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GLOW DISCHARGE TECHNIQUES FOR CONDITIONING HIGH VACUUM SYSTEMS[†]

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ABSTRACT

A review is given of glow discharge techniques which are useful for conditioning vacuum vessels for high vacuum applications. Substantial development of glow discharge techniques has been done for the purpose of in-situ conditioning of the large ultrahigh vacuum systems for particle accelerators and magnetic fusion devices. In these applications the glow discharge treatments remove impurities from vessel surfaces in order to minimize particle-induced desorption coefficients. Cleaning mechanisms involve a mixture of sputtering and ion- (or neutral) induced desorption effects depending on the gas mixture (Ar/O_2 vs. H_2) and excitation method (DC, RF, and ECR). The author will review the methodology of glow discharge conditioning, diagnostic measurements provided by residual gas and surface composition analysis, and applications to vessel conditioning and materials processing.

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1. Introduction

The ability of a glow discharge to enhance the chemical reactivity of the constitutive gases has given rise to numerous technical applications. Materials processing techniques involving glow-discharge-enhanced deposition¹ or etching^{2,3} processes have become essential for microcircuit fabrication. The subject of this review concerns a less well-known application of glow discharges: the cleaning and surface modification of vacuum vessels. Glow discharge cleaning (GDC) techniques have been developed primarily for the surface preparation of large vacuum vessels used with accelerator and magnetic fusion devices. However, GDC techniques are not limited to these historical applications and are useful for the surface preparation of any size vacuum vessel or constituent vacuum components used in ultrahigh vacuum (UHV) or vacuum systems requiring extreme cleanliness.

In this review the historical development of GDC techniques is briefly described (Section 2). In Section 3, typical experimental arrangements are described. The major part of the review (Section 4) is focused on the results of basic GDC studies and example applications from magnetic fusion devices and storage rings. These studies used quantitative residual gas and surface analysis measurements that illustrate the basic mechanisms and effectiveness of surface cleaning when GDC is applied to commonly used vacuum materials.

2. Historical Development of GDC

The use of glow discharges for surface cleaning can be traced to studies by Holland⁴ for the cleaning of glass and as an alternative technique to wet chemical cleaning for the preparation of substrates for thin film deposition.⁵ The first quantitative investigations of the use of glow discharge cleaning as a vacuum vessel conditioning technique were presented in

studies by Govier and McCracken⁶ (1970), Lambert and Comrie⁷ (1974), and Jones et al.⁸ (1974). The Govier and McCracken work involved measurements of the effect of GDC on vessel outgassing after a stainless steel vessel was subjected to DC-glow discharges in Ar, Ne, and He. Large changes in outgassing are not often observed as a result of GDC unless the measurements are mass resolved. The Govier and McCracken study did show that inert gas discharges did remove H₂O, CO, and CO₂ from the vessel, and in addition, that the discharge current is distributed fairly uniformly to the vessel wall in the experimental arrangement that is now viewed as standard for vessel conditioning: introduction of one or more electrodes within the vessel, which serve as the discharge anode, and use of the vessel walls as the cathode. This arrangement subjects the vessel wall to the ion bombardment which, as the later studies to be described have shown, removes surface and near-surface impurities from the vessel.

The GDC technique was given its first wide exposure to the vacuum technology community with the development of Ar and Ar/O₂ GDC techniques⁹⁻¹² for the surface treatment of the first of several large proton storage rings at CERN in the mid-1970's. The techniques developed for the Intersecting Storage Ring (ISR) at CERN were later adopted for use in other more recently constructed storage rings.¹³⁻¹⁵ Storage rings have several unique and stringent vacuum requirements. The operating pressure when the machine is in operation must be in the UHV range (typically less than 10^{-10} Torr) in order to minimize beam particle loss due to gas phase scattering. Beam lifetimes of the order of hours are required for storage rings. In electron and positron storage rings the vacuum chamber is subjected to intense synchrotron radiation, resulting in desorption of neutral gas from the surface. The beam

lifetime τ_B due mainly to bremsstrahlung on the residual gas nuclei can be expressed as

$$\tau_B \sim \frac{X_0}{MP} \quad (1)$$

where P is the gas pressure and M the molecular weight of the gas. In addition to the requirement of a low operating pressure on proton storage rings, ion-induced desorption coefficients¹⁷⁻¹⁹ on the vessel wall must be minimized in order to eliminate a beam-surface instability¹⁰ which can quench the beam. The requirements of UHV operating pressures and low photon-, electron-, and ion-induced desorption coefficients, combined with the impracticality of providing high linearly distributed pumping speeds in long conductance limited vacuum vessels, have forced the development in this field of effective surface cleaning techniques.

The development of GDC techniques in magnetic fusion research²⁰⁻²³ was also driven by a technical requirement to minimize particle-induced desorption coefficients and a practical requirement to find a cost-effective means of surface cleaning very large surface area vessels. In order to minimize radiation losses and hydrogenic depletion in high temperature fusion plasma experiments, nearly pure hydrogenic plasmas are desired. A figure of merit used in the field is the effective charge or Z_{eff} of the plasma defined as:

$$Z_{eff} = \frac{\sum \bar{n}_i Z_i^2}{\bar{n}_e} \quad (2)$$

where $\bar{n}_e$ is the average plasma electron density and $\bar{n}_i$ is the average plasma ion density for species i with atomic charge Z_i . With the widespread use of hydrogen discharge cleaning techniques²¹ in the period of 1975-1980, including

DC- and RF-driven glow discharges and pulse discharge cleaning techniques using the intrinsic tokamak magnetic systems, the attainment of low Z_{eff} plasmas became commonplace. Because of the strong dependence of Z_{eff} on the charge of the ion species, hydrogen became the gas of choice for discharge cleaning fusion devices. Early studies in the second-generation Soviet tokamaks^{24,25} focussed on inert gas GDC. However, problems with residual inert gas outgassing and the subsequent demonstration^{21,23} of the effectiveness of hydrogen GDC for the removal of the primary offending surface contaminants (C and O) led to the predominant use of hydrogenic gases as the reactant. Basic studies of the effects of hydrogen GDC were carried out by the author and his colleagues at Princeton^{20,21,26} for the conditioning of the large (36 m³) stainless steel vessel for the PDX tokamak (1978). The important results of these studies are summarized in Section 4. Similar studies involving small-scale lab tests and full-scale tests on the TFR²⁷ and TEXTOR^{23,28-31} tokamaks were performed by members of the Institute of Plasma Physics at KFA-Julich. This group pioneered the use of RF-assisted glow discharges for fusion vessels, which has the advantage over simple DC-driven discharges of lower pressure operation and higher impurity removal rates. A third general type of glow discharge, microwave driven at ECR (electron-cyclotron resonance) frequencies, was demonstrated by Sakamoto *et al.* on the JFT-2,³² JIPP T-II,³³ and TEXTOR³⁴ tokamaks and has the advantage of lower metallic sputtering because of the low plasma potentials of the ECR discharge.

In addition to the investigations cited above, studies of glow discharge cleaning of magnetic fusion devices have been published on the ASDEX,²² DIII,³⁵ DITE,³⁶ and HT-6B³⁷ tokamaks, and the TMX-U³⁸⁻⁴⁰ and GAMMA-10⁴¹ mirror machines. Of particular noteworthiness is the successful application of GDC for the initial cleaning of the very large vessels (~ 100 m³) for the

TFTR^{42,43} and JET^{44,45} tokamaks. A variation on the use of pure hydrogenic gases was the development of glow discharge carbonization schemes for tokamaks.⁴⁶ The technique involves the use of CH_4 -seeded H_2 discharges and results in the deposition of hard amorphous carbon films on the vessel surfaces. These films have certain advantages relative to shielding metallic vessel walls from sputtering during subsequent exposure to high temperature fusion plasmas.

With the increasing use of graphite first-wall structures on tokamaks (such as in TFTR, JET, and JT-60, where the graphite coverage is greater than 50%), the applications of GDC have evolved. Hydrogenic GDC is still employed for the initial cleanup of the vessel following atmospheric exposure. However, H_2 GDC of graphite results in a hydrogen-saturated near-surface region. When this condition is not desired, helium discharge techniques⁴⁷ are used to desaturate the carbon surfaces. The use of helium or hydrogen/deuterium glow discharges are planned for exchanging tritium from first-wall structures after the first use of tritium-fueled plasmas in TFTR.⁴⁸

Much of the reported work on GDC as noted above has concerned the cleaning of large vessels. However, the technology and benefits can be applied to any size vacuum system. At Princeton Plasma Physics Laboratory, GDC techniques are routinely used to condition small vessels (≤ 100 liters) when rapid removal of adsorbed H_2O is required, and when vessel surfaces or internal components have been contaminated by hydrocarbons.⁴⁹ The following sections describe some GDC techniques and elucidate the quantitative benefits of using them.

3. Experimental Arrangements

A typical experimental arrangement for glow discharge cleaning of a vacuum system involves one or more electrodes positioned within the vessel, depending on the size and geometry of the vessel. The electrode is powered by an excitation source which produces the glow discharge. The vessel structures to be cleaned (primarily the vessel wall) are grounded, as is the low potential side of the excitation source. The excitation source can be a DC power supply (600-1000 V), an RF power supply (200 kHz or 13.6 MHz are commonly used), a microwave power supply (1-3 GHz), or in some cases a parallel combination of DC and high frequency sources. The simplest arrangement is the DC system where the powered electrode (anode) can be a paddle or bar-shaped conductor sized to take the full current of the discharge without overheating. Anode structures have been designed on retractable probe assemblies or as permanently mounted fixtures in appendage ports of the vacuum vessel. This choice depends on the frequency of up-to-air cycles for the vessel and the desire to produce uniform discharge current distributions with the minimum number of anodes. In DC-powered systems a load resistor is usually put in series with the anode (unless a current-limiting power supply is used) in order to limit the current drawn by unipolar arcs which are quite common in the initial stage of glow discharge treatment of dirty surfaces. One of the serendipitous benefits of glow discharge cleaning is the arc conditioning which is observed during discharge exposure. The frequency of unipolar arcing typically decays exponentially as arc initiation sites are removed.^{20,23} Arc conditioning is a particular benefit if the vessel will be exposed to some plasma process following the cleaning procedure.

The use of high-frequency excitation sources for producing the glow discharge usually requires a more complicated excitation electrode; however,

the benefits may justify the additional complication for certain applications. For RF excitation the electrode typically consists of a coil structure with sufficient inductance to couple power into the discharge when an appropriately tuned matching circuit is used between the electrode and RF power supply. For ECR excitation the electrode structure is an antenna capable of propagating the applied frequency into the discharge, and can vary in complexity from a probe antenna coupled to the waveguide which delivers the power from the excitation source, to more sophisticated phased array antennae. ECR excitation requires the additional complication of an external magnetic field applied to the excitation volume. (For magnetic fusion devices the in-place magnetic systems are operated at steady-state, low field values for ECR propagation.)

Examples of GDC systems from two of the more well-known examples in the literature are shown in Figs. 1 and 2. Figure 1 shows the combined DC/RF-powered electrode system²³ used as a prototype for the cleaning systems installed in the TEXTOR³⁰ and JET⁴⁴ tokamaks. This electrode arrangement, a so-called "RG discharge" by the developers, has the advantage of operating at approximately twenty times lower pressures ($\sim 1 \times 10^{-3}$ Torr) than a comparable DC-only excited discharge. Figure 2 shows the rather simple electrode arrangement used at CERN^{12,15} for GDC treatment of individual segments of storage ring vacuum vessels prior to final assembly of the accelerator. The anode structure is simply a wire suspended down the center of the tubular vessel.

In addition to the electrode structure and excitation source, important experimental variables for a GDC system include specifications for the gas type, gas pressure, gas throughput (or pumping speed), and recommended ion dose to the vessel wall. Gas pressures have spanned the range 5×10^{-4} to $1 \times$

10^{-2} torr depending on gas type, excitation source, and electrode structure. For a fixed power input the ion flux to wall is not a strong function of the discharge pressure (see Section 4); however, the system pumping speed is usually a strong function of pressure. It is important for the removal of volatile contaminant species produced by the discharge to maintain a high gas throughput. Turbopumps are the pumps of choice for GDC because of their high pumping speeds for the usual GDC gases (Ar or H_2) and because of the constant pumping speed vs. throughput. With the use of turbopumps, glow discharges operating in the pressure range of $(1-5) \times 10^{-3}$ torr will maximize both the throughput of the fill gas and the pumping speed (and thus removal from the system) of the reaction products.

For H_2 GDC vessel current densities of $10-25 \text{ } \mu\text{A}/\text{cm}^2$ are typical,^{21,23} whereas somewhat higher current densities of $100 \text{ } \mu\text{A}/\text{cm}^2$ have been used in the Ar GDC applications (Ref. 11). The required ion dose (i.e., product of discharge current density and discharge time) for optimal cleanup of vacuum system contaminants is system dependent. However, the results of gas and surface studies described in the next sections have shown that ion doses in the range of $10^{18} - 10^{19} \text{ ions cm}^{-2}$ are necessary for cleanup of stainless steel vacuum systems.

4. Residual Gas and Surface Analysis Studies

Hydrogen Glow Discharge Cleaning

Quantitative residual gas analysis of the effects of hydrogen glow discharge cleaning have been carried out on a number of magnetic fusion devices,²¹ and in several of these experiments concurrent surface analysis measurements were performed.^{20,26,50,51} The combination of both measurement techniques has served to elucidate the basic mechanisms responsible for the

removal of surface contaminants. The residual gas analysis (RGA) measurements have been the most useful measurements for optimizing the discharge characteristics and monitoring the process since the actual production of volatile products (averaged over the entire vessel surface area) is measured directly. Actual quantitative measurement of the volatile reaction products of a glow discharge with an RGA is a non-trivial undertaking. Because of the relatively high operating pressures of the glow discharge, the RGA must sample the discharge products through a differentially pumped orifice.⁵² Particular attention must be paid to calibration of such a system,⁵³ and to the minimization of background signals^{54,55} resulting from the relatively high pressures of the discharge gas (H_2) in the detector chamber.

Figures 3-5 show examples of the residual behavior observed for H_2 discharge cleaning of stainless steel vessels. Figure 3a shows the observed time dependence of the primary residual gases produced during the initial discharge cleaning of the large (36 m^3) PDX torus, and 3b shows a subsequent cleaning run after substantial internal hardware was added to the vessel.²⁰ During both cleaning runs the vessel was at ambient temperature ($\sim 22^\circ\text{C}$) and the dominant residual gases produced were CH_4 , CO , and C_2H_4 . Subsequent studies²³ have shown that the H_2O production is small at room temperature, and have explained why no H_2O production was observed in the PDX experiments above the background levels in the RGA. Figure 4 shows the residual gas behavior observed for two vessel temperatures for the initial cleaning⁴² at $\sim 20^\circ\text{C}$, and a subsequent run⁴³ at 150°C for the large (86 m^3) stainless steel vessel for the TFTR device. In the TFTR experiments the RGA background levels for H_2O were reduced compared to the PDX measurements, and the production of H_2O was observed for both the ambient and higher temperature cleaning runs.

The data of Fig. 4 suggest a strong temperature dependence for the H_2O production. This dependence has been examined in detail by Noda *et al.*⁵⁶ and Waelbroeck *et al.*^{23,24} The work of Noda *et al.*⁵⁶ on the JIPP-TII torus is shown in Fig. 5 where the time and temperature dependence are shown for H_2O production during the application of a combined ECR and ac discharge. These data are plotted in an Arrhenius form in Fig. 6 along with the measurements obtained by Waelbroeck *et al.* from H_2 glow discharge cleaning of a relatively small (~ 100 liter) stainless steel test chamber. From the slopes of the Arrhenius plots, experimental activation energies for the rate-limiting reaction step are obtained. The results: 1.6 kcal/mole for the Noda *et al.*⁵⁶ data and 5.6 kcal/mole for the Waelbroeck *et al.*²³ data are rather low for a chemical process and are more characteristic of activation energies characteristic of diffusive processes.⁵⁷

In our original glow discharge studies in the PDX torus,²⁰ we argued that much of the residual gas data (Fig. 3) tended to show an approximate $1/t$ time dependence similar to the observations from simple thermal outgassing from steels. In the classic analysis of the latter situation given by Dayton,⁵⁸ outgassing of a single species (such as H_2O) from steels does not show a $t^{-1/2}$ dependence that would be expected if the outgassing were rate-limited from a simple diffusion process from the bulk metal. Because the surface of stainless steel is covered with a passivation oxide layer,⁵⁹ which allows a range of characteristic diffusion energies, D_a , Dayton shows that the summation of the D_a values leads to an overall $1/t$ dependence.

Recently Dimoff *et al.*⁶⁰ have reexamined the residual gas data shown in Figs. 3-6 in terms of fitting the time dependence to a general diffusion equation. Generally, Dimoff *et al.* find that the data can be fit to solutions of the diffusion equation (Fick's law) with two adjustable parameters: γ_e and

α^2/D ; where v_e is the system pumping speed and α^2/D characterizes the surface emissivity (α) and diffusivity (D). According to authors,⁶⁰ their model fits a wide range of data from quite different devices, accommodating a range, $\delta = 0.7 - 2.0$, of observed power law time dependences ($t^{-\delta}$) for the decay of RGA signals. Dimoff *et al.*⁶⁰ conclude that the rate limiting step for C and O volatilization in a H_2 glow discharge is a diffusion step.

In addition to providing basic reaction rate data, the residual gas analysis of glow discharge conditioning has provided data on the power and/or flux dependence of the volatilization reactions. Such information is useful for optimizing and scaling the cleaning process to various size vessels. For a given system and pumping speed for the volatile reaction products, the effectiveness of the cleaning process would be maximized by maximizing the partial pressure of the product. One can write a particle balance equation for the process which helps to illustrate the mechanisms which contribute to the increase or decrease of reaction product partial pressure. Because of the importance of oxygen as a primary impurity in fusion devices with steel vessels, this particle balance analysis has been applied most often to the kinetics of H_2O formation in the presence of hydrogen discharges. The rate of change of the H_2O partial pressure in a vessel with volume, V , can be written as

$$V \frac{dP(H_2O)}{dt} = (k_s - k_a) - k_i - S P(H_2O) \quad , \quad (3)$$

where k_s is the surface reaction rate for the formation of H_2O due to hydrogen (ion and atom) bombardment of the vessel wall; k_a is the rate of readsorption; k_i is the rate of ionization in the glow discharge; and the removal rate of H_2O from the vessel (i.e., the cleaning rate) is given by the product of the pumping speed, S , and $P(H_2O)$.

A useful and practical limiting solution for Eq. (3) occurs when the readsorption rate is small (i.e., high temperature walls), the reionization rate is small (i.e., low electron temperature discharge) and the system pumping speed S_p is large compared to k_a and k_i . In this limit and under quasi-steady-state conditions, the partial pressure of H_2O is proportional to the H-flux (r_H) to the vessel wall since k_s is proportional to r_H :

$$P(H_2O) = \frac{k_s}{S_p} \sim \frac{r_H}{S_p} \quad (4)$$

Figures 7 and 8 are examples of systems where the linearity of Eq. (4) is demonstrated. Figure 7 shows the dependence of $P(H_2O)$ in the JIPP-TII cleaning experiment⁵⁶ as a function of the discharge plasma electron density, n_e . (The hydrogenic ion flux to wall is proportional to plasma ion density which equals n_e .) The departure of the Fig. 7 data from linearity at higher n_e values occurs because H_2O ionization rates in the discharge are becoming important. This effect was discussed qualitatively by Oren and Taylor in their paper⁶¹ which first documented the importance of low temperature H_2 discharges for the removal of H_2O from steel vessels. A quantitative explanation of the H_2O ionization effects is given by Noda *et al.*⁵⁶ in their analysis of the data shown in Fig. 7, and a similar analysis was given by Waelbroeck *et al.*²³ in their analysis of the data shown in Fig. 8. In Fig. 8 the H_2O partial pressure in a H_2 glow discharge is plotted vs. discharge current for two different pumping conditions (with and without an appendage liquid nitrogen trap). Waelbroeck *et al.*²³ show that the H-atom flux to the wall is proportional to the discharge current. Surface probe measurements⁶² confirm that the hydrogenic ion (H^+ , H_2^+) flux to the wall is also proportional to the discharge current. For the data shown in Fig. 8 the discharge current

range is sufficiently small that H_2O ionization rates are small; however, only in the case shown with the appendage cryopump is linearity of pressure vs. discharge current maintained because the system pumping speed is sufficiently large compared to readsorption rates.

Surface Analysis Data

Further reaction kinetics data and basic information on reaction mechanisms have been obtained through in-situ surface analysis of steel samples exposed to H_2 glow discharges. These data were obtained through the use of unique experimental arrangements^{63,64} that allowed a sample of wall material to be translated from a vessel wall position to an analysis chamber without an air-exposure. Figure 9 shows changes in elemental surface composition of stainless steel² and Inconel⁵⁰ samples as a function of exposure to H_2 discharge cleaning. For clarity only the changes in the primary surface contaminants (C and O) are shown. Both samples are initially covered with the usual carbonaceous contamination that is characteristic of little or no surface treatment. Discharge cleaning effectively removes this layer to submonolayer levels, reducing the surface carbon concentrations to ~ 30 atomic percent with exposure times of an hour. The initial rate of carbon removal is 3×10^{12} C-atoms $cm^{-2} s^{-1}$ for an estimated H^+ flux of 1×10^{13} H^+ $cm^{-2} s^{-1}$ (PDX-case²¹). The removal rate of surface oxygen atoms is considerably slower: only after exposure of the passivation oxide from beneath the carbonaceous layer (after 1 hr.) is there a noticeable rate of oxygen removal of 3×10^{10} atoms $cm^{-2} s^{-1}$.

More sophisticated surface analysis by XPS and SIMS techniques of stainless steel samples exposed to H_2 discharge cleaning has provided insight into the processes involved in the reduction of the surface oxides.^{21,26,51}

XPS spectra show that only a partial reduction of oxide layer occurs during H_2 discharge cleaning: iron oxides (Fe_2O_3 and FeO) are progressively reduced to metallic Fe, whereas the chromium component of (Cr_2O_3) of the mixed oxide layer on steel is stable under low-energy hydrogen ion exposure.^{26,51}

This behavior is consistent with expected chemical stability of the various metal oxides on the basis of the equilibrium constants, $K_p = dP(H_2O)/P(H_2)$, for the reduction of these oxides in molecular hydrogen.⁶⁵ The flux of H atoms and ions from the discharge promotes the chemical reduction of the oxide. In most cases the energy of the incident hydrogen is near or below the sputtering threshold⁶⁶ (see below). This physical removal of the oxide is not an important process in H_2 discharge cleaning. With higher power discharges such that the impacting ion energy exceeds the sputtering threshold or with the use of more massive positive ions, D^+ or He^+ , sputtering effects can become important.

The surface analysis cited above supports a reaction model first proposed by Dietz *et al.*²⁸ for the reduction of steel oxides by H_2 discharges: The reaction first proceeds by hydrolysis of the metal oxide (MO):

followed by the subsequent reduction of the hydroxide to a free metal with the liberation of H_2O to the gas phase:

Assuming quasi-equilibrium for the forward and reverse reactions of Eq. (5), the rate equation for the production of H_2O can be written as

$$\frac{d}{dt} P(H_2O) = k [MO] [H]^2 \quad , \quad (7)$$

where $[MO]$ and $[H]$ represent the concentration of metal oxide and hydrogen in the near-surface region.

The presence of $FeOH$ is seen in both the XPS and SIMS spectra of H_2 -discharge-exposed steel surfaces,^{26,51} thus providing supporting evidence for the initial reaction step. Equation (7) can be put into a form useful for the overall H_2O balance equation given in Eq. (3) if we relate the hydrogen near surface concentration to the incident H-flux (r_H) by means of the recombination rate coefficient; k_r :

$$k_r = \frac{r_H}{[H]^2} \quad . \quad (8)$$

Thus, Eq. (7) now reads upon substitution of Eq. (8):

$$\frac{d}{dt} P(H_2O) = \frac{k}{k_r} [MO] r_H \quad . \quad (9)$$

Dietz et al.²⁸ and Waelbroeck et al.²³ have interpreted the observed saturation of H_2O production at wall temperatures $\sim 350^\circ C$ to be the result of the H-recombination on or within the oxide becoming rate-limiting.

The surface composition data shown in Fig. 5 suggest that neither the oxide removal nor the carbon removal proceeds to small values ($< 10^{-1}$ at %): at high fluences. The high fluence saturation of the oxygen surface concentration is due in part to the chemical stability of the chromium

component as shown above. However, the carbon saturation and possibly some fraction of the residual oxygen levels may be the result of a quasi-equilibrium which occurs in a real system at high fluence. The oxide layer on the vessel wall represents a large source of oxygen and carbon which can resupply the surface by diffusion. Another term can be added to the H_2O particle balance equation (Eq. 3) which represents the diffusive source, k_d :

$$A \frac{dn}{dt} = k_s + k_d - k_s \quad (10)$$

In this case the particle balance is written in terms of the change in the oxygen surface coverage (n), and the gas phase removal processes (k_i and SP) are ignored, so that a comparison can be made to surface composition data similar to that shown in Fig. 9. Figure 10 shows high fluence surface composition measurements taken with stainless steel samples exposed to thermal atomic hydrogen at several different sample temperatures.⁶⁷ In Fig. 11a, the oxygen coverage data shown in Fig. 10b are replotted along with a solution to Eq. (10) obtained with the appropriate boundary conditions.⁵⁴ Since the Fig. 10b measurements were taken at several sample temperatures, a value for the diffusion coefficient for O species could be extracted (Fig. 11b). The derived diffusion coefficient, $D = (0.4 - 1.2) \times 10^{-15} \text{ cm}^2 \text{ s}^{-1}$, indicates the order of magnitude necessary for diffusion to resupply the surface in order to maintain the observed (~ 5-10%) quasi-steady surface concentrations of C and O on H-bombarded surfaces. Secondly, the activation energy extracted from the Fig. 11b data ($\Delta E = 3.0 \pm 1 \text{ kcal/mole}$) is close to the activation energy derived by Dietz *et al.*²⁸ for the production of H_2O during H_2 discharge cleaning of stainless steel. This is further evidence for diffusion in the near-surface region being the rate-limiting step in H_2 discharge cleaning.

Energy Distributions of the Cathodic Flux for H_2 Discharges

As discussed above, one of the important advantages of H_2 glow discharges for cleaning applications is the chemical nature of the cleaning mechanism as opposed to the physical sputtering effects which dominate when heavier gases are used. There are many applications beyond the conditioning of fusion device vacuum vessels where glow discharge cleaning would be useful if sputtering effects could be minimized. For example, a vacuum system containing sensitive analytic instrumentation or a materials processing station would be jeopardized if the cleaning process transported metals from the vessel wall. In order to quantify this benefit, it is useful to look at actual metal sputtering measurements that have been done with H_2 glow discharges, and to examine the measurements of the cathodic flux energy distributions so that sputter yields can be accurately estimated.

Figure 12 shows the measurements of Mathewson *et al.*⁶⁸ of the energy distribution of (positive) ions arriving at the cathode (vessel wall) of a DC-driven glow discharge in H_2 . The measurements were made with a tandem 127° sector electrostatic analyzer - quadrupole mass analyzer, which sampled the ion flux effusing through a small (5 mm) aperture. Peaks in the ion energy distributions in Fig. 12 are present for the H^+ , H_2^+ , and H_2O^+ components of the ion flux. For these conditions (neutral pressure = 1.4×10^{-2} Torr, discharge current ~ 2.5 A), the molecular ion H_2^+ is the dominant peak at or near the anode potential, $V_p \approx 400$ V. When this ion impacts a surface, the energy would be equally apportioned to the two protons as the molecular ion dissociates upon impact. Therefore, each proton carries approximately $V_p/2 \approx 200$ eV of translational energy which is significantly below the sputtering threshold (~ 250 eV) for H^+ ions on steel surfaces.⁶⁶

A collaborative measurement⁶² of the cathodic flux in a H_2 glow discharge is shown in Fig. 13, which is a measurement of the implantation depth profile on a silicon target exposed at the vessel wall (cathode). The implantation profile can be unfolded to yield the impacting ion energy of the species hitting the target. Such unfolding has large uncertainties for the relatively small ion energies (< 400 V) and shallow depth profiles (< 10 nm) in this example. Nevertheless, the identified peak in the depth distribution is consistent with the peak proton flux corresponding to impacting ion energies of $\sim V_p/2 = 200$ V. It is also interesting to note that this result is independent of the neutral pressure over the range investigated (5-90 m Torr) probably because the ion mean-free-path is greater than the sheath dimensions for the entire pressure range. Therefore, other considerations can be used when choosing an operating pressure for H_2 discharge cleaning, such as the optimization of impurity pumping speeds.

A direct measurement of sputtering of metals from a vessel wall exposed to H_2 glow discharges has been made by Winter *et al.*⁶⁹ using the deposition probe technique.⁶² Figure 14 shows these results for deposition onto clean Si and Al samples as a function of H-atom fluence for exposure to both a pure H_2 and a $H_2/1\%$ CH_4 glow discharge. The dashed lines on the plot correspond to deposition that is strictly proportional to H-dose (i.e., no redeposition) and the slope gives a sputter yield for Inconel of $\sim 2 \times 10^{-3}$ atoms/incident ion for the (~ 225 eV) H^+ ions produced in an RF-assisted DC glow discharge. The sputter yield is increased by more than an order of magnitude with the addition of a small amount (1%) of a heavier gas (CH_4) in the discharge.

Argon and Ar/O₂ Glow Discharge Cleaning

Glow discharge cleaning techniques using argon and argon/oxygen gas mixtures have been developed largely by the vacuum groups associated with accelerators and storage rings, most notably the groups at CERN and Brookhaven. The need for glow discharge cleaning in accelerator applications parallels the need in magnetic fusion devices. Both applications require cost effective and efficient vessel conditioning techniques which can be applied to large area vacuum vessels in order to significantly decrease particle-induced desorption coefficients. For a number of reasons, glow discharge cleaning techniques applied to accelerators have emphasized first Ar discharge cleaning and then Ar/O₂ cleaning.

Historically, the early laboratory-scale studies of glow discharge cleaning emphasized noble gas discharges. The CERN group performed a large number of tests of various cleaning techniques on technical materials^{12,15,18} during the initial operation of the Intersecting Storage Ring (ISR). Included in these tests was an evaluation of argon discharge cleaning using a discharge arrangement such as shown in Fig. 2. The effectiveness was judged using a number of diagnostics including in-situ surface analysis and measurements of electron- and ion- (K⁺) induced desorption coefficients.

Figure 15 shows that for unbaked stainless steel surfaces, argon discharge cleaning (to a dose of $\sim 10^{18}$ cm⁻²) can reduce electron and ion desorption coefficients from initial values which are greater than one to final values in the range of 0.01 to 0.001. In-situ surface analysis measurements of discharge-cleaned samples showed that the surface carbon contamination was effectively removed after an ion dose of $\sim 10^{19}$ cm⁻². Rather than relying on purely physical sputtering to remove impurities from the vessel surfaces, the addition of O₂ enhances surface reactions forming CO

and CO_2 , thus accelerating the removal of surface carbon from the vessel.^{11,70} Figure 16 shows the integrated quantity of these volatile reaction products removed after the first and second application of Ar/O_2 discharge cleaning to a stainless steel vessel.¹⁵ As in the case of the quantitative measurements of H_2 discharges cleaning^{20,42} (Fig. 3, 4), the integrated removal of surface carbon and oxygen with the initial cleaning is of the order of 100 monolayers. It is also of interest to observe that the initial glow discharge treatment offers some degree of passivation with regard to subsequent air exposures. Figure 16b shows that the integrated impurity gas removal with the second cleaning after an air exposure is down by more than an order of magnitude compared to the initial cleaning (Fig. 16a).

Surface analysis of Ar/O_2 -cleaned metals shows an increased level of surface oxidation and minor amounts of implanted argon.¹⁵ The increased surface oxidation is apparently not a problem for storage ring applications because this enhancement does not seem to affect particle-induced desorption coefficients. Implanted argon can cause a problem in storage ring vessels that have small noble gas pumping speeds such as those devices designed with distributed non-evaporable getter pumps.¹³ In this case, a high temperature ($\sim 350^\circ\text{C}$) baking step is required prior to vessel use in order to degas the implanted argon.¹¹

O_2 and He/O_2 Discharge Cleaning

The majority of the discharge cleaning literature is concerned with the reactant systems described above: H_2 and Ar/O_2 (or Ar). The use of pure O_2 or He/O_2 glow discharges has received some attention in the literature because of the high surface carbon removal rates that can be generated in such discharges through the formation of CO and CO_2 reaction products. Pure O_2

discharges were examined in the cleaning studies in the PDX²⁰ and ORMAK⁷¹ tokamaks. The problems of increased surface oxidation, sputtering of the base metal, and possibly enhanced outgassing of oxygen-containing molecules outweighed any benefits of rapid carbon removal except in the cases of large hydrocarbon contamination. In the TEXTOR device, significant hydrocarbon contamination during the initial baking of the vessel was removed completely by a short (~ 2 hr.) exposure to a He/5% O₂ glow discharge.⁷² With careful monitoring with an RGA, the endpoint corresponding to complete removal of the oil could be detected and a switch-over to an H₂ glow discharge was performed for the final cleaning.

Pure O₂ discharges have also been applied very successfully to the problem of cleaning carbon contamination from the surfaces of optical components used in the UV and X-ray wavelengths.⁷³⁻⁷⁶

5. Summary

We have reviewed the various glow discharge techniques which have been applied to the cleaning and conditioning of vacuum vessels. Much of the development of the techniques and the understanding of the cleaning mechanisms has come from applications of H₂ glow discharge cleaning to magnetic fusion devices and Ar or Ar/O₂ glow discharge cleaning to storage rings. For both technologies the need for a cost-effective cleaning method for large surface area vessels was essential. Laboratory studies on small vessels and in-situ measurements on full-scale vessels have demonstrated that glow discharge cleaning can produce near atomically clean surfaces on technical materials with the consequence that particle-induced desorption coefficients are lowered by more than factors of 10³. Other benefits of glow discharge exposure include a decrease in vessel outgassing of common residual gas constituents

(hydrocarbons and H_2O) and unipolar arc conditioning which decreases the susceptibility of a vessel to subsequent arcing upon exposure to plasma processes. Although, much of the experience with glow discharge cleaning has been gained with large vessels, the techniques and the benefits can be applied to any size vacuum system.

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Figure Captions

- Fig. 1. Schematic diagram of the RF-assisted DC glow discharge system used for basic studies²³ and for design of the glow discharge cleaning systems used in the TEXTOR^{30,31} and JET^{44,45} tokamaks. The discharge is produced with an RF-biased coil (RFC) and a DC-biased auxiliary electrode (AE). See Ref. 23 for experimental details.
- Fig. 2. Schematic diagram of the DC glow discharge arrangement used for conditioning storage ring segments at CERN.⁹⁻¹¹
- Fig. 3. Residual gas behavior of the initial (a) H_2 glow discharge cleaning of the large (36 m^3) PDX vessel, and a subsequent (b) cleaning run after the addition of substantial internal hardware. For both runs the vessel was at ambient ($\sim 22^\circ\text{C}$) temperatures.²⁰
- Fig. 4. Residual gas behavior of the initial (a) H_2 glow discharge cleaning of the 86 m^3 TFTR vessel at ambient temperatures.⁴² (b) A subsequent cleaning run with the vessel at 150°C .⁴³
- Fig. 5. Behavior of the H_2O partial pressure, $P(H_2O)$, generated during H_2 discharge cleaning of the JIPP TII torus as a function of the vessel temperature.⁵⁶

- Fig. 6. Arrhenius plot of $P(H_2O)$ vs. the inverse wall temperature ($1/T_w$) for the JIPP-TII data⁵⁶ shown in Fig. 5, and as measured by Waelbroeck et al.²³ for H_2 discharge cleaning of a small stainless steel vessel. (The original plot of these data in Ref. 56 has an arithmetic error in the extracted activation energy which was corrected by Dimoff and Vijk.⁵⁷)
- Fig. 7. Dependence of $P(H_2O)$ vs. discharge plasma density in the JIPPT-II vessel.⁵⁶
- Fig. 8. Dependence of $P(H_2O)$ vs. discharge current for two different pumping conditions as measured by Waelbroeck et al.²³
- Fig. 9. Observed change in the elemental surface composition of hydrocarbon-covered stainless steel²¹ and Inconel⁵⁰ as a function of exposure to hydrogen discharge cleaning. (From Ref. 64 with permission.)
- Fig. 10. Surface composition data similar to Fig. 9 obtained by exposing a stainless steel substrate to a high fluence of thermal atomic hydrogen at several substrate temperatures. (From Ref. 67 with permission.)
- Fig. 11. (a) Logarithm of the relative oxygen coverage (n/n_o) minus the steady-state value (n_s/n_o) as a function of time for the oxygen coverage data of Fig. 10 at high hydrogen fluence ($\geq 1.5 \times 10^{19} \text{ cm}^{-2}$). (b) Values of the oxygen diffusivity obtained from the slopes of the fitted lines are plotted vs. $1/T$.⁵⁴

Fig. 12. Ion energy distributions of the cathodic flux of a DC-driven glow discharge in H_2 . The supply voltage (V_p) was - 400 V. The measurements were made on a 127° sector, electrostatic energy analyzer. (From Ref. 68 with permission.)

Fig. 13. Ion implantation depth profiles in a silicon target of the cathodic flux of a DC-driven glow discharge in H_2 , ($V_p = 390$ V). The depth profile was obtained by Secondary Ion Mass Spectrometry (SIMS). The peak of the depth distribution is consistent with a peak proton flux at ~ 200 V. The separate depth profiles correspond to different investigated discharge pressures (5-90 m Torr) at fixed discharge current. (From Ref. 62 with permission.)

Fig. 14. Deposited metal (Fe, Cr, Ni, and Mo components from the Inconel vacuum vessel) onto clean Si or Al samples exposed to RF-assisted DC glow discharges in H_2 and $H_2/1\% CH_4$. (From Ref. 46 with permission.)

Fig. 15. The effect of argon glow discharge cleaning on the electron and ion (K^+) desorption coefficients for stainless steel at ambient temperatures. (From Ref. 15 with permission.)

Fig. 16. (a) The composition and time dependence of the residual gases produced during the initial argon glow discharge cleaning of a stainless steel vessel. (b) Behavior following a second cleanup after an air exposure. (From Ref. 15 with permission.)

Fig. 1

Fig. 2

Fig. 3

Fig. 4

Fig. 5

Fig. 6

Fig. 7

Fig. 8

#81X0947

Fig. 9

Fig. 10

#8°X1161

Fig. 11

Fig. 12

#86X1822

Fig. 13

Fig. 14

Fig. 15

Fig. 16

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