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**AN UHV TRANSPORTABLE CONTAINER BASED ON A NEG PUMP**

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**Abstract**

Our work concerns the attainment of UHV pressures by using the non evaporable getter (NEG) pump in conjunction with turbomolecular or ion pump of small size. Studies related to surface science show that vacuum conditions in the range of UHV ( $10^{-10}$  mbar) are required for very reactive materials, such as niobium or titanium elements. It is a very common practice to perform some surface treatments on these materials and then facing with the problem to preserve the surface state so created from the atmospheric contamination for long times (storing, transport from one laboratory to another, etc...). The aim of this work is to find a vacuum system that is easy to construct, inexpensive and almost completely free from any kind of power supply.

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## 1 – INTRODUCTION

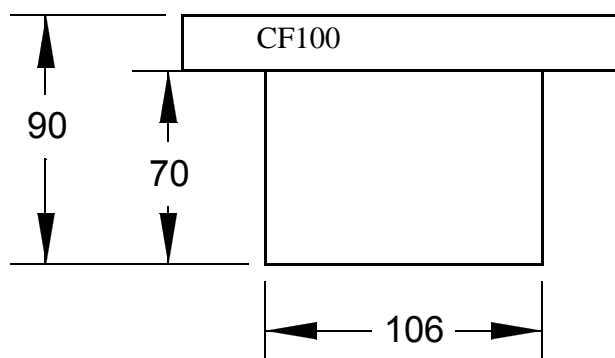
Nowadays the problem of the transport of samples or of particular systems among different laboratories is very common. In particular we had to face the problem of transporting under UHV conditions radio-frequency (RF) cavities made of Nb, that were prepared in one laboratory and were tested in another one. As Nb is one of the most reactive material, pressure of about  $10^{-10}$  mbar are necessary in order to keep unchanged with time the surface state, where the RF superconducting processes take place <sup>(i,ii)</sup>.

The solution to the problem of developing a transportable UHV device can be solved by using a small ion pump, supplied in a pulsed way by a battery (this system is not yet available), combined with a non evaporable getter (NEG) pump. If the requirements concerns small size chambers and it is possible to perform a rigorous bake out, then the ion pump can be replaced by a Penning gauge for UHV. This device takes two advantages: the read of the pressure inside the vacuum system and a good pumping action for those gases that the NEG pump cannot remove (mainly the inert gases).

The processes of operation of the getter materials and in particular of the NEG (Non Evaporable Getter) pumps involve mainly the superficial region of the material, through phenomena such as the adsorption, the diffusion, the desorption. It turns out therefore of fundamental importance the chemical characterization of the surface of the NEG pumps, in order to understand and to justify the behaviour of these alloys in "anomalous" conditions. In fact it is possible that, because of malfunctionments of the systems of low vacuum or for other reasons not directly controllable from the user, the getter material comes exposed to polluting atmospheres, and it is therefore necessary to know the consequences of this pollution, in order to being able, eventually, to operate an appropriate regeneration. In such way the various methods of surface analysis currently available become the more effective means to face such problems in a systematic manner. In the following paper we bring back an example of XPS (X-ray Photoelectron Spectroscopy) analysis carried out on getter films (St707), exposed for approximately 48 hours to the vacuum produced by a rotary pump.

## 2 - THE GETTER PUMPS

We tried to make getter pumps that could be useful not only for our specific application, but also for general problems in the vacuum field. Our goal is to obtain a system easy to use, inexpensive and compatible with the other pumping devices. In order to satisfy these requests, we decided to use a NEG pump.



**FIG. 1.** Schematic view of the outside of the getter pump mounted on the CF100 flange.

The choice of the flanges was subordinated to their use in the vacuum devices: the most widespread are the CF63 and the CF100, while the CF35 has a low pumping speed and a small capacity and the CF150 is rarely used. Then the system was projected for the CF100 (Fig. 1) and CF63 flanges and the tests were performed on the CF100.

The NEG pump was based on the commercially available St 707 getter alloy <sup>(iii)</sup>.

### 3 – EXPERIMENTAL

The experimental set-up is schematically shown in Fig. 2 and it is based on a metal vacuum system.

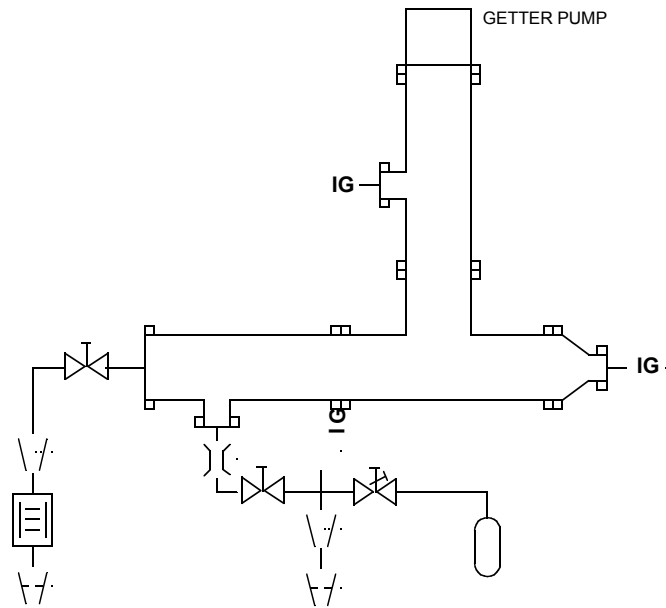
The low vacuum was ensured by the combination of a rotary pump with a sorption pump, mounted before a turbomolecular pump (Varian V70D), whose ultimate pressure is  $10^{-9}$  mbar.

The measurements of the pumping speed were performed by means of the “dynamic flow method”, by inserting in the main manifold a known pressure of a given gas across a known conductance and measuring the pressure inside the chamber <sup>(iv)</sup>.

The gas inlet system consisted of:

- a bottle filled with the purified gas;
- a leak valve connecting the gas bottle with the inlet apparatus;
- a ionization gauge Varian UHV24 connected to a control unit
- a known conductance (0.7 l/s for  $N_2$ , 1.9 l/s for He and 2.7 l/s for  $H_2$ ) between the dosing system and the main chamber.

The entire gas inlet apparatus is pumped by a combination of a rotary pump with a turbomolecular pump. The main manifold is equipped with two ionization gauges (Varian UHV24) and two sensors for the measure of the temperature of the chamber and of the NEG pump.

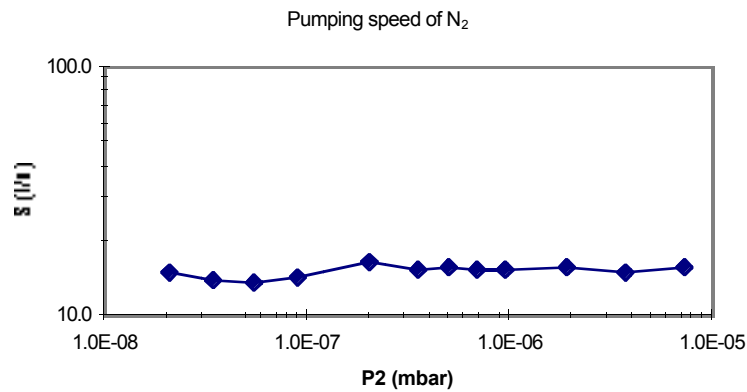


**FIG. 2.** The experimental apparatus used for the pumping speed measurements.

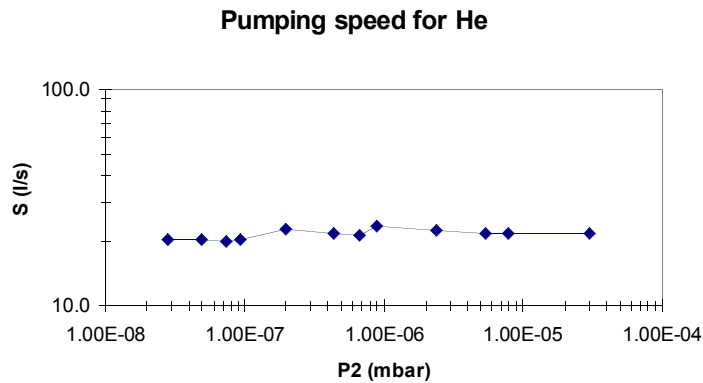
## 4 - RESULTS

### 4.1 - BAKE - OUT AT 150C

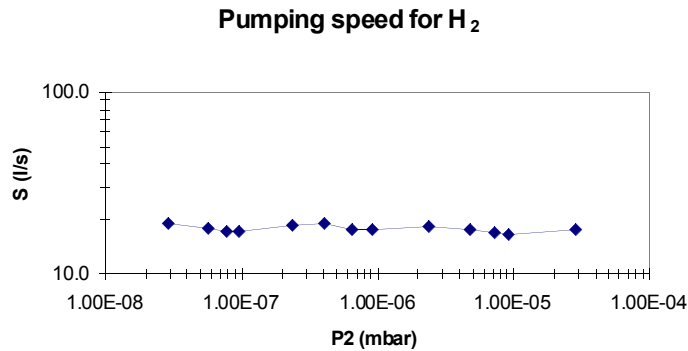
At first, we used the turbomolecular pump to reach a pressure of  $5 \times 10^{-7}$  mbar; then we baked the entire system (included the getter pump) at 150C for 24 hours, obtaining an ultimate pressure of  $5 \times 10^{-9}$  mbar. In these conditions we performed the measurements of the pumping speed of the turbo pump: by inserting  $N_2$ , He and  $H_2$  in the chamber (Figg. 3 - 5), we found pumping speed values in good agreement with the theoretical ones (Table 1).



**FIG. 3.** Pumping speed of  $N_2$  after the activation at 150C.



**FIG. 4.** Pumping speed of He after the activation at 150C.



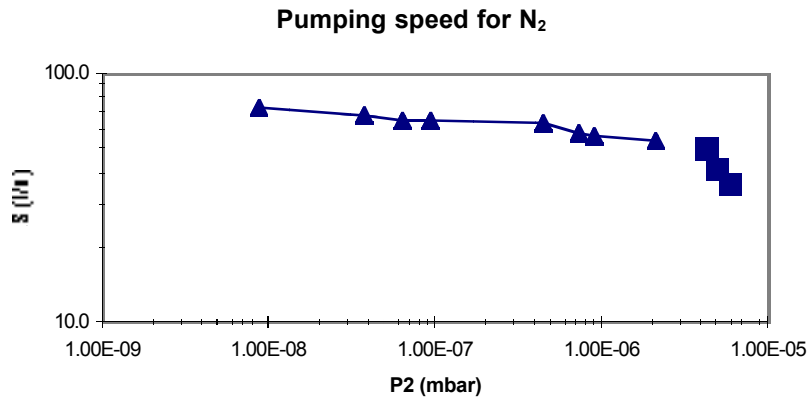
**FIG. 5.** Pumping speed of H<sub>2</sub> after the activation at 150C.

**TAB. 1.** - Calculated and measured pumping speeds inside the experimental chamber for the turbomolecular pump Varian V70D.

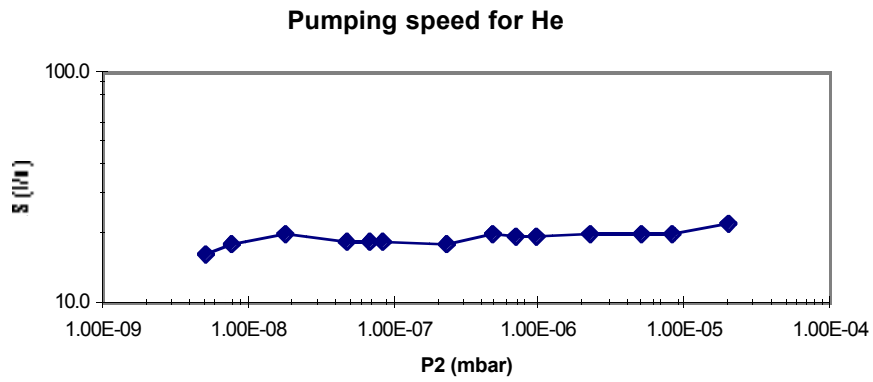
Gas	Theoretical pumping speed (l/s)	Measured pumping speed (l/s)
N <sub>2</sub>	20	15
He	21	21.3
H <sub>2</sub>	17.5	17.5

#### 4.2 - ACTIVATION AT 300C

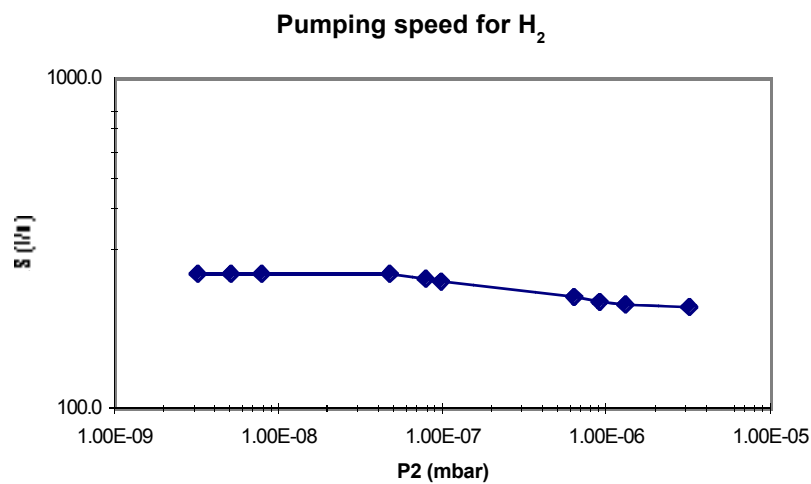
The partial activation of the NEG pump was carried out by heating the pump at 300C for 24 hours, keeping in the meantime the remaining of the system at 150C. At the end of the procedure of activation the system cooled down to room temperature in about 12 hours, reaching an ultimate vacuum of  $5.5 \times 10^{-10}$  mbar. The measurements of the pumping speed were performed with the leak valve between the gas inlet apparatus and the main system open. The results are reported in Figg. 6 - 8.



**FIG. 6.** Pumping speed of N<sub>2</sub> after the activation at 300C.



**FIG. 7.** Pumping speed of He after the activation at 300C.



**FIG. 8.** - Pumping speed of H<sub>2</sub> after the activation at 300C.

The increase of the pumping speed for  $N_2$  and  $H_2$  is evident, while the pumping speed for He remains unchanged (19.1 l/s), as this gas is not removed by the getter pump.

Moreover the curves representing the pumping speeds have the typical decreasing trend with the increasing quantity of adsorbed gas <sup>(4)</sup>; therefore we can extrapolate an approximate average pumping speed, that is 63 l/s for  $N_2$  and 240 l/s for  $H_2$ .

### 4.3 - ACTIVATION AT 350C

In the following activation we heated the NEG pump at 350C for 8 hours, always keeping the main chamber at 150C. The system got room temperature after 12 hours with an ultimate pressure of  $2 \times 10^{-10}$  mbar.

The pumping speed values are plotted in Figg. 9 - 10 and there is a clear increase of all the values.

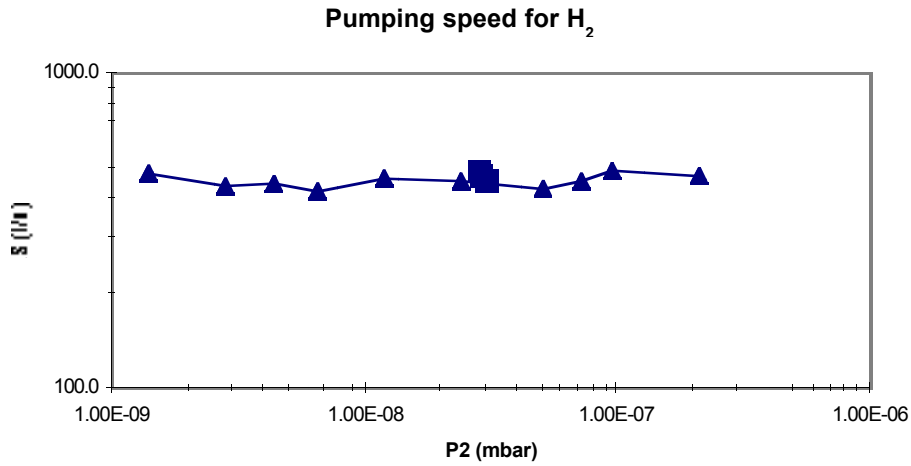


FIG. 9. - Pumping speed of  $H_2$  after the activation at 350C.

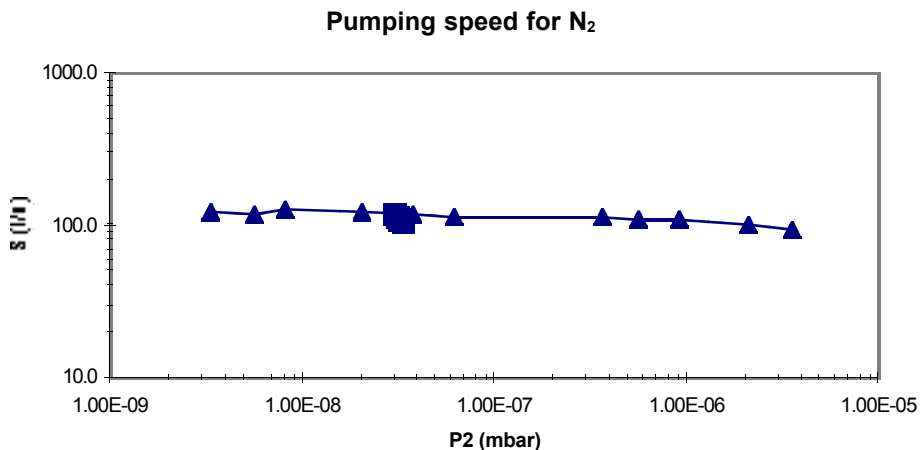


FIG. 10. - Pumping speed of  $N_2$  after the activation at 350C.

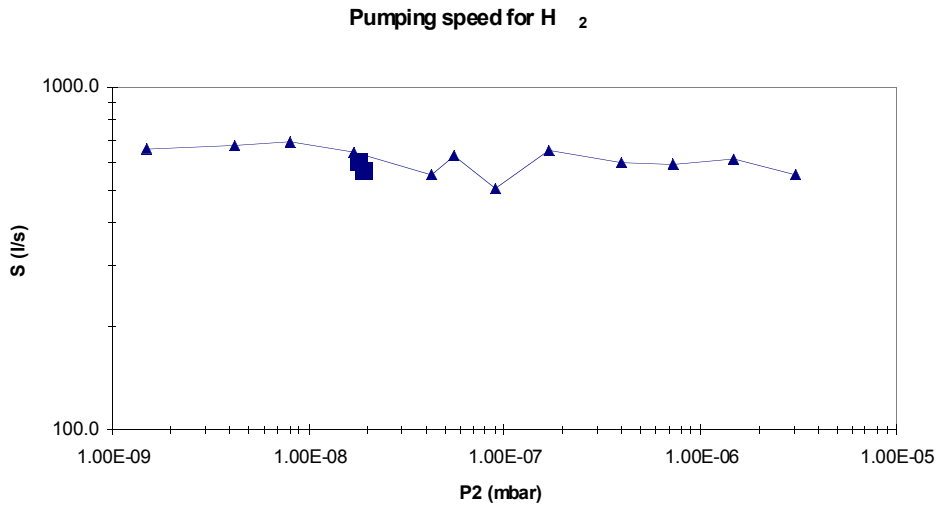
In order to verify the stability with time of our measurements, we inserted in the chamber a flux of gas at constant pressure and we measured the pumping speeds, that resulted almost

constant (this data are reported as ■ in Figg. 9 - 10). The average pumping speeds are is 115 l/s for N<sub>2</sub> and 450 l/s for H<sub>2</sub>.

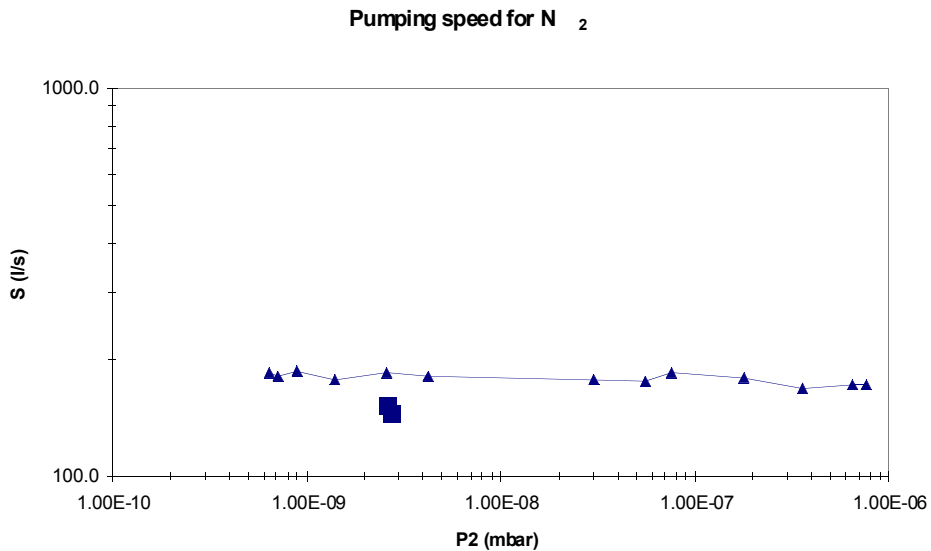
#### 4.4 - ACTIVATION AT 400C

The complete activation of the NEG pump was performed by heating the getter at 400C for 2 hours (the temperature of the main chamber was kept at 150C). The ultimate pressure reached by the entire system at room temperature after 12 hours was  $1.2 \times 10^{-10}$  mbar.

The trends of the pumping speed as a function of the pressure inside the main chamber are plotted in Figg. 11 - 12.



**FIG. 11. -** Pumping speed of H<sub>2</sub> after the activation at 400C.



**FIG. 12. -** Pumping speed of N<sub>2</sub> after the activation at 400C.



Also in this case we repeated the tests on the stability of the pumping speeds, that remained almost constant with time.

The average pumping speeds increased to 180 l/s for N<sub>2</sub> and 630 l/s for H<sub>2</sub>.

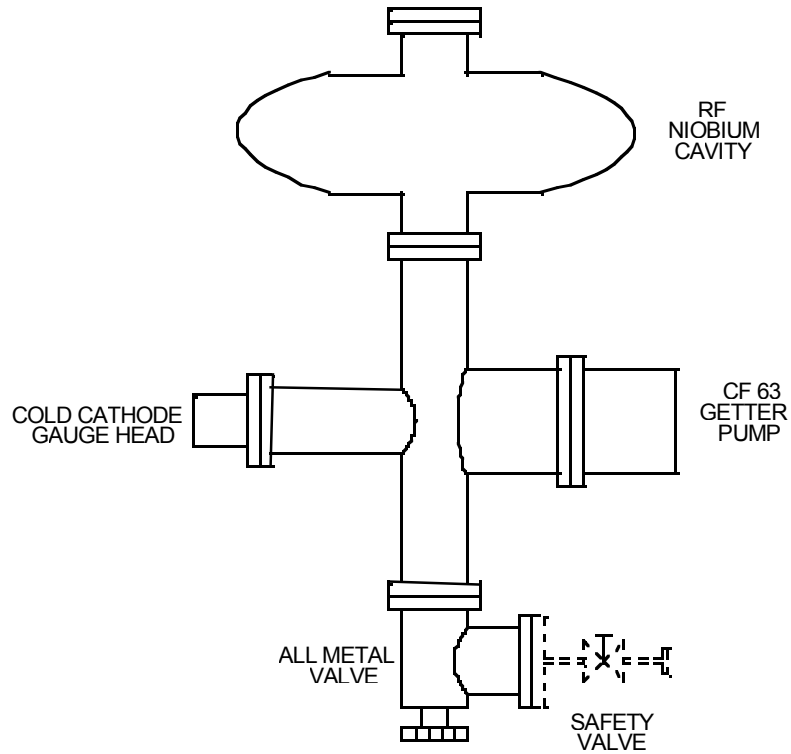
Recently a series of papers about the getter films obtained by sputtering and about the study of their characteristics as pumping systems have been published <sup>(v)</sup>. In these works the vacuum system around the getter pump was kept at room temperature during the activation of the getter film at different temperatures, in order to prevent the pumping action of the getter pump towards the gases produced by an eventual bakeout. This precaution had also the purpose to preserve the getter film from a too fast ageing, as the film thickness was only about 1 µm.

The goal of our work, on the contrary, is different, as we want to preserve the surface state of the system maintained under UHV condition from any kind of contamination for a long time. It is for this reason that we chose as operative condition of work to perform a bakeout at T=150C of the main manifold during the different phases of activation of the NEG pump. In this way we were able to minimise the contamination of the transported system from the gases that were emitted in consequence of the bakeout and that were removed by the NEG pump (it must be remembered that the samples or the systems kept under UHV are made of Nb, a very reactive material). Moreover the getter strip used in our apparatus has a thickness of 75 µm and therefore the ageing caused by an excessive pumping activity is less critical.

## **5. - THE UHV TRANSPORTABLE SYSTEM**

After the good and encouraging results obtained with the test system of Fig. 2, we pass to the construction of a real transportable system, effectively free from any kind of power supply. The system that we developed and tested is represented in Fig. 13.

The tests performed proved that this container maintain a pressure inferior to  $2 \times 10^{-10}$  mbar for periods of time longer than 15 days.



**FIG. 13. Scheme of the UHV transportable system used to transfer a RF cavity.**

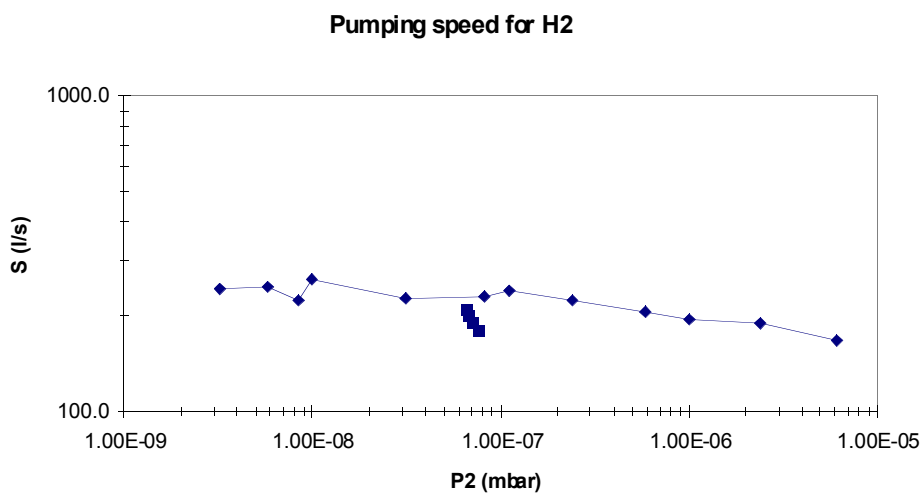
## **6. - CONCLUSION**

The system tested in this paper confirms that a simple getter pump can produce elevated pumping speeds with a lot of advantages: small size of the pump, easy and sure use, very low cost, possibility of developing UHV transportable systems. If this getter pump is used in conjunction with a turbomolecular pump, whose ultimate pressure is  $1 \times 10^{-9}$  mbar, it reduces this pressure of one order of magnitude, increasing noticeably the pumping speed.

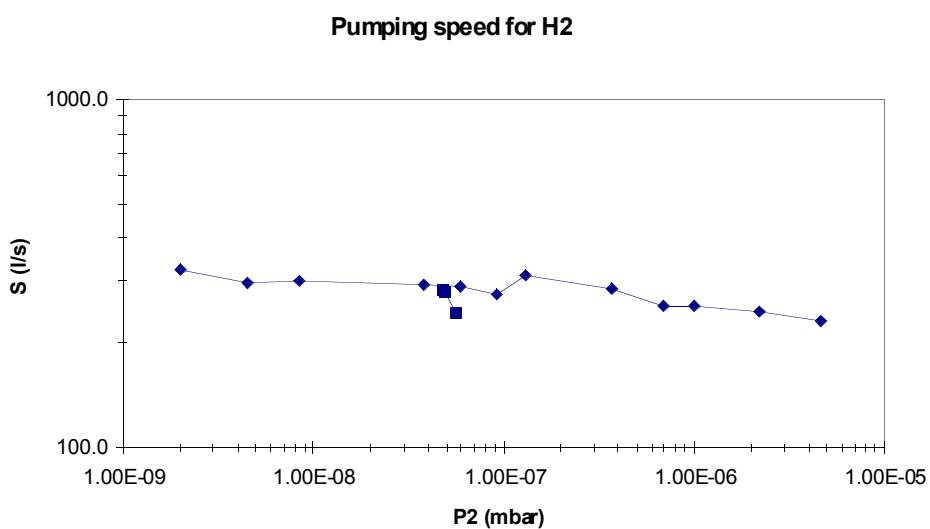
This system proves that for a wide range of applications (mainly for those system that can be heated at 400C) the use of large turbomolecular or ion pump can be substituted by the getter ones.

## **7. - AN OUTLINE ABOUT THE GETTER POISONING**

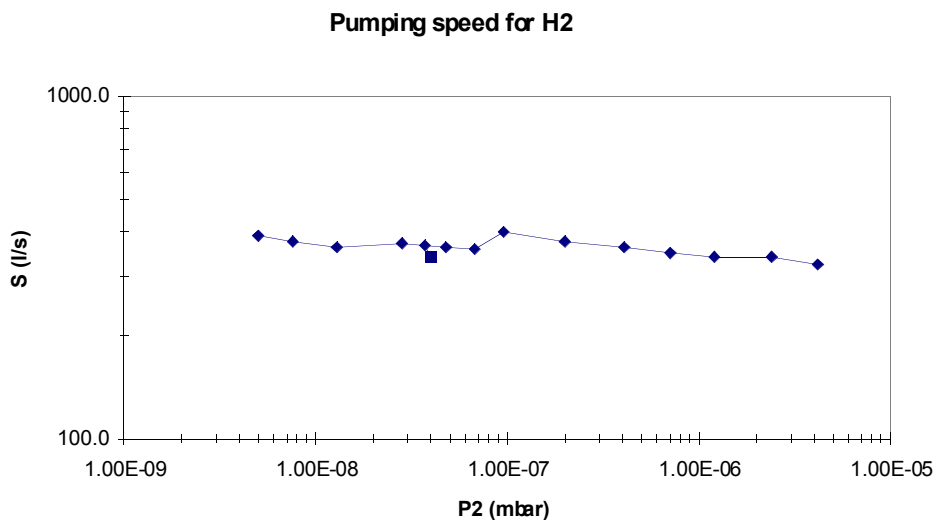
During the measurements we opened the vacuum chamber up to air and during the pumping down phase there was a lack of current, so that the turbo pump did not restart and the system remained in a low vacuum ( $p=1 \times 10^{-4}$  mbar, obtained by using only the rotary and the sorption pump) for 2 days. After a bakeout of the entire system, we repeated the pumping speed measurements for H<sub>2</sub>. The results (Fig. 14 - 17) show that there was a drop in the pumping speed, even if the ultimate vacuum was not influenced in an appreciable manner.



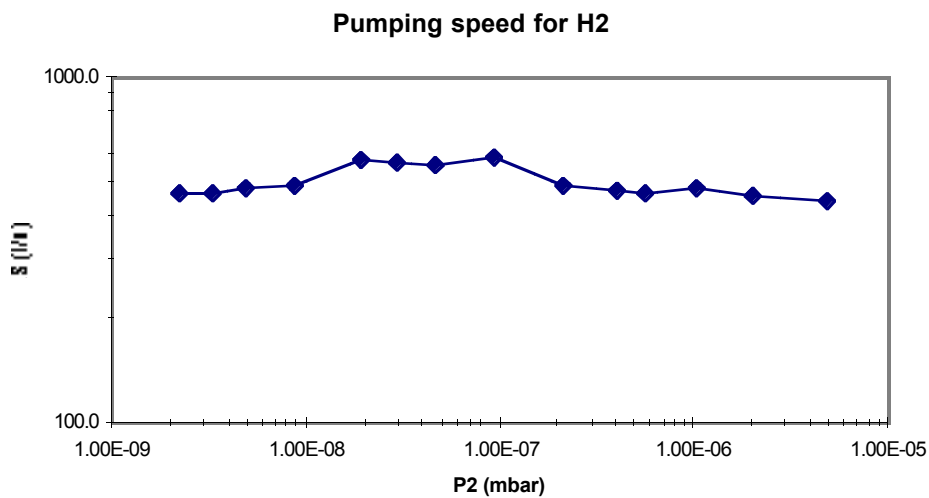
**FIG. 14.** Pumping speed of H<sub>2</sub> after the poisoning and the successive activation at 300C.



**FIG. 15.** Pumping speed of H<sub>2</sub> after the poisoning and the successive activation at 350C.



**FIG. 16.** Pumping speed of H<sub>2</sub> after the poisoning and the successive activation at 420C.



**FIG. 17.** Pumping speed of H<sub>2</sub> after the poisoning and the successive activation at 500C.

## 8. - DESCRIPTION OF THE SAMPLES FOR THE XPS ANALYSES

XPS analyses have been carried out on two samples of a getter strip (St707 type). The first sample analyzed has been taken from a new and never used strip; the second one comes from a strip used in a normal UHV system, that has been exposed to a low vacuum (produced by a system constituted by a rotary pump that has in series a sorption pump) for approximately 48 hours, after the first activation of the NEG pump and an exposure to

atmospheric pressure. Our hypothesis is that the second sample should show the effect of the pollution probably produced from the oils used inside the rotary pump.

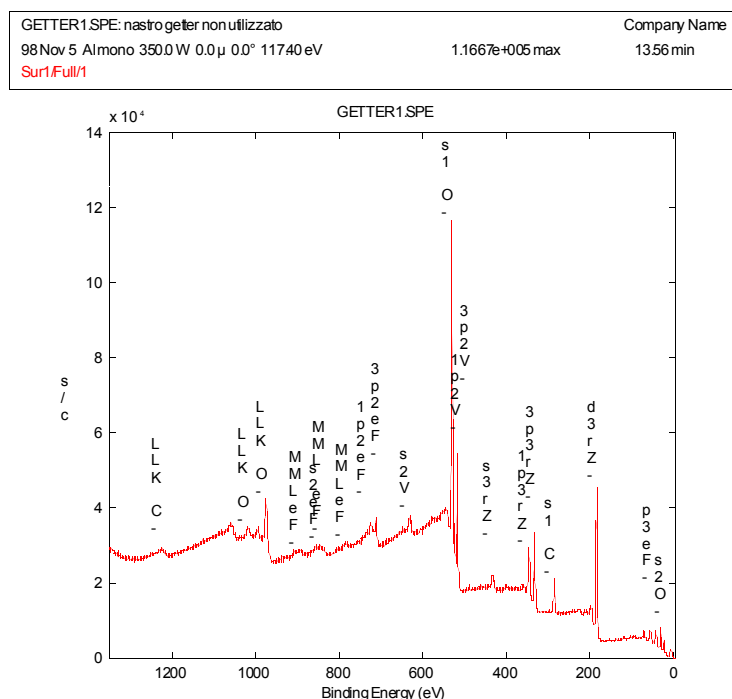
### **8.1 - DESCRIPTION OF THE XPS MEASUREMENTS**

The XPS spectroscopy allows to obtain the chemical composition of the analyzed sample, relatively to the first 10-20 atomic layers <sup>(vi)</sup>. In fact through the characterization of the energetic position of the main XPS lines in a spectrum, it is possible to determine not only the chemical elements present on the surface, but also the chemical state in which they are. Therefore this kind of analysis offers a complete chemical characterization of the surface state of the analyzed material. In our case for every sample taken in consideration we have carried out two different types of acquisitions: initially it has been acquired an XPS spectrum at low resolution and wide range (Survey Spectrum), that is on all the scale of binding energies at our disposal, with the aim to characterize the chemical elements present on the surface. Subsequently the acquisition of an XPS spectrum at high resolution has been performed over the energy ranges correspondent to the main lines of the characterized elements (Multiplex Spectrum), for being able to have structured spectra, on which the identification of the different chemical states is performed. By means of minimization procedures, in fact, it is possible to reproduce the line shape of a spectrum as superimposition of contributions coming from different chemical states relative to the same element.

From the intensity of the various XPS lines (calculated as the area under the peak over the background <sup>(6)</sup>) we can go back to the concentration in atoms of the various elements and obtain, therefore, a quantitative analysis, whose relative error is of the order of 5 - 10 %. Finally we remember that the sensitivity of an XPS analysis is about 1% in atoms and therefore an amount inferior to this quantity cannot be revealed by this technique.

### **8.2 - RESULTS**

The Survey Spectrum acquired on the sample of getter strip never used is represented in Fig. 18. The elements identified on the surface are: Zr, Fe, V, Al, C and O.



**FIG. 18.** - XPS Survey Spectrum on the never used getter strip sample.

The quantitative analysis carried out on such signals produces the atomic concentrations reported in Tab. 2 (in the second row the sensitivity factors of the technique for the different elements are reported).

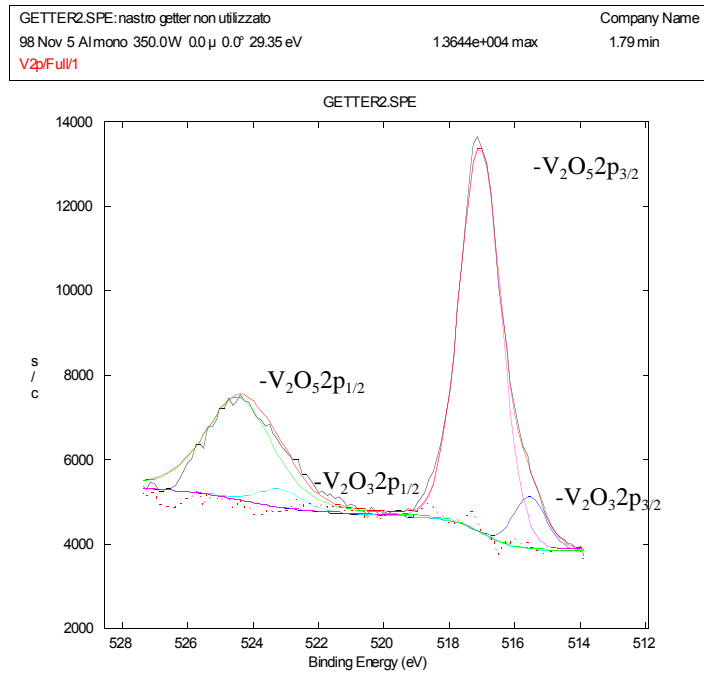
**TAB. 2.** Atomic concentrations deduced from the analysis of FIG. 18.

Atomic Concentration Table					
C1s	O1s	V2p	Fe2p	Zr3d	Al2p
0.314	0.733	2.184	2.946	2.767	0.312
18.89	52.51	10.35	5.79	11.14	1.32

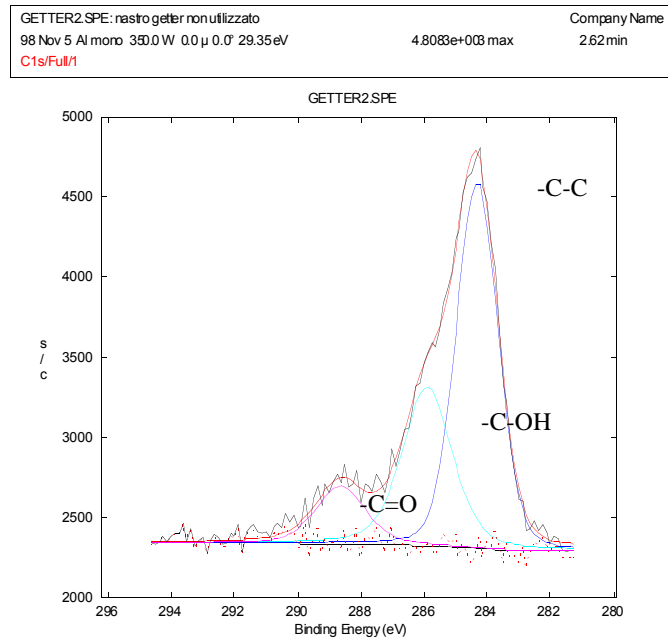
Successively a Multiplex Spectrum has been acquired on the C1s, O1s, V2p, Fe2p, Zr3d and Al2p. The deconvolution of the main lines of Fe, Zr and Al shows that such elements are in the state of maximum oxidation, correspondent respectively to the  $\text{Fe}_2\text{O}_3$ ,  $\text{ZrO}_2$  and  $\text{Al}_2\text{O}_3$  compounds. Viceversa for the V two different chemical states related to two different states of oxidation are identified  $\text{V}_2\text{O}_3$  ( $E_B=515.6 \text{ eV}^{(vii)}$ ) and  $\text{V}_2\text{O}_5$  ( $E_B=517.1 \text{ eV}^{(7)}$ ), like reported in Fig. 19.

The deconvolution operated on the C1s line (Fig. 20) is carried out by means of three signals: graphitic carbon (C - C bond,  $E_B=284.9 \text{ eV}^{(7)}$ ), C - OH bond ( $E_B=286.1 \text{ eV}^{(7)}$ ) and C=O bond ( $E_B=288.5 \text{ eV}^{(7)}$ ). Finally the deconvolution of the O1s line (Fig. 21) have been carried out by means of three bands: the band at  $E_B=530.1 \text{ eV}$  represents generically the bond

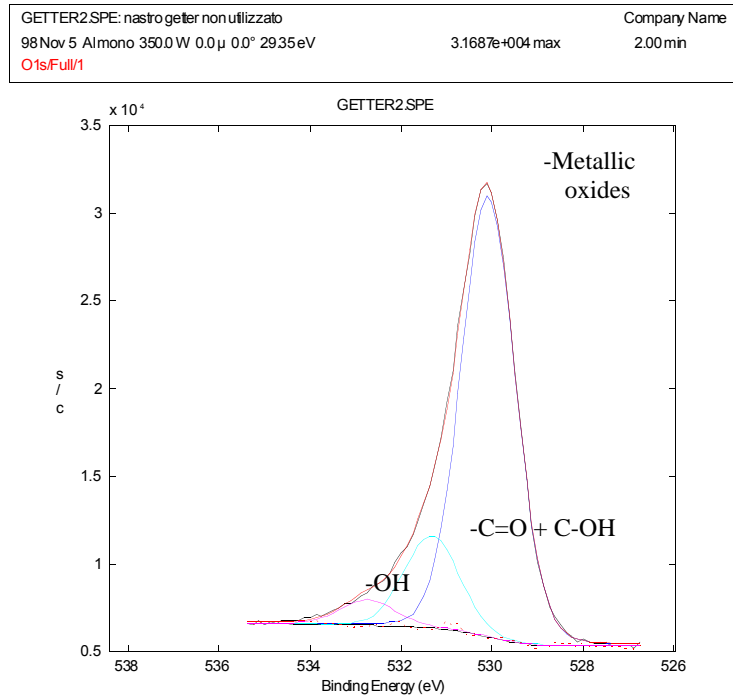
of the metallic oxide kind, the second band at  $E_B=531.3$  eV is produced by the C=O and C-OH bonds, while the third at  $E_B=532.7$  eV comes from the OH bond present in the water.



**FIG. 19.** Deconvolution of the V2p line of the new sample.

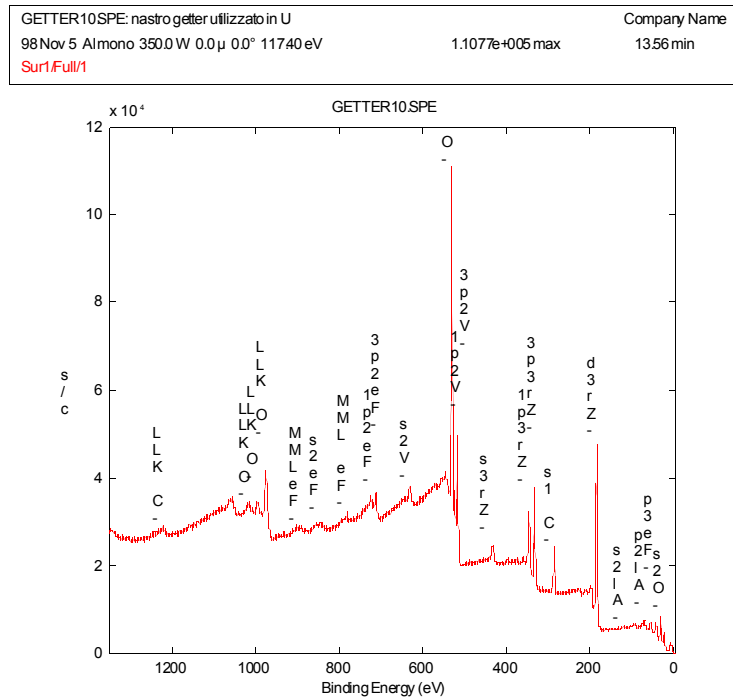


**FIG. 20.** Deconvolution of the C1s line of the new sample.



**FIG. 21.** Deconvolution of the O1s line of the new sample.

In Fig. 22 the Survey Spectrum relative to the sample of getter strip used in UHV is reported: the identified elements are the same ones of the new sample and the correspondent atomic concentrations are summarized in Tab. 3.



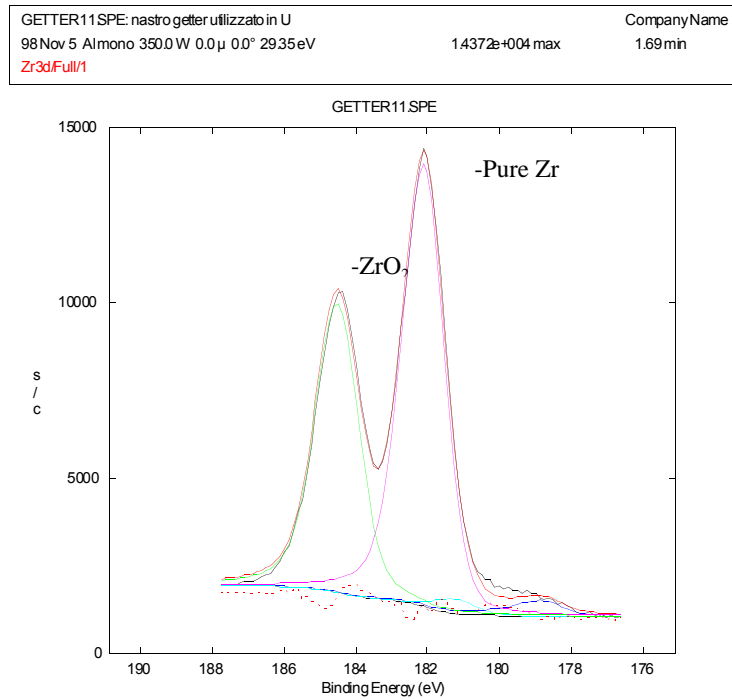
**FIG. 22.** - XPS Survey Spectrum on the getter strip sample used in UHV.



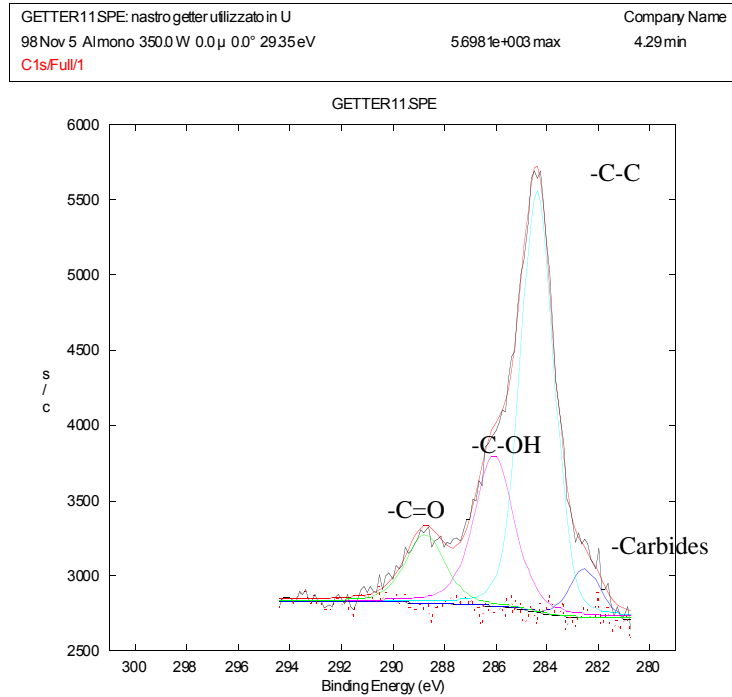
**TAB. 3.** – Atomic concentrations deduced from the analysis of FIG. 22.

Atomic Concentration Table					
C1s	O1s	V2p	Fe2p	Zr3d	Al2p
0.314	0.733	2.184	2.946	2.767	0.312
21.80	49.89	9.55	4.78	12.36	1.62

As far as the characterization of the chemical states concerns, it can be stated that at a qualitative level the situation remains unchanged for Fe, Al, V and O, while there are little different results from previous ones for Zr and C. In particular the deconvolution of the Zr (Fig. 23) puts in evidence the presence both of pure Zr ( $E_B=178.8$  eV <sup>(7)</sup>) and of the oxide  $ZrO_2$  ( $E_B=182.1$  eV <sup>(7)</sup>), while in the C1s line (Fig. 24) a new band regarding the previous ones is identified that corresponds to a bond of carbide type ( $E_B=282.5$  eV <sup>(7)</sup>).



**FIG. 23.** - Deconvolution of the Zr3d line of the getter strip sample used in UHV.



**FIG. 24.** - Deconvolution of the C1s line of the getter strip sample used in UHV.

According to the analysis carried out on the Multiplex Spectrums the following summary (Tab. 4) about the chemical composition of the surface of the getter strips is obtained.

**TAB. 4.** Chemical composition of the two getter strips analysed.

	Fe <sub>2</sub> O <sub>3</sub>	Zr	ZrO <sub>2</sub>	V <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>	C	C-OH + C=O	Carbides	H <sub>2</sub> O	Al <sub>2</sub> O <sub>3</sub>
<b>New getter</b>	7	0	27	1.5	11	25.8	20.8	0	6.9	1.3
<b>Getter used in UHV</b>	4.5	11.8	22.2	2.6	6.6	23.2	19	2.5	6	1.5

### 8.3 - DISCUSSION

From the comparison of the data obtained for the two samples, we can generically assert that in the sample used under vacuum the content in oxides and carbon diminishes with respect to the new strip, but not in a marked way. In particular relatively to the metals that forms the alloy there is a decreasing of the oxides with higher oxidation number and this allows the increase of the lower oxides and the appearance of the pure metal in the case of the Zr. Differently from what we believed, the exposure to the vacuum produced by a rotary pump has not produced an increase in the amount of the C-OH and C=O bonds, while a

meaningful difference is represented by the presence of a carbide type bond in the used strip. Finally the signal of Al present in both the samples could come from workings carried out on the alloy getter in phase of production.

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