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**OBTENTION OF PRESSURES IN THE 10^{-14} Torr RANGE
BY MEANS OF A Zr-V-Fe NON EVAPORABLE GETTER**

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ABSTRACT

Pressures in the low 10^{-14} Torr range have been reproducibly achieved in a 3 m long vacuum chamber. Pumping was provided by a 400 l s^{-1} sputter-ion pump, a titanium sublimation pump and 43.5 m of a Zr-V-Fe Non Evaporable Getter (NEG) strip.

Activation of NEG has been passively achieved during the initial bakeout of the vacuum system.

The pressure has been measured by means of a modified version of the Helmer gauge, designed and manufactured at CERN.

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1. INTRODUCTION

Achieving pressures lower than 10^{-12} Torr implies using low degassing materials and pumps of large pumping speed and low limit pressure. When assuming a surface degassing rate of 10^{-13} Torr ℓs^{-1} cm^{-2} , a reasonable figure for both vacuum fired stainless steel and Al alloys [1], the low 10^{-13} Torr range may be reached only by applying pumping speeds in excess of 1,000 ℓs^{-1} per m^2 of vacuum system surface area.

Whenever the vacuum system consists of a pipe of small diameter, as in the case of particle accelerators, linear pumps should be used to circumvent the obstacle of the conductance limitation. In this case NEG strips provide an ideal solution [2, 3], which has been adopted for the vacuum system of the Large Electron Positron Collider (LEP) at CERN [4, 5].

It has been shown that in this case pressures in the low 10^{-13} Torr range may be achieved by means of a NEG linear pump and sputter-ion pumps (SP) providing a speed of the order of 10 ℓs^{-1} per m^2 of vacuum chamber for CH_4 and rare gases (not pumped by NEG) [6].

The NEG pump of LEP relies on ohmic heating for activation at $740^\circ C$, and therefore needs space for the electrical insulation of the getter strip, resulting in a ratio of vacuum system to NEG strip surface areas of about 14. This value may be regarded as close to the possible minimum for this pumping solution.

A much more favourable situation may be obtained by means of a NEG strip (trade name by SAES Getters St 707[7]), which requires lower activation temperatures ($300^\circ C$ to $400^\circ C$) and consequently may be passively activated during the bakeout of the vacuum system. Since

the electrical insulation is not needed in this case, the NEG strip may be closely packed in contact with the vacuum chamber wall, resulting in less demanding space requirements and in larger pumping speeds. In the extreme case where the inner wall of the vacuum chamber is completely covered by a NEG layer, the vacuum system to NEG surface ratio could be as low as 0.5, i.e. about 30 times lower than in the case of LEP. Assuming a NEG pumping speed of the order of $1,000 \text{ } \ell\text{s}^{-1} \text{ m}^{-1}$ for H_2 (see par. 2), pressures in the low 10^{-14} Torr range might be obtained inside vacuum systems of any length. Obviously, the pumping speed for inert gases should be increased to about $100 \text{ } \ell\text{s}^{-1}$ per m^2 of vacuum system surface area to also reduce correspondingly their pressure.

This solution is very attractive because of its simplicity, and therefore some effort has been devoted at CERN to explore the performance of the passively activated St 707 NEG. Some preliminary results have been published [8] and some more recent data are given in the present paper.

2. THE St 707 NEG

The St 707 is a Zr (70%) - V (24.6%) - Fe (5.4%) alloy which may be activated at temperatures lower than 450°C [7]. Published results [8] show that an appreciable pumping speed is obtained after 300°C resistive activation, and that after 400°C resistive activation the pumping speeds for different gases are about a factor 2 lower than those of the St 101 NEG activated at 740°C . More relevant for the application discussed in this paper are the initial pumping speeds for hydrogen $\text{S}(\text{H}_2)$ after resistive activation at various temperatures.

Fig. 1 shows that $S(H_2)$ increases when increasing the activation temperature up to 400°C , then decreases until about 500°C and finally increases again to reach its highest value at the maximum applicable activation temperature (about 750°C). The origin of this behaviour, observed also for CO and N_2 , will be discussed in a future publication.

When passive activation is applied, the duration of activation coincides with that of the bakeout, which is typically of the order of 24 hours. The variation of $S(H_2)$, obtained after 300°C , 350°C and 400°C resistive activation, as a function of the activation time, is shown in fig. 2. These results indicate that a maximum pumping speed of about $1,000 \text{ l s}^{-1} \text{ m}^{-1}$ for H_2 is obtained after 24 hours at 350°C , while a 400°C bakeout may be dangerous because it may result in a decrease of pumping speed.

3. EXPERIMENTAL SET-UP AND PROCEDURE

The experimental chamber was a stainless steel tube (316 LN, vacuum fired at 950°C) of 3 m length and 160 mm inner diameter, pumped at one end by a sputter-ion pump (SP) of 400 l s^{-1} nominal speed and a titanium sublimation pump (SU) which may be cooled at liquid N_2 temperature. On the pumps side a Bayard-Alpert gauge (B.A.) of CERN design [9], a standard B.A. and a residual gas analyser (R.G.A.) were also installed, while a modified Helmer gauge [10] was mounted at the opposite chamber extremity. The inner surfaces of the chamber were fully covered by a 30 mm wide St 707 NEG strip (total length 43.5 m), wound on a very transparent stainless steel frame to facilitate its insertion in the chamber. In order to measure the degassing rate of the tube and of the measuring gauges, a diaphragm with a calibrated hole of 10 mm diameter was inserted between the chamber and the pumps,

prior to NEG insertion. Bakeouts were carried out at 350°C for 24 hours while pumping with a turbomolecular pumping station providing an effective pumping speed of 3 ℓs^{-1} for N_2 on the chamber. During bakeout the total pressure was monitored by the standard B.A. gauge, which was switched off at the end of bakeout.

4. RESULTS AND DISCUSSION

4.1. Degassing rates

In order to measure the degassing rate of the chamber and of the NEG frame, the vacuum system has been baked at 350°C without NEG strip and with the diaphragm in place. The ultimate pressure obtained in the chamber after bakeout was 6×10^{-12} Torr, while it was in the low 10^{-13} Torr on the pumps side. The resulting total degassing of the chamber was 2×10^{-10} Torr ℓs^{-1} (or about 10^{-14} Torr $\ell\text{s}^{-1} \text{cm}^{-2}$).

The degassing rates of the pressure gauges, measured during the same experiment, were 5×10^{-11} Torr ℓs^{-1} for the Helmer gauge (which is equipped with a Thoria coated tungsten filament) and 5×10^{-10} Torr ℓs^{-1} for the B.A. gauge (bare tungsten filament). These rates are in good agreement with results reported elsewhere [6, 11].

By repeating the same bakeout cycle (with diaphragm) in the presence of the NEG strip, an ultimate pressure of 2.5×10^{-13} Torr has been recorded, which has been imputed to a CH_4 degassing of 2×10^{-12} Torr ℓs^{-1} (or about 10^{-16} Torr $\ell\text{s}^{-1} \text{cm}^{-2}$).

4.2. Ultimate pressures without diaphragm

Fig. 3 shows the pressure evolution during and after bakeout. The step by step initial increase of the baking temperature has shown that NEG pumping begins at 250°C. It should be remembered, however, that the applied external pumping speed during bakeout is only about $3 \text{ } \ell\text{s}^{-1}$ and that, consequently, even a very small additional pumping would result in a large pressure variation.

After bakeout the pressure falls within 2 hours to below 10^{-12} Torr and then it keeps decreasing very slowly over a period of about 3 months, when it reaches the high 10^{-14} Torr range with the SU at room temperature and about 3.5×10^{-14} Torr with the SU cooled by liquid N_2 .

The pressure improvement consequent to N_2 cooling of the SU has progressively decreased with decreasing ultimate pressure. The gas species desorbed during SU heating to room temperature are CH_4 (about 75%) and Ar (about 25%).

By combining the various results reported above, the resulting pumping speed for CH_4 of the SP is about $40 \text{ } \ell\text{s}^{-1}$ at the ultimate pressure of about 2×10^{-13} Torr on the pump, while the speed of the SU (at 77 K) for the same gas is about $700 \text{ } \ell\text{s}^{-1}$. Taking into account the conductance of the chamber ($200 \text{ } \ell\text{s}^{-1}$ for CH_4) and assuming a uniform CH_4 degassing, the calculated contribution of this gas to the Helmer gauge reading is 6×10^{-15} Torr.

On the other hand, the N_2 equivalent contribution of H_2 estimated from the measured chamber degassing ($2 \times 10^{-10} \text{ Torr } \ell\text{s}^{-1}$) and NEG pumping speed ($1,000 \text{ } \ell\text{s}^{-1} \text{ m}^{-1}$) is about 2.5×10^{-15} Torr. By adding these two pressures, a value of

about 10^{-14} Torr is obtained, while the measured pressure was 3.5×10^{-14} Torr. This discrepancy is much larger than that which experimental uncertainty can justify.

4.3. Origin of the ultimate pressure discrepancy

Various possible causes of the above discrepancy have been analysed, as discussed below.

The 77 K pumping speed of the SU for CH_4 has been questioned. To clarify this point, a He bath cryopump [12] has been added to the vacuum system between the pumps and the chamber. The condensation pumping speeds of this pump are about $4,500 \text{ } \ell\text{s}^{-1}$ for H_2 and about $1,500 \text{ } \ell\text{s}^{-1}$ for CH_4 . On cooling the cryopump to 4.2 K, a further decrease of 2.5×10^{-15} Torr has been noticed on the Helmer gauge reading, which confirms both the previously calculated CH_4 pressure and the estimated speed of the SU at 77 K for this gas. In the presence of the cryopump, the estimated CH_4 pressure on the Helmer gauge was about 5×10^{-15} Torr.

In order to check if the H_2 dissociation pressure of the NEG could contribute to the ultimate pressure, the chamber and the NEG strip have been heated to 120°C . A pressure increase up to 1×10^{-12} Torr has been recorded. If this value is scaled down to room temperature according to the Sievert law, a contribution due to H_2 dissociation pressure of the NEG of about 10^{-17} Torr is obtained, showing that this hypothesis should also be disregarded. As mentioned in par. 4.1., the degassing rate of the Helmer gauge is about $5 \times 10^{-11} \text{ Torr } \ell\text{s}^{-1}$. This gauge was inserted in a tube of 60 mm bore, with the grid end at the connection between this tube and the chamber. When assuming that in this situation the pumping speed for H_2 on the gauge is about $1,000 \text{ } \ell\text{s}^{-1}$ the

resulting pressure on the gauge is 2.5×10^{-14} Torr, i.e. just about the difference observed.

The gauge degassing hypothesis was also confirmed by a parabolic increase of the gauge reading observed at the ultimate pressure when varying its electron emission current. The same variation carried out at 2×10^{-13} Torr showed an almost linear dependence.

5. CONCLUSIONS

The reported results show that ultimate pressures in the low 10^{-14} Torr range may be obtained reproducibly in a system operating at room temperature by means of a NEG linear pump passively activated during bakeout at 350°C . This result, obtained with a 3 m long chamber, could be obtained on indefinitely long vacuum systems as well, due to the modularity of the solution adopted. Some evidence is reported showing that the recorded ultimate pressure is mainly consequent degassing due to the gauge. In the absence of this degassing, pressures in the 10^{-15} Torr range should be obtained.

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Figure Captions

Fig. 1 Variation of the initial pumping speed for H_2 of the St 707 NEG as a function of the activation temperature (duration of activation 45'). The full line should be considered as a guide to the eye.

Fig. 2 Variation of the initial pumping speed for H_2 of a St 707 NEG as a function of the heating time and for various heating temperatures. The full line should be considered as a guide to the eye.

Fig. 3 Evolution of the total pressure during and after bakeout. Note the expanded pressure scales on the right side of the figure. The pressures during bakeout were measured by means of a standard B.A. gauge, the ultimate pressures by means of a modified Helmer gauge.

Fig. 1

Fig. 2

Fig. 3