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Achieving ultrahigh vacuum in an unbaked chamber with glow discharge conditioning

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Abstract. Glow discharge conditioning (GDC) has long been accepted as one of the basic wall conditioning techniques for achieving ultrahigh vacuum in an unbaked chamber. As a part of this fundamental experimental study, a test chamber has been fabricated from stainless steel 304 L with its inner surface electropolished on which a detailed investigation has been carried out. Both helium and hydrogen gases have been employed as discharge cleaning medium. The discharge cleaning was carried out at 0.1 A/m^2 current density with working pressure maintained at 1.0×10^{-2} mbar. It was experimentally observed that the pump-down time to attain the base pressure $\sim 10^{-8}$ mbar was reduced by 62% compared to the unbaked chamber being pumped to this ultimate vacuum. The results were similar irrespective of whether the discharge cleaning medium is either hydrogen or helium. It was also experimentally established that a better ultimate vacuum could be achieved as compared to theoretically calculated ultimate vacuum with the help of discharge cleaning.

Keywords. Ultrahigh vacuum; glow discharge conditioning; hydrogen; helium.

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1. Introduction

An ultimate vacuum in any leak-tight chamber with a given pumping speed and conductance is limited due to the outgassing from the inner walls of the chamber. The ultimate vacuum is rapidly achieved when the outgassing gas load gets progressively decreased. As the outgassing rate of a material is inversely proportional to time, high-temperature bake-out of the chamber walls for long duration is usually employed for achieving the ultimate vacuum. This however is difficult for large and complicated systems like magnetic fusion devices [1–5] equipped with a lot of in-vessel diagnostic apparatus and temperature-sensitive in-vessel components. Now-a-days, fusion devices are double walled with cryostat which also complicates the baking scheme. Thus, adoption of high-temperature baking is not always feasible in such devices. Additionally, there exists a permissible limit of the vessel's thermal expansion-induced stresses. Further, uneven baking may also result in steep temperature gradient and unacceptable thermal stresses.

Stainless steel is the most commonly used material for fabricating ultrahigh vacuum (UHV) chambers. It has been observed that when a stainless steel chamber is exposed to atmospheric pressure, water vapour gets sorbed onto its surface by physisorption and chemisorption in the form of several monolayers. Water vapour dissociates into hydrogen (H_2) and oxygen (O_2) under suitable conditions and then reacts with carbon, which is the principal surface contaminant in stainless steels, to produce CH_4, CO and CO_2 at the surfaces. These contaminants appear as major traces in the residual gas spectra of an unbaked stainless steel chamber. Thus, the UHV cannot be achieved and the impurities like oxygen and water vapour could not be brought down to the certain desired value despite continuous pumping. Such situations necessitate alternate processes that enable achieving the ultimate vacuum without baking for a definite period. Glow discharge conditioning (GDC) is one of the most widely adopted techniques for wall cleaning and it can be carried out using gases like hydrogen, helium and argon. Argon gas is ruled out

because it has higher sputtering yield which may lead to sputter coating of optical windows used for many diagnostic purposes especially in Tokamak devices. In hydrogen GDC, high chemical reactivity of the atomic hydrogen causes reduction of metal oxides and hydrogenation of carbonaeceous deposits to form volatile species which are desorbed from the surface both thermally and by particle impact and are evacuated from the device by the pumping system. In the case of helium GDC, the removal of impurities is not due to chemical reaction but due to ion-induced desorption and physical sputtering. These two gases are considered for their effect on wall conditioning in the present experimental investigation. Due to the wall surface interaction of hydrogen and helium, the adsorbed gas molecules gets reduced which in turn reduces the outgassing rate. This paper describes various physical effects of hydrogen and helium gases towards attaining ultimate vacuum in an experimental chamber.

2. Experimental set-up

The experimental set-up for the GDC under the present investigation is shown in figure 1. The vacuum chamber was fabricated from an electropolished 150 CF cylindrical tube of stainless steel (SS) 304 L material with a surface area of 0.93 m^2 exposed to vacuum. A turbomolecular pump (TMP) of 500 l/s (N_2 gas) was connected through a 150 CF gate valve and an aperture valve. In closed condition, the conductance of the aperture valve is 2.0 l/s (N_2 gas). This aperture valve protects TMP from high gas loads during the GDC experiments. A nude BA gauge and a residual gas analyser were connected at the different ports of this chamber. The chamber was also equipped with a combined gauge to monitor the hydrogen/helium gas base pressure for GDC. During GDC, gas was fed into the chamber using sapphire gas dosing valve.

A variable DC power supply of 0–1000 V was connected in series through a load resistor of 100 Ω to the SS anode and the vacuum chamber. The vacuum chamber was grounded electrically so that it will act as cathode for the glow discharge. A 35 CF glass window was provided to continuously monitor the glow during experiment. Double Langmuir probe was installed for measuring the density and the temperature of the discharge during the GDC. An additional valve was also provided for air and dry nitrogen gas purging into the vacuum chamber so that its condition could be altered for experimental purposes. PXI-based data acquisition was installed to continuously measure and store the experimental data for further analysis.

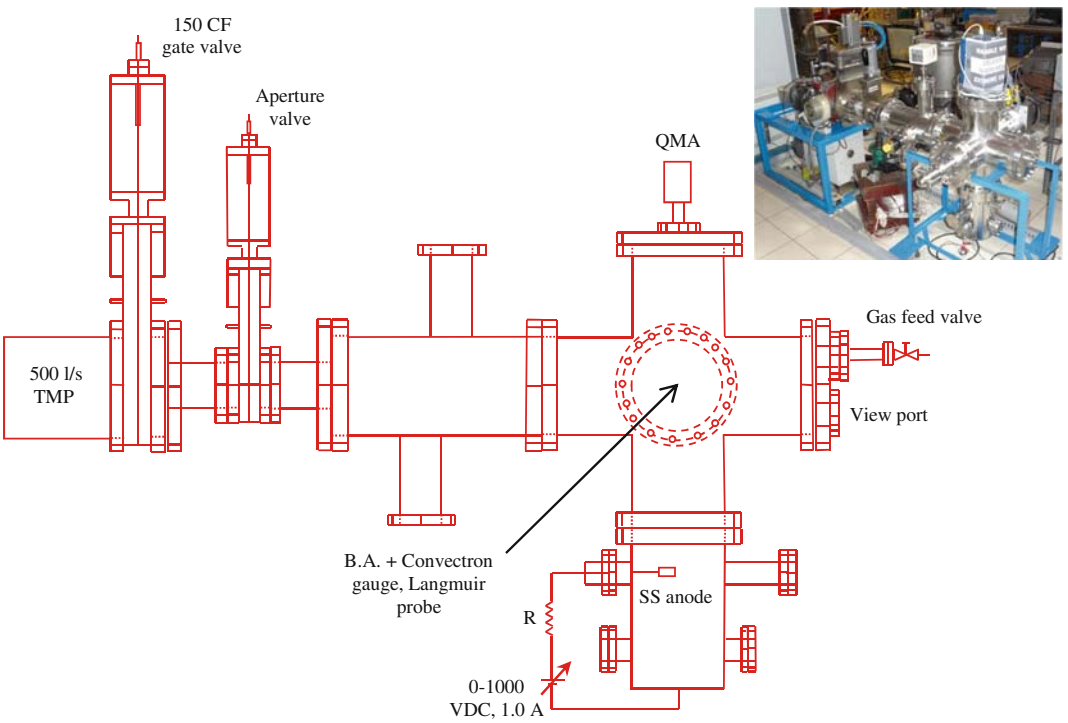

Figure 1. Schematic view of the experimental set-up for the UHV study.

3. Experimental method and results

Before installation, the vacuum chamber was cleaned with tap water to remove gross contamination. Thereafter it was properly cleaned with mild nitric acid (HNO_3) solution followed by a detergent. Finally, it was cleaned with demineralized water and then dried by blowing atmospheric air. After the complete installation of all the components, the vacuum chamber was leak tested in vacuum mode to ensure leak tightness to be $\leq 5.0 \times 10^{-9}$ mbar l/s at each joint and weld locations. Initial pumping of the vacuum chamber was then carried out till the expected ultimate vacuum was reached. Since the outgassing rate of the electropolished SS is 1.3×10^{-5} mbar l s^{-1} m^{-2} [6] and the effective pumping speed of the system is 110 l/s when the aperture valve is fully opened, the estimated ultimate vacuum is expected to be 1.1×10^{-7} mbar. The vacuum chamber was pumped from the atmospheric pressure and an ultimate vacuum of 8.2×10^{-8} mbar was achieved after 145 h of pumping. A residual gas analyser (RGA) spectra acquired during the initial pump-down from atmospheric pressure is shown in figure 2 indicating that the major contribution comes from H_2O (18 a.m.u.). The presence of CH_4, CO and CO_2 can also be seen at mass numbers 16, 28 and 44 respectively. The vacuum chamber was once again exposed to atmospheric condition for a week and then pumped down to 1.0×10^{-6} mbar which was usually achieved within 4 to 5 h of pumping. As soon as this pressure was achieved, the chamber was filled with helium gas up to 1.0×10^{-2} mbar. The anode voltage was increased slowly till the glow discharge breakdown was initiated. At 500 VDC and a current of 1.0 A, i.e. at the current density of 0.1 A/m^2, the glow discharge was found to be stable. This discharge was carried out for 5 h [7] and then the pump-down was carried out till an ultimate vacuum was achieved. During glow discharge, the glow temperature and density were measured using a double Langmuir probe. The electrotemperature of 2–3 eV and density between 1 and 5×10^{14}/m^2 were obtained during GDC. I–V characteristic of the double Langmuir probe is shown in figure 3.

Further, the similar procedure of air exposure of vacuum chamber was carried out and then GDC using hydrogen gas was carried out for the same time period. After hydrogen glow discharge, the vacuum chamber was pumped down till ultimate vacuum was achieved. The comparison between the ultimate vacuum achieved after helium and hydrogen gas glow discharge is shown in figure 4 with respect to the total

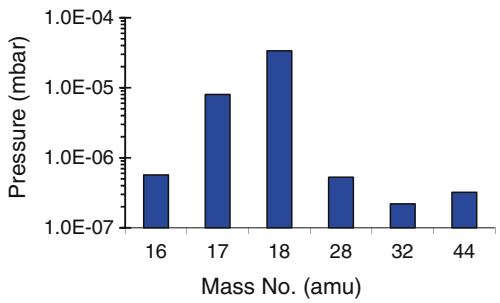

Figure 2. RGA spectra during initial pump down.

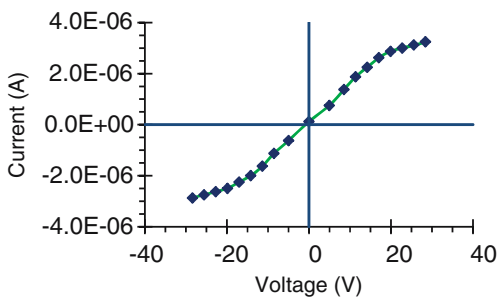

Figure 3. I–V characteristic of Langmuir probe.

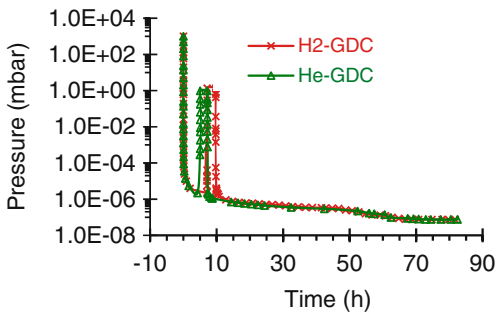

Figure 4. Ultimate vacuum with respect to time for helium and hydrogen glow discharges.

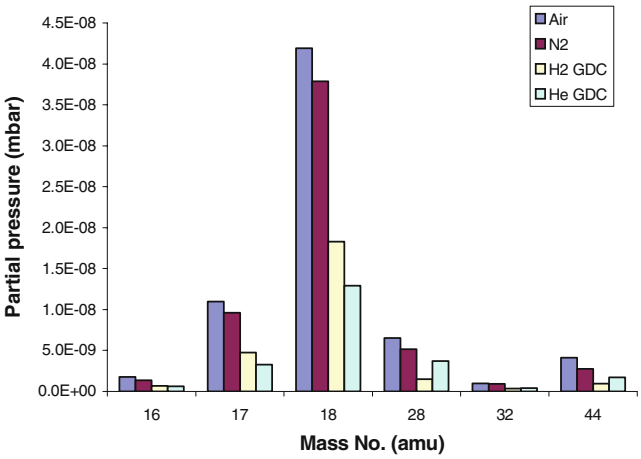

Figure 5. Partial pressure of different gases during different process.

measuring time. Figure 4 shows that an ultimate vacuum of 7.6×10^{-8} mbar was achieved after pumping for 71 h and 69 h in the case of both helium and hydrogen discharge cleaning. It is evident from the data that the ultimate base pressure is improved by 15% while the pump-down time is reduced by 62%. During all the four processes, residual gas analyses were carried out to know the effect on different gases. Figure 5 shows the partial pressure of individual gases during different processes. It is clear from the data that the main residual element within the system is water, the removal of which leads to the improvement of the ultimate vacuum and reduction in pump-down time.

4. Conclusion

Experimental evidence shows that in the case of air vented system with glow discharge conditioning, the pump-down time is reduced by 62% while ultimate vacuum is improved by 15% irrespective of whether helium or hydrogen gas is used for discharge cleaning in the test chamber. From these results we conclude that a fast pump-down of a stainless steel vacuum chamber to a pressure of $\sim 10^{-8}$ mbar can be done without high-temperature bake-out by hydrogen/helium gas glow discharge conditioning. In addition to discharge cleaning, an additional cryopump will help to achieve ultrahigh vacuum in an unbaked vacuum system where hydrogen and water vapour are dominant outgassing species.

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