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Estimation of Partial Pressure during Graphite Conditioning by Matrix Method

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Abstract. Plasma Facing Components (PFC) of SST-1 tokamak are designed to be compatible for UHV as it is kept in the main vacuum vessel. Graphite is the most widely used plasma facing material in present day tokamaks. High thermal shock resistance and low atomic number carbon are the most important properties of graphite for this application. However, graphite is porous and absorbs gases, which may be released during plasma operation. Graphite tiles are baked at high temperature of about 1000 °C in high vacuum (10^{-5} Torr) for several hours before installing them in the tokamak to remove the impurities (mainly water vapour and metal impurities), which may have been deposited during machining of the tiles. The measurements of the released gas (such as H_2, H_2O, CO, CO_2, Hydrocarbons, etc.) from graphite tiles during baking are accomplished with the help of a Quadrupole Mass Analyzer (QMA). Since, the output of this measurement is a mass spectrum and not the partial pressures of the residual gases, one needs to adopt some procedure to convert the spectrum to obtain the partial pressures. The conventional method of analysis is tedious and time consuming. We propose a new approach based on constructing a set of linear equations and solving them using matrix operations. This is a simple method compared to the conventional one and also eliminates the limitations of the conventional method. A Fortran program has been developed which identifies the likely gases present in the vacuum system and calculates their partial pressures from the data of the residual gas analyzers. Application of this method of calculating partial pressures from mass spectra data will be discussed in detail in this paper.

Introduction

Steady state Superconducting Tokamak (SST-1) is a large aspect ratio tokamak designed for a plasma discharge duration of \(\approx1000\) seconds to obtain fully steady state plasma with total input power upto 1.0 MW [1, 2]. Plasma Facing Components (PFC) of SST-1 are inside the main vacuum vessel (fig.1) and hence needed to be designed for ultra high vacuum (UHV) compatibility. Graphite tiles are used as an armour material which is baked at high temperature of about 1000 °C in high vacuum (10^{-5} Torr) for several hours before installing them in the tokamak. The tiles are baked in a specially designed vacuum furnace (inside the hot zone all materials including heating elements, insulation etc. are made of graphite) which is equipped with a Residual Gas Analyser (RGA) for qualitative and quantitative measurements of released gases (such as H_2, H_2O, CO, C_2O, Hydrocarbons, etc.) from graphite tiles during conditioning.

In any vacuum system it is not only necessary to know the total pressure of the system but also the specific gas components making up that total pressure, because the knowledge of total pressure is not sufficient to interpret the experimental data. These measurements are accomplished with the help of a Partial Pressure Analyzer (PPA). The PPA output does not directly give the partial pressures of these residual gases, but is a mass spectrum which contains peaks at different mass numbers (or mass to
charge ratios, \( m/e \)). We propose a new approach which is simple method compared to the conventional method. A Fortran program has been developed which solves the matrix equations identifies the likely gases present in the vacuum system and calculates their partial pressures. The details of this method used for calculating partial pressures from mass spectra data during graphite tile conditioning will be discussed in this paper.

**Figure 1.** Elevation view of PFC inside the vacuum vessel of SST-1 Tokamak

**Conventional Method**

In the conventional method of estimating the partial pressure of gas molecules [3], one has to identify a peak at some mass number and associate it with only one particular gas species. The amplitude of this peak can then be used to calculate the partial pressure (PP) of that gas species. The calculated partial pressure is used to estimate the contribution of that gas species at all other mass numbers in the spectrum from the known fragmentation pattern of that species. These contributions are subtracted from the spectrum to obtain a new spectrum. This procedure is repeated till all the significant peaks in the spectrum are accounted for. This procedure, when completed, would have estimated the partial pressures of the residual gases present in the vacuum system.

Fig. 3 shows a mass spectrum of the residual gases in a hypothetical stainless steel vacuum vessel [4]. In this case the mass range covered is from \( m/e=12 \) to \( m/e=58 \), and for the simplicity it is assumed that the mass peaks for the gases in the system will not be found outside this mass range. The peak height for each peak (in division) is shown in fig. 2. We expect \( \text{H}_2, \text{He}, \text{H}_2\text{O}, \text{O}_2, \text{CO}, \text{CO}_2, \text{ethanol} \) and acetone as this vacuum system is exposed to air, acetone and ethanol. In the conventional method, it is assumed that a particular peak is due to only one gas molecule. To estimate the PP by this method let us assume that the peak at \( m/e=58 \) in this mass spectrum (fig. 2) is due to only acetone (acetone has a fragmentation/cracking pattern here). So, the partial pressure of acetone can be calculated as:

\[
P_{j} = \frac{h_{j}}{S_{j} \times a_{i}^{j} \times W} \quad \text{……..(1)}
\]

Where \( h_{j} \) is the peak height contribution from gas \( j \) at mass number \( I \); \( S_{j} \) is the analyzer sensitivity; \( a_{i}^{j} \) is the fragmentation ratio or cracking pattern of the \( j^{th} \) gas species at \( i^{th} \) mass number.

\[
P_{\text{acetone}} = 33.5 \text{ div}/[(1.2 \times 10^{11} \text{ div/Torr}) \times 0.271] = 1.0 \times 10^{-9} \text{ Torr}
\]
Sensitivity of the analyzer for acetone = 1.2 x 10^{11} \text{ div/Torr}, and cracking pattern for acetone = 0.271 [4]. Once the partial pressure of acetone is known, we calculate the new heights for the mass numbers contributing for acetone (the mass numbers has the cracking pattern for acetone) using $P_{\text{acetone}} = 1.0 \times 10^{-9} \text{ Torr}$. These new heights will then be subtracted from the other peaks on the spectrum. Once we get the new peak heights, we take highest mass number which has a peak. Now consider any of the gas molecules which has a cracking pattern at this mass number and follow the same procedure. Suppose in the new peak heights, 46 is the highest mass number which has a peak (39.6 div). Lets consider this peak is due to ethanol (as ethanol has a fragmentation of 0.165 here). Now the PP of ethanol will be:

$$P_{\text{ethanol}} = 39.6 \text{ div}/(1.2 \times 10^{11} \text{ div/Torr} \times 0.165) = 2.0 \times 10^{-9} \text{ Torr}$$

Sensitivity of the analyzer for acetone = 1.2 x 10^{11} \text{ div/Torr} [4]. Now calculate the new heights for the mass number (in which ethanol has a fragmentation) and subtract that heights and get the remaining peaks in the mass spectrum (in the same way we did for acetone). In this way we get PP of other gas molecules are calculated as shown in fig. 3. Besides demanding knowledge of the vacuum system to identify the likely gas species that are present, the serious limitation is to one particular peak in the spectrum with one particular gas species only. If the same peak is selected for other gas species then the result will be different. This can be eliminated by our proposed new method, the matrix method which is discussed in detail in the next section.

**Figure 2.** Typical mass spectrum obtained from RGA [ref. 4]

**Figure 3.** Comparison of PPA by conventional and matrix method

**Matrix Method**

In the proposed new method of analysis, attributing the peaks to certain gas species is completely eliminated. All the suspected gas species may be assumed to be present and the new method predicts the partial pressures correctly. Besides removing this limitation, the new method is also simple to use. Like in the conventional method, we assume that any peak in the mass spectrum of a mixture of gases may consist of contributions from the molecular ions and/or fragment ions and the contributions add linearly, i.e., the observed peak amplitude is the sum of the individual peak amplitudes of the constituents of the mixture when they are alone present.
As described earlier, the peak at any mass number, say $i$, is a linear combination of individual peaks at the same mass number due to the constituent gases. This can be expressed as:

$$q_i = \sum_j a_j^i s_j p_j = a_1^i s_1 p_1 + a_2^i s_2 p_2 + \ldots + a_n^i s_n p_n$$

…….(2)

Where, $a_j^i$ is the fragmentation ratio or racking pattern of the $j^{th}$ gas species at $i^{th}$ mass number and is normalized to give $\sum_j a_j^i = 1$ for any index $j$. $s_j$ and $p_j$ are the sensitivity and the partial pressure of the $j^{th}$ gas species respectively. The fragmentation ratios are usually expressed with the maximum peak normalized to 10 or 100. In the matrix method, the fragmentation ratios are expressed with respect to the total as shown in table -1 for water.

| Mass No. | Fragmentation Ratio (Maximum peak normalized to 100) | Fragmentation Ratio used in the Matrix method |
|---------|---------------------------------------------------|---------------------------------------------|
| 14      | 7.2                                               | 7.2/108 = 0.0667                           |
| 28      | 100                                               | 0.9259                                     |
| 29      | 0.8                                               | 0.0074                                     |
| Total   | 108.0                                             | Total = 1.0                                |

* Fragmentation ratio data is provided by manufacturer of RGA, as provided by SRS, ref [4]

Thus, the fragmentation ratio for a particular gas molecule is always equal to 1.0 in the matrix method. Equation 1 may be written as:

$$q_i = a_1^i g_1 + a_2^i g_2 + \ldots + a_n^i g_n$$

…….(2)

where, $g_j = s_j p_j$ is the apparent partial pressure of the $j^{th}$ species. The set of equations for relevant mass numbers can be written as follows:

$$q_1 = a_1^1 g_1 + a_2^1 g_2 + \ldots + a_n^1 g_n$$

$$q_2 = a_1^2 g_1 + a_2^2 g_2 + \ldots + a_n^2 g_n$$

\[ \vdots \]

$$q_m = a_1^m g_1 + a_2^m g_2 + \ldots + a_n^m g_n$$

…….(3)

It should be noted that $m$ in the above equation corresponds to the largest mass number at which any of the $n$ gas species being considered can contribute to the partial pressure. It should also be noted, that there may be certain mass numbers at which none of the gases would contribute. The maximum number of equations is therefore less than or equal to $m$. The above equation can be written in the matrix form as:

$$A_{m \times n} \times G_{n \times 1} = Q_{m \times 1}$$

…….(4)

Where, $A_{m \times n}$ is the fragmentation ratio matrix, $G_{n \times 1}$ is a vector of the apparent partial pressures of the constituent gases and $Q_{m \times 1}$ is the column matrix of the mass spectrum from the PPA. Finally the gas concentration can be estimated by matrix operation of (multiply, transpose and inverse) of equations 2. The flow chart of the fortran program is shown in fig. 4. This is applied to estimate the partial pressure of different gas molecules during graphite tile baking as discussed in the next section.
Discussion and Conclusions

As is evident from the spectrum obtained from RGA during the graphite tile conditioning, the dominant species are water vapour, Nitrogen and Oxygen. The following observation can be made from these mass spectra.

i.) Iso-propyl alcohol (2-Propanol) is the major constituent at room temperature (RT) as the tiles were cleaned in ultrasonic bath in Iso-propyl alcohol solution.

ii.) Water is not bound strongly and decreases rapidly with time at all temperatures. It is generally the dominated desorbing species during a bakeout. The peak height decreases with the time.

iii.) Carbon monoxide is the major constituent above 600 ºC. The relative intensity of CO and CO₂ increases with temperature.

The new method described above has been tested on a number of spectra obtained during the Limiter graphite tile baking for SST-1 Tokamak. This spectrum is then used to find the relative concentrations using the matrix method. The method proposed is essentially solving a set of linear equations using matrix algebra. It is straight forward to calculate PP of the residual gases using this method. Fig. 5
shows the PP of gas molecules obtained from this matrix method. SRS RGA also estimates the PP of few gas molecules. In RGA we have to pre-define the gas molecules whose PP to be estimated. Whereas, in matrix method the PP of gas molecules are estimated from the peaks obtained in the mass spectra. Fig. 6 shows the comparison of PP of some gas molecules obtained from matrix method and SRS RGA.

Figure 5. Partial pressures of different gas molecules during graphite baking
| Temperature | Partial Pressure (Torr) | Code_RT | SRS_RT |
|-------------|------------------------|---------|--------|
| Room Temp   | 0.00E+00               |         |        |
| At 200 C    | 0.00E+00               |         |        |
| At 500 C    | 0.00E+00               |         |        |
| At 700 C    | 0.00E+00               |         |        |
| At 800 C    | 0.00E+00               |         |        |

**Figure 6.** Comparison of partial pressures obtained from code and SRS RGA

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