On Residual Gas Analysis during High Temperature Baking of Graphite Tiles

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Abstract. Steady-state Super-conducting Tokamak-1 (SST-1) is a medium size tokamak with major radius of 1.1 m and minor radius of 0.20 m. It is designed for plasma discharge duration of 1000 seconds to obtain fully steady-state plasma operation. Plasma Facing Components (PFC), consisting of divertors, passive stabilizers, baffles and poloidal limiters are also designed to be UHV compatible for steady state operation. All PFC are made up of graphite tiles mechanically attached to the copper alloy substrate. Graphite is one of the preferred first wall armour material in present day tokamaks. High thermal shock resistance and low atomic number of carbon are the most important properties of graphite for this application. High temperature vacuum baking of graphite tiles is the standard process to remove the impurities. Residual Gas Analyzer (RGA) has been used for qualitative and quantitative measurements of released gases from graphite tiles during baking. Surface Analysis of graphite tiles has also been done before and after baking. This paper describes the residual gas analysis during baking and surface analysis of graphite tiles.

1. Introduction
Carbon based materials, namely; Graphite and Carbon-Carbon Composites are the preferred armour materials in present day tokamaks. High thermal shock resistance and low atomic number of carbon are the most important properties of graphite for its application as an armour material in tokamaks. CFC has been found superior to fine grained graphite. The CFC material has higher thermal conductivity and higher strength and hence it is much less susceptible to damage from disruptions. During Plasma Operation in tokamaks, the plasma facing armour materials may reach high temperature. The result is that volatile compounds or impurities are introduced into the plasma. An expensive Tokamak conditioning period extending over weeks of discharge cleaning and hundreds of high power shots may be required to obtain acceptably clean plasmas. Generally graphite tiles are given a high temperature bake-out treatment prior to installation inside the tokamak to reduce the conditioning period. Thermal desorption is a part of the conditioning process. Removal of water vapour from graphite can be accomplished by a bake-out at about 350°C in vacuum. In order to remove all the absorbed gases it is necessary to bake the graphite at high temperature of about 1000°C in a vacuum furnace prior to installation inside the tokamak. Plasma Facing components are kept inside the main vacuum vessel of SST-1 tokamak and hence needed to be Ultra High Vacuum (UHV) compatible. PFC consists of divertors, passive stabilizers, baffles and limiters as shown in figure 1. All PFC modules are made of copper alloys on which graphite tiles are mechanically attached. High temperature outgassing experiments on different graphite tiles has been carried out, in order to
understand the outgassing behavior of graphite at various temperatures and to determine the bake-out and conditioning procedures. Experimental study has been carried out on the same grade (FP479) graphite that will be used in SST-1 tokamak. The density of this graphite is 1.82 g/cm³ and 9% open porosity. These studies have been performed in a vacuum furnace specially designed and commissioned for baking the graphite tiles. Residual Gas Analyzer (RGA) has been used to study the qualitative and quantitative behaviour of the outgassing species. Surface characterisation before and after baking has been done using Scanning Electron Microscope (SEM). Results of these studies will be discussed in detail in this paper.

2. Experimental details

A high vacuum furnace specially designed for graphite tile baking is employed in this work. It has a double walled chamber fabricated with SS-304. Inside the rectangular hot zone all materials including heating elements, insulation, etc. are made of graphite. Pumping system consists of a diffusion pump with pumping speed of 6000 liters/sec, which can evacuate the furnace chamber to 1x10⁻⁶ mbar. Roots and rotary pump combination was used for roughing and backing. Water-cooled chevron baffle and LN₂ trap is provided above the diffusion pump, which provides effective condensation of vapour to prevent back streaming of vapour into the chamber. All the valves like roughing valve, backing valve, high vacuum valve, holding valve and vent valve are electro-pneumatically operated and interlocked wherever necessary for failsafe operation. Schematic of the vacuum furnace and its pumping system are shown in figure 2. Prior to actual baking of the graphite tiles, the oven itself should be baked to 1000°C in high vacuum in order to avoid oven impurities depositing on the graphite tiles. Sample coupons were used to qualify the baking process. Graphite tiles are ultrasonically cleaned in alcohol to remove all the loose particles. It is then air dried to remove any entrapped alcohol. Cleaned tiles along with test coupons are kept inside the hot zone. After attaining a high vacuum (<1x10⁻⁵ mbar) baking is started as per pre-programmed temperature ramp rate and the outgassing species are analyzed using a residual gas analyzer (RGA). When the appropriate baking temperature (1000°C) is reached the charge is soaked at this temperature. At the end of the heat soak, the temperature is allowed to cool down to room temperature naturally under vacuum. Thermal desorption of outgassing species has been observed throughout the entire procedure.

3. RGA analysis

Observations of the residual gas composition during baking are summarized in this section. Quadrupole Mass Analysis (QMA) is a useful technique for monitoring out-gassed species in a vacuum system. QMA (Make: SRS; Model: RGA200) was employed in our outgassing studies. An RGA is a mass spectrometer consisting of a quadrupole probe. It works on the principle that a small fraction of the gas molecules are ionized and the resulting ions are separated, detected and measured according to their molecular masses. Transient behaviour of the residual gas during baking of empty
vacuum chamber and with graphite tile is observed. Water vapour seems to be the dominant impurity during thermal desorption and starts decreasing gradually. The remaining outgassing species are N₂, CO, CO₂ and H₂. The gas concentrations are given in percentages, which are normalised to the total pressure. Mass spectrum of different residual gas intensities during baking of graphite sample-1 shows that the concentration of H₂O starts decreasing from 65% at room temperature to 45% around 200°C. It starts rising suddenly and reaches a peak percentage of 60 at 300°C and then it starts decreasing gradually. The same thing happens in the case of empty furnace baking and with the graphite sample-2 baking. The concentration of CO₂ increases rapidly between temperatures 300°C to 500°C and then it starts decreasing slowly and rapidly above 900°C. Whereas in the case of CO, no intensity of CO was found till 500°C. But after 500°C the intensity rises steadily and decreases at 1000°C soaking. In the case of furnace baking without tiles the rise of CO happens slightly later around 600°C due to the absence of carbon load. In all the cases the concentration of H₂ increases at 700°C from 0%, gradually reaches 15% and then starts decreasing at 1000°C. There is no significant increase in intensity of oxygen. Temperature dependence of the outgassing of several species during baking of empty furnace and with graphite are shown in figures 3, 4 and 5. RGA mass spectrum obtained at room temperature and at 1000°C baking are shown in figure 6 and 7 respectively.

Figure 3. During baking of furnace without graphite tile.

Figure 4. During baking of graphite sample-1.
4. **Surface analysis**

Observations of changes on the surface of graphite test coupons exposed to conditioning procedures are summarized in this section. The surface characterization has been carried out with Scanning Electron Microscope (Leo s-440i model) for graphite test coupons before and after baking up to
1000°C. The test coupons were less than 1 cm² and not more than 0.5 cm thick. Surface analysis of these test coupons was performed with SEM to qualify the vacuum outgassing procedure for graphite tiles.\textsuperscript{[3]} Surface characterization of graphite tiles is important to establish the initial condition as baseline data for graphite tiles preconditioning process. Preconditioning is a term applied to the changes that occur in the near surface region of graphite materials as a result of high temperature vacuum bake-out procedure. The SEM images of graphite test coupons are given below. For comparison, an as-received sample of graphite is shown in figure 8. It is characterized by fairly uniform porosity. During the course of surface analysis, of the SEM images of baked samples, it was found that the pores have been cleaned out and the pores are more clearly visible as shown in figure 9.

![Figure 8. Graphite sample-1 (FP479)](image1)
![Figure 9. Graphite sample-1 (FP479)](image2)
![Figure 10. Graphite sample-2](image3)
![Figure 11. Graphite sample-2](image4)

5. Discussion
The desorption spectrum of graphite tile baking shows water as a dominant gas load, since numerous types of nuclear grade graphite absorb atmospheric H₂O rapidly during relatively short air exposures. Thermal desorption measurements shows that the peak desorption of H₂O, occurs around 300°C, while desorption of H₂O is comparatively smaller at higher temperatures, which ensures that temperature of 350°C are required for complete outgassing of water from graphite. There are several other peaks seen when water is present in vacuum chamber. This is due to the primary ions formed from water by electron bombardment are H₂O⁺, OH⁺, O⁺ and H⁺ giving mass spectrum with peaks at m/e= 18,17,16 and 1.\textsuperscript{[4]} O₂⁺ is formed by secondary reaction. CO⁺ and CO₂⁺ are formed due to the reaction of primary water ions with carbon or hydrocarbons. The decrease in CO₂⁺ with simultaneous increase in CO⁺ was found after 500°C. This is due to the fact that CO₂ reacts with the remaining hot carbon to give CO. No significant percentage of O₂ reveals the absence of oxygen gas inside the chamber, ensuring that the chamber is free from leak. As the furnace is vented with dry N₂ gas, the presence of N₂ gas is obvious. SEM images of as-received graphite samples are characterized by fairly uniform porosity. Much of the porosity is filled with graphite powder, which is produced, by machining and polishing of graphite. In the SEM images of baked samples, the pores have been
cleaned out, which can be produced by careful ultrasonic cleaning of graphite and high temperature vacuum baking.

6. Conclusion
This work shows the advantages of pre-conditioning of new tiles. As a result of pre-conditioning process, the major plasma contaminants were significantly reduced. Minimization of outgassing will lead to shorter conditioning times and cleaner plasmas. Prior to the standard bake-out procedures utilized it might be helpful to exhaustively clean the tiles ultrasonically to remove loose particles. Clearly, it could be cost effective to pre-condition new tiles prior to installation inside the tokamak without using weeks of machine time.

7. References
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