

Journal of Nuclear Materials 248 (1997) 78-84



Section 4. Impurity production and transport in plasma Advanced research of plasma wall interaction in China

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Abstract

Impurity control, edge plasma physics, acceptable recycling and proper plasma facing materials (PFMs) with long enough lifetime are the major concerns in plasma wall interaction (PWI) investigations in China. The wall conditioning using Ti getter, carbonization and boronization techniques have been conducted. Pump limiter and bias limiter were tested in HL-1 tokamak for impurity control. Low and middle-Z coatings have been developed and tested but the bonding between coating and matrix was not satisfactory enough. Graphite, doped graphite and C/C composite are now the main PFMs for current tokamaks in China. A significant modification of chemical sputtering (CS) resistance has been achieved using doped graphite. © 1997 Elsevier Science B.V.

1. Introduction

Plasma wall interaction (PWI) is an important problem in fusion plasma experimental devices and fusion reactors. The feasibility of tokamak operation depends strongly on the control of impurity generation and transport in edge plasma. The impurities, especially heavy impurities, could result in significant radiative power loss and even disruption. The hydrogen recycling is allowable and utilizable in many cases but too high recycling would bring difficulty on plasma density control in the tokamak machine. The recycling relates not only to the property of plasma facing materials (PFMs) but also to the condition in scrape-off layer (SOL) plasma. On the other hand, erosion of PFMs by sputtering and thermal shock during disruption, restrict their application condition and lifetime. So PWI would result in two things: plasma quality deterioration and PFM degradation, which are of concern in fusion research.

The experimental research of PWI has been conducted using both tokamak machines and specially designed devices in China. HL-1 and its modification machine, HL-1M, are the main tokamaks for PWI research. Some small experimental devices for chemical sputtering (CS) and radiation enhanced sublimation (RES), ion driven permeation and thermal shock investigation have also been built up recently. A systematic research on PWI has been progressing in China.

2. The control of the impurity generated from the wall and hydrogen recycling

2.1. The control of the impurity generated from the wall

The vacuum vessels of HL-1 and HL-1M are made of stainless steel. There is not any low-Z (atomic number) PFM in HL-1 while the graphite tiles only cover 15% of the inner surface of HL-1M. So wall conditioning seems necessary for reducing the impurity generation on the wall. A metal film gettering technique was adopted in the initial stage of HL-1 operation, where the getter was titanium. The evaporated Ti film on the inner surface of the vessel acts as a pump with high conductance of the pumping area to the volume. The sticking coefficients for O₂, N₂ and D₂ on evaporated Ti have been measured [1]. The measured sticking coefficients for O2 and N2 rapidly dropped when one monolayer of the gas forms on the film surface while the coefficient for D_2 stays at a high level even if more than one equivalent monolayer has been formed. The reason is the diffusion of hydrogenic species into the bulk

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of Ti and their high solubility in Ti. It is believed that the hydrides (TiH_2) are formed, and meanwhile the hydrogen concentration may reach up to $10^{29}/m^3$ yielding a capacity of about 1 MPa/cm³ for hydrogen storage. The evaporated Ti film covered $\sim 30\%$ of the HL-1 wall surface with ~ 18 monolayers of Ti, which reduced the light impurities and led to a great improvement in the plasma operation parameters: the loop voltage was decreased by a factor of 2 and the plasma current was increased by 20%; intensities of O III, O IV and O V were reduced by factors of 2-5 and the total radiation losses were decreased by a factor of 3 [2]. The Z_{eff} in most discharges reduced from 4 to 2. But Ti-getter was not effective to heavy impurities and Ti itself may also contaminate the plasma.

Carbonization which can effectively suppress heavy impurities was performed in HL-1 tokamak some time later [3]. The in situ carbonization is really a good idea for present fusion experimental devices where the metal is still facing the plasma directly. The research was started at TEXTOR [4] and it was then adopted at many other tokamaks in the world. Alternative current (ac) discharge in mixed gas $(CH_4 + H_2)$ was used for carbonization in HL-1 vacuum vessel. The advantage of the ac discharge in comparison with the glow discharge which is usually adopted in many tokamaks is that the impurities in the carbon layer formed by the former is lower. The reason is that the ion energy in ac discharge is lower than that in glow discharge and so there are no sputtering products arising from the metal surface in the former. But the carbon deposition rate is a little lower in that case. The thickness of the carbon layer deposited on the vessel surface was about 30 nm. The metal impurity level in the plasma after carbonization was decreased by 80% while the energy confinement time of the plasma was increased by 20%. The carbonization seems effective for reducing the heavy impurities but oxygen and carbon impurities arose again.

The boronization was performed in HL-1M in 1995, meanwhile carborane $(C_2B_{10}H_{12})$ [5] with very low toxicity, rather than B_2H_6 , was used in the boronization [6]. The boronization was also firstly carried out using B_2H_6 at TEXTOR in 1989 and a series of good results were achieved due to the boronization [7], but, unfortunately, B_2H_6 is very poisonous. The boronization technique with $C_2B_{10}H_{12}$ is developed by Russian scientists and has been successfully used in T-3M and T-11M tokamaks.

The mass spectra of three main impurity species were taken before and after the boronization where the fractions of CH₄, H₂O and CO were decreased by factors of 60-75%, 67-97% and 84-96%, respectively. The reduction of CH_4 means that the a-B/C:H film possesses a very strong CS resistance. On the other hand, boron has a very high chemical affinity to oxygen and so solid B₂O₃ would be formed with chemical affinity as high as -1193kJ/mol. The a-B/C:H film not only reduces the oxygen release from the surface but also blocks H₂O desorbtion 2375: without boronization, shot 2465: with boronization).

from the bulk graphite. So the H₂O, CO and other oxygen containing impurities in plasma are sharply decreased.

The boronization of the vacuum vessel of HL-1M improved the vacuum condition in it. Before boronization, the discharge cleaning was necessary to be made every night to keep the base vacuum of $(1-2) \times 10^{-4}$ Pa, while, after boronization, the base vacuum of $(5-6) \times 10^{-5}$ Pa can be easily kept for a long time even though the discharge cleaning has been canceled. Similarly, the HL-1M can directly start the ohm discharge without the discharge cleaning in case of the boronization, whilst the discharge cleaning for, at least, more than 15 h was usually necessary before start of the ohm discharge in case of no boronization being performed in the vessel.

The plasma quality and parameters were obviously enhanced after the boronization. The radiation losses due to heavy impurities during ohm discharge before and after boronization are given in Fig. 1, where the total radiation losses were decreased by factors of 1-2. Besides, the loop voltage was dropped from 1.5-2 V to 1 V, while the energy confinement time was increased by 35-40% since the light impurities were significantly suppressed and heavy impurities almost disappeared after boronization. The boronization improved the hydrogen recycling condition, and the plasma density became easily controllable. The central electron temperature increased by 35-40% and the plasma density peak increased by 50%, both due to the boronization. The density of edge plasma decreased and its temperature enhanced while the parallel flow Mach number arose by 40-60% with the marked reduction of edge turbulence after boronization. Both stability and repeatability of the ohm discharge were improved.

When low hybrid current drive (LHCD) power increased over 100 kW before boronization, the plasma current used to be often quenched because of the impurity flood due to PWI. But after boronization, the LHCD power was enhanced up to 400 kW and plasma current quench has never been observed up till now.

Fig. 1. Time evolution of the radiated power from plasma (shot



2.2. The hydrogen recycling

The hydrogen recycling exists in almost every tokamak. Of course, not only hydrogen but also impurities join the recycling due to the PWI. The gas puffing was the main fueling method in the initial stage of HL-1 operation. The measured electron density n_e dropped immediately when the hydrogen gas puffing was switched off, and the particle density decay time τ_p^* was only 170 ms [8]. When He-puffing discharge was used, however, the electron density still rose for ~ 60 ms after He-puffing switched off, and then dropped slowly. The corresponding $\tau_{\rm p}^*$ was as long as 510 ms. It is suggested that the helium not only is ionized, contributing electrons directly, but also induces the desorption of hydrogen on the wall, results in strong wall fueling. So the amount of helium feed was less than that of H₂-puffing by a factor of 5 for the same n_e in both cases. After carbonization the averaged electron line density \bar{n}_{e} and τ_{p}^{*} increased shot by shot from shot number 9184 to 9187 (\bar{n}_{e} : from 3.2 to 4.8 × 10¹⁹/m²; τ_{p}^{*} : from 170 to 420 ms), that means the wall fueling is also strong and extends the current rise phase into the flat top phase. Many discharges in HL-1 show that the hydrogen recycling coefficient R and particle confinement time $\tau_{\rm p}$ increase by 20% comparing with that before carbonization [3]. The boronization has been performed in HL-1M and the R decreases markedly, and plasma density control becomes easier than that before boronization.

3. Impurity influx control in SOL

A major concern for getting a reactor fusion plasma condition is the control of impurity influx to the central plasma. The impurity level in the plasma center is determined by the impurity production at edge plasma due to the PWI, and by the transport of the impurities in both edge and central plasma. To pump out the particles in SOL is a good idea for reducing edge recycling and impurity influx to the central plasma region. HL-1 was equipped with a movable pump limiter since 1990. It is located at the outer side of the torus midplane and can be moved radially from $r_{\rm pl} = 0.23$ to $r_{\rm pl} = 0.18$ m, where $r_{\rm pl}$ is the pump limiter radial, which is just the position of limiter head from the plasma center. The operation of pump limiter showed that the recycling coefficient *R* was decreased by 20% and particle confinement time enhanced to $\tau_{\rm p} = 60-90$ ms at electron density $n_{\rm e} = (2-4) \times 10^{19}/{\rm m}^3$, and if the pump limiter moved away, the $\tau_{\rm p}$ was only 30–70 ms at the same $n_{\rm e}$ (see Fig. 2) [9].

On the other hand, the radial electric field would be beneficial to improvement of plasma parameters. The biased pump limiters were adopted in many tokamaks and the significant enhancement of plasma quality was observed [10–12]. The biased pump limiter experiments were performed at HL-1 tokamak [13]. Both positive and negative biases were tested in the experiments and, generally speaking, the effect of positive bias on the plasma parameters is more significant in comparison with the negative one. In the former case (+200 V bias), the averaged central line density and energy confinement time $\tau_{\rm E}$ were increased by 50% and 20-40%, respectively, the particle confinement time τ_{p} , estimated from particle flux signals measured with Langmuir probes, was increased by 50-100%, and the particle flux signal from the probe located near the edge plasma was decreased by 50%. The electrical field profile in SOL can be affected by limiter biases and a radial potential barrier (or $\mathbf{E} \times \mathbf{B}_{T}$ field) may be formed so that the edge plasma flow to the wall is impeded. So the particle flux is decreased, then the $n_{\rm e}, \tau_{\rm p}$ and $\tau_{\rm E}$ are increased accordingly. The O VI signal from edge plasma was increased by 60% and meanwhile the loop voltage was increased by 20%. The oxygen impurity increased when the limiter bias turned on. The oxygen may come from the wall and somewhat relates to the bombardment of runaway electrons. The C III and Cr I emission lines near the neutralizer plate of the pump limiter obviously increased for both positive and negative limiter biases. C and



Fig. 2. τ_p and R vs. n_e at HL-1 tokamak with and without pump limiter.

Cr impurities may come from the sputtering of ions or electrons accelerated by radial electric field near the limiter.

4. Erosion and redeposition of coatings in the edge plasma

Many impurities come from high heat flux components (HHFCs), such as limiter, divertor plate and neutral beam dump plate, which are directly bombarded by energetic charged and neutral particles. The carbonization and boronization are of no use for preventing the impurity production on their surfaces. Low or middle-Z coatings, e.g., B_4C , TiC and SiC, are one of the choices for HHFCs. The following problems are their plasma erosion resistance and high heat flux thermal shock resistance. An experimental investigation on erosion of TiC and SiC coatings in edge plasma has been conducted in HL-1 tokamak [14].

TiC-coated graphite specimens were put into the HL-1 edge plasma, where electron density was $4.1 \times 10^{17} / m^3$. The fluence of $1.9 \times 10^{23}/m^2$ was reached on the TiC coating surface which faced to the plasma center. It is shown that the most TiC coating has exfoliated after plasma irradiation and the sputtering and chemical erosion seem very serious in these area due to the high oxygen content, which decreases the bond force between coating and substrate and accelerates the chemical erosion of the coating. Crystal parameters of graphite and TiC coating were measured by means of XDS before and after plasma irradiation, respectively. The crystal structure of TiC was still regular hexahedron and the distance between crystal surfaces (d) slightly changed after plasma irradiation. A TiC coated specimen was put into a position whose radius (in minor radius direction) was r = 0.198 m, which was just inside the poloidal limiter. The TiC coating was exfoliated entirely at this position but many titanium atoms were found in the depth of 1 µm beneath the graphite surface (see Fig. 3). It is indicated that the preferential sputtering of C and recoil implantation of Ti occurred in TiC coating in this case. TiC coating would be beneficial to heavy impurities control in fusion experimental devices. but TiC coating with thickness of $\sim 10 \ \mu m$ seems too thin for limiter application.

SiC coated graphite specimen was put into SOL plasma to be irradiated up to a fluence of $2 \times 10^{24}/m^2$. It was found that a thick redeposited layer covered the specimen surface after its exposure in SOL plasma. The XPS analysis of irradiated and unirradiated SiC coating surfaces was conducted respectively. In a sharp contrast with the peaks before the exposure in SOL plasma, the peaks of oxygen and carbon grew up and Si peak became lower obviously after exposure and molybdenum peak appeared. It meant that the redeposition was the dominant process on the surface in SOL plasma. The redeposition depth profile on SiC coated graphite was measured and it was found that a



Fig. 3. AES depth analysis results of TiC coated graphite before and after plasma irradiation on the surface of toroidal limiter. where 1 - C (272 eV), 2 - Ti (386 eV), 3 - Ti(417 eV), 4 - O(510 eV). (a) TiC coated graphite before plasma irradiation. (b) graphite substrate where the TiC coating has exfoliated entirely after plasma irradiation.

lot of carbon, oxygen, and metals existed on the surface. The carbon atoms came from movable limiter which was made of graphite while molybdenum came from the fixed limiter made of Mo in the tokamak. The XPS analysis was made on SiC coating surface before and after plasma irradiation. It can be seen that the structure of SiC coating has not any obvious change after plasma irradiation.

The thermal shock is another concern for their application on HHFC surface. The chemical vapor deposition (CVD) TiC coatings on graphite and Mo, respectively, have also been tested using Nd:YAG pulsed laser beam [15,16]. The pulsed energy density of the laser was up to 2 MJ/m² and the duration of the pulse was 0.1–1 ms. It was found that the damage thresholds of TiC-coated graphite and Mo were 20 and 25 MW s^{1/2} m⁻², respectively. Some microcracks and exfoliation were found on the coatings after laser beam impingement.

The coatings can endure higher heat flux and thermal shock in some condition but the bond force between coating and substrate is not satisfactory and so the exfoliation arise very often. In this point of view, the bulk materials, e.g., high quality graphite and C/C composite, would be better.

5. Modification of chemical sputtering for carbon base PFMs

It is well known that the graphite and other carbon base materials have been used as low-Z PFMs in fusion plasma experimental devices for a long time and they were very attractive because of their good thermo-physical properties. Unfortunately, graphite is heavily eroded by hydrogen ions due to CS in the temperature range of 700-1000 K and the RES at the temperature above 1100 K, which is easily exceeded in present large tokamaks and reactor designs. A great number of carbon atoms entering the plasma by means of CS and RES causes the carbon flood in the plasma and so restricts the increment of plasma energy. Some B-, Ti- and Si-doped graphites were developed for suppressing CS and RES at elevated temperature and they were, in fact, a kind of ceramic (e.g., B_4C , TiC and SiC) doped/mixed graphite [17-21]. The experimental results show that the CS is suppressed and some reduction of RES is also observed at many laboratories.

Thermo-physical parameters of two and three dopant mixed graphite recently developed in China are given in Table 1, where G-5 is the pure graphite for comparison [22]. GBS-105, GBT-864 and GBTS-941 have been selected as specimen material for CS experiments and they were cut into small pieces, which were numbered $2^{\#}$, $3^{\#}$ and 4[#] respectively, while pure graphite (specimen number 1[#]) was also selected for comparison. CS product was methane (CD_4) under both 1 keV and 50 eV bombard energy and the signal of CD4 was collected by quadrupole mass spectrometer (QMS). The QMS signal vs. specimen temperature under 1 keV and 50 eV D⁺ bombardment for specimen 1[#], 2[#], 3[#] and 4[#] are summarized in Fig. 4. When 1 keV D⁺ was incident on the graphite and doped graphite specimens, the CS peaks of three kinds of doped graphites were similar while their peak intensities were same, just equal to 1/5 of CS peak for pure graphite. But their location shifted towards the lower temperature and

 Table 1

 Thermophysical parameters of graphite and two or three dopants-mixed graphites



Fig. 4. Chemical sputtering of graphite and three kinds of B-, Siand Ti-doped graphites under the bombardment of D^+ with energy of (a) 1 keV and (b) 50 eV.

the shifts between that of graphite and doped graphite were about 110–130 K. So the CS has been suppressed effectively in this case. When the energy of incident D^+ beam was decreased to 50 eV, the CS peaks were broadened for both graphite and doped graphites and the peaks of doped graphites decreased by 23, 30 and 19% for specimens $2^{\#}$,

Material type	Specimen number for chemical sputtering	Content of dopants (wt%)			Density	Porosity	Bend	Thermal	Thermal
		B ₄ C	Ti	Si	(g/cm ³)	(%)	strength (MPa)	expansion coefficient $(10^{-6}/K)$	(W/m K)
G-5	l [#]	0	0	0	1.83	< 10	34.2		72.0
GBS-105	2#	10	0	5	2.10	4.70	102.9	3.06	39.6
GBTS-864	3#	8	6	4	2.17	4.93	123.3	2.96	59.40
GBTS-941	4#	9	4.8	1.6	2.12	5.33			68.40

The better CS resistance of doped graphite is related to its microstructure. Three types of highly pure graphite and one doped graphite were analyzed before and after hydrogen ion irradiation using Raman spectrometer (RS). Three pure graphite tested in the experiment were ISO880U (made in Japan), G-5 and T901 (made in China) while doped graphite was GBS-105. The behaviors of ISO880U, T901 and G-5 were similar to each other before and after H⁺ bombardment. There were two peaks in Raman spectrum: peak G (1.580 cm⁻¹) corresponds to the sp² hybrid bond of carbon atoms in graphite crystal and the peak D (1.355 cm^{-1}) corresponds to the sp³ hybrid bond in the graphite. The peak D is also related to the defect in the graphite, such that the ratio of I_D/I_G can indicate the completeness of the graphite crystal, where $I_{\rm D}$ and $I_{\rm G}$ are intensity of the peak D and G respectively. The ratio of I_D/I_G deduced from the Raman spectrum of G-5 and GBS-105 before and after H⁺ irradiation vs. irradiation dose was obtained. The I_D/I_G ratios of G-5 and GBS-105 before irradiation were ~ 0.3 and ~ 0.6 respectively and it means that more defects existed in GBS-105 due to adding of dopants in comparison with that of G-5. But after irradiation, $I_{\rm D}/I_{\rm G}$ of G-5 increased sharply and it became larger with the increase of the irradiation dose, up to ~1 at last, but I_D/I_G of GBS-105 only increased slightly with the increase of the irradiation dose. So it is obvious that more defects would arise in the pure graphite after H⁺ irradiation while only less defects due to H⁺ irradiation can be observed in doped graphite. These results are consistent with that obtained at other laboratories [24]. The doped graphite seems more stable under hydrogen ion beam irradiation.

6. The thermal shock resistance of carbon/carbon composite

Plasma disruptions, which are observed in almost all tokamaks, are among the most severest events in fusion devices. The plasma disruption will make PFMs to experience intense thermal shock during both plasma quench and current quench phases. The time scale for two phases are 1-3 ms and 10-20 ms respectively. A high heat flux will deposit on the surface of PFM in the plasma quench phase, while not only energy deposition but also a huge electromagnetic force would be induced in first wall and PFMs in current quench phase. The energy deposition during plasma disruption is estimated as high as 1-10 MJ/m² for near term large tokamaks and it would be higher for fusion

reactors because the energy deposition is highly localized on a small random area of PFM surface. The plasma disruption has been considered as an important factor limiting the lifetime of PFMs. Carbon/carbon (C/C) composite possesses higher strength and good thermophysical properties and so is a good PFM for tokamaks. Thermal shock experimental research has been performed in China using three kinds of laser beams: ruby laser, Nd:YAG laser and Nd-glass laser [16,25,26]. The laser pulse duration was 0.1–1.0 ms and the energy density deposited on the specimen surface is up to 13 MJ/m². The weight losses of four types of three-dimensional C/C composites were measured. The current C/C composites do not seem good enough but new better C/C composites have been developed recently.

7. Beryllium as PFM

Beryllium was introduced into JET first in the form of an evaporated layer on the whole inner surface of the vacuum vessel, which was largely covered with graphite. Be belt limiters and antenna screens were added later and were used together with Be evaporation [27,28]. Now Be has been considered as the main PFM for ITER. Be is a good PFM but it has not been adopted for current tokamaks in China because of its drastic toxicity. A research of Be as a PFM has been started with international cooperation. Some Be specimens made in China were put into Petten reactor for irradiation and now the irradiation has been finished. The next step of the research is thermal shock tests in an intense electron beam facility using these irradiated Be specimens. Now the thermal shock tests are under way.

8. Conclusions

Impurity control, edge plasma physics, acceptable recycling and proper PFM with long enough lifetime are the major concerns on PWI investigation in China. The wall conditioning using Ti getter, carbonization and boronization techniques have been performed and good results have been obtained. Pump limiter and bias limiter have been adopted in HL-1 tokamak and experimental results indicated that they were useful for impurity control. Low and middle-Z coatings have been developed for HHFC but the bonding between coating and matrix is not satisfactory enough. Graphite, doped graphite and C/C composite are now the main PFM for current tokamaks in China. Chemical sputtering of doped graphite has successfully been suppressed. Be is a good PFM and the experimental research of Be is conducted smoothly with international cooperation. Unfortunately, Be has not been used in current tokamaks in China because of its drastic toxicity.

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