Cleaning of HT-7 Tokamak Exposed First Mirrors by Radio Frequency Magnetron Sputtering Plasma∗

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Abstract The stainless steel (SS) first mirror pre-exposed in the deposition-dominated environment of the HT-7 tokamak was cleaned in the newly built radio frequency (RF) magnetron sputtering plasma device. The deposition layer on the FM surface formed during the exposure was successfully removed by argon plasma with a RF power of about 80 W and a gas pressure of 0.087 Pa for 30 min. The total reflectivity of the mirrors was recovered up to 90% in the wavelength range of 300-800 nm, while the diffuse reflectivity showed a little increase, which was attributed to the increase of surface roughness in sputtering, and residual contaminants. The FMs made from single crystal materials could help to achieve a desired recovery of specular reflectivity in the future.

Keywords: radio frequency, plasma sputtering, first mirror, cleaning

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(Some figures may appear in colour only in the online journal)

1 Introduction

First mirrors (FMs) are the vital elements of the optical and laser diagnostic systems in a large scale tokamak such as the international thermonuclear experimental reactor (ITER), which will have more than 80 FMs directly facing the plasma [1]. The re-deposition of the eroded first wall materials and the deposition of the wall conditioning materials may form a contamination layer on the surface of the FMs, which will not only sharply decrease the FM’s reflectivity, but also influence the accuracy and validity of the diagnostic signal, or even the security of the tokamak operation. A 20 nm deposition layer is thick enough to decrease the reflectivity of the FM [2]. However, the deposition layer on the surface of the FMs exposed in a tokamak is often much thicker than that [3–5].

To prolong the lifetime of the FMs, FM cleaning is proposed and investigated worldwide [6–11]. Two main methods are used: laser cleaning, which uses pulsed laser radiation to remove the deposition layer by ablation or delamination, and plasma cleaning, which sputters the deposition layer by layer to recover the FM surface. The laser cleaning did not give satisfactory results at the visible wavelength range when the tokamak exposed FMs were cleaned, as reported in Refs. [6] and [12], while the plasma cleaning is identified as an effective method which can clean any type of material if the plasma parameters are properly chosen. Two plasma sources, radio frequency (RF) plasma and electron cyclotron resonance (ECR) plasma have been investigated [11,12]. The ECR hydrogenic plasma can give an acceptable result when cleaning the laboratory and soft tokamak hydrocarbon films [11], but the ECR device structure is relatively complex. The RF Ar plasma cannot effectively clean the FM because the surface oxidation of FM competes with the plasma sputtering during the cleaning [12]. Compared with the RF plasma, the magnetron sputtering plasma can sputter the target with a more rapid speed, which may reduce the oxidation of the FM during the cleaning process. In addition, the device has a simpler structure than the ECR device because only the magnetron target head is required to be placed in a vacuum chamber. Moreover, a highly uniform target treatment could be obtained by changing the distribution of the magnet field.

So the RF magnetron sputtering plasma device was built and used to clean the HT-7 tokamak exposed FM, and preliminary results are described in the present paper.

2 Sample preparation and exposure

To study the cleaning effect of this method on a tokamak exposed FM, a 316L SS (C≤0.03%, Si≤1.00%, Mn≤2.0%, P≤0.035%, S≤0.03%, Ni: 12.0%-15.0%, Cr:
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16.0%-18.0%, Mo: 2.0%-3.0%) sample was pre-exposed in the HT-7 tokamak which has a SS main chamber first wall and graphite limiter. The SS mirror has a dimension of 40 mm×35 mm×3 mm with a resulting surface roughness of 1.9 nm. It was placed in the low field side near poloidal limiter with a distance of 25 mm from the last closed flux surface, where the FM would experience a deposition dominated exposure [13]. The sample was exposed to 3706 pluses of plasma discharge (plasma current of 80-180 kA, electron density of (1.5-3.0)×10^{19} m^{-3}), 3 hours siliconization, 3 hours boronization, 124 hours of helium glow discharges, and 14 hours of deuterium glow discharges during 2009-2010 campaign.

3 Experimental set-up for cleaning

The RF magnetron sputtering plasma is one kind of glow discharge plasma and has a controllable sputtering process. The schematic of the RF magnetron sputtering plasma cleaning device in the laboratory is shown in Fig. 1. The system consists of an RF power source, a vacuum chamber, a mechanical pump system, a molecular pump system, and a working gas supply system. The frequency of the power supply system, with a maximum output power of 500 W, is 13.56 MHz. In general, the vacuum chamber was pumped to a background gas pressure of 10^{-2} Pa by the mechanical pump and the molecular pump before the experiment. The gas pressure was increased to a proper value through increasing the flow velocity of the working gas before starting the plasma discharge, which was achieved by adjusting the input RF power and the capacitances of the matching network. While cleaning the HT-7 tokamak exposed FM, Ar was selected as the working gas with a gas flow of 3 standard-state cubic centimeters per minute (sccm) and a working gas pressure of about 0.087 Pa to get a stable plasma discharge and an efficient sputtering process. In order to clean the FM effectively, the RF power was chosen to be 80 W, which produced a negative self-bias on the FM of about 130 V. The electron density and temperature of the magnetron sputtering plasma were respectively estimated to be (1-8)×10^{15} m^{-3} and 5-10 eV by referring to previous experiments. The total cleaning time was 30 min.

4 Results and discussion

Photographs of the SS FM before exposure, after exposure but before cleaning and after cleaning are presented in Fig. 2. The FM was clean and homogeneous before exposure in the HT-7 tokamak (as seen in Fig. 2(a)). After exposure, an inhomogeneous colorful deposition layer was formed on the FM surface (seen in Fig. 2(b)) which was ascribed to the deposition of the wall conditioning materials and the re-deposition of the sputtered first wall materials. Fig. 2(c) shows the photo of the FM after cleaning by the RF magnetron sputtering plasma, from which one could see no visual deposition on the FM surface, indicating that the RF magnetron sputtering plasma can effectively remove the tokamak deposition impurities. The surface of the cleaned FM showed a slight inhomogeneity compared with that of the FM before exposure.

Fig.2 FM surface photos: (a) before exposure, (b) after exposure but before cleaning and (c) after cleaning

Fig.1 Experimental setup for FM cleaning with RF magnetron sputtering plasma
The total and diffuse reflectivity of the FM shown in Fig. 3 were measured by a spectrophotometer in the wavelength range between 300 nm and 800 nm; in this range the FM reflectivity is very difficult to recover. The total reflectivity significantly decreased after exposure (seen in Fig. 3(a)), which was almost half of the original value and caused by the absorption of the contaminated deposition layer seen in Fig. 2(b). However, after cleaning, the total reflectivity is obviously improved. Compared with the original one, the total reflectivity was recovered by up to 90% at the wavelength range from 300 nm to 800 nm, which is better than the cleaning results using the Q-switched Nd:YAG laser and the excimer laser reported in Refs. [6] and [8]. It indicates that the magnetron sputtering plasma can effectively clean the multicolor tokamak deposition layer (seen in Fig. 2(c)). Fig. 3(b) shows the diffuse reflectivity of the FM before and after cleaning. It could be noted that the diffuse reflectivity increased a little after cleaning, which could make a contribution to the recovery of the total reflectivity due to the sputtering effect of the Ar plasma on FM. When an Ar plasma is used to clean the FM, the FM surface would also be sputtered and damaged. The small scale micro-relief and the stepped structure (seen in Fig. 5(b)) could develop due to the polycrystalline structure of the FM itself. As a result, the surface roughness of the cleaned FM would increase. The surface roughness of the cleaned FM is 3.95 nm measured by the atom force microscope which is a little higher than that of the FM before exposure.

Fig. 4 shows the surface morphology of the FM before exposure, after exposure but before cleaning and after cleaning. One can clearly see that the surface of the FM before exposure is clean and smooth. After plasma exposure in the HT-7 tokamak, dense particles with micrometers in size were deposited on the surface of the FM (seen in Fig. 4(b)), from which we can speculate that the thickness of the deposition layer is in the order of micrometers. However, the deposited particles are unseen on the surface of the cleaned FM in Fig. 4(c), which indicates that the deposition layer on FM surface formed during the exposure in the HT-7 tokamak was successfully removed by the magnetron sputtering plasma as discussed before. However, the micro-relief and stepped structure were found on the surface of the cleaned FM, which is obviously a feature caused by the sputtering effect. At the same time, a small amount of residual contaminant existed on the surface of the cleaned FM (white particles and dark spots in Fig. 4(c)). These sputtering features and residual contaminants could be the reasons for the increase of the diffuse reflectivity after cleaning and the difference of total reflectivity between the original and the cleaned FM.

Fig. 5 shows the elemental compositions of the FM before and after cleaning measured by energy dispersive spectroscopy (EDS) analysis. The main contaminant elements of the exposed FM are carbon, boron, oxygen and silicon as indicated in Fig. 5(a). The elemental compositions of the stepped structure and the micro-relief on the cleaned FM are mainly iron, nickel, silicon, chrome and molybdenum (seen in Fig. 5(b) line 1), which are the main elements of the SS FM itself. Carbon was detected on the dark spot which was shown.
in Fig. 5(b) line 2. It may be the composition of the SS, or the penetration of the carbon during the exposure in the HT-7 tokamak before cleaning, or else the re-deposition of the carbon contaminants during the cleaning. The 316L SS FM contains about 0.03% carbon. And the SS FM could also be implanted with carbon during the plasma exposure as in Ref. [14]. In addition, carbon is one of the main compositions of the deposition layer. The removal of the deposition layer could produce a high content of the deposits on the main chamber of the device, which might lead to small re-deposition of the deposits. The high absorption of carbon could be the reason for the difference of total reflectivity between the original and the cleaned FM. The elements for the white particles are mainly the SS composition elements (seen in Fig. 5(b) line 3). The intensity of the detected elements is similar to that of the stepped structure which indicates that the white particles could be the inner bulk defect of the FM itself.

The specular reflectivity of the FM is very important for the transmission of radiation from the plasma towards the detectors in diagnostics. It could be given by the difference between total and diffuse reflectivity: $R_{\text{Spec}} = R_{\text{Tot}} - R_{\text{Diff}}$ [15]. To get a high specular reflectivity, the diffuse reflectivity of the cleaned FM must be controlled effectively by reducing the micro-relief and stepped structure level. The FMs made from monocrystalline materials have a better performance than those made from polycrystalline materials under a sputtering-dominated condition [14], which demonstrated that using single crystal mirrors could help to achieve the desired recovery of specular reflectivity, evidencing the successful non-destructive cleaning.

5 Conclusion

An RF magnetron sputtering plasma cleaning method was developed for fusion device FMs. After 30 min of cleaning, the multicolor deposition layer containing boron, oxygen, silicon and carbon on the surface of the FM exposed in the HT-7 tokamak was successfully removed by the Ar plasma. The total reflectivity of the cleaned FM was recovered by up to 90% in the wavelength range of 300-800 nm. This is the wavelength range in which the reflectivity is difficult to recover. However, it should be noticed that it is the specular reflectivity which must be recovered. Using single crystal mirrors would help to achieve the desired recovery of specular reflectivity in future.

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