

## ULTRAHIGH VACUUM TECHNOLOGY FOR STORAGE RINGS

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### Summary

Present UHV technology permits producing pressures of  $10^{-9}$  to  $10^{-11}$  torr in the aperture-limited vacuum chambers of storage rings. Very disturbing pressure rises occur in electron storage rings from synchrotron light and in proton storage rings from ion bombardment. This has prompted the development of special cleaning and bakeout procedures on chambers before installation and *in situ*. In some cases, cleaning and baking alone are not sufficient and a pumping action distributed all along the pipes must be provided for. A controversy exists whether one should use, for future superconducting storage rings, a cold (4 K), cool (77 K) or warm (293 K) bore vacuum chamber. Experiments are in progress to supply the basic facts needed for taking decisions.

### 1. Introduction

Electron and proton storage rings require for operation ultrahigh vacuum - pressures of  $10^{-8}$  to  $10^{-11}$  torr or better. To produce ultrahigh vacuum has been a matter of routine for many years and even the additional complication that storage rings are strongly aperture-limited and that pumping stations cannot be installed as frequently as one would like is not a serious problem for the achievement of the required static pressure, that means the pressure in absence of beams.

All the difficulties with the vacuum system of storage rings come from the interaction of the circulating beams with the chamber walls. These interactions are of various kinds: circulating electron beams produce intense synchrotron light which strikes the chamber walls. This firstly increases the wall temperature provoking enhanced thermal outgassing. Furthermore, it produces secondary electrons which, when hitting the chamber wall again, desorb molecules resting adsorbed on the surface. A circulating proton beam does not emit synchrotron light of enough intensity but it interacts with the chamber wall in a different way;<sup>1</sup> the protons create, by collision with the residual gas molecules, positive ions which are repelled by the electrostatic potential of the beam and bombard the chamber walls where they release surface molecules. This mechanism has a positive feedback: the released gas contributes to the ionisation and to the release of gas. The effect is dependent on the beam current. There exists a critical current at which the pressure becomes unstable and tends towards infinity. In a well cleaned chamber, however, a circulating proton beam decreases the residual gas pressure. The condition is that the sticking probability of the incident ion is larger than the probability of desorbing a neutral molecule.

There exist two principal methods of fighting the beam induced pressure rises: one method is the conditioning of the chambers in order to reduce thermal and induced gas release and the other is the provision of some sort of distributed pumping speed in addition to the normal lumped pumps. Often both methods are combined.

### 2. Chamber conditioning

The aim of any chamber conditioning is to reduce the rate of gas desorption. We have here to distinguish between thermal, electron induced and ion induced desorption where these three parameters are only loosely coupled to each other. Conditioning of chambers can be done during manufacturing, in the laboratory before installation and *in situ* after installation.

#### 2.1 Bakeout and annealing

The most commonly applied *in-situ* conditioning is bakeout at temperatures between 150 and 350°C for periods ranging from a few hours to several days, depending on the requirements and possibilities. Bakeout is very efficient in reducing the thermal desorption. A typical bakeout of 300°C for 24 h reduces the desorption from stainless steel to  $10^{-12}$  Tl/s cm<sup>2</sup> or less, where  $\approx 99\%$  of the remaining gas being desorbed is hydrogen diffusing from the bulk of the metal to the surface and from there into the vacuum. The remaining desorption of heavier gases, like N<sub>2</sub>, CO, H<sub>2</sub>O etc. is  $10^{-14}$  Tl/s cm<sup>2</sup> or even much less.

A reduction of the hydrogen desorption down to also  $10^{-14}$  Tl/s cm<sup>2</sup> can be achieved by pre-annealing the completed chamber, subassemblies or the sheets from which the chamber is manufactured in a vacuum oven at about 1000°C for a few hours.<sup>2</sup> The only problem is to find a suitable oven. At CERN we have installed for this purpose a big oven with a useful volume of 3.5 m length and 1 m width where the pressure is kept at  $5 \times 10^{-6}$  torr at 1000°C and against a H<sub>2</sub> load of  $2 \times 10^{-2}$  Tl/s. The partial pressure of hydrogen and its compounds during annealing is a crucial parameter for the ultimate thermal desorption rate which can be achieved.

On small samples in the laboratory, thermal gas desorption rates of less than  $10^{-15}$  Tl/s cm<sup>2</sup> could be achieved. But it would be difficult to obtain the same with full size chambers. Besides, at least for storage rings, rates of less than  $10^{-13}$  Tl/s cm are unlikely to be ever needed.

Bakeout is, unfortunately, rather inefficient in reducing the electron induced desorption, and still less the ion induced desorption. See, for example, Figure 1, which shows the result of several subsequent bakeout cycles each of 24 h at temperatures between 150 and 300°C on a sample of aluminium alloy 5086.<sup>3</sup> What has been measured is the desorption yield of CO molecules per incident K<sup>+</sup> ion of 1.4 keV and the total desorption yield of all kinds of molecules per incident electron of 600 eV. The desorption rate of CO molecules is the most critical figure in assessing the vacuum stability of proton storage rings. Although hydrogen is sometimes released with even high probability, it has smaller ionisation cross-section for high energy protons and it is also less powerful in desorbing further molecules. K<sup>+</sup> ions have been used because they can be produced by a very simple ion source.<sup>4</sup> The energy of 1.4 keV is representative for ions produced in a 20 A proton beam. The electron energy of 600 eV has been chosen because it gives the the highest desorption yield from aluminium. The rate

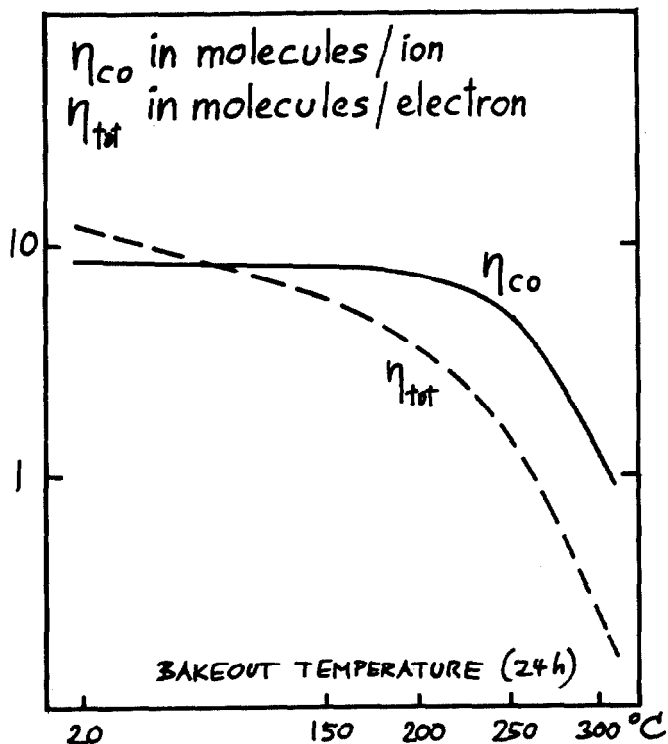


Figure 1 : Desorption yield of CO molecules by  $K^+$  ions of 1.4 keV and of all molecules by electrons of 600 eV after 24 h bakeout at different temperatures. Sample : Aluminium 5086

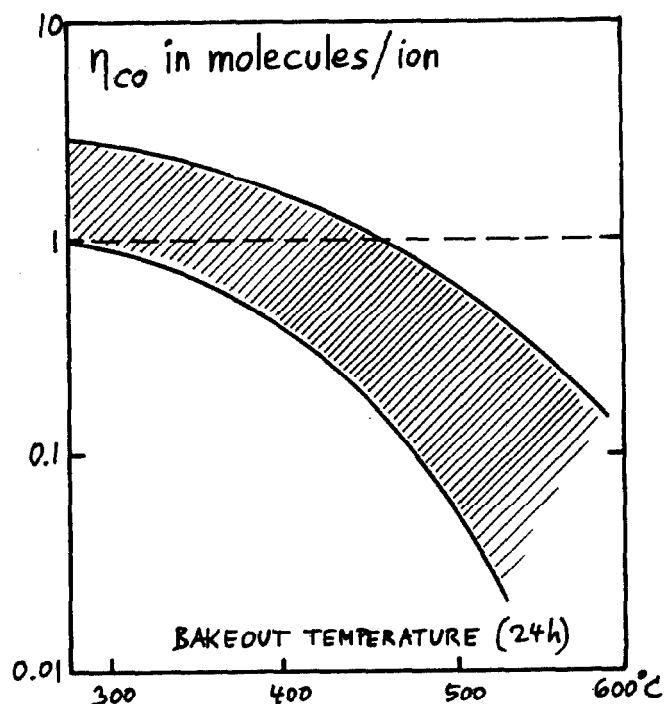


Figure 2 : Desorption yield of CO molecules by  $K^+$  ions of 1.4 keV after 24 h bakeout at different temperatures. Samples : 2 x stainless steel, 2 x Inconel 718, 1 x Inconel 600, 1 x titanium

for electrons of a few electron volts as those responsible for the pressure rises in electron machines is orders of magnitude smaller but the qualitative behaviour is the same. As one can see, the ion-induced desorption does not change up to 250°C in sharp contrast to the thermal desorption which is, after a bake at this temperature, already down by  $\approx 3$  orders of magnitude. The electron-induced desorption does improve up to 250°C, but the important decrease occurs only above this temperature.

Fig. 2 shows the measured ion induced desorption after bakeout at higher temperatures for different materials including stainless steel, titanium and inconel.<sup>3</sup> The results are presented in a shaded band because the differences between different material are not larger than the scatter between the samples of one and the same material. One has to go as high as 450°C in order to reduce the CO yield from all materials below unity. It should be mentioned here that in proton storage rings a desorption yield of less than unity must be aimed at because only then the pumping effect of the ions being shot into the wall exceeds the desorption yield and the vacuum can never become unstable but shows, to the contrary, beam pumping.

Practical experience on the ISR has shown that in some cases a bake at 350°C is sufficient to make the desorption yield smaller than unity which is not in contradiction with our laboratory measurements. On other places, however, in spite of baking at 350°C, the ion desorption yields remain as high as 4 and more efficient cures are needed.

## 2.2 Glow discharge cleaning

What is obviously needed is the cleaning of the surface by a bombardment with particles of energies exceeding the binding energies of  $\approx 1$  to 10 eV of the many molecules which survive a bakeout. This can be done by the well known glow discharge cleaning which is usually applied as laboratory treatment preceding the installation.<sup>5</sup> At CERN we are using a mixture of 90% Ar and 10% O<sub>2</sub> at  $2 \times 10^{-2}$  torr. The voltage of + 350 V is applied to a wire stretched, for this purpose, through the middle of the chamber, while the latter is kept at a temperature of 300°C. It has been found necessary for good results to apply a dose of  $> 10^{18}$  ions/cm<sup>2</sup>. After the treatment a small residual partial pressure of argon can be detected which is, however, eliminated by a 300-350°C bakeout subsequent to the glow discharge treatment.

Measurements have been made at CERN in order to find out what a glow discharge is actually doing to a surface.<sup>6</sup> Fig. 3 shows as an example the evolution of the partial pressures during the cleaning of a chamber during a discharge in argon of  $2 \times 10^{-2}$  torr. The stainless steel chamber had previously been baked for 24 h at 300°C. The integral under the curve for CO corresponds to 28 monolayers of this gas, which have survived the 300°C bakeout. The total amount of released H<sub>2</sub> and CH<sub>4</sub> is less than one per cent of that of CO. Auger electron spectroscopy confirms an important reduction of the carbon content of the upper surface under the influence of the discharge, in particular if 10% of oxygen is added to the argon.<sup>7</sup>

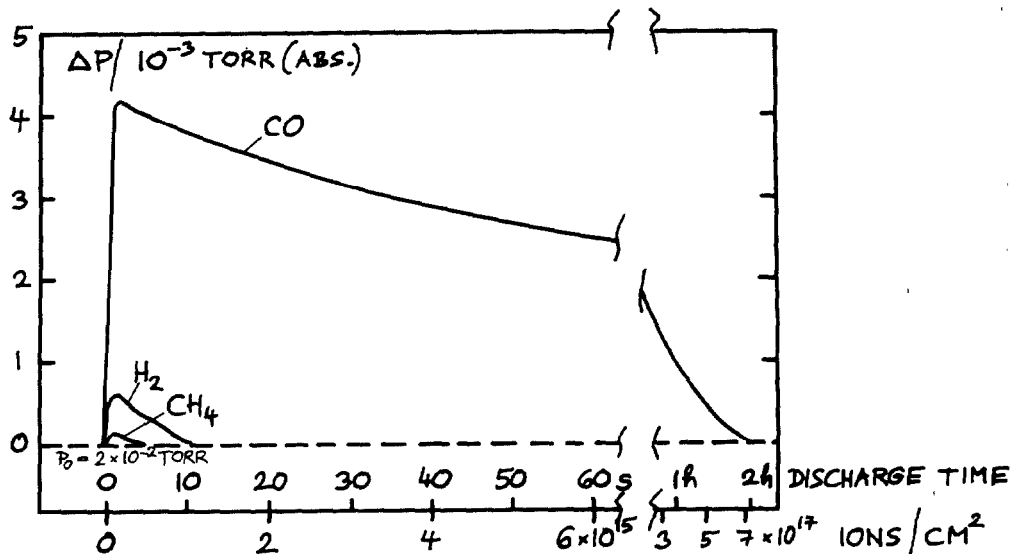


Figure 3 : Partial pressure evolution during glow discharge cleaning

The following table summarises the results of some laboratory tests on the efficiency of glow discharge cleaning on three different metals.<sup>8</sup> For the tests a beam of 2 keV  $^{15}\text{N}_2^+$  ions ( $M = 30$ ) has been used in order not to confuse them with the desorbed gases. The desorption yield for CO, the important parameter for the stability of proton storage rings, which had been above unity after the standard bakeout, is reduced to  $< 0.01$ , the detection threshold of the apparatus. The subsequent bakeout carried out in order to remove the residual argon has the side effect of the reappearance of a small CO desorption yield. The system has then been exposed to air for 12 h in order to simulate the transport of a chamber from the laboratory to the machine for installation. After the following *in-situ* bakeout, the CO desorption is furthermore increased but stays for all samples below the critical level of unity.

Table I

CO Desorption Yield ( $\eta_{\text{CO}}$ ) in molecules/ion from three different materials after a sequence of treatments

After :	stainless steel	pure aluminium	titanium alloy
cleaning	10	8.5	9
bakeout 24 h 300°C	1.8	1.2	1.8
discharge $10^{18}$ ions/cm <sup>2</sup>	< 0.01	< 0.01	< 0.01
bakeout 24 h 300°C	0.15	0.05	0.12
air 12 h 1 bar	5	5.5	4
bakeout 24 h 300°C	0.9	0.4	0.7

The differences between the three materials are not significant. One must also keep in mind the aluminium which seemed here to be the best, can never be baked at 300°C when it forms a chamber and not a small sample.

The results of Table I are in good agreement with practical experience in operating the ISR. A complete sector of  $\sim 100$  m length has been treated by GD cleaning and is showing, since then, "beam pumping", that is, a decrease of pressure in presence of high intensity proton beams. Three subsequent exposures to air followed by 300°C bakeout could not deteriorate this performance.

The cleanest chamber is obtained if the GD cleaning is done *in situ* without exposing the chamber to air after the treatment. It is possible to design vacuum chambers with built-in GD electrodes, for instance in the form of a moveable wire as described elsewhere at this conference.<sup>9</sup>

Another very interesting approach is that of the DESY team working on the construction of the PETRA storage ring. They are using the centre electrode of their distributed sputter-ion pumps as anode for *in-situ* GD cleaning.<sup>18</sup>

### 2.3 Beam conditioning

A running period of electron accelerators and storage rings normally starts with one or two days of conditioning, during which time the machine is operated with full intensity while one is waiting for the vacuum to improve down to a level acceptable for the physics program. This is a cleaning process which differs not too much from glow discharge cleaning. Unfortunately, no similar effect of self-cleaning could be seen in the ISR. The density of the ion flux produced by 25 A at  $10^{-11}$  torr is only  $10^6$  ions/s cm<sup>2</sup>. That is enough to cause trouble, but by far not enough for cleaning. It would take 30'000 years of operation to reach the standard cleaning dose of  $10^{18}$  ions/cm<sup>2</sup>.

### 2.4 Electropolishing

In the old past we had abandoned electropolishing as cleaning and conditioning treatment after we had noticed that it charges the surface with hydrogen, thus resulting in an increased thermal hydrogen desorption. Looking more recently at surfaces under a scanning electron microscope, we were struck with how smooth and

faultless an electropolished surface looks as compared with all surfaces treated otherwise. We are now in the process of studying again the electron and ion induced desorption from electropolished stainless steel and inonel after having reduced the hydrogen charge by high temperature annealing.<sup>10</sup> The results so far are encouraging.

### 2.5 Multipactoring

We had recently made by surprise the observation that very sharp pressure rises can occur in a well cleaned aluminium chamber when avalanches of secondary electrons are triggered by the RF field produced by a bunched proton beam. This annoying phenomenon is known under the name of multipactoring. The idea has then been born that artificially produced multipactoring would also be an efficient way of conditioning vacuum chambers, either in the laboratory or *in situ*.<sup>11</sup> The advantage with respect to GD cleaning would be that no gas fill is required. On the other hand, electron induced desorption is selective and might clean a surface only partially.

### 2.6 Differences between materials

It is an old and recurring question whether one chamber material is superior to others in the sense that it gives lower desorption rates or is easier to condition than others.

With respect to ion and electron induced desorption, the answer is simply that there are no differences within the scatter margins. This statement is based on the measurement of electron and ion induced gas desorption carried out on a vast number of samples of stainless steel 316 LN, Inconel 600, Inconel 625, Inconel 718, pure copper, pure titanium, Ti 6 Al 4 V, pure aluminium, Al alloy 5086 and others. This is not really surprising if one keeps in mind that even after a 300°C bakeout a chamber wall is still covered with at least 20 monolayers of adsorbed gas. *In-situ* glow discharge cleaning breaks down this layer so much that the impinging ions or electrons have a real chance to strike metal atoms, but in this case the induced desorption is for all metals too small to be measured and to be compared. Exposing, after the glow discharge cleaning, the chamber to air, installing, pumping and baking it, results again in a gas layer of some monolayers with the only difference that this layer contains less CO than before. But, the ions or electrons have again little chance of seeing the underlying metal.

Some practical experiments on the ISR, however, do not fully agree with the laboratory measurements and general considerations discussed above: several titanium chambers without GD cleaning show consistently beam pumping ( $\eta < 1$ ), which is rare on stainless steel chambers under the same conditions.

The differences between materials are considerable in what kind of conditioning is possible and what not. Aluminium can be baked under vacuum to no more than 200°C, stainless steel to at least 400°C, inonel even to 600°C. Aluminium and titanium cannot be annealed at 1000°C to eliminate hydrogen. The first one will melt and the second would suffer from recrystallisation. This can be done with stainless steel and inonel where it is worthwhile mentioning that the residual hydrogen desorption from inonel is lower than that from stainless steel.<sup>12</sup>

## 3. Distributed pumping

An efficient way to take care of beam induced gas loads - particularly in chambers with small aperture - is to provide one sort of distributed pumping all along the chamber in addition to the lumped pumps. Different possibilities exist which will be discussed hereafter.

### 3.1 Sputter-ion pumps

Most electron storage rings (SPEAR,<sup>13</sup> PEP,<sup>14</sup> CESR,<sup>15</sup> ADONE,<sup>16</sup> DORIS,<sup>17</sup> PETRA,<sup>18</sup> VEPP-2M,<sup>19</sup> VEPP-3) have been built or are being built with distributed sputter-ion pumps incorporated into the chamber design and making use of the magnetic field of the bending magnets. Electron storage rings have magnets of, fortunately, only moderate field strength and space is not difficult to find for the necessary extension of the chamber cross-section. The advantage of sputter-ion pumps is that they pump more or less all gases and that they have a practically unlimited capacity so that they can easily swallow the strong initial outgassing when an electron storage ring is filled with beams for the first time after exposure to air. The pumping speed for nitrogen ranges from 100 to 400  $\ell/\text{sm}$ . This relatively high pumping speed permits the operation of machines with only moderate bakeout or without bakeout at all. All distributed sputter-ion pumps are, for reasons of simplicity, of the diode type, because this type needs only one voltage holding electrode instead of two in the case of triode pumps. If tantalum is partly used as cathode material, the pumping speed for argon can be enhanced. The size of the pump cell must be adapted for maximum pumping speed to the strength of the magnetic field in which it has to operate.

No experience exists with distributed sputter-ion pumps on proton storage rings. This possibility is, however, being discussed among others for projected proton storage rings with superconducting magnets. If space can be found inside the extremely costly aperture of superconducting magnets, one would still have to develop a pump which works reliably in the high magnetic field of 4 to 5 tesla. In the optimisation of parameters one should aim not so much for maximum linear pumping speed as in the case of electron storage rings, but for a very low base pressure. It should be remembered that the best commercial lumped sputter-ion pumps reach a base pressure below  $10^{-11}$  reliably only in combination with a titanium sublimation pump.

### 3.2 Sublimation pumping

A standard titanium sublimation pump consists of a filament of 1.5 mm diameter and 150 mm length made of an alloy of 85% Ti and 15% Mo. For activation the filament is heated for  $\approx 1$  min by an AC current of  $\approx 50$  A up to a temperature close to the melting point so that a fresh layer of titanium is deposited by sublimation on the nearest chamber wall which then acts as a getter for most gases. At the operating pressure of proton storage rings of  $10^{-11}$  torr such a getter layer will practically never become saturated and has to be renewed only once after each exposure to air. There are no principle difficulties in installing several such filaments inside an extended section of a vacuum chamber. In electron storage rings with an operating pressure of up to  $10^{-8}$  torr they cannot compete with distributed sputter-ion pumps because sublimation pumps are saturated after adsorption of one monolayer of gas. In proton storage rings, however, they would have the advantage of offering

a good pumping speed in the range of  $10^{-12}$  torr which is not possible to achieve with sputter-ion pumps alone. But there are also disadvantages such as the heavy, permanently installed cable for the 50 A filament current, the heat dissipation inside the vacuum chamber and the problem that the exchange of a burned-out filament requires demounting the whole chamber.

### 3.3 Sputtering from a titanium wire

The difficulties mentioned with Ti sublimation pumps can be eliminated by applying a method which has recently been developed at CERN and which is described in more detail in a separate contribution to this conference.<sup>9</sup> In this method, a titanium wire is mounted inside the chamber and serves as cathode of an argon glow discharge. By transfer of titanium by sputtering from the wire to the walls, a getter film is formed which remains active after the subsequent pump down and bakeout. In the case where there is enough space, the Ti wire might be permanently installed. Otherwise, the wire can be mounted on retractable supports and pulled against the chamber wall once the discharge is finished. It is expected that the titanium film will remain active as long as it is the case for a sublimation pump. If necessary, the reconditioning of a sector by repeating the discharge sputtering could be done within 24 hours.

### 3.4 Non-evaporable getter

Another way of providing distributed pumping speed could be the use of ribbons of so-called non-evaporable getter (NEG).<sup>20</sup> Contrary to more known getter materials which provide pumping only when sublimated, NEGs are activated by heating up to 600-750°C. During the activation the gas saturated top layer diffuses into the bulk and leaves empty places on the surface for the chemical adsorption of H<sub>2</sub>, N<sub>2</sub>, O<sub>2</sub>, CO, CO<sub>2</sub>, etc. A commercially available ribbon of 20 mm width made out of constantan and coated with an alloy of 84% Zr and 16% Al could be installed inside a slightly enlarged vacuum chamber of a proton storage ring. This ribbon could be heated for 24 h at ~ 600°C providing at the same time the heat for the bakeout of the chamber which would stay at about 300°C, depending on insulation and geometry. After the bakeout, at room temperature, the NEG can give a pumping speed of about 300 l/sm for most gases except, of course, for inert gases and methane. At the typical pressure of 10<sup>-11</sup> torr of proton storage rings, the lifetime of the pumping would be practically unlimited. Laboratory tests are in progress at CERN to study this possibility.

### 3.5 Cold Bore

All design studies for future proton storage rings are based on superconducting magnets. It is obviously very tempting to keep also the vacuum chamber wall at liquid helium temperature. The vacuum chamber itself is then acting as a cryopump, the residual gas density is very low, bakeout is unnecessary and one saves in inner aperture of the superconducting magnets.<sup>21,22,23,24</sup> It is, however, not easy to assess how stable the vacuum in a cold bore will be against ion induced pressure rises as they are seen in the ISR. What frightens most people is the fact that the ion induced desorption from condensed hydrogen can be as high as 10<sup>4</sup> molecules/ion.<sup>25</sup> This must, however, be balanced against the high re-adsorption probability which must be close or equal to unity. A simple analysis<sup>26</sup> shows that the critical current in a cold bore of circular section can be estimated from

$$I_{\text{crit}} = \frac{\pi}{4} \frac{D\bar{v}\alpha}{\sigma\eta}$$

where  $I_{\text{crit}}$  is measured in protons/second,  $D$  is the tube diameter,  $\bar{v}$  the average velocity of the desorbed molecules,  $\alpha$  their sticking coefficient,  $\sigma$  the ionisation cross-section and  $\eta$  the ion induced desorption coefficient. The average velocity  $\bar{v}$  is not known and is often, in a pessimistic approach, assumed to be equivalent to a kinetic energy of 4.2 K. The coefficient  $\eta$  depends on the amount of adsorbed gas and on the energy of the incident ions. Most people seem to agree, firstly that only H<sub>2</sub> and He present a problem, and secondly, that H<sub>2</sub> is problematic only for coverage of the order of one monolayer or more. Only an experiment can decide whether H<sub>2</sub> layers of a monolayer or more are stable or unstable. A possibility to avoid the H<sub>2</sub> difficulty altogether is to operate the vacuum chamber at a temperature of 10 K or more.<sup>26</sup> The H<sub>2</sub> adsorption would then remain much below a monolayer, but all gases heavier than He would still be efficiently pumped.

A first small proton accelerator and storage ring with cold bore vacuum chambers is under construction at Berkeley under the name of ESCAR and is expected to give important information.<sup>27</sup> An experimental cold bore section has been built at CERN to be tested with high intensity beam in the ISR. It is being reported on elsewhere at this conference.<sup>28</sup>

## 4. Conclusion

The present state of ultrahigh vacuum technology offers a large scale of methods and is able to satisfy practically all requirements on the vacuum conditions of future storage rings under consideration.

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