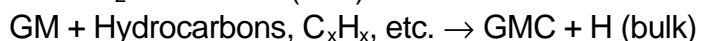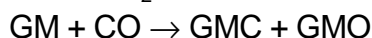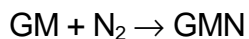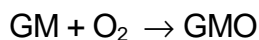# How To Use Getters and Getter Pumps

Gettering is a vacuum pumping technology that has been with us, in many forms, for almost as long as vacuum technology itself has been in existence. Historically, especially in the early days of electron tubes, vacuum practitioners were concerned with "clean-up" of the residual gases and needed to "get" them. Obviously, then, the gases would be gotten by getters. This would mean tying up gases by some means that would result in removing them from the vacuum vessel. This would put getter pumps within the overall category of capture pumps along with cryopumps and sputter-ion pumps. Capture pumps can be taken as an analog of a garbage can. Gettering, as a category, must also be broken down into physical getters and chemical getters, or some nomenclature problems will result.

Physical getters are often found in cryostats or dewars where a zeolite material such as molecular sieve is used to physically absorb and hold water vapor. If, as is often the case, the molecular sieve is placed against a surface at liquid nitrogen temperature with good thermal contact, it will absorb and hold the common air gases as well. This is the same pumping technique used by cryosorption roughing pumps where the pumped gases will be pumped and held as long as the sieve is maintained at a low enough temperature.

Chemical getters, though, are what is most often meant when the term "getter" is used. A chemical getter provides a pumping action by a chemical reaction where a chemically active gas combines with a chemically active metal to form a low vapor pressure solid compound. The chemically active metal can be either an element or an alloy that, for convenience, can be called a getter metal (GM) which provides pumping actions which can be shown in the following simplified chemical equations:

$GM + O_2 \rightarrow GMO$
$GM + N_2 \rightarrow GMN$
$GM + CO_2 \rightarrow CO + GMO \rightarrow GMC + GMO$
$GM + CO \rightarrow GMC + GMO$
$GM + H_2O \rightarrow H + GMO \rightarrow GMO + H \text{ (bulk)}$
$GM + H_2 \rightarrow GM + H \text{ (bulk)}$
$GM + \text{Hydrocarbons, } C_xH_x, \text{ etc.} \rightarrow GMC + H \text{ (bulk)}$
$GM + He, Ne, Ar, Kr, Xe \text{ (inert gases)} \rightarrow \text{No Reaction}$
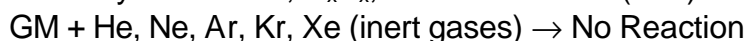
The reactions of the chemically active gases with the getter metal are fairly straightforward. They all form a low vapor pressure ceramic compound where the active gas is permanently removed from the vacuum chamber. The inert gases are not pumped at all due to the obvious reason that they are inert and will not react with the getter metal. Hydrogen (H_2), though, is the odd gas out in that it does not react to form a chemical compound but merely dissolves in the getter metal to form a solid solution. Chemical getters can be broken down into two separate categories; evaporable and non-evaporable.
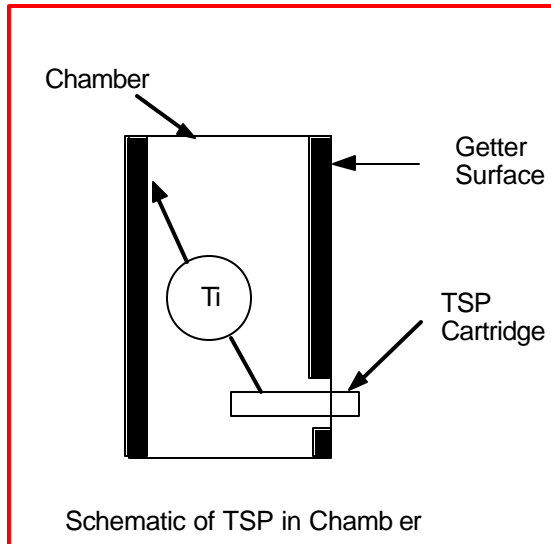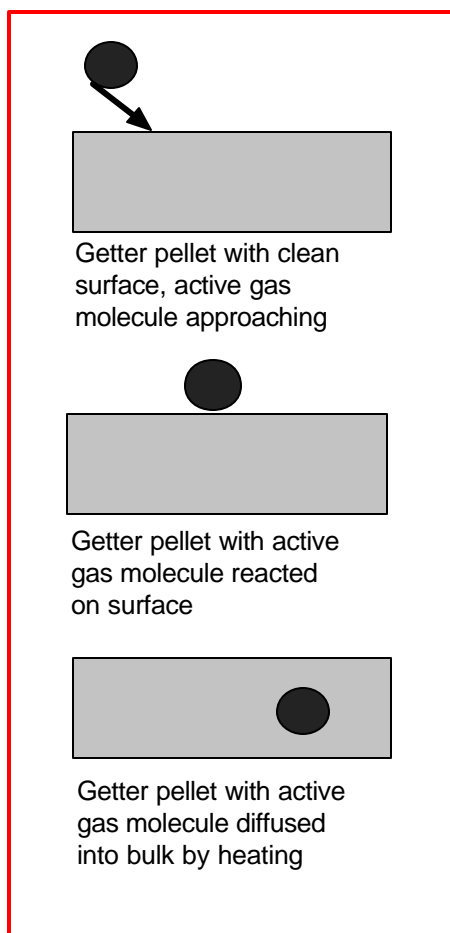
Evaporable getters are often used in electron tubes where a mirror-like metal coating can be easily observed on the inside of the glass envelope. When the tube has been pumped down and ready for tip-off to seal it, a slug of getter metal, often barium or a barium alloy, is heated to a high enough temperature to evaporate it so that it can subsequently condense on the tube's inner surfaces. There it forms a high surface area reactive coating that will remain as an *in situ* pump after the tube is pinched off. Another common example is the titanium sublimation pump (TSP.) A TSP operates by heating either a titanium/molybdenum alloy filament directly or a slug of titanium indirectly by radiation from a filament to about 1450° C where the titanium sublimes. Sublimation is defined as going directly from the solid state to the vapor state without passing through the liquid state. A common example of sublimation is hanging out a wet piece of cloth to dry in the winter where the water freezes but still evaporates. Once sublimed into the vapor state, the titanium is allowed to condense on an internal array or on a portion of the inner surface of the chamber. As it condenses into a thin film, the titanium becomes a pump for active gases and H_2. The pumping speed of the condensed film is directly proportional to the total surface area of the film. The pumping speed of a fresh film will decline as it reacts (pumps) the gas in the chamber, and the lifetime of the film is dependent upon the gas load it is required to pump. The higher the gas load, the shorter the lifetime will be. Under high gas load conditions, the titanium source will need to periodically replenish the film to maintain pumping speed, and this can be a problem. The titanium source will have become saturated with hydrogen when it cooled after reaching sublimation temperature, and re-heating releases the hydrogen. This up and down temperature cycle will result in pressure spikes as further cycling continues. The solution for this potential problem is to maintain the source at an elevated, but below sublimation temperature, at all times. Additional problems result from the layering of fresh titanium over saturated titanium. The film layers become highly stressed to the point that cracking and

Chamber

Getter Surface

Ti

TSP Cartridge

Schematic of TSP in Chamber

peeling can result. H_2, dissolving in the titanium, adds to this effect since the bulk volume of the film increases with the amount of dissolved H_2. Both of these effects can cause particles to form within the vacuum chamber with disastrous results to some processes. Conversely, TSPs are an efficient and economical source of high speed and clean pumping, and this is especially true for ultrahigh vacuum applications where the low gas load will allow very long film lifetimes.

Non-evaporable getters (NEG), as can be assumed from their name, remain in the solid state instead of being evaporated and condensed on a surface. This family of getters is usually, but not always, alloys of zirconium. Although they can be in any solid form, they are most often found as either chunks or pellets, or they are found as thin films bonded to metallic substrates.

Getter pellet with clean surface, active gas molecule approaching

Getter pellet with active gas molecule reacted on surface

Getter pellet with active gas molecule diffused into bulk by heating

Getter pellets can react with gas molecules, whichusually form a low vapor pressure ceramic compound.

Activation is a process that's required for NEGs. When the getter material is exposed to air for handling or loading into a system or device, the material's surface will "skin over" with reacted gases. This means that the NEG will be totally enclosed in an envelope of oxides, nitrides, etc. as shown by the gettering equations shown above. Additionally, the bulk of the material will be saturated with dissolved H_2. Since, under these conditions, the getter material will be essentially inert, it will not provide an active getter-pumping surface. Activation, then, is the process to prepare the getter surface for pumping. This is done *in situ* by heating under vacuum after being installed. This process is two-fold. During heating, the reacted "skin" layer will diffuse into the NEGs bulk in an attempt to achieve a constant concentration equilibrium throughout the bulk and the H_2 will be driven out of solid solution into the chamber where it can be pumped away by an appropriate high vacuum pump. The time and temperature required for activation will vary with the specific getter alloy, and the procedure recommended by the manufacturer should be followed. The vacuum level required will depend upon the actual application, but a pressure of at least 10^{-4} torr is usually required and pumping time must be long enough to ensure that the released H_2 is pumped away. Any H_2 remaining in the chamber will be re-pumped by the getter, and this will limit the amount of H_2 the getter can subsequently pump. As the newly

activated NEG cools, the newly clean and active surface will begin to react with the active gases and the bulk will begin to dissolve H_2 into the bulk.

Pumping action, following activation, follows the reactions shown in the chemical equations. Gases will be removed from the vessel by reaction on the NEG's surface until enough gas is reacted to once again "skin over" the surface. At this point, pumping action for active gases ceases and the reacted "skin" begins to act as a barrier for further dissolution of H_2. The time required for this to happen is a function of the gas load to be pumped. In ultrahigh vacuum applications, this might be years, but might be minutes or hours in a high vacuum process with its attendant high gas loads. Gas loads that would result in a unacceptably short period of surface saturation are usually dealt with by raising the temperature of the NEG to some point above room temperature. The higher temperature will result in pumping and diffusion into the bulk taking place at the same time as sort of a continuous activation cycle. The higher the temperature, the faster the diffusion. As in most engineering considerations, compromise is required. A higher temperature that would result in very fast diffusion also reduces the amount of H_2 that can be contained in solid solution. Depending upon the actual NEG alloy in use, a compromise temperature must be chosen that trades-off diffusion rate for H_2 retention. The high H_2 Pumping speed often allows NEGs to be used as a specific H_2 booster pump in tandem with cryo or turbo pumps.

Batch systems that are cycled repeatedly to atmospheric pressure require careful thought. Each time the NEG material sees air or any other active gas such as N_2,, re-activation is required. Additionally, if the gas loads are high enough to require that the material be heated in order to meet the gas loads, the material has to be cooled to room temperature before exposure. When an active gas encounters a hot NEG material, an exothermic reaction occurs that can run away in terms of temperature and present a very real safety problem. In a cycled system, the chamber can be released to argon instead of an active gas, or the getter can be contained within a separate housing that can be isolated from the chamber by an appropriate valve.

The use of O-rings in getter-pumped systems presents a very specific problem. Since the O-rings are slightly gas-permeable, some ambient air will continually permeate into the chamber. This small level of gas load presents no problem in terms of the active air gases, but air contains about 0.9% of inert argon. Although this seems like such a minuscule amount of gas that it can be ignored, the argon will enter the chamber on a continuous basis. This means that a single ISO/KF-10 Viton O-ring will allow the argon pressure to rise from about 1×10^{-6} torr to above 1×10^{-4} torr in 100 hours in a one liter chamber or 10 hours in a 100 ml chamber. Obviously, using metallic gaskets instead of Viton is often a requirement. Another option is to provide an additional pump, such as a small appendage sputter-ion pump, that can handle the small argon gas load.

Getters and getter pumps can provide very economical and compact pumping in many applications if they are used properly. Understanding the pumping process and the behavior of the getter material is essential if good results are to be expected and attained.