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CONDITIONING OF ION SOURCES FOR MASS SPECTROMETRY OF PLASMAS

By

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CONDITIONING OF ION SOURCES FOR MASS SPECTROMETRY OF PLASMAS

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ABSTRACT

Mass spectrometry is a useful diagnostic technique for monitoring plasma species and plasma-surface interactions. In order to maximize the sensitivity of measurements of hydrogen-fueled fusion plasmas or hydrogen-based discharge cleaning and etching plasmas, the ion sources of mass spectrometers are operated at or near the high pressure limit of 10^{-4} Torr (10^{-2} Pa). Such high ambient pressures of hydrogen give rise to high background levels of residual gases such as H_2O , CO , and CH_4 , due to surface reactions on the ion source electrodes. For a commonly used ion source configuration, the residual gas production is a linear function of the ambient H_2 pressure. Hydrogen conditioning can reduce the absolute residual gas levels. Steady-state residual gas production is observed in a conditioned ion source, which is related to a balance of diffusion and sorption on the electrode surfaces.

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200

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I. INTRODUCTION

Mass spectrometry is a useful diagnostic technique for plasma systems. The technique is applied to both fusion plasmas¹ and process plasmas² for monitoring of plasma species and analysis of plasma-surface interactions. Because of the high operating pressures of fusion plasmas (10^{-4} - 10^2 Torr) and process plasmas (10^{-3} - 1 Torr), it is usually necessary to pump differentially diagnostic mass spectrometers to maintain the pressure below the operational limits imposed by gas scattering. In order to maximize the sensitivity, the differential pumping ratio is kept small and the mass spectrometer is operated close to the high pressure limit.³⁻⁵ As a result, the interaction of the major plasma species within the ion source is accentuated, which can lead to significant increases in background levels and loss in dynamic range of measurement. This paper focuses on a common problem encountered in hydrogen-fueled fusion plasmas and in hydrogen-based discharge cleaning or etching plasmas. In a typical situation, the combination of an electron-bombardment ionizer and quadrupole mass filter is used to monitor volatile species, usually H_2O , CO_x , and C_mH_n , which result from H-plasma interactions with surface oxides and carbon compounds. Because of high ambient hydrogen pressures in the ion source, atomic hydrogen will be formed on the hot filament, subsequently bombarding the electrode surfaces producing H_2O , CO_x , and C_mH_n . In some practical situations, the background level can exceed the plasma production of these species.^{4,5}

In this paper, we have studied the background impurity problem in some detail. The existing literature concerned with impurity production by hydrogen in hot filament devices is discussed in the following section. In Sec. III, we present measurements of the impurity production for a typical ion source configuration and further, demonstrate the effects of conditioning to

minimize impurity production. In Sec. IV, we interpret the quasi-steady-state impurity levels which are observed in a well-conditioned ion source in terms of the interaction of atomic hydrogen with residual oxygen and carbon within the passivation oxide layer on stainless steel.

II. LITERATURE SURVEY

In the era of glass vacuum systems, there were a number of published studies concerned with the impurities generated when a Bayard-Alpert gauge or omegatron-type mass spectrometer was operated in hydrogen. In a pioneering study by Hickmott,⁶ impurity levels of CO, H₂O, and CH₄ in the range of 1-10% were detected in a low pumping speed system. Hickmott attributed the impurity production to the interaction of H⁺ which formed on the hot tungsten filament⁷⁻¹⁰ with the glass surfaces of the surrounding vacuum vessel. He discounted the tungsten filament as the direct source of the impurities because of two observations: 1) the CO and CH₄ production was essentially the same for three filaments with different bulk carbon impurity levels; and 2) the observed impurity levels were quasi-steady-state and did not decrease after conditioning the filament for five days at 2200 K in H₂ at a pressure of 5×10^{-6} Torr. Hickmott allowed the possibility that the tungsten filament could be involved in the transport of carbon in a "water cycle" as described by Langmuir.⁷ The problem was re-examined several years later by Singleton¹¹ who showed that with a higher pumping speed system, the impurity levels are negligible for filament temperatures below 1425 K, and the levels rise above background for higher filament temperatures and for H₂ pressures higher than 1×10^{-8} Torr. These two early studies were followed by several qualitative investigations using omegatron-type mass spectrometers.^{12,13}

There has been little discussion of hydrogen produced impurities in the

recent literature, in particular, the environment of the problem has evolved with the predominance in contemporary UHV technology of stainless steel vacuum systems and residual gas analyzers based on quadrupole mass filters and electron-bombardment ion sources.¹⁴

Quantitative studies of impurity production with hydrogen in a modern quadrupole instrument were performed by Dietz et al.^{3,15} In the application of a quadrupole mass spectrometer (QMS) for monitoring hydrogen discharge cleaning in fusion devices, Dietz et al. pointed out the need for extensive conditioning of the ion source in hydrogen in order to get reproducible results, and also showed that the impurity production was linear with the ambient hydrogen pressure.

In our studies of hydrogen discharge cleaning of the two tokamak devices PLT¹⁶ and PDX,⁴ we indicated the level of the background impurity production for a similar QMS. For typically encountered conditions, the increase in H₂O background could exceed the partial pressure increases expected from application of the discharge. In a related study with the same type of QMS, Ellefson et al.¹⁷ demonstrated that hydrogen discharge cleaning of the QMS ion source, in situ, was useful for decreasing the hydrogen induced background levels.

III. MEASUREMENTS

The measurements to be described pertain to one particular ion source/QMS combination. The ion source (Extranuclear M/N 041-1) is constructed of 304 SS electrodes, aligned axially with the axis of the quadrupole mass filter (Extranuclear M/N 162-4), and insulated with alumina ceramic bushings and ball bearings. The ionization region is a cylindrical region (approximately 1.2 cm long by 2.5 cm diameter) containing a 0.1 mm

diameter tungsten filament as the electron source, and a Au-plated Ni grid for ion focusing. The remainder of the ion source is a three-element Einzel lens separated from the ionization region by a 0.3 mm aperture.

The QMS was installed in an appendage vacuum system on a UHV test stand which has been previously described.¹⁸ The system volume is relatively small (4.5 l) to minimize the surface area of stainless steel in the vicinity of the QMS. The system is pumped by a nominal 150 l/s turbopump and attains base pressures in the 10^{-10} Torr range after bakeout at 150 C for several days. Research grade hydrogen gas can be introduced to the QMS enclosure via a regulated inlet system. Total gas pressures are measured with a nude Bayard-Alpert gauge. A capacitance manometer on the system is used to calibrate both the ion gauge pressure and QMS partial pressure measurements. Finally, an insulated stainless steel anode is included for glow discharge conditioning of the QMS.

Figure 1 shows the measured change in partial pressures (ΔP_i) of the predominate impurities produced in the QMS system as a function of the hydrogen pressure (P_2) for two conditioning treatments. The measured values of ΔP_i are seen to be proportional to P_2 for all pressures corresponding to molecular flow conditions. At high pressures ($> 10^{-3}$ Torr), the dependence of ΔP_i on P_2 departs from linearity due to gas-phase molecular scattering.¹⁹ The absolute level of ΔP_i depends upon the degree of conditioning. The largest values of ΔP_i plotted in Fig. 1 were obtained after the ion source was given a "standard" conditioning treatment. This treatment was simply operation of the ion source at ambient base pressures ($\sim 10^{-8}$ Torr) for a typical emission level (1 mA) for a period of many days (> 10). Without such rudimentary conditioning, values of ΔP_i could exceed the "standard" values by more than an order of magnitude, and the dependence of ΔP_i on P_2 would be sublinear and

irreproducible.

Figure 1 also shows the impurity production in the ion source following a surface modification of the ion source electrodes. For this case, the electrode surfaces were sputter-coated with approximately 0.5 μm of Cr and then vacuum baked at 600° C for 12 hours prior to reassembly and installation in the vacuum system. Subsequently, a glow discharge conditioning (GDC) treatment was applied in situ. The GDC treatment involved exposing the entire QMS assembly to a H_2 glow discharge at 50 m Torr for 10 hours. The ion source was grounded during the treatment to encourage H^+ ion bombardment. The average ion current to the ionization region electrode was 10 mA. The choice of this conditioning treatment for the ion source is based on our experience with H_2 discharge cleaning of stainless steel. Surface studies^{20,21} have indicated that carbon is effectively removed by the GDC treatment, and that reduction of the FeO_x components of the passivation oxide layer is the predominant source of H_2O during H-bombardment. In contrast, the Cr_2O_3 component was found to be stable with H-bombardment at ambient temperatures.

The GDC treatment reduced the carbon impurity production (CO and CH_4) by approximately a factor of five, and reduced the H_2O production by a factor of three. These results indicate that reactions on the electrode surfaces, rather than direct emission of impurities dissolved within the filament, are the source of the observed impurities.

Figure 2 shows that the filament temperature, and not the emission level is a critical parameter with regard to the impurity production. The ratio $\text{H}_2\text{O}^+/I_e$ is seen to be constant as a function of electron emission (I_e) except at very low emission currents ($< 10^{-2}$ mA) where the filament temperature is calculated to fall below 1800 K.

IV. DISCUSSION

It has been our experience with the Extranuclear 041-1 ion source, that after conditioning, quasi-steady-state levels of impurities are produced with exposure to constant hydrogen pressures, and that the absolute impurity level can be changed only by modifying the electrode surfaces or significantly decreasing the filament temperature. A satisfactory model of the impurity production mechanism should explain the following observations:

- 1) the observed abundance of volatile species ($P_{H_2O} > P_{CO} > P_{CH_4}$);
- 2) the linear dependence of the impurity production with H_2 pressure;
- 3) the constancy of the ratio $\Delta P_i/P_2$ for given surface conditions; and
- 4) the quasi-steady-state impurity production observed at constant H_2 pressure.

As a basic premise for a model, we propose that atomic hydrogen, that is formed by dissociation on the hot tungsten filament, reacts with carbon and oxygen on the stainless steel surfaces of the surrounding electrodes forming the observed residual gases. We propose that the observed quasi-steady-state impurity gas evolution represents a balance between H^0 -induced desorption, re-adsorption from the gas phase, and diffusion from the bulk.

First, in support of observation 1, it is known from the studies of hydrogen discharge cleaning of vacuum vessels,³⁻⁵ that H^0 (or H_n^+) bombardment

of stainless steel will generate volatile H_2O , CO , and CH_4 . The studies of Waelbroeck *et al.*¹⁵ show $P_{H_2O} > P_{CO} > P_{CH_4}$ after initial H-discharge exposure and for stainless steel surface temperatures greater than $100^\circ C$. Based on the power input to the Extranuclear ionizer, the equilibrium bulk temperature for the structure is estimated to be within $(180 \pm 30)^\circ C$ for 1 mA emission.

Observation 2, the linear pressure dependence, is consistent with residual gas production being proportional to the rate of formation of H° . The analyses of Ehrlich,²² and later Hickmott,⁸ show that the production of H° by dissociation on a hot tungsten filament is linear with P_2 for sufficiently high filament temperatures, $T_f \gtrsim 1800$ K. The value of the dissociation probability (α) is constant at high temperatures ($T_f > 2200$ K), but will begin to decrease at a threshold temperature (2000-2200 K) which is dependent on the surface conditions.⁹⁻¹⁰ The filament temperature for 1 mA emission in the Extranuclear ion source is calculated to be 2060 K, on the basis of the filament resistance.²³

The production of hydrogen ions, H_2^+ and H^+ , within the ion sources would also be linearly proportional to the H_2 pressure. However, the estimated H_2^+ flux to the electrode surfaces facing the ionization region is smaller by a factor $\gtrsim 10^4$ than the H° flux for an emission current of 1 mA. In addition, if ion-induced or electron-induced desorption were the source of the residual gases, then a strictly linear dependence of P_i with emission current would be observed, contrary to the behavior shown in Fig. 2.

Observation 3 and 4 are dependent on the details of the surface reactions which generate the volatile products H_2O , CO , and CH_4 . Thus far, only the H_2O ²⁴ and CH_4 ²⁵ reactions have been discussed in the literature. Considering that H_2O is the dominant residual gas product, an oxygen balance equation can be written as follows:

$$\frac{dp_i}{dt} = A (\eta \phi_H - s v_i p_i) - S_i p_i \quad (1)$$

where $\eta \phi_H$ is the desorbed H_2O flux, given by the product of an apparent reaction probability, η , and the incident H° flux, ϕ_H ; $s v_i p_i$ is the adsorption flux from the gas phase, given by the product of the sticking coefficient, s , the specific arrival rate per Torr, v_i , and the partial pressure, p_i ; and $S_i p_i$ is the gas-phase removal rate from the system, where S_i is pumping speed. For the steady-state conditions corresponding to the measurement conditions: $dp_i/dt = 0$ and Eq. (1) can be rearranged to:

$$\frac{p_i}{p_2} = \frac{\eta \alpha v_H}{S/A + s v_i} \quad (2)$$

where the relation $\phi_H = \alpha v_H p_2$ has been substituted for the linear dependence of H° formation with respect to p_2 . (For the geometry of the Extranuclear ion source, and an assumed value of $\alpha = 0.1$, the value of $v_H = 1 \times 10^{19} \text{ cm}^{-2} \text{ s}^{-1} \text{ Torr}^{-1}$.)

Substituting for the measured quantities in Eq. (2) gives an estimate of the required reaction probability to account for the observed ratio of $\Delta p_i/p_2 = 10^{-3}$: the bombarded surface area, $A \approx 10 \text{ cm}^2$ (a more exact estimate would include a surface roughness factor in the evaluation of A and the filament area in the evaluation of v_H); the value of S is estimated to be 50 l/s based on pumping-speed measurements for N_2 ; and the value of the sticking coefficient is quite uncertain, but is probably in the range of 0.01-0.1 for $T = 200^\circ\text{C}$. With these estimates, the required value of η is $(1-3) \times 10^{-3}$ which is consistent with the value for the reaction probability measured on oxidized

stainless steel by Dietz et al.²⁴ who found $\eta > 3 \times 10^{-3}$ for $T > 10J^\circ C$.

Tschersich and von Seggern²⁶ have measured the change in surface composition of stainless steel during H^+ bombardment using AES. Figure 3 shows Tschersich's measurements of the surface concentration of oxygen and carbon as a function of H^+ fluence for a 304 stainless steel sample at several temperatures. For low fluence ($< 10^{18} \text{ cm}^{-2}$), there are fast removal processes evident for both surface carbon and oxygen. Similar removal rates have been observed on stainless steel exposed to H_2 discharge-cleaning plasmas.²¹ It is reasonable to assume that these low fluence processes are concerned with the removal of adsorbed concentrations of carbon and oxygen. Such processes give rise to the copious, but transient, residual gas production that is present in an unconditioned ion source during initial operation. However, following extensive H^+ bombardment (i.e., conditioning) the nature of the stainless steel surface, and in turn, the level of residual gas production is evidently quite different. As shown in Fig. 3, the surface concentrations at high H^+ fluence show residual levels (2-6% for O, and 5-10% for C) that are slowly varying or nearly constant with fluence. These residual levels, which are most likely maintained by diffusion from the passivation oxide layer and re-adsorption from the gas phase, are responsible for the quasi-steady-state residual gas production under subsequent H^+ bombardment.

This situation can be modelled if we consider an oxygen balance equation from the standpoint of the change of oxygen coverage, n , of the surface:

$$\frac{dn}{dt} = F_g + F_d - F_h \quad (3)$$

where F_g , F_d , and F_h are the fluxes of oxygen atoms at the surface due to adsorption from the gas phase, diffusion from the bulk, and desorption by atomic hydrogen. Following the notation used in the gas-phase balance equation (Eq. 1), the adsorption flux is equal to $F_g = sv_1P_1$, assuming that the adsorption is independent of the coverage for values of n which are small compared to monolayer coverage, n_o . The desorbed flux is equal to $F_h = n\phi_H$. The diffusive flux at the surface ($x=0$) is obtained from Fick's Law, with the simplifying assumptions that the bulk source of oxygen is diffusion from the passivation oxide layer (thickness, $l = 2.10^{-6}$ cm), and that a single-valued diffusion constant, D , is valid:

$$F_d = -D \left. \frac{\partial c}{\partial x} \right|_{x=0}$$

The concentration of oxygen $c(x,t)$ is a solution to the one-dimensional diffusion equation²⁷ with the following boundary conditions: the initial concentration, c_o , is uniform for $t = 0$ and $0 \leq x \leq l$; c_o is considered to be large compared to the bulk concentration, i.e., $c = 0$ for $x = l$ and $t > 0$; and $D \partial c / \partial x = F_g$ for $x = 0$ and $t = 0$. This last condition represents the flux balance at the surface prior to H^0 -induced desorption.

A solution to a similar problem, the diffusion of oxygen into a Ni surface during inert-gas sputtering, has been considered by Kirchheim and Hofman.²⁸ In the time limit appropriate to the present problem, where t is large compared to the two characteristic times, the desorption time constant $\tau_h = n_o / \eta \phi_H$, and the diffusion time constant, $\tau_d = 4l^2 / \pi^2 D$, the limiting form of Eq. (3) reduces to:

$$\frac{n}{n_o} - \frac{2F_g}{\eta \phi_H} = \left(\frac{Dc_o}{l\eta \phi_H} + \frac{2F_g}{\pi \eta \phi_H} \right) \exp \left(-\frac{\pi^2 D t}{4l^2} \right) \quad (4)$$

where n/n_0 is the relative surface coverage, and $2F_g/n\phi_H$ represents a limiting steady-state value of the relative coverage, n_g/n_0 .

Figure 4a shows the oxygen coverage data of Fig. 3a at high fluence, ($> 1.5 \times 10^{19} \text{ cm}^{-2}$), converted to a function of time for comparison with Eq. (4) by dividing the fluence coordinate by the constant value of the H^+ flux that was appropriate for this interval, ($\phi_H = 1.05 \times 10^{16} \text{ cm}^{-2} \text{ s}^{-1}$). From the slopes of the lines in Fig. 4a, values for D can be calculated for the three experimental temperatures. These values of D are plotted in Fig. 4b versus $1/T$ to extract an Arrhenius activation energy ($\Delta E = 3 \pm 1 \text{ kcal/mole}$) for the diffusion process. The calculated values of D necessary to maintain oxygen flux balance, $(0.4-1.2) \times 10^{-15} \text{ cm}^2 \text{ s}^{-1}$, are not unreasonable in comparison to literature value of the diffusion coefficient of oxygen in transition metals.²⁸ (Measurements of oxygen diffusion in the mixed oxide, $\text{Fe}_2\text{O}_3/\text{Cr}_2\text{O}_3/\text{NiO}$, of the passivation layer on stainless steel do not exist.) Considering the probable uncertainty of the derived values of the activation energy, it is noteworthy that the value derived from Fig. 4b ($\approx 3 \text{ kcal}$) is close to the activation energy derived by Waelbroeck *et al.*¹⁵ (5.8 kcal) for the production of H_2O during H_2 discharge cleaning of stainless steel.

The more complicated temperature dependence exhibited by the surface carbon data (Fig. 3b) cannot be modelled by Eq. (4). This discrepancy may be the result of the experimental method. Since the sample was cooled at periodic intervals during the H^+ bombardment to allow the surface analysis, diffusive time-constants which are significantly shorter than the thermal time-constant would mask the simple time dependence of Eq. (4).

V. CONCLUSIONS

A plausible mechanism has been described for the residual gas production in an electron-bombardment ion source operated at high H_2 pressures. Quasi-steady-state levels of predominantly H_2O , CO , and CH_4 are produced by H^+ -induced desorption from stainless steel electrode surfaces. Surface reaction rates are maintained by residual surface oxygen and carbon coverages that are replenished by diffusion from the passivation oxide layer and by re-adsorption from the gas phase. Extensive conditioning of the ion source, including Cr-plating of the electrodes followed by H_2 glow discharge cleaning, yielded only modest reductions in the residual gas production (by a factor of three for H_2O and a factor of five for CO , CH_4).

Although 304 stainless steel is an excellent vacuum material from the standpoint of its outgassing characteristics and corrosion resistance, it is not the material of choice for the construction of ion sources to be operated at high H_2 pressures. The passivation oxide layer evidently presents a large reservoir of accessible oxygen and carbon for residual gas formation that cannot be significantly reduced during normal operational periods of the ion source. In addition to minimizing the quantity of electrode material within the ionization volume, a better choice for electrode construction would be a material with a thin or nonexistent passivation oxide layer (such as a noble metal coating with a permeation barrier sub-coating) or a material with an oxide that is easily reduced with exposure to hydrogen at moderate temperatures (such as Ni).

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FIGURE CAPTIONS

- FIG. 1 Change in partial pressure of the dominant residual gases produced in the Extranuclear 041 ion source as a function of the H_2 pressure for the standard ion source (open points), and after Cr-plating and H_2 glow discharge conditioning of the electrodes (filled points).
- FIG. 2 Mass spectrometer output signal for H_2O^+ (18 amu) as a function of electron emission current for a constant H_2 pressure of 1×10^{-4} Torr. The filament temperature is indicated on the top scale.
- FIG. 3 Change in relative surface concentration of oxygen (a) and carbon (b) on a stainless steel sample as a function of the incident fluence of thermal H^+ . (Data courtesy of K.G. Tschersich, Ref. 26).
- FIG. 4 (a) Logarithm of the relative oxygen coverage minus the steady-state value as function of time for the high fluence ($> 1.5 \times 10^{19} \text{ cm}^{-2}$) oxygen coverage data of Fig. 3a. (b) Values of the oxygen diffusivity obtained from the slopes of the fitted lines are plotted versus the inverse temperature.

Fig. 1

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Fig. 2

Fig. 3

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Fig. 4

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