ITER AND FUSION REACTOR ASPECTS

IMPACT OF LONG-TERM SPUTTERING ON MIRROR SAMPLES FABRICATED FROM DIFFERENT KIND TUNGSTEN

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The results are presented on effects of long term sputtering of mirror-like samples made of pure tungsten and tungsten with dopants (Y, K-Al, La). The dynamics of surface relief and optical characteristics were measured depending on the depth of erosion under Ar ion bombardment; the rate of sputtering was estimated by the use of mass loss measurements. Difference was found in the character of surface relief, rate of degradation of reflectance and relation between specular and diffusive components of reflected light for different kind W samples.

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INTRODUCTION

Tungsten is becoming to be one of the main fusion reactor materials. The tokamak ASDEX is the first all-W-first-wall device; in tokamak JET all divertor area is protected by W armour protection; the second W-first-wall device WEST in France is in preparation to start the operation. In ITER W-coated protection will be 150 m^2: 50 m^2 – divertor receiving plates and 100 m^2 – total area of dome and baffle parts of the divertor. The environment conditions for both these W components will be principally different: receiving plates will be subject to high energy plasma fluxes with low energy ions, whereas dome and baffle parts – to sputtering by charge exchange atoms (mainly D and T atoms) [1] of a wide energy distribution with mean atom energy several hundred eV [2].

Here we present comparative results obtained with mirror samples made of several kind tungsten produced in China and subjected to long-term sputtering in the course of experimental program modelling the behaviour of dome and baffle W protection in ITER.

1. EXPERIMENTAL TECHNIQUES AND SAMPLES

The effects of long term sputtering on development of surface relief was studied for pure tungsten (PW) and tungsten with alloying additions: 0.1%K+0.1%Al (W-K), 2wt%La_2O_3 (W-La), 1wt%Y_2O_3 (W-1Y) and 2wt%Y_2O_3 (W-2Y). There were two samples of every kind excluding a W-La sample. All samples have size of ~10x10x2 mm. Before the experiments, all samples were polished to a quite high optical quality with an aim to use the optical methods to characterize the microrelief that develops under sputtering. To shorten the time of experiment, Ar ions (energy 600 eV) were used as projectiles instead of hydrogen isotope ions, because with such substitution of projectiles the characteristics of the developing microrelief do not differ significantly after high enough thickness of sputtered layer ($\Delta h \geq 2$ μm).

Sputtering procedures were provided in many steps, and after each step the measurements of mass loss, the reflectance at normal incidence, and the correlation between specular and diffusive component of the light reflected from the mirror under the test were carried out. Besides, the surface of samples was photographed in optical, interference, and electron scanning microscopes. Sputtering procedures lasted during the time needed for sputtering of every sample to the depth ~4 μm. This value is approximately equals to the depth estimated for sputtering by charge exchange atoms of the W armour protection of the baffle and dome parts of divertor by the end of ITER operation.

1.1. PREPARATION OF SAMPLES

1.1.1. THE MANUFACTURING TECHNOLOGY FOR PW AND W-2Y, W-2LA, W-K

The plates of industrially available pure tungsten and industrially available tungsten alloys were provided by Beijing Tian-long Tungsten & Molybdenum Co., Ltd. (TLWM). The production route for this material consists of four steps: 1) compact of W powders to square geometry by cold isostatic pressing at a pressure of 220 MPa; 2) sintering densification in flowing hydrogen atmosphere at temperatures above 2000 ºC for 4h; 3) one-way rolling for obtaining a plate with a deformation of about 75 %; 4) removal of residual stresses by a stress relief treatment to obtain better mechanical properties, e.g., strength and toughness.

1.1.2. THE MANUFACTURING TECHNOLOGY FOR W-1Y

The samples of W-1Y were fabricated in laboratory. Powders of W (with an average particle size of 2.0 μm and a purity of 99.9 %) and Y (48 μm, 99.9 %) were used as starting materials and mixed together according to the nominal composition of W-1Y (in wt%). Then the mixtured powders of W and Y were charged into a vessel made of WC together with WC balls for mechanical alloying (MA) in a planetary ball mill for 30 h with a rotate speed of 380 rpm. The inner atmosphere of the vessels for MA was a purified Ar gas. For the densification of mechanically alloyed W-1Y powders, they were placed into a graphite tool and subjected to sintering in vacuum. The samples were first held at 1100 ºC for 2 min and then sintered at 1600 ºC for 3 min. The applied pressure was 50 MPa.

1.2. SPUTTERING PROCEDURES

The sputtering procedures were provided in the stand DSM-2 described in [3]. As an ion source, plasma of SHF discharge produced in conditions of electron cyclotron resonance is used. The plasma density n_e is ~10^{10} cm^{-3}, electron temperature $T_e \approx 5$ eV with argon as working gas. According to omegatron type gas analyzer the main component of impurity is water with the total impurity level negligibly less in comparison with argon. The plasma ions were accelerated by applying a negative potential (-600 V) to the watercooled mirror holder. The density of ion current to the mirror surface was of the order of 1 mA/cm^2.

1.3. OPTICAL MEASUREMENTS AND SURFACE ANALYSIS

The reflectance (*R*) at normal incidence of the light (range 220…650 nm) was measured before and after every plasma exposure by means of a homemade scheme suggested in [4]. Besides, after every sputtering procedure the method described in [5-7] was applied for comparing the intensity of specular (R_{spec}) and diffusive (R_{diff}) components in the image of a bright source (П-like shape for an ideal mirror) after reflection of the light beam (λ=550 nm) from the test mirror. The appearance of wings in the image is connected with an imperfection of the test mirror. When the mirror surface becomes rougher, i.e., due to sputtering, the intensity of wings increases with corresponding decrease of a specular (central) part of the image. Thus, the ratio between intensity of wings and central part is a measure of the roughness of reflecting surface.

The data on light distribution intensity and a contrast (ratio of central part of the image profile to the wings) give possibility to clear up: does there exist the sputtering depth, after which the optical characteristics of the surface stop to change when sputtering procedures is being continued, or not?

Additionally, in the course of sputtering procedures the surface of every sample was regularly photographed in optical (OM) and interferometry microscopes (IM). After finishing sputtering procedures ($\Delta h \approx 4$ μm), surface of all specimens was analyzed by means of scanning electron microscope (SEM).

2. RESULTS

2.1. PURE W

Because of high identity of results for both pure W samples, the data for only one of them are presented. Fig. 1 shows the OM and IM photos when the layer of Δh = 4.2 μm in thickness was sputter eroded. The height of steps between some neighbor grains achieves ~1.6 μm (see Fig.1,b). Such a step structure of surface relief is caused by high difference of sputtering yield for grains with different orientation relatively mirror sample surface. As was shown in [8], tungsten grains oriented along axis [111] are the most resistant to sputtering whereas those oriented along the [110] direction are weakest.

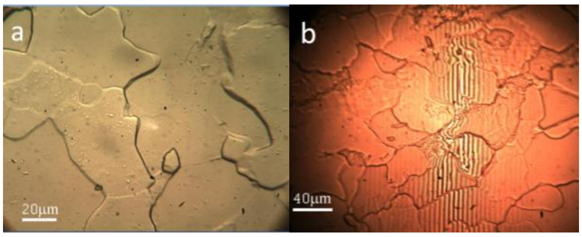

Fig. 1. The images of final surface states for the ***PW*** *#1 sample: a) – OM; b) – IM*

Fig. 2 demonstrates the behavior of some optical characteristics depending on Δh. As seen, *R* degrades rather slowly Fig. 2,a. This can be explained by practical lack of in-grain roughness even after finishing of sputtering actions and due to normal incidence of light to every separate grain during optical measurements.

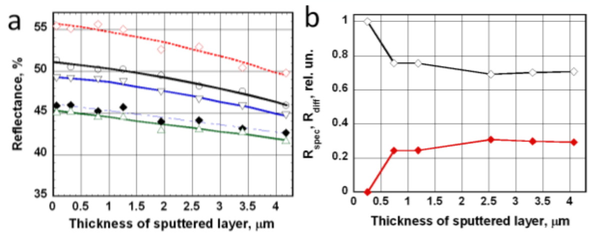

Fig. 2. a – dependence on Δh *of reflectance at wavelengths:* **o** *– 220 nm,* ♦ *– 250 nm,* Δ *– 300 nm,* ∇ *– 500 nm,* ◊ *– 600 nm; b – dependence on* Δh *of specular (◊) and diffuse (♦) parts of the profile*

As follows from Fig. 2 the relationship between R_{spec} and R_{diff} became practically fixed after $\Delta h \approx 1$ μm. Near this Δh value, there is observed also a definite stabilization of the surface "macro-characteristics" responsible for sharing reflectance to specular and diffusive parts. This result is important from the viewpoint of the use of tungsten as a plasma facing material of baffle and dome of the ITER divertor that will be subjected to charge exchange atoms of a very broad energy distribution [2].

2.2. W-1Y

The behavior of both these specimens is very much similar, but with significant difference in comparison with behavior of pure tungsten samples. All photos (one example is presented in Fig. 3) do not show any grain borders, and the characteristic longitudinal size of inhomogeneities is of the order ~2 μm. A strong difference is observed for dependence of reflectance on Δh (Fig. 4) in comparison with PW samples. The $R(\Delta h)$ is decreasing for the whole range of sputtering process with gradual decreasing rate. The lowest *R* values are 8…15 %, depending on the wavelength.

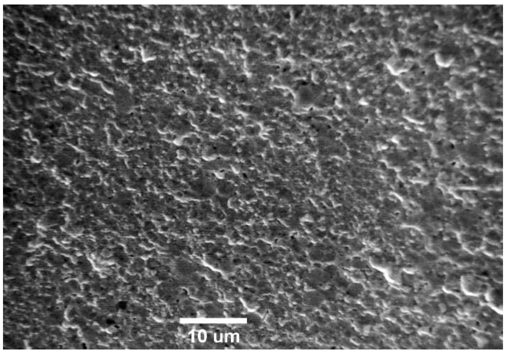

Fig. 3. SEM photos of the W-IY #1 surface – after sputtering the layer with thickness 4.25 μm

It is worthy to note a very important difference in Δh dependences of relationship R_{spec} and R_{diff}, shown in Fig. 4. After sputtered layer thickness reached ~2 µm, the diffusive component became to exceed the specular component. In generally for this case, the constant value of the contrast in this case (not shown) indicates on much wider angle of scattering of the diffusive part in comparison with the range of measurements (±0.5 mm from the center of the image).

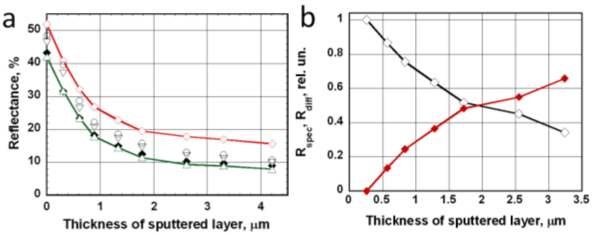

Fig. 4. a – dependence on Δh of reflectance at different wavelengths; b – dependence on Δh of specular and diffuse parts of the profile. Symbols are same as in Fig. 2

2.3. W-2Y

In spite of same dopant, there is no agreement between behavior of these specimens and behavior of the preceding pair. All characteristics behave noticeably differently with increasing Δh. The relief of both W-2Y samples is much closer to the relief on the surface of pure W samples (Fig. 5 and Fig. 1), i.e., with an evidence of a step structure which, however, is characterized by some in-grain relief as distinct from pure W samples. The reflectance decreased faster, the rate of R_{spec}/R_{diff} change is qualitatively in agreement with those for pure W samples, however, the contrast is factor three less at the beginning and drops further after sputtering was started (not shown).

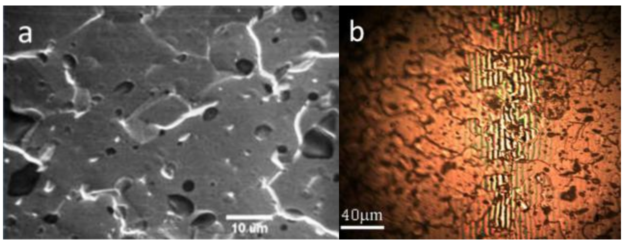

Fig. 5. Photos of the W-2Y #1 surface after sputtering the layer with Δh=4.07 μm; a) – SEM; b) – IM

2.4. W-K

For both W mirrors with K and Al additions the rate of reflectance degradation in the course of sputtering process (Fig. 6), was rather similar to that for W specimens with 2 % yttrium adding. The qualitative correlation is also observed for relationship between specular and diffusive components, however, no correlation was observed of this relationship and dependence of contrast on Δh.

The relief (Fig. 7) developed has a specific texture which is well seen at high magnification, but has not been studied in detail yet.

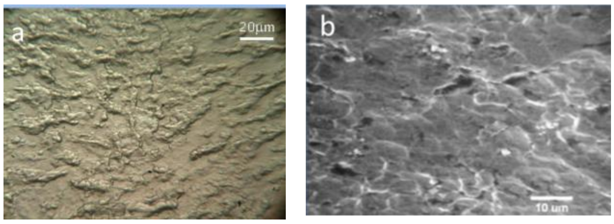

Fig. 6. Photos of the W-K surface after sputtering the layer with thickness Δh=4.2 μm; a) – OM; b) – SEM

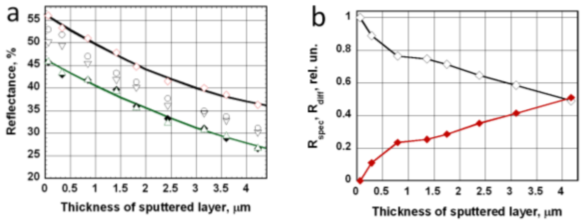

Fig. 7. a – dependence on Δh of reflectance at different wavelengths; b – dependence on Δh of specular and diffuse parts of the profile. Same symbols as in Fig. 2

2.5. W-2LA

The quality of initial surface of this specimen was far from optical quality (Fig. 8). Sputtering actions resulted in further development of roughness accompanied bycontinued degradation of optical characteristics. The image profile is not of a П-like shape, therefore it is difficult to divide the intensity of reflected light into specular and diffusive components, when sputtered layer thickness increases.

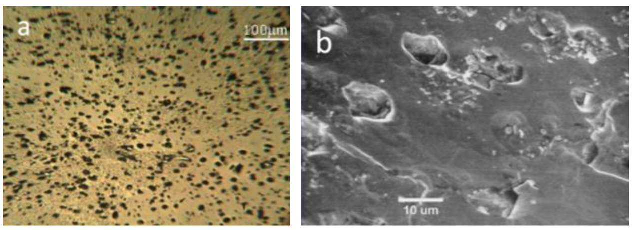

Fig. 8. Photos of the W-2La surface: a – initial state (OM), b – final state (SEM) with Δh = 3.97 μm

2.6. WEIGHT LOSS

The measurements of weight loss of specimens were carried out before and after every sputtering action with an aim to provide comparison of resistance to sputtering of different W samples under identical experimental conditions. The accuracy of weight measurement was ~25 μg for typical weight change 0.5...1.0 mg after each exposure in plasma. Because there was some scattering in the specimen sizes, all obtained data were scaled to 1 cm^2. The results found in such a way are shown in Fig. 9 for all abovementioned specimens. When calculating the sputtering rate we ignore the difference in the specific density of material and took its value equals to 19 g/cm^3.

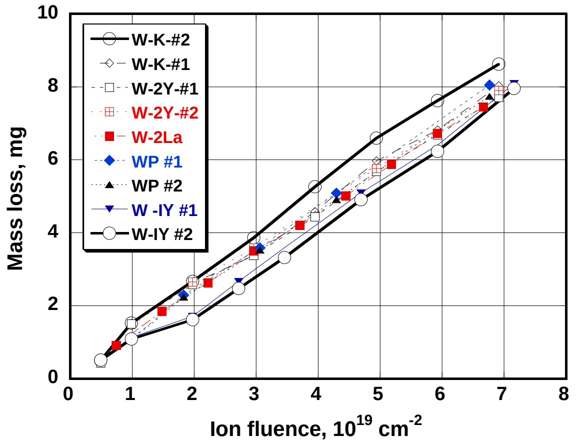

Fig. 9. Dependence on ion fluence of the weight loss normalized to 1 cm^2 of the surface for all W specimens

As seen, the mass loss data for some specimens are rather different. The highest resistance to sputtering showed one of the W-1Y sample with a quite low difference between both such kind samples. The least resistant was one of those doped with 0.1% K and 0.1% Al (namely, W-K-#2), with a quite noticeable difference of sputtering rate among both these samples. The maximal difference between the strongest (W-K-#2) and weakest (W-1Y#2) sputtering rate reached ~20 % at high enough ion fluence, $>5 \cdot 10^{19}$ cm^2. However, the variation of loss weight between majority of specimens (i.e., excluding W-K-#2) is much lower, close to 10 % in average.

CONCLUSIONS

When analyzing all data we may conclude that for pure tungsten specimens (PW) the reflectance at normal incidence do not degrade significantly, at least after sputtering the layer of ~4 μm in thickness. The most part of their in-grain surfaces is continuing to be smooth till the end of sputtering procedures. The relationship of specular and diffusive parts of reflected light is stabilized after sputtering layer thickness reached ~1 μm and does not change with following sputtering. This means, to a certain extent, stabilization of the ability to absorb electromagnetic radiation emanating by burning plasma. This fact can be important from the viewpoint of the use of tungsten as plasma facing materials in divertor of ITER (baffle and dome), where sputtering by charge exchange atoms will predominate over other mechanisms of plasma-surface interaction. Comparing obtained results, according to this criterion the best of all described specimens is the specimen made of **pure tungsten**.

The relief on WP specimens has a typical step structure with the step heights ≤1.6 μm, what is in agreement with the value (up to 1.8 μm) measured by laser prophilometry for recrystallized W specimen after sputtering to ~4 μm [8].

The mirror specimens made from doped tungsten behave very differently from pure tungsten. Their reflectance is decreasing faster with increasing the sputtering depth due to rise of roughness. The roughness developed due to sputtering for most of doped samples is characterized by a rather chaotic relief with exclusion for the samples with 2% of Y addition, where some similarity of a step structure can be seen.

A quite big difference was found for sputtering rate: up to ~20 %, with the most resistant – the tungsten specimens doped with 1 % of yttrium (W-1Y), possibly due to preferential orientation of majority of their grains in the plain [111], which was found in [8] to be much more resistant to sputtering.

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ПОВЕДЕНИЕ ПРИ ДЛИТЕЛЬНОМ РАСПЫЛЕНИИ ОБРАЗЦОВ ЗЕРКАЛ ИЗ РАЗНОГО ТИПА ВОЛЬФРАМА

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Проведено сравнение поведения образцов зеркал из чистого вольфрама и вольфрама с добавками (Y, K-Al, La) при длительной бомбардировке ионами аргона. Измерялись оптические характеристики поверхности и динамика её рельефа в зависимости от толщины распылённого слоя, которая определялась по потере массы. Обнаружено большое различие в характере рельефа, скорости деградации коэффициента отражения и соотношения зеркального и диффузного компонент отражённого света для зеркалец из вольфрама разного типа.

ПОВЕДІНКА ПРИ ДОВГОТРИВАЛОМУ РОЗПИЛЕННІ ЗРАЗКІВ ДЗЕРКАЛ З ВОЛЬФРАМУ РІЗНОГО ТИПУ

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Проведено порівняння поведінки зразків дзеркал з чистого вольфраму и вольфраму с домішками (Y, K-Al, La) при довготривалому бомбардуванні іонами аргону. Вимірювались оптичні характеристики поверхні і динаміка її рельєфу в залежності від товщини розпиленого шару, яка знаходилась по втраті маси зразка. Знайдено велику різницю в характері рельєфу, швидкості деградації коефіцієнту відбиття і співвідношення дзеркального і дифузного компонентів відбитого світла для дзеркалець з різного типу вольфраму.