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The longer life and high performance of lithium containing coatings developed by ICRF in the HT-7 superconducting tokamak ☆

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Abstract

A new wall conditioning method, lithium containing silicon coatings (α -C:H/Li–Si) in situ realized by means of ion cyclotron range of frequency plasma assisted chemical vapor deposition, has been successfully developed in the HT-7 superconducting tokamak, which leads to not only the effective suppression of carbon and oxygen impurities, but also lower hydrogen recycling than siliconization. After the wall conditioning, the impurity level in the vacuum vessel of HT-7 device measured by QMS and spectroscopy was largely reduced and obviously lower than siliconization and even approaches lithium in situ coatings. The depth profile of deposition was investigated by Auger surface analysis. The decreases of edge plasma temperature and electron density showed that the plasma confinement has been significantly improved comparing with siliconization, and is sustained for nearly 350 shots. © 2003 Elsevier Science B.V. All rights reserved.

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1. Introduction

Wall conditioning has played and is continuing to play an essential role on the path of controlled nuclear fusion research towards ignition. Many of the advances in controlled nuclear fusion have been closely linked to progress in our abilities to control the impurity influx and hydrogen recycling. Among the in situ coating materials for plasma facing walls, lithium has the lowest atomic number and highest reactivity, so lithium coatings may be the best wall conditioning method. Several methods have been used to introduce lithium into plasmas: pellet injection [1–4], evaporation [5–7], lithium borohydride discharge [8], and laser-assisted lithium aerosol injection [9]. A great success of lithium wall conditioning in TFTR has been reported since 1992, where deposition of a few milligrams of lithium on the bumper limiter leads to a significant increase the fusion triple product ($n_e \tau_E T_i$) and considerable improvement of the energy confinement time from $\tau_E = 0.075$ s (L mode) to $\tau_E = 0.33$ s with very peaked density profiles [10].

Lithium wall conditioning effects appear as a very low hydrogen recycling (low edge density), reduction of oxygen impurity in a hot core plasma region, significant suppression of carbon impurity, and improvement of energy confinement, all of these effects have been observed clearly in TFTR and other devices [11]. However, it should be noted that the lifetime of lithium coatings is very short, which rapid deterioration may be related to the physical and chemical properties, such as low melting point, low hardness, strong chemical reactivity

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and high mobility in metals, leading to rapid damage of the coating under plasma irradiation [12]. Thus, there has been an open question of how to prolong its effective service time.

Recently, ion cyclotron range of frequency (ICRF) wall conditioning technique was successfully tested and developed in the HT-7 superconducting tokamak [13,14]; this special technique can be used for the wall conditioning of future large superconducting tokamaks with high magnetic fields like ITER. ICRF siliconization in HT-7 device has provided a good wall condition for oxygen gettering and powerful edge radiation, and has a relatively long lifetime [15]. But the effective services of lithium wall conditioning are transient, only one or two shots, so lithium containing silicon coatings, which combine the superiorities of relatively long lifetime of siliconization and high performance of lithium coatings in situ, should be developed.

2. Experimental setup

HT-7 is a medium sized iron core superconducting tokamak with a limiter configuration. The last closed flux surface is defined by the main carbon limiter with a complete circular ring along the poloidal direction. The main parameters of HT-7 device are: major radius R = 1.22 mm, minor radius a = 0.27 m, $B_{\rm T} = 1-2.5$ T, $I_{\rm p} = 100-250$ kA, line averaged density $n_{\rm e} = (1-8) \times 10^{19}$ m^{-3} , $T_e = 0.6-3.2$ keV, $T_i = 0.3-1.8$ keV, discharge duration $\tau_d = 10-60$ s, deuterium as working gas. A stainless steel liner is installed in the vacuum chamber with radius of 0.32 m. The normal operation base vacuum is $(0.4-2) \times 10^{-5}$ Pa. The lower hybrid current driven (LHCD) grill protectors are made of molybdenum. The ICRF system can be operated under pulsed mode or CW mode. Three kinds of RF antenna configurations are tested; a1/2 turn long antenna at high field is the best for wall conditioning and used in this experiment [16].

A new wall conditioning system has been installed in HT-7 superconducting tokamak as showing in Fig. 1. Lithium was introduced by better-controlled evaporation method, a special cover with a nozzle was installed on the oven which can spray lithium vapor very uniformly along the toroidal direction. Before the experiment, a few blocks of solid lithium (99.9% purity), typically 1-2 g, were put into a small stainless steel oven $(\sim 4 \text{ cm}^3)$ under argon gas flow to prevent the lithium surface oxidation. The oven can be sent into the HT-7 vacuum vessel by a magnetic transport system, located 5 cm above the bottom of the inner vessel. The oven can be heated to 400-500 °C in vacuum, either with dc resistive heating of a tungsten spiral wire or with RF inductive heating (13.56 MHz, 100-200 W). The temperature of the oven was measured and monitored by

Fig. 1. The arrangement of the ICRF lithium containing silicon in situ coatings system.

Main poloidal carbon limiter

thermocouples, and the evaporation rate can be better controlled using two temperature controlled sublimators. A Stanford quadruple mass spectroscope (RGA200) was used to measure the component of residual gas in the vacuum chamber before and after wall conditioning. Many small samples with different substrates (stainless steel, graphite) were placed axially along the side wall and the radially on the top plate of the inner vessel. After wall conditioning, the samples were taken out, immediately put into an argon-purged container and send to the USTC for analyses, lithium was quantitatively profiled by AES combined with Ar ion beam etching. H, D, Li, C, O, Si, and metals were also profiled by XPS, SIMS.

Prior to ICRF lithium containing silicon coatings, a half hour of careful ICRF discharge cleaning with helium was applied in order to further enhance the performance, which to a great extent removed background oxygen impurities such as metal oxides and oxygen containing gases (H₂O, O₂, CO, etc.), depleted the plasma interactive walls of previously implanted fuel particle and reduced the hydrogen retention in graphite. The whole process of lithium containing silicon coatings is similar to ICRF siliconization in HT-7 [15], during the process of siliconization, the lithium vapor was inducted together. Silane (SiH₄) is diluted by more than 90% with Helium (He) and the total pressure is controlled at $(2-8) \times 10^{-2}$ Pa. The toroidal magnetic field is about 1.5-2.2 T, the ICRF power is 15-20 kW with 1 s on and 1 s off. After careful discharge cleaning, the lithium vapor and silane gas mixed with helium was induced into the HT-7 tokamak under the RF plasma in order to carry out the wall conditioning. The injected gas atom and molecules are decomposed and disassociated into Li^+ , Si^+ and SiH_x^+ (x = 1–3) and carbon ions, which get energy from the ICRF plasma and are co-deposited together on the first wall of the device to form the



RF antenna

Li vapor

2# pumping duct

500¥s

Х П

SiH4

 $\begin{array}{c} X \\ D_2 \end{array}$ He

amorphous lithium containing silicon coatings (α -C:H/ Li–Si). The whole proceedings lasted for 1–2 h. To improve the deposition of silicon film of high quality, the liner temperature was kept at 150 °C.

ICRF plasma parameters were measured by different diagnostics. The hydrogen ion temperature is 1-2 keV with a high-energy tail up to several tens of keV. The ion temperature is a very important parameter because it governs the energy of the silane, lithium and carbon ions that impact on the wall, and which also plays an important role in obtaining a hard and high quality film. Plasma density is in range of $0.5-3 \times 10^{17}$ m⁻³. The electron temperature is in the range 3-8 eV within an injection power of 10-20 kW.

3. Results and discussion

Before and after wall conditioning, residual gas analysis (RGA) was carried out and the results are shown as Fig. 2. After the ICRF lithium containing silicon coating, the water peak of the vacuum chamber was reduced by 50%, CH₄ was reduced by 20%, CO was reduced by 150% and CO₂ was reduced by more than six times. It could be easily found from the results that lithium containing silicon coatings also have a very strong ability to getter residual gases and react with most residual gases (H₂O, O₂, CO and CH₄) in fusion devices through chemical reactions. The impurity level is obviously lower than siliconization [15] and even approaches the level of lithium in situ coatings.

In order to obtain information on the state of lithium co-deposited with siliconization on graphite and stainless steel after lithium containing silicon coating, small samples were transferred by argon protection to the structure analyzing center of USTC for analyzing, the depth profile was measured and some differences between the substrate of SS and isotropic graphite can be seen in Fig. 3(a) and (b). There is about 60 nm lithium



Fig. 2. QMS results before and after ICRF lithium containing silicon in situ coatings.



Fig. 3. (a) The depth profile of lithium containing silicon in situ coatings on SS by AES, (b) the depth profile of lithium containing silicon in situ coatings on IG by AES.

mixed silicon and carbon coatings on the SS substrate, while in the same case of graphite, which also exhibits about 50 nm lithium mixed silicon and carbon coatings on the IG substrate. From the results of Fig. 3(a), even in the substrate of SS, there is low lithium content due to high mobility of lithium in metals; but the high lithium content in carbon substrate can be seen from Fig. 3(b) due to high porosity of graphite and can also form graphite intercalation compounds with lithium [17]. The carbon of mixed coatings come from the residual gases and sputtering of carbon wall materials during ICRF wall conditioning.

The better plasma performance discharges were obtained after ICRF lithium containing silicon coating in HT-7 device. Loop voltage dropped by 25–40%, Z_{eff} was reduced nearly 30%, the recycling and the total radiation loss dropped by 25% comparing with siliconization. CIII and OVI were 30% lower than those with siliconization as can be seen in Fig. 4. The decreases of the plasma temperature and the electron density in the edge showed that the confinement of the plasma has been improved compared with siliconization.

It is important to investigate the effects of the lithium containing silicon coatings on hydrogen recycling and



Fig. 4. Comparison of CIII and OVI line radiation intensity vs the line averaged density with similar discharge parameters after siliconization and Li and Si in situ coatings.



Fig. 5. Li I line intensity as a function of discharge numbers after Li and Si in situ coatings.

the behavior of intrinsic impurity. To analyze these effects that is necessary to measure shot-to-shot variations of lithium, electron density and impurity. Neutral lithium emission at 670.8 nm is measured with an interference filter tuned to Li I light and used to study the behavior of recycled lithium after wall conditioning. Li I line intensity as a function of discharge numbers after Li and Si in situ coatings shown as in Fig. 5, and it can easily be seen from these mixed coatings that the service time is sustained for more than 350 shots as is certified also with other diagnostics and experimental results.

4. Summary

Lithium containing silicon coatings (α -C:H/Li–Si) developed by ICRF, a new wall conditioning method, on the first wall of the HT-7 superconducting tokamak

has been made a try. After careful ICRF wall cleaning, sufficient lithium was introduced using better-controlled vaporization method together with siliconization. The impurity level is reduced significantly and better plasma performance was obtained after ICRF lithium containing silicon coatings. This new conditioning method has high performance and long lifetime. The mechanism of recycling reduction and C and O impurity suppression, and energy confinement improvement is still under further investigation.

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