Heating effect on deposition of hydrocarbon film and characteristics of metallic mirrors in ITER relevant conditions

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1. Introduction

Most of ITER optical diagnostics require relay mirrors, the first of which will be exposed under severe conditions: gamma-neutron radiation, bombardment by charge exchange atoms (CXA) and deposition of materials eroded from in-vessel components. Due to effect of the nuclear environment the first mirror (FM) have to be made of a metal. Redeposition of the divetor plates faced by carbon could be a reason of mirror deterioration [1, 2]. FM heating to the temperatures, at which a chemical erosion of hydrogenated carbon (C:H) films by hydrogen atoms will be prevail over deposition, may prevent this effect [3]. We have being performed a study the heating effect on the deposition efficiency and reflectivity of metallic mirrors using a direct current (dc) magnetron device.

2. Experiments on magnetron deposition

Several SS and Cu mirrors were exposed during 2 hours to the dc magnetron discharge in the

mixture of 43% Ar, 35% CH₂D₂ and 22% D₂ at temperature range from 150°C up to 400°C. The layout of experiment is presented in Fig.1. Two mirrors (10×10 mm^2 , 4 mm of thickness) could be tested at once. Thin SS foil screened a part of samples during deposition. The mirrors were installed on a heater and, after pumping of the vacuum chamber down to 10^{-5} Pa, heated to a test temperature. Then magnetron discharge (I=100 mA, $U_{cath}=370 \text{ V}, n_{e}\sim 10^{10} \text{ cm}^{-3}, T_{e}\approx 8 \text{ eV})$ was ignited at pressure of 3 Pa. The diameter of the graphite cathode was 20 mm. The



Fig.1. Layout of experiment: 1 – vacuum vessel; 2 – moving table; 3 – magnetron; 4 – graphite cathode; 5 – magnetron discharge; 6, 7 – Cu and SS mirrors; 8 – screened parts of mirrors; 9 – thermocouple; 10 – heater

average energy of ions bombarding the cathode was about of 0.75 U_{cath} (≈ 280 eV). The temperature of mirrors during deposition was monitored by a thermocouple. Besides, in a

number of cases heated mirrors were exposed in the gas mixture without magnetron discharge.

3. Analysis methods

The surface elemental composition of mirrors was tested by means of simple SIMS and AUGER analyzers before and after exposure. The reflectance of mirrors was measured by a spectrophotometer and film thickness by means a profiler at several points at the boundary of the opened and the screened surfaces. The surface morphology and microstructure of deposits were investigated with a JEOL scanning electron microscope.

The backscattering of 2.3 MeV protons (BSP) and Rutherford backscattering (RBS) of 2.3 MeV He⁺ ions were used for accurate analysis of the deposits' composition on four SS mirrors. The hydrogen isotopes' depth profiles were measured with Elastic Recoil Detector Analyses (ERDA) using 1.9 MeV He⁺-ion beam at 10° incident angle upon the sample. To obtain absolute atom concentrations the energy spectra measurements were done using the polyethylene calibration etalons. The accuracy of the measurements is estimated as 15%.

4. Main results

It was found that at temperatures 150°-300°C a continuous growth of hydrogenated carbon films was observed. As a result the reflectance spectra of the mirrors were strongly distorted by the interference as shown in Fig.2. Visually the deposits are very similar to the films that were observed on the mirrors after exposure in T-10 tokamak inside of the diagnostic port in front of the graphite limiter [1]. The growth of C:D films was mitigated at higher



Fig.2. Reflectance of SS mirrors: initial and with deposits

temperatures (350, 400°C). However, thin deposits were still detected on the surfaces of hot mirrors that leads to 10-20% reflectance drop for both SS and Cu mirrors. The dependence of film thickness versus the temperature of SS mirrors is shown in Fig.3. According to BSP data C:D films basically consist of carbon (\approx 50%), hydrogen isotopes (\approx 40%), oxygen (5-7%) and nitrogen (2-3%) impurities. This result is confirmed by the SIMS measurement when strong picks of carbon and light hydrocarbons were recorded.

Calculated (D+H)/C ratio inside the C:D films is about 0.8. ERDA gives practically constant D and H concentration distributions over the thickness of the film as shown in Fig.4.

After exposure at 350°C and 400°C the concentration of O and N atoms increases as much as about 10 times in deposits arisen on SS mirrors (see Fig.5) as compared with C:D films. It should be



700 600 500 400 nm 300 É 200 100 0 150 200 250 400 300 350 T, 0C

Fig.3. The dependence of film thickness versus the temperature of SS mirrors



Fig.4. D and H profiles inside the C:D film on SS mirror exposed at 180°C

Fig.5. BSP data for deposit on SS mirror exposed at $350^{\circ}C$

noted that deuterium is practically absent in these deposits. One of the explanations of the effect is that the erosion and other chemical processes at the surface, like carbide and oxide formation, became more intensive at elevated temperatures, thus changing the film composition.

The copper mirrors' reflectivity also decreases after exposure at high temperatures. In this case the SIMS analysis has shown increase of only carbon peaks, and copper oxide peak is decreased. It could be explained so, that the copper's carbide and oxide are not stable at these temperatures. Probably, very thin carbon deposit arises on Cu mirror in the experimental conditions. The study of Cu mirrors will be continued. In particular, the experiments with higher deuterium and less methane concentrations in gas mixture will be done. Possibly, it will be lead to stronger chemical erosion of carbon.

Finally let us note that after exposure during 2 hours at 400°C in the same gas mixture without magnetron discharge SS mirror was covered by hydrocarbon film that resulted in reflectance degradation of the mirror. At the same experimental conditions the reflectance of Cu mirror decreased only in infra red spectral range. After exposure at 150°C without magnetron discharge the reflectance of both types of mirrors was not change.

5. Modelling calculation

Simple numerical modeling has been performed for analyses of the experimental conditions. The flux of carbon atoms from graphite cathode was estimated as $F_C \sim 10^{17} \text{ s}^{-1}$ owing to its physical sputtering by Ar ions. The average energy of sputtered C atoms was about of 7.4 eV. SRIM 2003 code was used for estimation of C atom flux density on the mirror at the experiment geometry. The flux density was about 7×10^{14} atoms/cm²s.

6. Conclusion

The growth rate of hydrogenated carbon films is basically determined by competition of deposition and chemical erosion [3]. For FM in ITER the result will depend on such parameters as CXA specific fluxes and energetic spectrum, gas composition and pressure, location and temperature of the mirror. Some from these parameters depend on tokamak discharge mode. So, the temperature dependence is turning to be a complicated one, but in general a mirror heating mitigates the growth of hydrogenated carbon films that was demonstrated in the present experiments.

On the other hand a chemical activity of hot metallic surface strongly increases, that result in modification of the mirror surface. Moreover reflectance degradation of the hot mirrors takes place even without plasma at relatively high methane concentration.

It could be concluded that the right choice of material is very important for preservation of the mirror quality. Numerical simulation and experiments for modeling of a FM performance in ITER relevant conditions, that have to take into account both fluxes of plasma impurities on surfaces of the mirror and composition of cold gas, are necessary steps towards decision of the first mirror problem.

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