



CapaciTorr® Pumps MK5 Series - General Information

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CapaciTorr Pumps - MK5 Series

GENERAL INFORMATION

CapaciTorr Pumps, a type of vacuum pump manufactured by Saes Getters S.p.A., sorb active gases with a nonevaporable getter (NEG) material. They are used alone or in combination with other vacuum pumps in several applications in the high and ultrahigh vacuum field (HV and UHV).

WORKING PRINCIPLES

The pumping process sorbs by chemical reaction **active gas molecules** impinging on the clean metal surface of each particle of getter material contained in the pump cartridge. A clean metal surface means a metal surface not covered by oxide or carbide layers which result when the getter material is exposed to air during manufacturing, installation or air venting steps.

The method used to obtain this clean metal surface distinguishes the nonevaporable getter pumps from others:

The getter material **does not need to be evaporated**, as does titanium in a titanium sublimation pump. It only needs to be heated, under dynamic vacuum, to diffuse the passivation layer covering the surface of each getter particle into the bulk of the getter material. The passivation layer consists mainly of oxides and carbides. This heat treatment, called **activation**, **enhances the kinetics of this diffusion process, kinetics which are negligible at room temperature**.

In this way the getter material maintains its original configuration of metal particles sintered together to form a porous body. The activation process is illustrated schematically in figure 1, in which a single getter particle is considered.

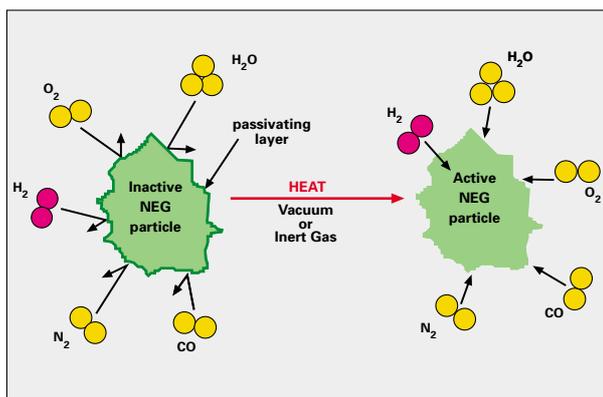


Figure 1

Once activated, the sorption process or pumping action of a CapaciTorr Pump can be split into three main steps. The first step is the dissociation of the gas molecule, which occurs on the surface. The second step, **surface sorption**, always occurs for any type of active gas. The third step is **bulk diffusion** which may or may not occur according to the specific working temperature and gas.

When bulk diffusion does not take place, the successive sorption of the gas molecules progressively saturates the surface of the getter material until sorption ceases. To restore the sorption process, it is necessary to remove this passivation layer by heating and diffusing the gas molecules sorbed onto the surface into the bulk of the getter material. This process is called **reactivation**.

During the life of the device, depending on the operating temperature of the pump, successive reactivations of the cartridge are possible to restore pumping performance until the full cartridge capacity has been reached.

GETTER MATERIALS

The getter materials used in the CapaciTorr pumps MK5 series are a Zr-V-Fe material with the trade name St172 (a mixture of Zr powder and St 707 getter alloy), and a Ti-V material with the trade name of St 185.

These getter materials, originally in powder form, are consolidated to form the getter elements of the getter pump, which can be either disks or blades, through a process of mild compression followed by a controlled sintering step. This manufacturing procedure results in very good mechanical characteristics and very high porosity, which results in higher sorption performances.

St 172-based CapaciTorr pumps are ideal for best performances in terms of speed and they are particularly well suited for high gas load applications where it is necessary to increase the pump operating temperature to boost the material sorption capacity.

The St 185 based CapaciTorr pumps are ideal when best mechanical performances are requested. These alloys pump all the active gases and are especially efficient in pumping hydrogen and its isotopes. The active gases (CO, O₂, N₂) are permanently sorbed by these getter alloys, while hydrogen and its isotopes can be removed from the alloys in accordance with Sieverts' Law.

PUMPING CHARACTERISTICS

Behavior of the getter materials

It is possible to identify four main families of gases with respect to their interaction with the getter materials. The first family consists of hydrogen and its isotopes, which are sorbed reversibly. The second family consists of other active gases such as CO, CO₂, O₂ and N₂ which are sorbed irreversibly. The third family consists of gases, which are sorbed, in a combination of the two above described ways such as water and hydrocarbons. The fourth family consists of rare gases, which are not sorbed by the getter material.

Hydrogen

Hydrogen does not form a stable combination with the getter alloy but diffuses rapidly into the bulk of the active material, where it is stored as a solid solution. A given concentration of hydrogen inside the getter alloy corresponds to an equilibrium pressure of hydrogen which is strongly dependent on temperature. This dependence is described by Sieverts' Law given by the following expression:

$$\text{Log } P = A + 2 \text{ Log } q - \frac{B}{T}$$

Where: **q** = is the concentration, in liter•Torr/gram of alloy

P = is the equilibrium pressure, in Torr

T = is the getter temperature, in K

A and **B** are experimentally determined constants that depends on the getter material type.

For St172 A =4.45 and B =5730

For St185 A =4.536 and B =5719

CapaciTorr pumps can be used as heat controlled reversible pumps, which pump hydrogen at low temperature and release it at high temperature.

The equilibrium curves for St 172 and St 185 are reported in Fig. 2 and Fig. 3.

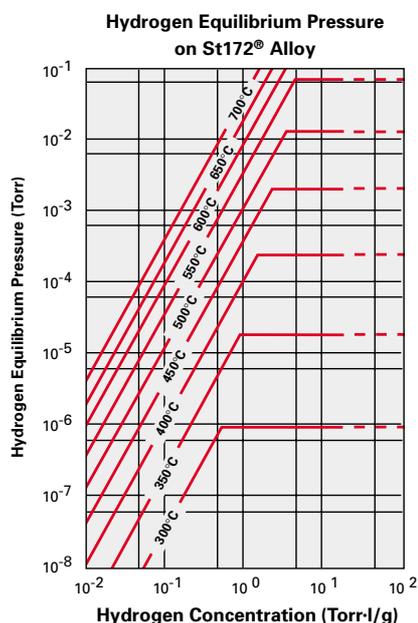


Figure 2

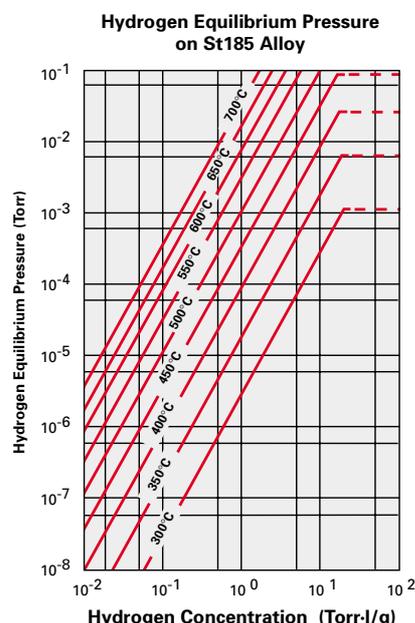


Figure 3

Other Active Gases

Other active gases such as carbon monoxide, carbon dioxide, nitrogen and oxygen are chemisorbed irreversibly by the getter material. The chemical bonds of the gas molecule are first broken on the surface of the getter and then the various constituents are sorbed as atoms, forming oxides, carbides and nitrides. The strength of the chemical bonds between the getter and these elements is so strong that even if the getter material is heated to temperatures of the order of 1000°C, no gases are released into the vacuum environment. On the contrary, high temperature treatments promote the diffusion of the gas species into the bulk of the getter material, cleaning the surface for further sorption.

Water Vapor and Hydrocarbons

Water vapor and hydrocarbon chemical bonds are cracked on the surface of the getter material. Hydrogen, oxygen and carbon are then sorbed as explained above. However, sorption efficiency of hydrocarbons at temperatures under 500°C is very low.

Rare Gases

Rare gases are not sorbed by the getter materials. CapaciTorr pumps can be therefore used to remove impurities of active gases in rare gas filled devices.

Pumping Performances

Due to the working principle of the getter material and its typical behavior toward different gas families, the pumping performance (pumping speed and gettering capacity) of CapaciTorr pumps depends on the type of gas sorbed and the operating conditions (temperature) of the getter cartridge.

The pumping performance of CapaciTorr pumps is measured according to the ASTM standard F798-82, which specifically refers to nonevaporable getter devices. The procedure is an application of the well-known dynamic flow method, also used for measurements in other standards. The test is carried out at constant pressure over the getter device. It is important to notice that the testing pressure does not affect the pumping speed of the pump when the pressure is below 10⁻⁵ Torr.

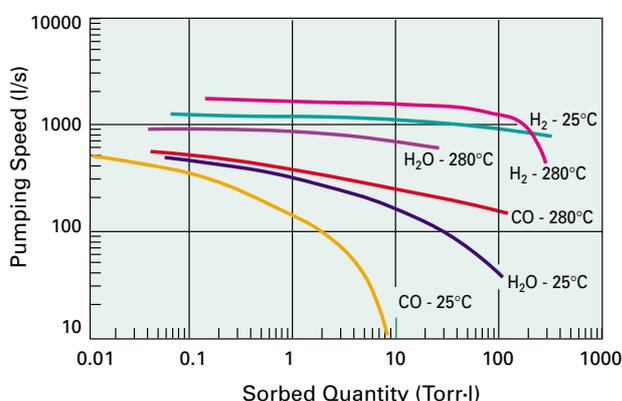


Figure 4

Typical pumping speed curves of a generic NEG pump

In fig. 4, the typical pumping speed curves of a general getter pump for H₂, CO and H₂O are shown. The pumping speed value is plotted versus the quantity of gas sorbed during the test. As mentioned above, it would be meaningless and misleading to plot it versus the pressure as in sputter ion pumps. The different sorption mechanisms of the getter material towards H₂ (high diffusion rate both at high and room temperature) and CO (low diffusion rate at room temperature, high diffusion rate at high temperature) are also evident in the curves.

The effect of the diffusion phenomena for the sorption of CO is interesting to note. When diffusion is not present, i.e. operating at room temperature, only the surface of the getter is available for the sorption of the gas. Therefore the capacity is limited and the pumping speed drops to low values when the surface becomes saturated. In this case, many reactivations will be necessary to use the total capacity of the getter cartridge. When diffusion is present, i.e. (operating at high temperature), the bulk of the getter material is available for gas sorption. The capacity for a single activation process is much larger and an almost constant value of the pumping speed is maintained for a much longer time.

There is not a big difference in sorption performance of H₂, which diffuses quickly into the bulk of the getter material, even at room temperature.

Oxygen and nitrogen in the bulk diffusion regime are sorbed with pumping speeds of approximately: 65% for O₂ and 15% for N₂ with respect to that of H₂.

Hydrocarbons are sorbed only at elevated temperatures and with very low efficiency.

Activation of a CapaciTorr pump

As previously mentioned the non-evaporable getter materials used in the CapaciTorr pumps develop their pumping characteristics after an activation process, i.e. after heating them to a suitable high temperature under dynamic vacuum for an appropriate time.

The heat treatment diffuses the thin protective layer, formed on the surface of the getter particles by air exposure during manufacture, into the bulk and makes the surface of the getter material clean and able to sorb the gas molecules of the vacuum system in which it is operated.

The efficiency of this activation process is related to the diffusion coefficient of the specific getter material, which in turn depends on an exponential function of the temperature. It is also related to the square root of time, as all the diffusion processes.

The standard activation conditions for the CapaciTorr pump that uses St 172 getter material is set at 450 °C for 45 minutes and for the pumps that use St 185 Getter material at 500 °C for 45 minutes. These conditions can be modified considering that lower temperatures should be compensated by longer times and vice versa. The activation process should be carried out after a pump down of the vacuum system to a pressure of 10⁻⁴ Torr or less. After having reached this pressure range the heater can be energized to reach the desired temperature.

During the heating phase, gases desorb from the getter cartridge. This is due to the physisorbed gases which form the external monolayers covering the surface of the getter material, while chemisorbed layers are diffused into the bulk of the getter material.

Desorbed gases include H₂O, CO, CO₂, CH₄, and eventually H₂, due to the behavior of the getter material toward this gas. In order to minimize this gas evolution, baking the system, with the getter cartridge maintained at a relatively higher temperature than the other components of the system has been found to be effective. This procedure minimizes the migration of the gases desorbed from the wall of the system toward the getter cartridge, which has a real surface area much larger than the system walls themselves.

During the activation it is advisable not to exceed pressures in the 10⁻³ Torr range to avoid phenomena of corrosion of the heater wire, RF discharges between the heater and other pump elements and contamination of the gettering material due to excessive sorption of active gases during the activation process.

For this purpose and depending on the backing pump pumping speed, it may be advisable to activate the getter cartridge by successive steps, not applying the full power to the heater at the beginning of the activation process. In this case, reaching the activation temperature may take a few hours, compared to the 30 minutes usually needed if full power is applied at the beginning.

Operation of CapaciTorr pumps

When activated, the CapaciTorr pumps can be operated at various temperatures according to the load of active gases, taking into consideration two main facts. Firstly, the higher the temperature, the higher the diffusivity of the sorbed gases into the bulk of the getter material. Secondly, the higher the temperature, the higher the H₂ equilibrium pressure. High temperature operation is preferred when high gas loads are present. In this way it is possible to maintain a high diffusion rate and consequently a constant pumping speed for all the active gases. Temperatures in the range of 250 – 350 °C are typically suggested for St 185 based cartridges. Temperatures in the range of 150 – 250 °C are typically suggested for St 172 based cartridges. High temperature is usually not compatible with UHV operation due to desorption of hydrogen from the getter material.

Room temperature operation is indicated when the gas load is low and when it is mandatory to maintain the operating pressure below 10^{-8} Torr. When pumping high purity hydrogen, operating at room temperature is also possible with high gas loads, due to the high diffusivity of H_2 at that temperature. In this latter case, a careful evaluation of the gas load and the quantity of gas sorbed are necessary to avoid possible embrittlement.

Reactivation of CapaciTorr Pumps

The reactivation of the getter cartridge of a CapaciTorr Pump is necessary when the cartridge is exposed to air or when its pumping speed falls below acceptable limits. In both cases the surface of the getter material becomes covered by a passivation layer of mainly carbides and oxides. If reactivation is preceded by an air exposure and a successive pump down cycle, it should follow the same procedure and has the same characteristic of the first activation. If the reactivation follows normal in vacuum operation without air venting, it may be shorter and carried out at a lower temperature (indicatively 25% lower). Moreover during a reactivation which follows normal in vacuum operation the only gas released is hydrogen.

Through successive reactivations it is possible to use the entire capacity of the getter material. When the pumping speed no longer recovers sufficiently after reactivation, the cartridge must be replaced.

Regeneration of SORB-AC Cartridge Pumps

Hydrogen and hydrogen isotopes sorbed by the cartridge can be released from the getter material through a regeneration treatment.

Regeneration is necessary when:

- The pumping speed for hydrogen isotopes has fallen below acceptable limits because the equilibrium pressure has been approached.
- Equilibrium is far from being reached but the hydrogen or hydrogen isotopes quantity pumped is approaching the embrittlement limit (20 Torr•l/g for St 172 and 30 Torr•l/g for St 185).

Embrittlement of the material takes place when the quantity of hydrogen sorbed in the getter material is high enough to modify its mechanical characteristics causing it to lose its mechanical integrity. The process of hydrogen sorption and successive desorption (regeneration), can be visualized on the Sieverts' plots of each specific alloy (see fig.5).

The temperature increase of the getter alloy establishes a high hydrogen equilibrium pressure, allowing hydrogen removal by means of a standard backing pump. From the plot in fig. 5 one can see that the regeneration process will be more efficacious at higher temperature. The time necessary for the regeneration of a cartridge is given by the expression:

$$t = M/F(1/q_f - 1/q_i)10^{(A-B/T)}$$

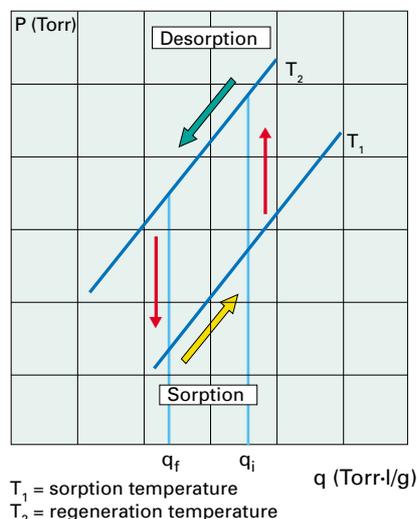


Figure 5

Where: t = is the regeneration time
 M = is the mass of the getter material in grams
 F = is the speed in l/s of the backing pump
 q_f = is the final quantity of H_2 in the getter material in Torr·l/g
 q_i = is the initial quantity of H_2 in the getter material in Torr·l/g
 A and B are the Sieverts' constants for the specific getter material
 T = is the regeneration temperature in K

Due to the exponential shape of the regeneration curve, a significant amount of time is saved when the regeneration is not programmed to be complete (100% of the sorbed hydrogen released again) but only partial (for example 90%). Thus if a given amount of hydrogen must be sorbed in each cycle (for instance 10 liter torr/g) it is much better to operate in the $q_f = 2$ $q_i = 12$ mode than in the $q_f = 0$ $q_i = 10$ mode.

SPECIAL INSTRUCTIONS

Air venting

Opening the vacuum chamber where CapaciTorr pumps are mounted should be performed only when the getter material is at room temperature or at least below 50°C for St 172 based cartridges and 150 °C for St 185 based cartridges. After each air exposure, a new reactivation of the getter cartridge is required.

A progressive reduction of pumping speed for hydrogen and active gases is observed after successive exposures to air. Fig. 6 shows the reduction of pumping speed of a CapaciTorr B 1300-2 pump after successive air ventings. Similar behavior has been observed for St 172 based CapaciTorr pumps. An activation at higher temperatures can be effective in improving the recovery of the sorption performances after successive air ventings.

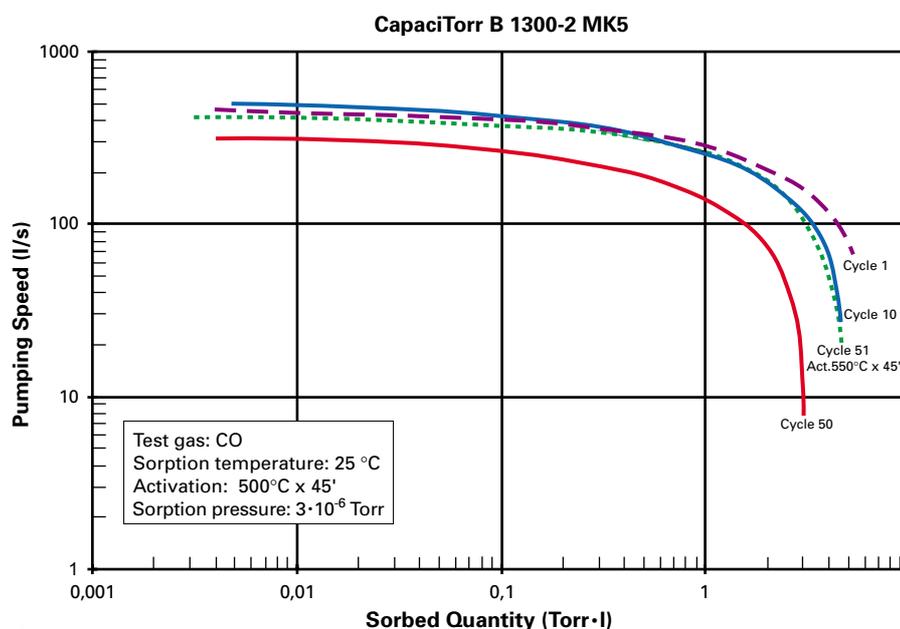


Figure 6

If dry nitrogen is used instead of air, the pumping speed reduction, after the same number of exposures, appears to be diminished. This is because, for the first cycles, the effect of the reactivation which follows a nitrogen exposure is greater than the new contamination caused by exposure to nitrogen. Further improvement is obtained when pure argon is used as a protective gas during maintenance operations.

Vacuum failure during activation or regeneration

During activation or regeneration of CapaciTorr cartridge pumps, air must not be allowed to suddenly enter the vacuum system. Such an occurrence could cause burning, i.e. a quick oxidation, of the getter material.

This happens if the temperature of the cartridge at the moment of vacuum failure is above 100°C in the case of St172 cartridges and above 300°C in the case of St 185 cartridges.

The burning is slow and progressive, not explosive.

Should a serious vacuum failure take place when the temperature of the cartridge is high although below the above indicated values, permanent damage of varying degrees will occur according to temperature, but not burning.

In this case, pumping characteristics of the getter material would be affected to a greater or lesser extent depending on temperature and time. Also, in these cases, a more efficient recovery of gettering efficiency can be obtained by using a reactivation procedure at a temperature higher than the normally adopted value.

Mechanical Shocks

Due to the mechanical characteristics of the insulating elements of the heater (ceramic spacers) particular care must be observed in handling during assembly (and removal) of the pumping system. Accidental dropping and similar mechanical shocks could result in breakage of the insulating elements with possible short circuits of the electrical path.

The SAES Getters Group manufacturing companies are ISO9001 certified, the Asian and Italian companies are ISO14001 certified also. Full information about our certifications for each company of the Group is available on our website at: www.saesgetters.com

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The logo consists of a red square containing the word "saes" in white lowercase letters above the word "getters" in white lowercase letters.

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