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Tungsten self-sputtering yield with different incidence angles and target temperatures

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Abstract

The self-sputtering of different types of tungsten due to 1 keV W^+ bombardment at temperatures of 25°C and 600°C and incident angles in the range of 30–60° was studied by means of the weight loss method. The experimental data at room temperature agreed reasonably with the results of TRIM calculations. Enhanced self-sputtering yields due to beam-induced desorption of WO_2 were found at a temperature of 600°C. The weight loss of W–Cu composite is larger than that of the CVD-W and ps-W under the same irradiation conditions due to the selective removal of copper. © 1998 Elsevier Science B.V. All rights reserved.

1. Introduction

Tungsten has been selected as one of the armor material candidates of the ITER plasma facing components. The advantages of the tungsten armor can be characterized as follows: (1) low sputtering yield against light ions, (2) high melting point that will result in lower erosion against high heat loads. One of the critical issues of tungsten as the plasma facing materials is that the self-sputtering yield is still large at low energy. In spite of the fact that sputtering yields on tungsten have been widely investigated experimentally, only few experimental data of the incident angular dependence for the tungsten self-sputtering yields have been reported [1–3]. Further experimental database is also been limited for specified incident angles and temperatures that will be relevant for ITER conditions. The intention of the present study is to extend the experimental tungsten self-sputtering database for different angles of incidence and target temperature, that will be necessary for the ITER design.

2. Experimental setup

The sputtering experiments were performed in the modified self-sputtering testing accelerator (SSP) [1,4]. The schematic of SSP is shown in Fig. 1. A sputter ion source, is used to produce a W^+ ion beam. To run the source a support gas, normally argon, is used. By the application of a negative potential to the W electrode in the arc chamber, some ions can be attracted towards it that results in the introduction of neutral sputtered atoms into the arc plasma, where they can be ionized. The use of molybdenum for the extraction slit and a 0.8 mm diameter tungsten wire for the filament results in increasing the operation time up to 80 min. The extraction electrode and the beam drift tube are operated at a potential V_E up to 10 kV below that of the ion source. The extracted and mass-separated ion beam with an energy $eV_E = 10$ keV is then decelerated down to the impinging ion energy $eV_A = 1$ keV just in front of the sample target that is grounded. The 1 keV W^+ ion flux density was 0.63×10^{17} m^{-2} s^{-1}.

The base pressure in the target chamber was 2×10^{-6} Pa. During ion source operation the pressure rises to about 2×10^{-5} Pa mainly due to the argon gas.

The target samples were powder-sintered W (ps-W) and chemical vapor deposited W (CVD-W) from Tokyo

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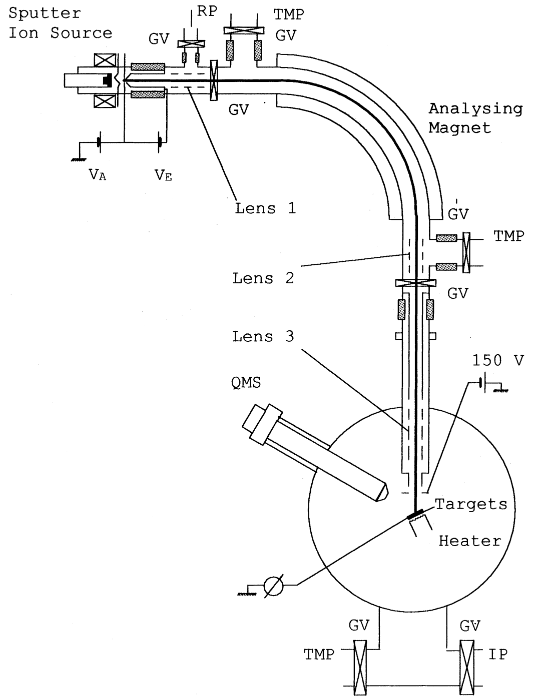

Fig. 1. Experimental setup: GV, Gate Valve; TMP, Turbo-Molecular Pump; RP, Rotary Pump; IP, Ion Pump; QMS, Quadrupole Mass Spectrometer.

Tungsten and W–Cu composition (30 wt% of Cu) from Sumitomo-Denko with a typical size of 10 mm × 10 mm × 0.9 mm. The sputtering yield was determined by the weight loss method. The target weight change and the integrated beam current were measured by a Mettler ME22 microbalance with an absolute accuracy better than 1 μg and by a microammeter with an absolute accuracy better than 15%, respectively. The weight change of the target is a result of the weight decreasing due to sputtering and the weight increasing due to impinging ions which are not reflected:

$$\Delta m = -S_{\mathrm{W\text{-}W}} \times (M_{\mathrm{W}}N/N_0) \times \alpha + (1 - R_{\mathrm{W\text{-}W}}) \times (M_{\mathrm{W}}N/N_0), \tag{1}$$

where Δm is the weight change of the target, $S_{\mathrm{W\text{-}W}}$, M_{W}, $R_{\mathrm{W\text{-}W}}$, are the sputtering yield, the target atomic mass and the reflection coefficient for W, respectively, N_0 is Avogadro's number, N the number of incoming projectile atoms. Since with increasing angles of incidence, measured from the surface normal, the reflection coefficient increases, and can no longer be neglected as for normal impact. Thus the weight loss method allows us to estimate only the total gross yield

$$Y = S_{\mathrm{W\text{-}W}} + R_{\mathrm{W\text{-}W}} = 1 - \Delta m \times (N_0/M_{\mathrm{W}}N). \tag{2}$$

3. Experimental results and discussion

3.1. Estimation of the self-sputtering yield for ps-W and CVD-W

The gross yield Y obtained by using Eq. (2) and the measured Δm are shown in Fig. 2. The solid line represents the calculated values of $S_{\mathrm{W\text{-}W}}$ + $R_{\mathrm{W+W}}$ by using TRIM92. The present experimental data at room temperature as well as available published data [2,3] agree reasonably well with the results of TRIM calculations. On one hand this agreement provides support that the surface binding energy of 8.68 eV for tungsten used in the calculation is close to the real value. The gross yield of the tungsten buildup layer, where the surface binding energy is obviously lower, is higher than calculated [3], see the dotted curve in Fig. 2. On the other hand it is not obvious why the effect of surface topography was not observed in contrast to other published data [5].

There are several parameters or processes that can be responsible for the change of the sputtering yield at 600°C, among them are the surface roughness, chemical sputtering by oxygen, surface chemical composition.

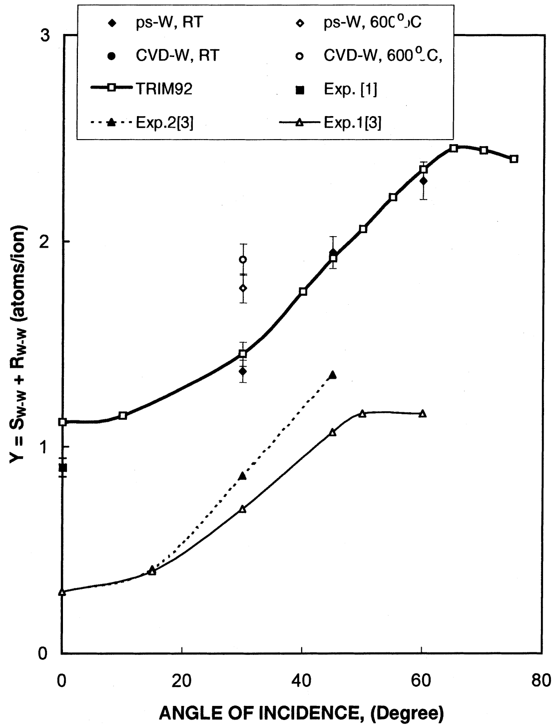

Fig. 2. Gross yield of tungsten self-sputtering due to 1 keV W^+ ion bombardment as a function of the incidence angle at different temperatures. The gross yield for 350 eV ions is below unity, see Experiment 1 of [3]. In this case a buildup of layer occurs. The yield of such a buildup layer is shown as the dashed curve (Experiment 2 of [3]).

The sputtering yield is inversely proportional to the surface binding energy usually regarded equal to the heat of sublimation. It was shown in Ref. [6] that only near the melting point the heat of sublimation decreased to 80–90% of that value at room temperature, depending on the material. All specimens were cut into plates and then polished mechanically to a mirror-like surface. We can expect that at the beginning of ion bombardment all specimens have almost the same surface roughness. The oxygen flux density J_G due to adsorption from the gas phase on the specimen surface in the present experiments is about 2×10^{12} O atoms cm^{-2} s^{-1}. The rate of desorption of tungsten in the form of W_xO_y species is negligible at this oxygen flux and a temperatures of 600°C [7], but an enhanced sputtering yield due to beam-induced desorption of MoO_2 and cascade sputtering of MoO was observed at 485°C during oxygen exposure [8]. It seems a conceivable mechanism of this process may be acceptable also to explain the experimental data for tungsten: the thermal desorption of WO_2 from a beam-loosened surface compound at a rate faster than collisional emission. Indirect confirmation of such assumption was observed by SIMS (see Fig. 3). The most intensive secondary positive ion signals (except for Na^+, K^+, Ca^+) were W^+, WO^+, WO_2^+ for ps-W and CVD-W specimens. Since the oxygen flux to the surface was not enough to keep a stable WO concentration, the intensities of WO^+ and WO_2^+ signals decrease during ion bombardment at higher temperatures.

3.2. Sputtering of W–Cu composition by 1 keV W^+ ions

The W–Cu composition is prepared by impregnation of porous W with Cu. As a result at the specimen surface there are regions with pure W and pure Cu. At present experimental conditions the sputtering yield of Cu is more than twice as large as the tungsten self-sputtering yield. Fig. 4 shows the calculated weight loss for a different percentage of Cu area on the surface and experimental results for ps-W, CVD-W and W–Cu. In a theoretical estimation results of TRIM calculations of the sputtering yield and reflection coefficient for pure W and Cu have been used. The weight change of the W–Cu target is a result of weight decreasing due to sputtering and weight increasing due to impinging ions that have not been reflected

$$\begin{aligned}\Delta m = &- S_{\text{W-W}} \times (M_{\text{W}}N/N_0) \times \alpha + (1 - R_{\text{W-W}}) \\ &\times (M_{\text{W}}N/N_0) \times \alpha - S_{\text{W-Cu}} \times (M_{\text{Cu}}N/N_0) \times \beta \\ &+ (1 - R_{\text{W-Cu}}) \times (M_{\text{W}}N/N_0) \times \beta, \qquad (3)\end{aligned}$$

where $S_{\text{W-Cu}}$, M_{Cu}, $R_{\text{W-Cu}}$ are the sputtering yield, the target atomic mass and the reflection coefficient for pure Cu respectively, and α, β are the part of surface occupied by W and Cu respectively.

It can be seen that the experimental weight loss is larger than the calculated one at room temperature. In-situ SIMS analysis revealed that the Cu concentration decreases at the surface due to preferential sputtering, see Fig. 5. On the contrary, ion bombardment at 600°C results in an increasing Cu^+ ion signal. Therefore the sample weight loss under RT ion bombardment occurred almost due to the tungsten self-sputtering. It has been shown [5] that experimentally measured sputtering

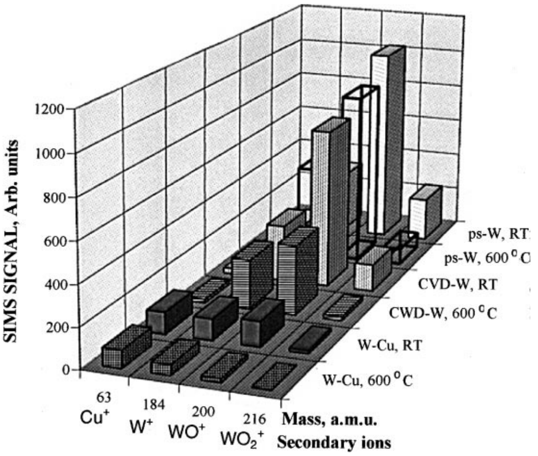

Fig. 3. Intensity SIMS signal of secondary ions Cu^+, W^+, WO^+, WO_2^+ for the different specimens irradiated with 1 keV W^+ ions. Angles of incidence and detection are 30° and 40° with respect to the surface normal, respectively.

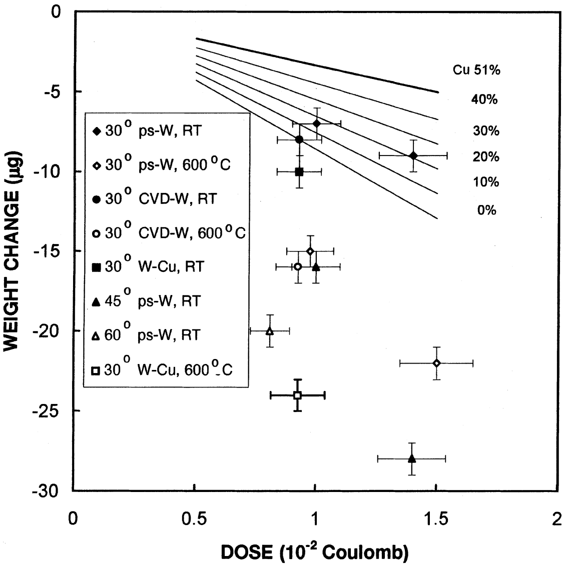

Fig. 4. The calculated weight loss due to 1 keV W^+ ion bombardment of W–Cu composition for different percentages of Cu area on the surface and experimental results for the different specimens. The incidence angle, the type and temperature of the specimen are indicated near the symbol in figure.

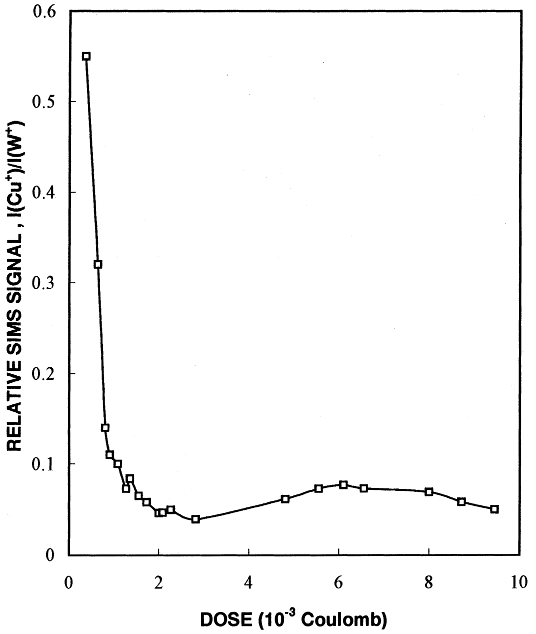

Fig. 5. The dependency of the SIMS relative signal on dose under 1 keV W^+ ion bombardment of W–Cu composition. Angles of incidence and detection are 30° and 40° with respect to the surface normal, respectively.

yields for materials with a significant surface roughness are higher than results of TRIM calculations. The development of surface roughness due to ion bombardment, that was observed for W–Cu in Ref. [9], may be responsible for the weight loss larger than in the case of CVD-W. Abnormally high values of the sputtering yields of pure W, pure Cu, and W–Cu composition with the polyenergetic flux of hydrogen particles has been observed in Ref. [10].

The tendency of increasing sputtering yields with increasing target temperatures will be examined in experiments at higher temperatures (1000°C) and is currently under way.

4. Conclusions

An experimental investigation of tungsten self-sputtering as a function of the incident angle over the range from 30° to 60° for ps-W at room temperature has been performed. The experimental data agree reasonably well with results of TRIM calculations.

The measured values of self-sputtering yields for CVD-W and ps-W are almost the same both at RT and 600°C. The self-sputtering yield at a temperature of 600°C was larger than that at room temperature due to beam-induced desorption of WO_2.

It was also found that the weight loss of the W–Cu composite is larger than that of the CVD-W and ps-W under the same dose at room temperature due to the selective removal of copper and further propagation of surface roughness.

References

[1] M. Saidoh, K. Sone, Jpn. J. Appl. Phys. 22 (9) (1983) 1361.
[2] W. Eckstein, J. Laszlo, J. Nucl. Mater. 183 (1991) 19.
[3] C.H. Wu, E. Hechtl, J. Nucl. Mater. 196–198 (1992) 569.
[4] Fusion research and Development Center: Annual report 1980, Rep. JAERI-M 82-027, 1982, p. 140.
[5] Y.N. Zhukova, E.S. Mashkova, V.A. Molchanov, V.M. Sotnikov, W. Eckstein, The dependence of the sputtering yield at different angles of incidence for the polycrystalline surface with structure. Book of abstract XI International Conference Ion–Surface Interaction 1993, Moscow, vol. I, pp. 88–90.
[6] R. Hultgren, J.P. Desai, D.T. Hawkins, M. Gleiser, K.K. Kelley, D.D. Wagman, Selected Values of the Thermodynamic Properties of the Elements, Am. Soc. Metals, Metals Park, OH 1973.
[7] C.H. Wu, J. Nucl. Mater. 145–147 (1987) 448.
[8] M. Saidoh, H. Gnaser, W.O. Hofer, Appl. Phys. A 40 (1986) 197.
[9] L.I. Ivanov, A.P. Komissarov, A.V. Polyakov, Fiz. Khim. Obrab. Mater. 1 (1994) 5.
[10] G.G. Bondarenko, Ya.Ya. Udris, J. Nucl. Mater. 233–237 (1996) 688.