ion fluence.

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ЭФФЕКТ ВОЗДЕЙСТВИЯ ВОДЫ НА ОПТИЧЕСКИЕ СВОЙСТВА ОБРАЗЦОВ МЕТАЛЛИЧЕСКИХ ЗЕРКАЛ

В.Г. Коновалов, С.И. Солодовченко, В.С. Войценя, И.В. Рыжков, А.Ф. Штань, М.Н. Махов

Изучены эффекты воздействия воды или водяного пара на оптические свойства образцов зеркал, изготовленных из разных металлов: молибдена, вольфрама, нержавеющей стали, никеля, меди, родия, бериллия и аморфных сплавов двух композиций. Было обнаружено, что материал зеркала играет решающую роль в степени воздействия воды или водяного пара на деградацию зеркала. Наиболее сильно деградировали Mo- и Cu- образцы, а самая низкая деградация наблюдалась для зеркал SS и Rh/SS. Коэффициент отражения может быть восстановлен до практически начального уровня, если после воздействия воды зеркала подвергаются воздействию ионов Ar-плазмы с энергией ≥100 эВ до ионного флюенса >10^{19} ионов/см^2.

ЕФЕКТ ВПЛИВУ ВОДИ НА ОПТИЧНІ ВЛАСТИВОСТІ ЗРАЗКІВ МЕТАЛЕВИХ ДЗЕРКАЛ

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Вивчено ефекти впливу води або водяної пари на оптичні властивості зразків дзеркал, виготовлених з різних металів: молібдену, вольфраму, нержавіючої сталі, нікелю, міді, родію, берилію і аморфних сплавів двох композицій. Було виявлено, що матеріал дзеркала відіграє вирішальну роль у мірі впливу води або водяної пари на деградацію дзеркала. Найбільш сильно деградували Mo- і Cu- зразки, а найнижча деградація спостерігалася для дзеркал SS і Rh/SS. Коефіцієнт віддзеркалення може бути відновлений до практично початкового рівня, якщо після впливу води дзеркала піддаються впливу іонів Ar-плазми з енергією ≥100 еВ до іонного флюєнса >10^{19} іонів/см^2.