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R. Giannantonio, M. Succi, and C. Solcia
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A preliminary study on the pumping performance of a cryopump combined with a high-capacity non-evaporable getter (NEG) pump was performed. The pumping speed, the pump-down time, and the gas composition in a standard UHV chamber were evaluated using the cryopump alone and combined with a high capacity getter pump made from sintered porous getter material. In order to exploit the high specificity of the getter pump for hydrogen and water vapor, the cited quantities were measured both before and after a baking treatment. It was found that the ultimate pressure in the baked chamber could be greatly improved using the getter pump and that pump-down times of the unbaked chamber were consistently reduced due to the very high pumping speed of NEGs for water vapor.

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I. INTRODUCTION

A. Combination of non-evaporable getter pumps with conventional pumping systems

The number of applications where extremely low pressures are to be achieved is increasing. Instances where UHV conditions are necessary are, for example, those related to semiconductor processing, surface science, and high energy physics, where the need for a vacuum of the order of $10^{-11}$ Torr exists.

The choice of the pumping system is of great importance in attaining these vacuum levels. Conventional pumping equipment, based on ion pumps, cryopumps, and turbomolecular pumps can in principle achieve sufficiently low ultimate pressures, provided that the vacuum system is realized with suitable materials and is submitted to a prolonged baking treatment.\(^1\)

In the UHV pressure range, however, the cited pumping systems fail to pump the main gaseous species in the system, i.e., H\(_2\). Their combination with a non-evaporable getter (NEG) pump, offering utmost sorption characteristics for H\(_2\), may therefore turn out to be useful when the overall pumping speed and the ultimate vacuum have to be further improved.

It is thus possible to take advantage of the synergistic action arising from the combination of a NEG pump, used to remove reactive gases such as CO, CO\(_2\), N\(_2\), H\(_2\), and H\(_2\)O, with a conventional pumping system able to pump inert gases such as He, Ne, Ar, and hydrocarbons.

Recently, several articles have appeared on the coupling of NEG pumps with ion and sputter-ion pumps\(^2\)–\(^7\) and, to a lesser extent, with turbomolecular pumps\(^8\) demonstrating the growing interest in this approach.

B. Combination of NEG pumps with cryopumps

Cryopumps are the preferred choice among the above mentioned pumping systems in applications where a very high pumping speed and a very “clean” vacuum is needed, such as in the semiconductor industry. The cryopumps generally used for semiconductor manufacturing are of the Gifford–Mac Mahon type, usually operating at 12–14 K. At these temperatures, the ultimate pressure attainable in the vacuum chamber is limited by the equilibrium pressure of hydrogen adsorbed on the high-surface material, e.g., charcoal, molecular sieve, etc., located in the inner walls of the 2nd stage cryopanel. For example, when no more than half a monolayer of hydrogen is adsorbed on the surface of coconut charcoal, the equilibrium pressure of H\(_2\) at 14 K is $>10^{-10}$ Torr.\(^9\) A NEG pump should therefore prove to be useful in lowering the ultimate pressure, since the H\(_2\) equilibrium pressure on NEG alloys is much lower than on charcoal.\(^10\)

Further, a very common problem arising during in situ thermal treatments is due to the heating of the cryopanels caused by the heat load: the temperature of the cryopanels can rise up to 20 K so that hydrogen can desorb from the charcoal into the vacuum chamber. To prevent an excessive overpressure in the chamber it is possible to (a) reduce the heating of the cryopanels using a baffle or (b) use a NEG pump to quickly eliminate the evolved hydrogen. In case (a) the reduced conductance from the pump to the vacuum chamber will cause a lower pumping speed, i.e., a higher ultimate pressure. In case (b) no appreciable variation of the base pressure should be observed.

Limited information and actual results have indeed been published on combinations of NEG pumps with cryopumps. Preliminary results concerning the behavior of a vacuum system where both a cryopump and a NEG pump are used are reported here in terms of ultimate attainable vacuum, residual gas composition, and pump-down times.

II. EXPERIMENTAL SETUP

A. UHV system

The UHV system used in this work is shown schematically in Fig. 1. It consists of a stainless steel cylindrical chamber having a volume of 24.5 l and an inner surface area of 4500 cm\(^2\); the volume and the surface area of the tubulations are 15.5 l and 5500 cm\(^2\), respectively. The chamber can
be opened at one side to position a getter pump through a 12 in. CF flange. The other side is connected through an 8 in. CF gate valve to the cryopump. Measurements of absolute pressure inside the chamber were made using both a Bayard–Alpert ionization gauge and an extractor gauge (Leybold IE-414 and IE-514, respectively, connected to a control unit, IM-520). Partial pressure measurements were performed with a quadrupole mass spectrometer equipped with 90° scanning electron microscopy (SEM) (Balzers QMA 125/QMG 421 C). The introduction of gases in the system was done through a sampling chamber where pressure could be monitored with two capacitive manometers covering the 10^{-4}–1 Torr and 10^{-1}–1000 Torr pressure ranges (MKS Baratron). The main chamber is also connected to a rough pumping system consisting of a 60 l/s turbomolecular pump (Balzers) backed by a 12 m^3/h rotary pump (Edwards). Back-diffusion of oils from the rotary pump was prevented using a catalytic trap (Balzers). All of the cited instrumentation was interfaced to a computer to achieve complete automation, i.e., good repeatability of measurement cycles.

### B. Cryopump

The cryopump used in this work was a Leybold RPK 800 S2, a closed-loop, gaseous helium cryopump with a 2nd stage temperature of 14 K. The main features of this pump are collected in Table I.

### C. Getter pump

The high-capacity (HC) NEG pump, installed inside the UHV chamber in a so called immersed configuration, was a standard CapaciTorr® pump, made and commercialized by SAES Getters. The main characteristics of this high-capacity getter pump are collected in Table II. Gettering activity is due to two cartridges each containing 54 fins prepared by mixing and sintering proper amounts of St707 (Zr 70% V 24.5% Fe 5.5%) and Zr powder. Each fin weigh 7 g and exposes 1 m² of (apparent) surface area to the gases. The characterization and a detailed description of the optimized geometry of the pump have already been published elsewhere.

### III. RESULTS AND DISCUSSION

Measurements were made in the chamber pumped by the cryopump alone and combined with the HC NEG pump.

Aiming at measuring the ultimate pressure and the gas composition in the baked chamber, the following experimental procedure was used. First, the system was first submitted to a bake-out process at 200 °C for 3 days. If present, the getter pump was held at this same temperature for the same period (with the exception of the activation step) to avoid both an excessive contamination and a significant activation of the getter material. The baking treatment was performed with valves V1 and V3 closed and with valve V2 open (see Fig. 1), i.e., pumping the chamber with the turbomolecular pump alone (this was made to avoid overheating of the cryo-panels with consequent desorption of hydrogen followed by readsoption of the same gas on the walls of the chamber during cool-down). Second, if present, the HC NEG pump was activated at 540 °C for 45 min. Third, after sufficient time elapsed so that the whole system (chamber+HC NEG pump) was at room temperature (about 12 h) the residual pressure and gas composition were measured.

### Table I. Cryopump characteristics.

<table>
<thead>
<tr>
<th>Pump type</th>
<th>Leybold RPK 800 S2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pumping speed (l/s)</td>
<td>H₂O 650</td>
</tr>
<tr>
<td></td>
<td>H₂ 500</td>
</tr>
<tr>
<td></td>
<td>CO 200</td>
</tr>
<tr>
<td>Capacity (bar×1 CO)</td>
<td>20</td>
</tr>
<tr>
<td>Activation</td>
<td>45 min at 540 °C</td>
</tr>
<tr>
<td>Getter material</td>
<td>Porous Zr–V–Fe alloy</td>
</tr>
</tbody>
</table>
The mean results of several sets of tests performed in the baked chamber with and without the getter pump are summarized in Table III.

At the end of each measurement cycle, the rise of pressure in the chamber, which was kept isolated by closing valve V1, was recorded with both the ionization gauge and the mass spectrometer. Typical variations of the partial pressures of the main gases, while the chamber remained static for 30 min with and without HC NEG pump are shown in Figs. 2(a) and 2(b), respectively. The specific outgassing rate of the chamber could be calculated from the pressure rate-of-rise experiments to be about $4 \times 10^{-12}$ Torr l s$^{-1}$ cm$^{-2}$.

It is worth noting that the net decrease in the ultimate pressure, from $8 \times 10^{-10}$ to $5 \times 10^{-11}$ Torr, when using the getter pump in addition to the cryopump, is mainly due to the very high pumping speed for H$_2$ of the HC NEG pump. From the pump-down data and the calculated outgassing rate, this pumping speed can be estimated as greater than 750 l s$^{-1}$. It can be noted in Fig. 2(b) that the ion current at m/e=15 slightly rises when closing valve V1. The very small amount of hydrocarbons (mainly CH$_4$) that give rise to this peak are however pumped by the HC NEG pump; their surface concentration on the getter material is as low as $1.5 \times 10^{-8}$ molecules/surface atom. It is also worth noting that the rise of pressure from the base value of $5 \times 10^{-11}$ to $2 \times 10^{-9}$ Torr is essentially due to the backstreaming of helium from the cryopump into the chamber through the Viton seal of the gate valve V1.

Further experiments were made to test the ability of the cryopump to pump water vapor with and without the aid of the HC NEG pump in the unbaked chamber. In Fig. 3 the typical partial pressure profiles of H$_2$ and H$_2$O (i.e., the main gases) under pump-down in the unbaked chamber in the unbaked chamber with and without NEG pump.

<table>
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Gas composition

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presence of the HC NEG pump are shown. The procedure used was the following:

(i) one atmosphere of air was introduced into the system;
(ii) the system was left at this pressure in static conditions for 30 min; (iii) pump-down was started initially using the turbomolecular pump alone; (iv) when the pressure was lower than $1 \times 10^{-4}$ Torr, valve V2 was closed and valve V1 was opened (at time $t = 0$) with the system under cryopumping; (v) the chamber was cryopumped for 18 h; (vi) HC NEG pump activation followed at 540 °C for 2 h; and (vii) after waiting for the getter pump to cool to room temperature (which required 2 h), a further heating of the pump at 150 °C for 2 h was performed. The striking result emerging from Fig. 3, is that after the NEG pump activation, the partial pressure of H$_2$O, here expressed as the electrometer ion current ($I_e$), decreases, between times $t = 18$ h and $t = 28$ h, by a factor of 40. Since the temperature of the inner walls of the chamber is never higher than 80 °C, even during the NEG activation (i.e., assuming that the outgassing rate of water changed only by means of the rather usual $t^{-1}$ law$^{12}$), the gettering action of the HC NEG pump increases the total pumping speed for H$_2$O by about a factor of 25. This remarkable effect is both due to the “nude” mounting of the HC NEG pump inside the chamber and, obviously, to the peculiar pumping action of the getter pump with respect to water vapor, as clearly shown in Fig. 4. Note that a sorption curve of H$_2$O on the HC NEG pump at 25 °C and $3 \times 10^{-6}$ Torr is reported (sorption measurements made according to Ref. 13). It is also interesting to note that the heating of the HC NEG pump at 150 °C for 2 h also enhances the speed of water vapor pumping: the effect of temperature on the pumping speed for H$_2$O of a NEG pump (but not of a HC NEG pump) has indeed already been observed and discussed elsewhere$^{14}$.

As a consequence of the above mentioned phenomena, the time required to reach a pressure of $2.5 \times 10^{-9}$ Torr in the unbaked chamber, without activating the NEG pump, was
>50 h while under the pumping action of CapaciTorr®, this same pressure was reached in <30 h.

**IV. CONCLUSIONS**

The addition of a HC NEG pump in a system equipped with a cryopump has proven to be effective in reducing the ultimate pressure by one order of magnitude. The improvement is mainly due to the increase of the pumping speed for hydrogen and water vapor, thanks to the high sorption efficiency of NEG material for these gas species. The dynamics of hydrogen exchange between the cryopanels and the NEG alloy needs to be further investigated.

The use of a HC NEG pump allows for the reduction of the pump-down time to reach e.g., $10^{-9}$ Torr in the unbaked chamber due to the very high pumping speed of the HC NEG pump for water vapor.

The pressure rate-of-rise in the static system is greatly lowered in the presence of the HC NEG pump. This could prevent excessive surface contamination of samples in surface science and semiconductor manufacturing systems possibly caused by a temporary black-out or cryopump failure.


10 SAES Getters, St707 and St172 Catalogs, October 1989.
13 ASTM procedures F798-82.