INVESTIGATIONS BY TECHNIQUES OF ELECTRON STIMULATED DESORPTION

OF THE MERITS OF GLOW DISCHARGE CLEANING

OF THE VACUUM CHAMBER OF THE ISR

by

A.W. [Jones, E. Jones and E.M. Williams *)

Geneva, 6th October 1972

*Visiter from University of Liverpool, Dept. of Electrical Engineering and Electronics.
CONTENTS

1. Introduction.

2. Experimental technique and system.

3. Preliminary investigations with the diagnostic technique.

4. Observation of desorption efficiency with treated and untreated surfaces.

5. Discussion and conclusions.

6. References.
1. **Introduction**

The importance of the contribution of the surface of vacuum chambers of the ISR to the stability of the residual gas density in the presence of proton beams is readily apparent from the criterion for stability discussed by Fischer\(^1\). In a recent report Jones \textit{et al}\(^2\) have described experiments with a 14 m section of the ISR in which use was made of the clearing electrodes to operate a glow discharge during the bake-out cycle. The philosophy as envisaged in this report is to induce desorption by ion impact of the more lightly bound gas at the surface and to substitute in its place a layer of the more inert argon gas used in the discharge. The present communication describes an extension of this work; improvements have been made to the procedure for sampling the surface using electrons, and, further, sampling has been carried out simultaneously in regions treated and untreated by glow discharge. A direct measure has thus been possible of the value of gas discharge cleaning in reducing the reactivity of the surface to electron beams.

2. **Experimental technique and system**

The technique of electron stimulated desorption (E.S.D.) forming the basis of the present diagnostic technique, consists in bombarding the surface with electrons at a low current density, typically up to 10 \(\mu\)A cm\(^{-2}\), and energy of around 200 eV. The action of the electrons at the surface induces electronic transitions of the adsorbate with subsequent desorption in neutral and charged form. In the present experiments the pressure increase accompanying E.S.D. is measured. This information is used to determine the efficiency of desorption \(\eta\), i.e. the number of gas atoms desorbed per incident electron.

The temporal behaviour of pressure \(p(\text{torr})\) in a desorption cell of volume \(V\) (litres) may be described according to the differential equation:

\[
\frac{Vdp}{dt} = \frac{nQiAe}{e} - pS + qA, \tag{1}
\]

with

\[
\frac{dn}{dt} = -\frac{nQi}{A'e} + p_0 \omega s(n), \tag{2}
\]
where \( n \) is the surface coverage (atoms cm\(^{-2}\)).

\( Q \) is the total desorption cross-section (cm\(^2\)) such that \( nQ \) represents the probability of desorption \( n \).

\( a \) is a constant to convert from number of atoms to torr litres (\( 3.6 \times 10^{19} \)).

\( S \) is the speed of pumps at the desorption cell (ls\(^{-1}\)).

\( \Gamma \) is the electron current (A).

\( e \) is the electronic charge (1.6 \times 10^{-19} C).

\( q \) is the specific influx of gas through the walls of the desorption cell (torr ls\(^{-1}\) cm\(^{-2}\)).

\( A \) is the total wall area of the cell (cm\(^2\)).

\( A' \) is the wall area bombarded by electrons (cm\(^2\)).

\( \omega \) is the specific rate of collision between atoms of the gas and the surface of the cell (atoms cm\(^{-2}\) s\(^{-1}\) torr\(^{-1}\)).

\( s(n) \) is the sticking probability.

A solution to equation (1) is most easily obtainable by assuming that \( n \) is effectively constant over the range of pressure increase during E.S.D. - in practice this means that the electron current density at the surface should be low. Under these conditions the solution to equation (1) is

\[
p(t) - p_o = \frac{nQI^-a}{eS} \left[ 1 - \exp\left(-\frac{S}{V} t\right) \right] \quad (3)
\]

where

\[
p_o = \frac{qA}{S} \quad (4)
\]

\( p_o \) is the pressure in the cell prior to the onset of E.S.D. at \( t=0 \). As \( t \to \infty \), the increase in pressure above \( p_o \) attains an equilibrium value \( \Delta p \), where

\[
\Delta p = p_{eq} - p_o = \frac{nQI^-a}{eS} \quad (5)
\]

Finally, with the termination of E.S.D. the pressure decays according to:

\[
p(t) - p_o = \frac{nQI^-a}{eS} \exp\left[-\left(\frac{S}{V}\right) t\right] \quad (6)
\]
Rearranging (5) gives the desorption efficiency $\eta$ as

$$\eta = nQ = \frac{\Delta p e S}{I^a}$$  \hspace{1cm} (7)

which provides the most convenient and direct method of determining $\eta$ from a knowledge of $I^a$, $\Delta p$ and $S$.

The 14m section of the ISR vacuum chamber used in the experiments is shown schematically in Figure 1. Facilities are provided for pressure measurement in line with the pumping units, comprising ion and sublimation pumps, which are situated some distance from the regions of the vacuum system where E.S.D. is carried out. The arrangement may be thought of as made up of a desorption cell connected to a pump and pressure measuring system by a pipe of finite conductance. When determining $\eta$ from equation (7) it may be noted that the parameters $\Delta p$ and $S$ appear as a product and that this product, representing a quantity of gas, will be the same in the desorption cell as at the region of the pump. The actual experimental system is slightly more complex than this since there are two pumping units (sublimation pump 2 was not made use of in the course of this work). The pressure variations during E.S.D. were monitored at UHV gauge 1 and in the case of probe A, sampling in the region treated by glow discharge, the contribution of the pumping unit in line with UHV gauge 2 is less than 1% and can therefore be neglected. For probe B on the other hand, sampling in the non-discharged region, the value of $\Delta p$ observed at UHV gauge 1 should be increased by 14% to allow for the gas load pumped by the unit in line with UHV gauge 2.

3. Preliminary investigations with the diagnostic technique

An example of the pressure pulse observed with E.S.D. is given in Figure 2. This result exhibits all the features of equations (3), (5) and (6) and was measured with an electron current of 50 $\mu$A at energy 220 eV bombarding an area estimated as some 5 cm$^2$. Analysis of this result yields a value for $\eta$ of some $10^{-3}$ atom/electron which, in turn,
assuming a surface coverage of $10^{15}$ atoms cm$^{-2}$ implies that the desorption of surface gas brought about by the sampling process is of the order 0.05% of a monolayer per minute. This low level of perturbation is consistent with the finding of a direct proportionality between $\Delta p$ and $I^{-}$ for, typically, electron currents up to $\approx 100$ $\mu$A.

Analysis of the form of the initial rise of pressure using equation (3) yields a value of pumping speed of $150 \xi \text{s}^{-1}$. The result of Figure 2 was obtained at a stage in the experiments when the ion pumps only were in operation with a base pressure of $1.8 \times 10^{-10}$ torr. Thus the derived value is to be compared with the nominal value of $400 \xi \text{s}^{-1}$ for the speed of the ion pump.

Uncertainties in pump speed and also with regard to absolute pressure determinations introduce systematic errors in forming the product $pS$ in equation (7). This is not of too great a concern in the present case, however, since a priori the interest is in comparing the desorption efficiency $\eta$ in the two sampling positions. There is greater interest in formulating as accurately as possible the relative change in pumping speed when the sublimation pumps are used to supplement the ion pumps. The procedure adopted in the present work has been to express pumping in terms of the pumping speed of the ion pump by comparing the 0-90% rise time of the pressure pulse obtained with ion pumps only (Figure 2) with that observed with the auxiliary sublimation pumps in action. (It is of interest to note that speeds determined in this way were found to range up to 3.5 times the pumping speed of the ion pump). In adopting this procedure the speed of the ion pump was taken as $400 \xi \text{s}^{-1}$ and the pressure increments $\Delta p$ were expressed simply as nitrogen equivalents.

4. Observation of desorption efficiency $\eta$ with treated and untreated surfaces

Figure 3 summarises the results of measurements of $\eta$ for both treated (glow discharged) and untreated surfaces performed as a function of time after bakeout with in all cases the electron energy set at 220 eV.
The base pressure in the system during the course of the measurements (corrected for X-ray contribution) is indicated towards the top of the figure.

Sampling was first carried out 20 h after bakeout with the ion pumps only in operation and with a base pressure of $1.8 \times 10^{-10}$ torr. There is evident at this stage a value of $\eta$ for the treated area one order of magnitude lower than that for the untreated surface. In the course of the next $\approx 100$ h the base pressure was reduced to $1 \times 10^{-10}$ torr and the difference between the desorption efficiencies for the two regions is seen to increase. At 135 h the sublimation pumps were first fired, and then fired again at 160 and 255 h. These actions bring about a reduction of the base pressure and the value of base pressure attained at 260 h is $2 \times 10^{-11}$ torr. Throughout this activity the desorption efficiency for the untreated surface remains practically unchanged. In contrast, in the case of the treated surface there is a tendency for $\eta$ to follow variations in the base pressure. This tendency became more apparent with successive firings of the sublimation pumps and is most probably linked with the decrease in the gas load with repeated firings.

After the measurement at 260 h the treated surface was electron scrubbed at 220 V, 60 mA for 1 h. This causes $\eta$ for the treated surface to decrease and by 280 h, with the base pressure at $2.2 \times 10^{-11}$ torr, the measured value of $\eta$ is $1.2 \times 10^{-6}$ - an order of magnitude lower than the measurement prior to scrubbing. The action of scrubbing the treated area caused the pressure in the system to increase, to a maximum of $6 \times 10^{-10}$ torr, which most probably accounts for the slight increase in $\eta$ for the untreated area observed at this point. After the measurements at 280 h the untreated area was scrubbed at 1 kV, 1 mA; notwithstanding the large amount of gas produced in this process (pressure increase up to $10^{-9}$ torr) the scrubbing action after 1 h served only to decrease $\eta$ by some 40%. In view of this the process was continued for a further 1 h at 220 V, 60 mA and $\frac{1}{2}$ h at 220 V, 100 mA. At the end of this treatment the desorption efficiency measured at a base pressure of $7 \times 10^{-11}$ torr is some 75% below the initial unscrubbed level. The pressure increase in the
system during scrubbing of the untreated area is reflected in a clear increase of the desorption efficiency for the treated surface at this stage. However, with the further firing of the sublimation pumps the value of $\eta$ corresponding to the treated surface decreases whilst that of the untreated surface remains constant.

At this point the vacuum system was exposed to room air for 24 h and the system then evacuated and baked following conventional procedures. Sampling was carried out some 45 h after bakeout with the ion and sublimation pumps in operation and with the base pressure at a level of $2.5 \times 10^{-11}$ torr. The results of these measurements are included in Figure 3 and show that the desorption efficiency for the treated surface is maintained at the precontamination level, while $\eta$ for the untreated surface is restored to the level measured prior to any attempt at electron scrubbing.

In the earlier work of Jones et al.\textsuperscript{2} it was observed that a glow discharge at ambient temperature followed by a normal bakeout did not produce any significant cleaning in the untreated regions. To investigate this conclusion the probe located in the treated region was moved to a new position over an untreated surface and the experiments repeated with now a glow discharge in this new region at ambient temperature followed by bakeout. Measurements at a stage = 20 h after bakeout (pressure $1.7 \times 10^{-10}$ torr) gave a value of $\eta$ for the newly treated surface of $3.7 \times 10^{-4}$, with the untreated surface unchanged at the predicted value of $8 \times 10^{-4}$, i.e. a change of one half an order of magnitude on the treated surface in comparison with the change of one order of magnitude observed at an equivalent stage with the discharge at elevated temperature. But with the first firing of the sublimation pumps $\eta$ for the treated area was increased to a level equal to that of the untreated surface, thus offsetting the advantage gained by the discharge cleaning. It may be stressed that the gas load imposed on the treated surface in this case was quite comparable with that encountered in the course of the earlier measurements of Figure 3.
5. Discussion and conclusions

The procedure of discharge cleaning during bakeout followed by electron scrubbing provides a means of reducing the electron desorption efficiency of the surface of the vacuum chambers of the ISR by some two and a half orders of magnitude. Moreover, the surface treated in this way retains its low desorption characteristic even after contamination with room air. Discharge cleaning at elevated temperature without subsequent electron scrubbing provides a gain of some one and a half orders of magnitude.

The desorption efficiency for the normal, untreated surface is \(\approx 10^{-3}\) and it is of interest to note that values of this order have previously been determined in other accelerator systems under similar conditions\(^{3,4}\). Electron scrubbing taken by itself does not appear to be a very efficient method of surface cleaning, particularly as contact with room air brings about recontamination. It appears that the action of a discharge during bakeout promotes the surface to a form in which the desorption characteristics respond more significantly to i) electron scrubbing and ii) changes in base pressure in the system. This later behaviour is consistent with the notion of an adsorption isotherm between the residual gas and the surface.

It is surprising that despite the relatively large desorption probability in the case of the untreated surface, prolonged electron scrubbing has greater effect in the treated area. Possible explanations may be, firstly, the occurrence of a process of surface migration in the untreated area, i.e. diffusion of gas over the surface to replenish the gas being desorbed, and secondly, that in the case of the untreated surface the ad-layers are really thick. These ideas raise the question as to the surface composition and as to how the composition is changed by the discharge. The answer can only be speculative at this stage, although it is of interest to note some recent evidence from studies of metal surfaces, in particular stainless steel surfaces, using Auger Electron Spectroscopy (AES)\(^{5,6,7}\) and Appearance Potential Spectroscopy (APS)\(^8\). With stainless
steel (type 304) after bakeout the spectra show the main contaminant as carbon with peak strength either comparable with or greater than that of the parent metals. Moreover, there is increasing evidence with regard to the chemical interpretation of the carbon signal to indicate that the carbon exists in the form of graphite. This material is of course an excellent sorber of gas, and it is found to persist even after heating for extended periods at 500°C. Measurements with APS confirm that the graphite is removed by ion bombardment. It may well be the case that with gas discharge cleaning of vacuum chambers the significant action is in removing the surface graphite.

The key question remains as to significance of a reduction in electron desorption efficiency for desorption phenomena involving ion bombardment. A general characteristic of E.S.D. is that desorption efficiencies are greater for the least tightly bound adsorption states. Thus a reduction in desorption efficiency can be looked upon as a reduction of the contribution of the less strongly bound gas. This can but help to reduce the amount of gas desorbed under ion impact, although the full extent of the amelioration can only be judged by direct experiment. It is, however, very encouraging to note from the criterion for stability discussed by Fischer\(^1\) that a reduction in ion desorption efficiency in the run away regions by a factor of around one quarter would allow the design current of 20 A to be attained in the ISR.

6. References

2) Jones, A.W., Jones, E. and Schuhbäck, H., Private Communication in form of ISR Performance Report, 28 August 1972
FIG. 1. SCHEMATIC REPRESENTATION OF THE TEST FACILITY
FIG. 2. PRESSURE PULSE WITH E.S.D.