CNIC-01531
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TOKAMAK 第一壁等离子体表面工程

PLASMA SURFACE ENGINEERING IN FIRST WALL OF TOKAMAK

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摘　要

介绍了 HL-1M 内壁的硼化、硅化和锂涂层处理，氢的再循环和壁处理对杂质控制、中心辐射能量损失的影响。实验结果表明，壁处理对等离子体约束是非常有益的，对硅化壁，获得了 4 s 的长脉冲放电，且具有很好的重复性；而对于硼化壁，仅获得 2.1 s 的长脉冲放电，且重复性很差；锂涂层对于降低氢的再循环和减少杂质水平表现出更大的优越性。另一方面，作为 HL-2A 和未来聚变堆的应用，开发了一系列的硼、钛、硅掺杂石墨和碳化硼掺杂的 C/C 复合材料，该文也对化学溅射，氚滞留和再循环，高热负荷的一些实验结果进行了评述，同时还介绍了 SiC，TiC 和 B_4C 涂层，以及 B_4C-C，SiC-C，B_4C-Cu，Mo-Cu 和 W-Cu 梯度功能材料。

Plasma Surface Engineering in First Wall of Tokamak

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ABSTRUCT

The boronization, siliconization and lithium coating of the inner wall of HL-1M are introduced, the hydrogen recycling and the influence to impurities controlled and core radiation energy loss are discussed. Experiments prove that these wall treatments are very useful for the plasma confinement, a 4 s reproducible long pulse discharge is obtained for siliconized wall, but the plasma pulse length only achieves 2.1 s and its reproducibility is very poor for boronized wall. Lithium coating is the best method of the wall treatments for lowering hydrogen recycling and decreasing the impurities level. For the applications of HL-2A and the future fusion device, a series of B, Ti, Si-doped graphite and B_4C-C/C composites have been developed, some experimental results about chemical sputtering，tritium retention and recycling, as well as high heat loads are reviewed. Meanwhile, SiC, TiC and B_4C coating, and B_4C-C, SiC-C, B_4C-Cu, Mo-Cu and W-Cu functionally graded materials are also introduced.

INTRODUCTION

Tokamak is the major controlled nuclear fusion device; the design and construction of the first wall of Tokamak is one of the most important issues. The inner wall of Tokamak machine consists of plasma facing materials, such as graphite, C/C composite and beryllium, and structural materials, such as 316 stainless steel and ferrite steel. Up to now, graphite was mostly widely used as plasma facing material. For the special parts of the first wall, such as divertor and limiter, high heat flux components must be adopted because much higher thermal load will occur at these positions, therefore, high strength, high melting point materials are always preferential. In the research of high heat flux components, SiC, TiC, B_4C coatings and W-Cu, Mo-Cu mixtures are paid more attentions.

In HL-1M tokamak, the first wall is 316 ss, only 6% area covered by graphite blocks. During glow discharge, a lot of carbon impurity and heavy metal impurities will be released into the plasma, which will induce the loss of plasma energy and influence the properties of plasma confinement. Therefore, some wall treatments are necessary. In this paper, the boronization, siliconization and lithium coating of the inner wall of HL-1M are introduced and their influences on tokamak plasma are discussed.

Mentioned above, boronization, siliconization and lithium coating of the inner wall can effectively improve the properties of plasma confinement, the modification of carbon materials was also carried out. Recent several years, B, Ti, Si-doped graphite and B_4C-C/C composites have been developed. Experimental results show that they are great superior to high purity graphite on restraining chemical sputtering, decreasing tritium retention and recycling, and resisting thermal shock. In the present paper, the properties of B, Ti, Si-doped graphite will be reviewed; some experimental results both in modified facility and HL-1M tokamak machine can be obtained. Meanwhile, some high heat flux components materials, such as SiC, TiC coatings and B_4C-C, B_4C-Cu, Mo-Cu and W-Cu functionally graded materials (FGM) are also discussed.

1 THE WALL COATING OF HL-1M

1. 1 Boronization

The inner wall of vacuum chamber of HL-1M consists of stainless steel with

only 6% area covered by graphite blocks. When HL-1M operates with high parameters, B_T =3 T, I_p=300 kA and 2 MW high power auxiliary heating, impurity control and a low hydrogen recycling of the inner wall are needed. One way to resolve the problem is wall treating by boronization, siliconization and lithium coating [1～3].

The boronization is carried out in a He glow discharge with $C_2B_{10}H_{12}$ addition, operation conditions is as follows [4]:

DC current is 1.0～1.5 A with a single electrode;

DC voltage is 800～1000 V;

Pressure is 10^{-3}～10^{-4} Torr (1 Torr = 133.3224 Pa);

Ratio of $C_2B_{10}H_{12}$ vapor to He pressure is 0.1∶1.0;

Temperature of vacuum vessels of the boronization system is 80～100 °C;

Deposition time is about 1 h.

After the boronization of the inner wall, a-B/C:H film was formed on the wall surface, the thickness of the coating is 50～70 nm and the ratio of boron to carbon is 1∶6.

1. 2 Siliconization [4, 5]

The process is similar to boronization and uses DC glow discharge with a mixture of (5%～10%)SiH_4+(95%～90%)He. The thickness of a-C/Si:H film is about 100 nm, major components in the coating are Si, C and SiC by means of X-ray Photoelectron Spectroscopy (XPS) analysis.

1. 3 Lithium coating [4, 5]

Lithium is deposited on the inner wall of HL-1M by plasma-assisted deposition, a few blocks of solid lithium weighted at 2 g are put in an oven under Ar gas flow to prevent the lithium from oxidation and the oven can be moved radially inwards and outwards. Solid lithium blocks was heated up to 600 °C, evaporated into a He glow discharge where it is ionized and finally deposited on the wall the as the discharge cathode. The discharge pressure (p_{H_2}+p_{li}) is 35×10^{-2} Pa and the current density is 10～12 μA/cm^2 with 400～500 V anode voltage. The deposition runs for about 10 min at room temperature, about 1 g lithium is deposited on the wall, the average film thickness estimated is about 10 nm.

1. 4 Hydrogen retention and recycling

Fig. 1 shows neutral gas pressure changing with time in scrape-off layer after different wall conditioning during the Tokamak discharge, it shows hydrogen recycling performance in the edge plasma. From Fig. 1, it can be seen that hydrogen

recycling of boronized wall is obviously higher than that of siliconized wall and lithium coating, and hydrogen recycling of siliconized wall is a little higher than that of lithium coating wall. When the additional hydrogen gas was puffed in, the phenomenon above can be observed more clearly.

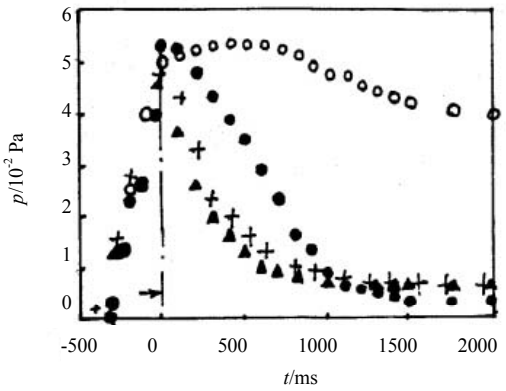

Fig. 1 Neutral gas pressure changing with time in SOL layer after different wall surface treatment

t=0 discharge is beginning. (O) No discharge, (●) Boronization, (+) Siliconization, (▲) Lithium coating.

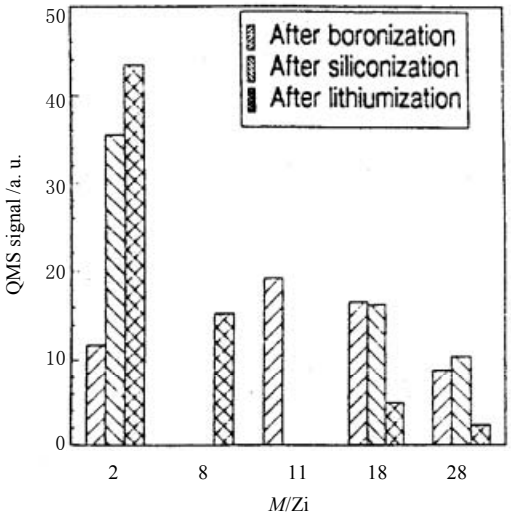

Fig. 2 QMS signal in main chamber during pulse blow gas after different wall surface treatment

In order to test the absorption ability of the wall surface to hydrogen by different wall treating without discharge, equal amounts of hydrogen gas (p_{H_2}=5.3×10^{-2} Pa, Δt=30 ms) were puffed into the vacuum chamber just after every one wall treating, the gas compositions in vacuum chamber were measured by QMS (shown in Fig. 2). As can be seen from Fig. 2, a-C/Si:H film, lithium coating in particular, is inert to hydrogen molecules, but B/C film has a strong absorption capacity, which is regarded as physical absorption. For siliconized and lithiumized wall, there is no chemical reaction occurred between Si, Li and H_2 at room temperature. That is why hydrogen recycling during a Tokamak discharge from boronized wall is higher than that from siliconized or lithiumized wall. The another reason for the lower hydrogen recycling by siliconization and lithium coating treating is the strong chemical affinity of silicon and lithium for hydrogen ions or

atoms. In fact, during a Tokamak discharge, the strong pumped effect to hydrogen has been observed for siliconiztion and lithium coating wall treating [4, 5].

1. 5 The influence on Tokamak discharge by different wall treating

Because the boronization materials contain some quanlities of carbon, when Tokamak discharge occurred, the boron atoms in B/C film are preferential sputtering, unable wall conditions appear after several boronization cycles. As a result, the wall outgassing and impurity intensity are sharply increasing, leading to disruptive shots when pellets are injected in the pellets fueling experiment or in the LHCD experiment. In the long pulse discharge experiments, only 2.1 s plasma pulse length was achieved and its reproducibility is very poor. But the wall conditions stay stable over a long time and the wall gassing and impurity are controlled effectively by siliconization. In the pellets fueling experiments, almost no disruption shots occur. The driving power in the LHCD experiments rises up to 850 kW and the reproducible long pulse discharges with 4 s plasma pulse length are obtained (as seen in Fig. 3).

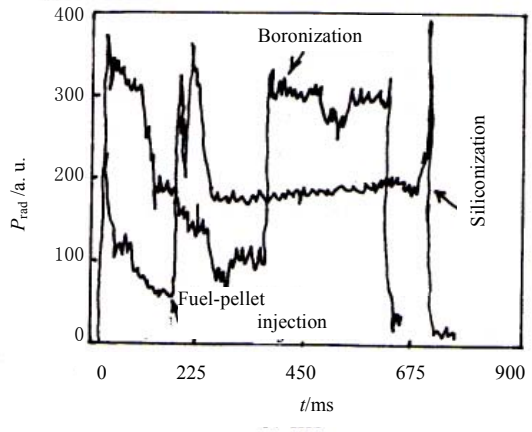

Fig.3 Plasma core heat radiation loss after fuel-pellet injection in different wall conditions

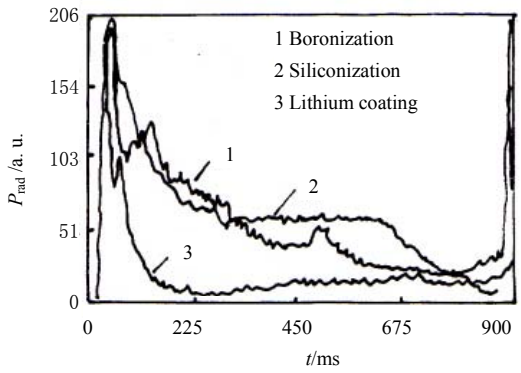

Fig.4 Plasma core heat radiation loss in the first shot after different wall surface treatment

The primary discharge experiment by lithium coating demonstrates its great superior, Fig. 4 shows the core radiation power loss in the first shot after the different wall treating under the same discharge parameters. From Fig. 4, it can be seen that the core radiation power loss reduces to the lowest level with a fresh lithium coating, but the superiority lasts only several shots, which indicates how to prolong the lifetime of lithium coating is a problem to be resolved.

2 PLASMA FACING MATERIALS

2. 1 B, Ti, Si-doped graphite and B_4C-C/C composites

A series of B, Ti, Si-doped graphite have been developed by hot pressing, the average density is up to 2～2.17 g/cm^3, which is close to the limited graphite density 2.26 g/cm3. The porosity of B, Ti, Si-doped graphite is lower than that of high purity graphite. The bend strength of B, Ti, Si-doped graphite is higher than that of high purity graphite by 2～3 times, and the thermal conductivity of the doped graphite is a little lower than that of high purity graphite. The physical and thermomechanical properties are listed in Table 1. The concentration of boron, titanium or silicon is 3%～20%, respectively. Microstructure analysis reveals that only about 2% B, Ti or Si can be dissolved in the graphite matrix while the additional amounts exist with compounds, such B_4C, SiC or TiB_2.

Table 1 Physical properties of some doped graphite

Materials type	Content of Dopants (mass percentage) %	Density g/cm^3	Porosity %	Bend Strength MPa	Thermal expansion coefficient 10^{-6}/K	Thermal conduct. W/m•K
TPMS		1.83	<10	34.2	2.95	72.0
TPMSBS-10	B 10%+Si 5%	2.10	4.70	102.9	3.06	39.6
TPMSBST-8-6	B 8+Ti 6+Si 4	2.17	4.93	123.3	2.96	59.4
TPMSBST-9-4	B 9+Ti 4.8+Si1.6	2.12	5.33	123.2	2.95	68.4
GB110	B 10	2.00	6.14	81.1		48.6
GBT78	B 7+Ti 8	2.13	5.7	74.3	3.38	71.7
GBT155	B 15+Ti 5	2.12	5.23	90.9		75.3
GBT205	B 20+Ti 5	2.09	6.34	104.1	4.78	71.25
GTB10610	B 10+Ti 6	2.18	4.18	67		48.6
GBS33	B 3+ Si 3	2.0	6.5			100.4
RG-Ti	Made by Russia	2.20				400
USB15	Made by Russia	1.8	12.0			100

3D C/C composites are made by means of hot isostatic press, its average density is 1.94 g/cm^3, the porosity is about 3%～6%. The concentration of boron in B_4C-C/C composites is 3 to 7 (mass percentage), the physical properties of C/C composites are concluded in Table 2. The thermal conductivity of C/C (4#) is close

to the level of C/C composites abroad and is very homogeneous.

Table 2 Physical properties of C/C composites

Material type	Composition	Density	Porosity	Thermal conduct
		$g \cdot cm^{-3}$	%	$W \bullet (m \bullet K)^{-1}$
C/C (1#)	Pitch fibers	1.89	6.6	174/164
C/C (2#)	Graphite fibers	1.96	4.0	133/128
C/C (3#)	Graphite fibers	1.94	3.66	119/81
C/C (4#)	Graphite fibers	2.03	3.16	197/190
B_4C-C/C (6#)	7(mass percentage) B_4C	2.07	3.05	125/122
B_4C-C/C (12#)	10(mass percentage) B_4C	2.09	3.0	120/118
AEROLOR	Made by France	1.80		300/85
CX-20028	Made by Japan	1.74		325/186

2. 2 Chemical sputtering [6]

When the B, Ti, Si-doped graphite was bombarded by deuterium ions with 1 keV energy at elevated temperature, the methane yield was measured by QMS, the experimental results are shown in Fig. 5. From Fig. 5, it can be seen that the methane yields CD_4 of the doped graphite are much lower than that of high purity graphite TPMS and the peak temperatures of CD_4 for the doped graphite moved towards to the lower temperature with the increasing of boron. Comparing with the experimental results of chemical sputtering of RG-Ti, RG-Ti-B and USB15 made by Russia, chemical sputtering yields of B, Ti, Si-doped graphite made by China are lower than that of RG-Ti and RG-Ti-B and slightly higher than that of USB15.

2. 3 Hydrogen retention and recycling

As mentioned above, some amounts of boron increased in graphite can effectively reduce the chemical sputtering yields, it is believed that hydrogen retention and recycling of B, Ti, Si-doped graphite should be decreased as well. A thermal desorption experiment was carried out, the TDS of GB110 graphite (10 (mass percentage) B), high purity graphite ISO880U and B_4C coating on copper were measured (see Fig. 6). The peak temperature of methane obviously moved to lower temperature direction for GB110 and B_4C coating. The mechanism of thermal desorption of methane in boron doped graphite was investigated, there are three process which contribute to methane release in boron doped graphite [7]. (1) Methane forms at the internal pore surface and freely diffuses through the internal channels.

(2) Methane forms by the chemical reaction between the hydrogen atoms trapped in B_4C precipitates and the carbon atoms from B_4C compounds. (3) Methane forms in the matrix lattices and desorbs by bulk diffusion.

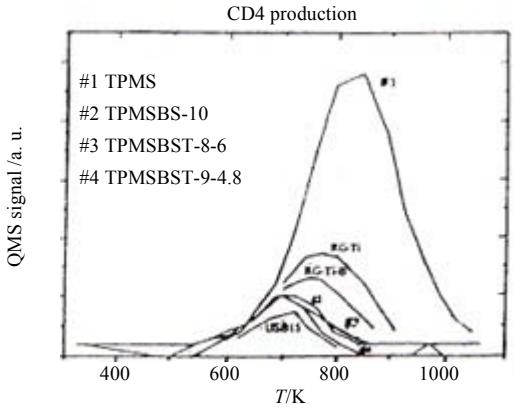

Fig.5 Chemical sputtering yields of B-, Ti-and Si doped graphite. RG-Ti, RG-Ti-B and USB15 made by Russia

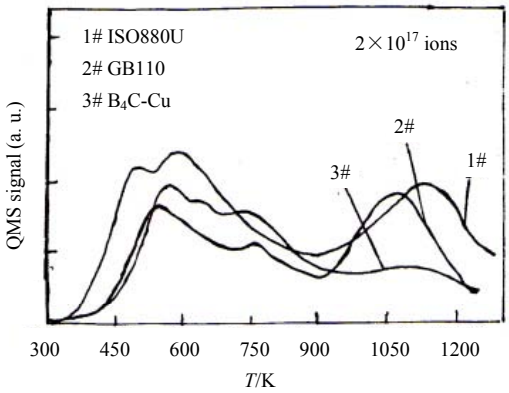

Fig.6 Thermal desorption spectra of methane for different carbon materials

2. 4 Thermal shock

In the future fusion reactor, one of the functions of plasma facing materials is to remove the thermal deposition from plasma; therefore, high heat load on the plasma facing materials is the most important issue. In the past ten years, the experimental facilities of high heat load were constructed and thermal shock tests were carried on with pulse laser and electron beam, a lot of experimental results were obtained [8, 9]. According to a simple plate-shaped model [10], fracture failure will occur when the surface temperature just exceeds the minimum critical value $\Delta T_c=\sigma_F (1-\nu)/S\alpha E$, where σ_F denotes the critical stress associated with the fracture failure mode, ν is Poisson's ratio, α is the linear thermal expansion coefficient, E is Young's modulus, $\Delta T_c = T-T_0$ is the surface temperature change relative to the initial state, and S is a factor depending on the geometry and kind of stress. Relative ΔT_c values can serve as a basis of comparison of the performance of different materials under thermal shock stressing with fixed geometry. From the energy balance equation, one can obtain the expression of $\Delta T_c = K\Phi t^{1/2}$, where Φ is the energy density (kW/cm^2) and t is the pulse length, K is a constant only depending on the physical properties of the materials, except for shear and bending failures for which it can be expected to depend somewhat on the beam diameter. Fig. 7 shows the weight loss of B, Ti, Si

doped graphite with $\Phi t^{1/2}$ by single pulsed laser impingement. Fig. 8 shows the weight loss (total weight lose/irradiation area) of C/C composites with $\Phi t^{1/2}$ by five times pulsed laser impingement, in this figure, the experimental result of high purity graphite ISO−880U (made by Japan) is also showed for comparison. A Nd:YAG laser beam with 0.1 ms pulse length is used in both cases.

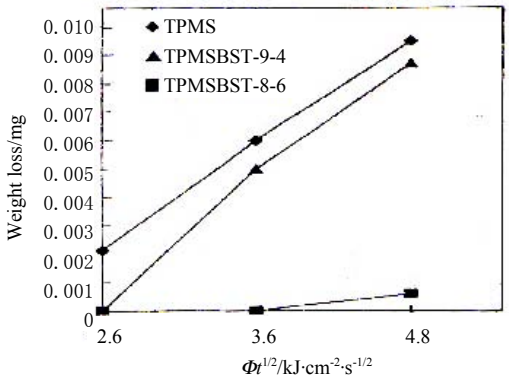

Fig. 7 The weight losses as the function of $\Phi t^{1/2}$ for three kinds of graphites

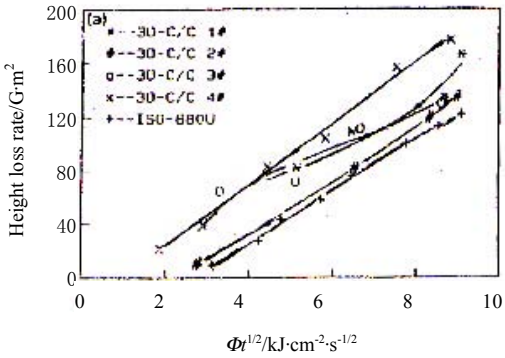

Fig. 8 Weight loss after five laser shots for 3D-C/C composites

3 HIGH HEAT FLUX COMPONENT MATERIALS

3. 1 SiC, TiC and B_4C coating

SiC, TiC and B_4C coatings on graphite or copper matrix are made by means of Chemical Vapor Deposition (CVD) and Plasma Spraying technique. The thickness of these coatings are 10～1000 μm. The erosion experiments of edge plasma are performed both in electron beam thermal shock facility and HL-1M [8, 11]. Experimental results show that the cracks occur in the surface of the coating as well as between the coatings and the substrates under thermal load of 100 MW/m^2 with 10 ms pulse length. It demonstrates that how to strengthen the bonding force between the coatings and substrates is the key issue for the applications of coatings.

3. 2 Functionally graded materials (FGM)

In order to strengthen the bonding force between the coatings and substrates and avoid the great dismatch of thermal expansion coefficient between coatings and substrates, B_4C-C, B_4C-Cu, SiC-C, Mo-Cu and W-Cu functionally graded materials have been developed, recently. The chemical sputtering, tritium retention and recycling, high heat load experiments have been performed, especially, the

resistibility to thermal shocks for FGM is great superior to coating materials. For example, after the shocks of 2 ms electron pulses with 0.5 keV energy and 0.4 A current by 30 times, great cracks, even fracture were found not only in the surface but between the surface and the substrate in the B_4C-Cu samples which made by plasma spraying immediately (photograph 1S and 1C in Fig. 9), where the thickness of the coating is 160 μm. But only surface erosions were found in the FGM samples and almost no cracks occurred on the surface or in the inner of the samples (photograph 2S and 2C in Fig.9), the thickness of B_4C surface coating and the graded layer is about 260 μm.

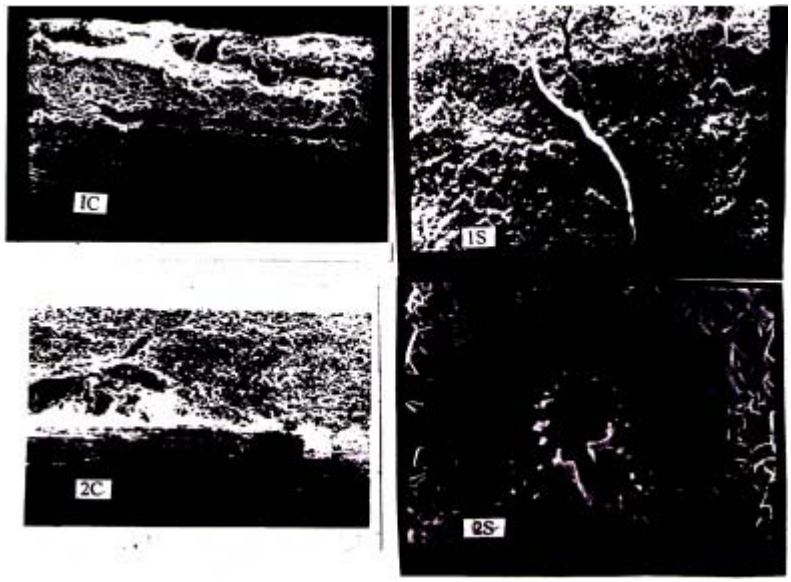

Fig.9 Thermal load behaviors of B_4C-Cu coating and B_4C-B_4C+Cu-Cu FGM specimen

1——B_4C coating specimen; 2——represents FGM specimen;

S——surface; C——section.

4 CONCLUSIONS

In this paper, the two main issues concerned of the first wall engineering of Tokamak machines are discussed, which are the inner wall treating technique by boronization, siliconization and lithium coating, as well as plasma facing materials and high heat flux component materials development. After the inner wall conditioning, the hydrogen recycling and the core irradiation energy are lowered, the property of plasma confinement are improved and long pulse discharges are

obtained. A series of B, Ti, Si-doped graphite and B_4C-C/C composites are developed, experimental results indicate that some amounts of B, Ti and Si are helpful to restraining chemical sputtering， lowering hydrogen retention and recycling, and resisting heat load. Some coating materials, such as SiC, TiC, and B_4C-C or B_4C-Cu coatings materials with graded medium layers are developed, the functionally graded materials SiC-C, Mo-Cu and W-Cu are also developed by means of SHS and HIP. Primary experiments indicate FGMs are very hopeful for plasma facing materials and high heat flux components applications.

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