Tungsten Deposition on Graphite using Plasma Enhanced Chemical Vapour Deposition.

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Abstract: The tokamak concept is the frontrunner for achieving controlled thermonuclear reaction on earth, an environment friendly way to solve future energy crisis. Although much progress has been made in controlling the heated fusion plasmas (temperature ~ 150 million degrees) in tokamaks, technological issues related to plasma wall interaction topic still need focused attention. In future, reactor grade tokamak operational scenarios, the reactor wall and target plates are expected to experience a heat load of 10 MW/m² and even more during the unfortunate events of ELM’s and disruptions. Tungsten remains a suitable choice for the wall and target plates. It can withstand high temperatures, its ductile to brittle temperature is fairly low and it has low sputtering yield and low fuel retention capabilities. However, it is difficult to machine tungsten and hence usages of tungsten coated surfaces are mostly desirable. To produce tungsten coated graphite tiles for the above-mentioned purpose, a coating reactor has been designed, developed and made operational at the SVITS, Indore. Tungsten coating on graphite has been attempted and successfully carried out by using radio frequency induced plasma enhanced chemical vapour deposition (rf-PECVD) for the first time in India. Tungsten hexa-fluoride has been used as a pre-cursor gas. Energy Dispersive X-ray spectroscopy (EDS) clearly showed the presence of tungsten coating on the graphite samples. This paper presents the details of successful operation and achievement of tungsten coating in the reactor at SVITS.

1. Introduction

Recent trends on consumption and demands of energy among developing as well as in developed countries have alarming effects on global climate. Till now, world population is mostly dependent on thermal power, hydropower, natural gases, natural hydrocarbons, nuclear power (fission), wind energy, and very recently solar energy for their energy needs. Global climatic changes has adversely affected the pattern of rain fall, wind flow and the natural resources like coal, natural gas, fuel hydrocarbon has started depleted at an alarming rate and expected to be finished by the end of this century. This trend is going to create a chaotic situation in future in order to maintain the energy needs. Scientists from interdisciplinary fields all over the world are working tirelessly to device new sources of energy for meeting the energy demands suitably met and several international
collaborations are initiated by extending the scientific as well as technical know-how to each other. Nuclear power is known as the most efficient way to produce huge amount of energy. Nuclear fusion has definite advantage over fission and promises clean and environment friendly solution to present energy demands. Fusion of two light hydrogen isotopes (D-T) in a magnetically confined reactor can generate energy equal to mass difference which can be efficiently extracted from the fusion reactor and convert it into electrical energy. To fuse the two light hydrogen isotopes, they need to be heated to very high temperature \( \sim 10^8 \) K. But, maintaining such high temperature in fusion reactors is not an easy task. In present day scenario, virtually no material exist that can directly withstand this temperature and hence a magnetic cage is required to hold this superheated gas known as plasma. In the past few decades several machines such as tokamaks, stellarators, spheromaks, reverse field pinch etc. have come into existence [1-9]. Out of them, tokamak is the frontrunner to be converted into a fusion reactor and has yielded a fusion power with D -D phase reactions. In a larger step, USA, Russia, China, South Korea, Japan, India and European Union has decided to collaboratively fund and develop world’s largest tokamak called International Thermonuclear Experimental Reactor (ITER) which is in its construction phase in Cadarache, France [10]. The machine is expected to be operational in mid 2020s and expected to yield a fusion power ten times to its input power. Although, the superheated plasmas are trapped in the magnetic cage, the trap is not full proof and the heated particles leave the trap and fall on the plasma facing components damaging them seriously. Hence, the first wall and the target material choice is very crucial for any fusion reactor and ITER is designed with Beryllium as its first wall and tungsten as target material i.e. divertor. The plasma-material interaction effects influence the operation, safety and performance of the fusion reactors. Till now, most of the tokamaks are operated with stainless steel and carbon as first wall and target materials as Molybdenum, carbon, tungsten, titanium, Beryllium, carbon etc. and researchers have gained enough experience on the behaviour of these materials in plasma environment [11-12]. However, suitable choice of first wall materials still remains a subject of research with material as well as fusion scientists.

As fusion power remains a candidate for supplying future energy needs to the human community, reactor design and its interruption free working is being aimed with high priority. A fusion machine depends upon all branches of physics to engineering knowledge to be put together for its success. Starting from thick stainless steel reactor body, support structures, superconducting magnetic coils, cryogenic fluids, major electrical components (DC & Rf), beam sources for heating, vacuum pumps, plasma facing surface etc. are required to be put into order for successful operation of the reactor. The most crucial engineering and physics challenge to the fusion community has been identified as the interaction between plasma and wall material (PWI) [12]. The core plasma is required to be maintained at high temperature and with minimal impurity penetration from the walls. This requires edge plasma to be acting as a good thermal as well as physical barrier so that impurities from the walls cannot travel to the core plasma. The wall material also experiences high heat flux, particle flux, transients like edge localised modes (ELM), disruptions. This leads to substantial damage of the plasma facing components in forms of physical erosion i.e. particle erosion, layer melting, and material evaporation and chemical erosion i.e. formation of macro hydrocarbons amongst many other processes.

Plasma facing components (PFCs) are primarily divided into two categories depending upon their functionalities. They are classified as 1) target materials, i.e. limiter and divertor, 2) first wall materials. In the limiter configuration, a solid surface is used to define the plasma radius and to avoid direct contact of hot plasma on the vacuum vessel wall. In the divertor configuration, the plasma boundary is defined by specially designed magnetic fields and the particle flux coming out of the main plasma is diverted to a remote location of the tokamak. The limiter and divertor plates are called the target plates which see the majority heat flux and particle that are diffused cross field in the Scrape Off Layer (SOL) from the core plasma and direct neutron bombardment generated as a fusion by product. Although the target plates take the maximum plasma heat and particle load, the plasma particles and heat also fall on vacuum vessel wall, generally known as the first wall. The choice of the target and the
first wall materials are therefore very important in order to run the fusion reactor successfully. In section 2, we will discuss on the different choice of suitable material for plasma facing component (PFC) and review choice of tungsten, section 3 will describe the methodologies that can be adopted to carry out an in-situ or ex-situ repair of the tungsten and will explain plasma assisted chemical vapour deposition technique suitability, section 4 will describe the radio frequency assisted coating reactor at SVITS, in section 5, we shall be presenting preliminary results on tungsten coating characterization.

2. Material choice for fusion reactors
Cleanliness and the purity of the core plasma are largely governed by the choice of the target and wall materials. The criterion for choice of target materials is different than the wall materials as the nature of particle and heat flux seen by them is different. The target materials experience a high level of heat flux than the wall materials and hence they need special design and material characteristics. The plasma interaction with the target and wall material impact the life time of the plasma facing components and fusion fuel retention in these materials remain a safety concern. In this section, we shall briefly discuss the life time of the plasma facing components under different plasma conditions. The lifetime of the plasma facing components is determined by the erosion processes and the erosion is initiated by physical sputtering, chemical erosion, material loss due to uncontrolled plasma impact (ELM), plasma disruptions leading to the bulk melting and evaporation. It is well known that in order to sustain the fusion reaction, the concentration of species with high mass number in the core plasma should be very low. So obvious choice for wall materials is the materials having low mass number and carbon is a prospectus candidate. However, there are several limitations with carbon i.e. carbon reacts chemically with oxygen, hydrogen, deuterium and tritium thus fuel retention is high and reactor depletes the fuel. Being low atomic mass its sputtering yield is high compared to the metals. For example, in ITER plasma discharges (Q = 10), typical particle flux reaching the divertor region will be $10^{24} \text{m}^{-2} \text{s}^{-1}$ leading to a large particle incidence ($\sim 10^{26} \text{m}^{-2}$) per plasma shot with divertor plasma densities $\sim 10^{21} \text{m}^{-3}$. This could lead to the local temperature rise on the divertor to $\sim 1500^\circ K$. With the given statistics, various codes have predicted the life time of the wall and target materials. Table 1 shows the estimated numbers predicted by DIVIMP code\(^{(1)}\) for wall material and ERO code\(^{(2)}\) for divertor. During steady state operation the walls and target will experience intensive particle and heat loads $\sim 10 \text{MW/m}^2$ and transient loads $\sim 20 \text{MW/m}^2$.

Table 2 shows the properties of the target materials presently under consideration although ITER has finalized its first wall and target materials. Hence, proper choice is still remains a burning subject of research. Numerical calculations have predicted that 280 tons of iron will be eroded from ITER under it’s normal operation conditions and this rate would be of much higher order if carbon, boron and beryllium is used.

Table 1. Estimated life times for plasma facing components.

| Material | Erosion rate (nm/s) | Erosion Source (atoms/s) | Eroded material (g/shot) | Life time (Shot) |
|----------|---------------------|--------------------------|--------------------------|-----------------|
| Be\(^{(1)}\) | 0.1 | $8 \times 10^{21}$ | 48 | 20000 |
| W\(^{(1)}\) | 0.01 | $2 \times 10^{20}$ | 26 | 200000 |
| CFC\(^{(2)}\) | 100 | $4 \times 10^{22}$ | 330 | 200 |
| W\(^{(2)}\) | 2 | $4 \times 10^{20}$ | 48 | 10000 |
Table 2. Probable candidates for target materials in a fusion reactor [13].

| Material | Density ($10^3$ kg/m$^3$) | Melting temp ($^\circ$C) | Thermal Conduc. (W.m$^{-1}$.K$^{-1}$) | Therm. Expa. Coeff ($10^{-6}$. K$^{-1}$) | Tensile Strength (MPa) |
|----------|-----------------------------|--------------------------|--------------------------------------|---------------------------------------|------------------------|
| Be$^{(1)}$ | 1.85 | 1283 | 187 | 11.3 | 350 |
| Graphite$^{(2)}$ | 1.85 | --- | 80 | 3.4 | 50 |
| B$_4$C$^{(3)}$ | 2.51 | 2450 | 29 – 67 | 5.6 | 350 |
| SIC$^{(4)}$ | 3.2 | --- | 67 | 2.9 | 360 |
| Cu$^{(5)}$ | 8.25 | 1083 | 391 | 16.7 | 206 |
| SS316L$^{(6)}$ | 7.96 | 1400 | 14.6 | 16.2 | 525 |
| TZM$^{(7)}$ | 10.2 | 2620 | 125 | 5.3 | 600 |
| W$^{(8)}$ | 19.3 | 3410 | 144 | 4.4 | 600 |

3. Choice of tungsten (W)
For many reasons, tungsten (W) is considered as superior material amongst the various materials suitable for the target plates in tokamaks. Tungsten has a high melting point and could act as the best plasma facing component if plasma temperature near the target plates remains less than 50 eV. Plasma contamination is suppressed with tungsten especially with low edge temperatures and having a very low chemical sputtering affinity with hydrogen as fuel. Thereafter, sputtered tungsten has a lower ionization potential and lower velocity than sputtered carbon thus ionized tungsten tends to re-deposit near to their point of origin. Tritium inventory in tungsten is expected to be very small. Tungsten recrystallization occurs at fairly low temperatures (1100 $^\circ$C – 1300 $^\circ$C) than its melting point and its melting is only anticipated by thermal quench loads with type I Edge Localized Modes. Current plasma wall research on tungsten focuses on the melt layer behavior under ITER like disruption scenarios.

Despite its superior properties tungsten will suffer erosion under unfavorable plasma conditions. Repairing of the affected area ex-situ i.e. inside the tokamak without breaking its vacuum, will consume a lot of machine operation time. Moreover, in small scale machines pure tungsten component addition generates complications in PWI studies. For example, ASDEX – upgrade uses graphite tiles with few micron coating of tungsten for PWI studies [14]. Similar experiments are being conducted in JET – ITER like wall experiments [15]. First wall of the test blanket modules (TBM) for ITER may be considered to be coated with tungsten for better stability and performance. For such several reasons tungsten coating on various substrate materials are being tried for its future requirements [16].

4. Tungsten coating methodology
Tungsten coating can be suitably employed using physical vapor deposition (PVD), chemical vapor deposition (CVD), magnetron sputtering, plasma spraying both in vacuum and atmosphere, and plasma assisted chemical vapor deposition techniques (PACVD). PVD is a tedious and most difficult process to perform on tungsten as it has a high melting temperature. Chemical vapor deposition requires a precursor material preferably a halide (F) based, having low dissociation temperature but the substrate temperature requires being elevated. CVD in presence of a reactant gas to F i.e. Hydrogen tend to cool the system. Magnetron sputtering is an effective technique but in a laboratory
scale a very small area only could be coated but it is efficient in producing a higher coating thickness. JET divertor tiles have been coated with magnetron sputtering technique. Plasma spraying is one of the effective methods to deposit tungsten melted grains on a substrate but it is associated with several issues. It is a violent process involving arc plasmas in atmosphere or in a vacuum chamber and required very high process temperature. The coating process is rapid thus micro as well as macro cracks develop through out the coating volume and coating becomes non-uniform. Difference of thermal conductivity of the substrate and the tungsten leads to weak interlayer bonding as the deposition process itself leads to sputter the deposits. In atmospheric plasma spraying, impurity impregnation cannot be avoided and maintaining surface uniformity will be challenging. Plasma assisted chemical vapor deposition is considered one of the effective method as it can be done at a fairly low temperature plasmas produced mainly by application of radio frequency waves. Plasma it self acts as a catalyst to the chemical reaction and ionized particles can be preferentially guided by electric fields and deposited on the substrates. It’s a fairly slow process but degree of uniformity and sticking coefficient of the surface is high.

4.1 Plasma enhanced chemical vapor deposition (PECVD)
Fractionally ionized plasmas have great interests in thin film coating and material processing. In such plasmas, the energy exchange between the electron and neutral gas is very inefficient. So, the bulk plasma is more positive than any object (substrate) present and in contact with plasma. The voltage between the plasma and substrate drops at the sheath region and the ions in the sheath feel an electrostatic force and are accelerated towards the substrate. Thus, the substrate exposed to plasma receives energetic ion bombardment. CVD performed in a plasma environment leads to increase in the nucleation, growth kinetics, and hence density of the deposited film and also plays an important role in the process of sputtering of the impurities called Plasma Enhanced Chemical Vapor Deposition (PECVD).

PECVD has been developed to combine the good adhesion of CVD and low temperatures of physical vapor deposition (PVD). This process avoids typical drawbacks like requirement of high temperature, temperature related deformations, poor adhesion etc. The plasma environment in PECVD performs mainly two basic functions; reactive chemical species are formed by cracking of relatively stable molecules via electron impact collisions and supplies energetic radiation such as positive ions, meta stable species, electrons and photons. Increased ion bombardment tends to make the film denser and cause film stress to become more compressive. PECVD performed with RF plasma is called rf-PECVD. Furthermore, PECVD can be performed at low pressure and temperature than CVD. In PECVD, ions suffer more collisions in a gas phase and lose most of their energy before they reach the cathode. Hence, effects due to sputtering on the substrate are minimized. Substrate heating due to the PECVD process is very small and the substrate temperature is mainly controlled by external heating. In PECVD, higher flow rate and RF power can increase the deposition rate and also the uniformity but at the same time there will be waste of reactant gas because of non ionization. Thus process parameters play a critical role in film formation, growth and characteristics.

In plasma assisted chemical vapor deposition, low temperature plasma is produced using different plasma production methods. For tungsten coating a precursor gas/solid containing tungsten is used. The precursor gases/solids are broken down in the plasma, which acts as a catalyst to the reaction. Tungsten ions form in the plasma get attracted towards the cathode and get deposited. The rate of deposition can be controlled or enhanced by the input RF power and applied negative bias on the substrate plate. Tungsten ions get deposited on the substrate with good adhesion and get redistributed over the surface with good step coverage in PACVD process. In this process a fairly large area can be coated by choosing a pair of larger diameter capacitive plate in the reactor chamber.
4.2 Coating reactor at SVITS

Under the National Fusion Program (NFP) of Board of Research Fusion Science and Technology (BRFST) [17], tungsten coating program using PECVD has been taken up at SVITS [18] for developing tungsten coated graphite tiles to be used for Indian tokamaks, ADITYA upgrade tokamak and SST-1 tokamak. Glow discharge plasma is produced by applying a radio frequency between two parallel capacitive plates in this reactor. For deposition of tungsten, tungsten hexafluoride (WF$_6$) gas is used as precursor gas, which is mixed with hydrogen (H$_2$) neutral gas and fed to a reaction chamber.

$$3H_2(g) + WF_6(g) \rightarrow W + 6HF(g)$$

The coating system is designed, fabricated and installed in Shri Vaishnav Institute of Technology & science (SVITS), Indore. The vacuum chamber is a stainless steel (SS304) cylindrical chamber with diameter 360 mm and height 300 mm. The vacuum vessel consists of various CF ports (35CF & 63CF) used for plasma viewing, fixing of vacuum gauges, diagnostics etc. The system is supported on a support structure made of Aluminum hollow square bars. The system is being pumped by a turbo and rotary pump combination. The turbo pump is having a pumping speed of 60 litres/sec and is connected to the vacuum vessel through a gate valve. The turbo pump is roughed through a diaphragm pump inbuilt in the pumping unit itself. The main vacuum system is pumped to the roughing vacuum with a rotary pump of capacity 12 m$^3$/hr. The vacuum on the system is being monitored by combination of pirani and cold cathode ionization gauges. The system is provided with an electro-magnetically actuated vent valve for venting. The vacuum chamber consists of two circular metallic discs (diameter = 15 cm, thickness = 1 cm) placed at the top and bottom flanges and are used as plasma electrodes. The disks are connected to a DC power supply (1kV, 1Amp) as well as to a RF source (13.56 MHz, 600W) with auto matching network used for plasma generation. The electrical connections are taken out with appropriate high voltage RF feed-through. A high precision quadrupole mass analyzer (QMA 300 AMU, mass resolution of 0.5 AMU) is attached to the system for monitoring of impurities as well as the reactant gas presence. Mass flow controller for the WF$_6$ gas is installed for calibrated gas flow to the reaction chamber and hydrogen gas is introduced via a precision leak valve. The system is pumped down to $2 \times 10^{-6}$ mbar and coating pressure regime is maintained at $\sim 10^{-3}$ to $10^{-4}$ mbar. Suitable arrangement with KOH solution is used to neutralise the HF gas. Figure 1 shows the schematic of the experimental system with the connected subsystems.

Figure 1. Experimental coating reactor installation at SVITS showing vacuum chamber, table top turbo pump, quadrupole mass analyser, vacuum gauge, rf power supply etc.
5. Experiment

Table 3 shows the plasma and system parameters in the experimental system.

| Parameter                  | Value          |
|----------------------------|----------------|
| Test gas                   | Argon          |
| RF power                   | 0 – 200W       |
| Frequency                  | 13.56MHz       |
| Bias                       | 200 VDC        |
| Substrate temperature      | 250 °C         |
| Ultimate pressure          | $2 \times 10^{-6}$ mbar |
| Working pressure           | $5 \times 10^{-4}$ mbar |
| Plasma density             | $10^{10} - 10^{11}$ cm$^{-3}$ |
| Coating Gas mixture        | H$_2$ + (5%) WF$_6$ |

Before initiation of the coating process, a uniform plasma region is identified between the parallel electrodes by producing Argon plasma. Potential and density measurements were carried out using a single Langmuir probe compensated for 13.56 MHz. It is observed from the figure that the plasma has a uniform region between -8cm < R < 8cm. Figure 3 shows the sample arrangement on the cathode i.e. bottom plate electrode. The substrate is pure polished graphite tiles to be used as limiter tiles in Aditya tokamak [19]. The sample tiles are cleaned with detergent solution and rinsed thoroughly with water before final cleaning with iso-propyl alcohol. The samples were initially baked to 250 °C under high vacuum in the coating reactor so that the trapped moisture is evaporated. The evaporation process is monitored on the QMA and it is allowed to bake until the H$_2$O traces are bare minimum to the initially recorded values. In the second step, hydrogen plasma is created at RF power of 60W and the samples are exposed to the plasma for 60 minutes for plasma surface cleaning and micro level surface modification. The plasma is simultaneously monitored with a spectrometer working at visible spectral range. In the third stage, tungsten hexafluoride (WF$_6$) gas is injected to the reactor chamber in a controlled manner through a calibrated mass flow controller. Controlled gas injection is required because WF$_6$ is a heavy gas and it may lead to a substantial change in the plasma resistance between
the electrodes and hence plasma may get disrupted. With the injection of the gas, WF$_x$ (x = 1 to 5) isotope ion traces are observed on the QMA and a typical spectrum is shown in figure 4. Figure 4 shows the traces of tungsten found in natural abundance. Figure 5 shows the picture of mixture of tungsten hexafluoride and hydrogen plasma. During the coating process hydrogen reacts with fluorine gas and produces a huge amount of acidic hydrogen fluoride (HF) gas which is required to be efficiently removed from the reactor volume before opening the chamber for removal of the samples. HF signal is continuously monitored throughout the experiments. The exhaust line of the pumping system is put into a potassium hydroxide (chemical base) solution and the HF is efficiently decomposed to solid potassium fluoride which is not so reactive.

$$KOH + HF = KF + H_2O$$

Figure 3. Figure showing the arrangement of the graphite tiles on the heater surface mounted on the bottom electrode. The heater assembly and the bottom plate are electrically conducting.

Figure 4. Figure shows the analog scan for the traces of tungsten isotopes detected by the high precision residual gas analyser.
Figure 5. Picture shows the first plasma of WF$_6$ + H$_2$ gas mixture and the sample pieces are exposed to the plasma.

6. Results & Discussion
The coated samples are tested for quality of tungsten deposits and stability. Figure 6 shows the EDX trace for elemental analysis and tungsten rich signal is observed. Apart from the tungsten traces, oxygen, carbon and fluorine traces are also present in the coating. The origin of the carbon and oxygen signals is due to the beam interaction with graphite and from the plasma system respectively. The fluorine is embedded from the precursor gas. The tungsten coating thickness achieved is the order of 300 nm. It is considered that with increase in the coating thickness (few μm), the beam interaction with substrate will decrease and the carbon trace will be reduced. The fluorine trace suppression would require more concentration of hydrogen into the coating reactor and reduced inflow of the WF$_6$. This is more a process optimization engineering.

Figure 6. EDX scan of the tungsten coating with beam power maintained at 15 kV.
The above tests on the coated samples clearly establish the successful tungsten coating with the help of plasma enhanced chemical vapour deposition at SVIT, Indore, which is achieved first time in India. The state of art technology has been established and the process requires minor optimization for improvisation of the coating properties and thickness. Initial characterization results are very satisfactory and promising to utilising this technique further for our own in-house requirements. Cross-sectional analysis of the coatings suggests achievement of a thickness of ~ 300 nm and it is possible to produce micro–meter thick coatings by enhancing the reaction rate by increasing the RF power and deposition time. The coatings will be further tested for its stability in IPR LASER and electron beam facility for its stability.

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