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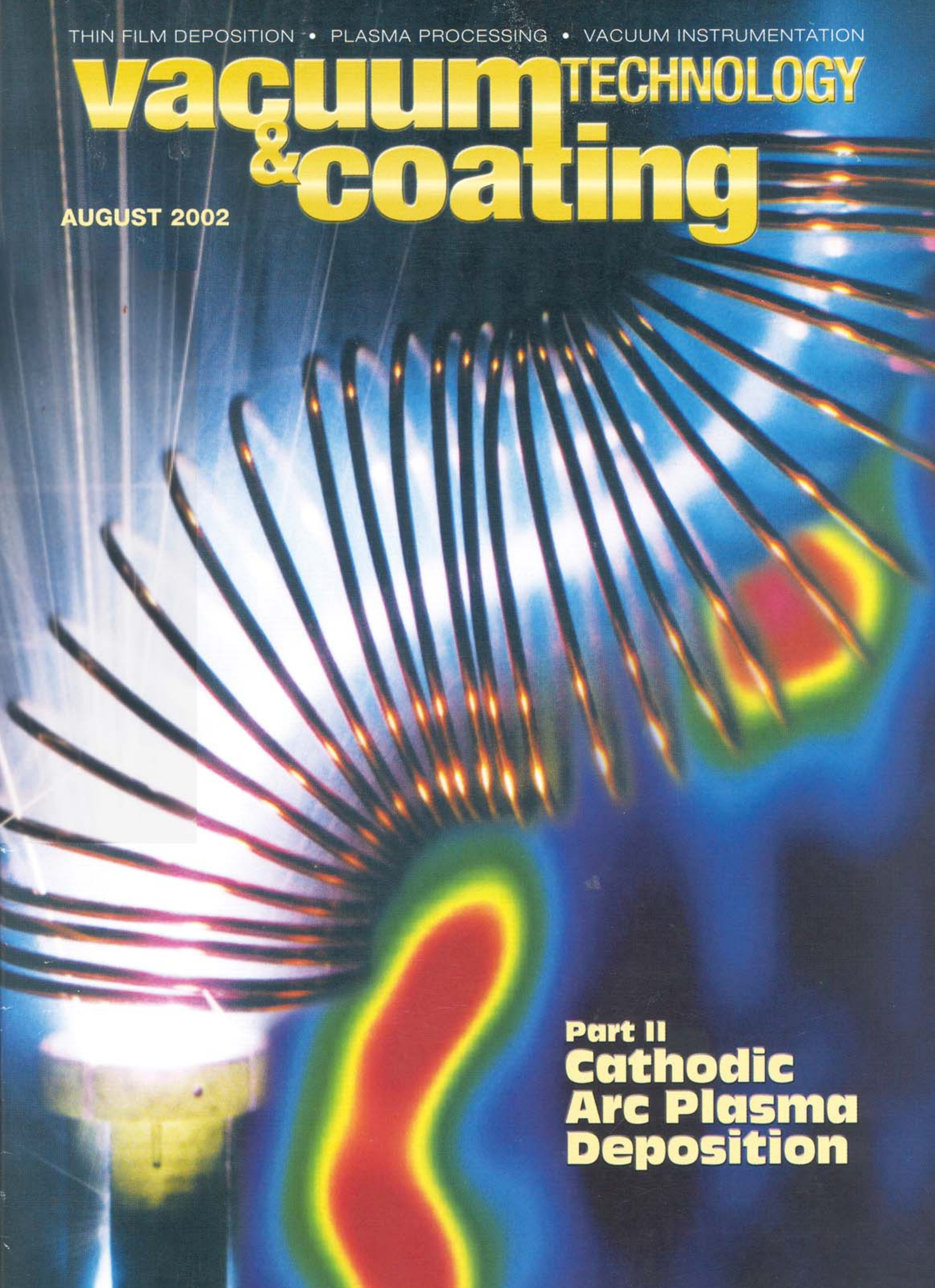

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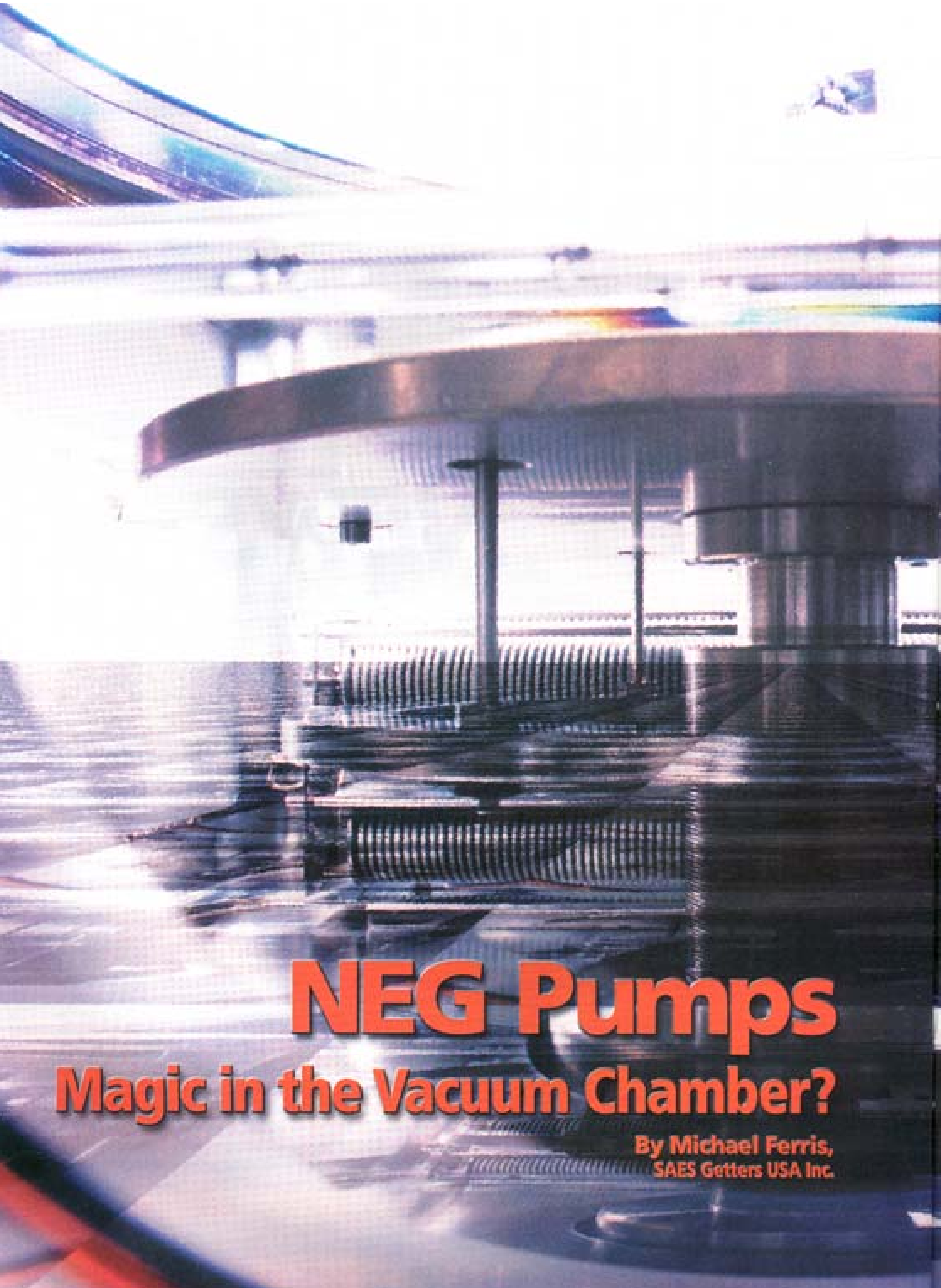

NEG Pumps
Magic in the Vacuum Chamber?

By Michael Ferris,
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They are called pumps, but they have no moving parts. They usually are not powered. Then why have them at all? The simple fact is that in many applications these "pumps" can make the difference between a process working or not working, or they can significantly improve base pressure and chamber purity during a process. They also can maintain vacuum during a power outage. They are non-evaporable getter (NEG) pumps. NEG pumps use reactive metals such as zirconium or titanium as their active elements to achieve their magic.

NEG pumps are not as universally known as turbomolecular or cryo pumps, but they fill a variety of gaps in vacuum pumping technology. A good example is that they pump when the power is off. How many other pumps do that? Another example is their affinity for hydrogen. NEG pumps pump hydrogen extremely well in the UHV pressure range. Their pumping speed is constant from about 10^{-5} Torr down to UHV.

In this article I will attempt to give an introduction to NEG pumping technology and discuss the applications and its methods of use.

Why a NEG?

No one adds a pump that does not give a benefit, so what benefits can a NEG pump offer that others cannot? NEG pumps have some very interesting capabilities that make them attractive in many circumstances. As mentioned above, they maintain constant pumping speed over a wide pressure range. They pump hydrogen better than other kinds of pumps. They usually operate without power. They are clean. They are lightweight and compact for their pumping capabilities. They can help provide better ultimate vacuum than is achievable without them. They are vibration-free. They operate unaffected by magnetic fields, and do not generate magnetic fields. When comparing a NEG pump to a titanium sublimation pump (TSP), the NEG does not need to evaporate a film, which can be an issue because the films can develop stresses and flake off the deposition surface. Another advantage of a NEG pump over a TSP is that the NEG can be put into a much smaller space than a TSP. The NEG also does not need a surface that can be sacrificed to titanium deposition.

Just Going Through a Phase Change

How do NEG pumps work? Simply stated, NEG pumps work by chemical reaction and phase change. Things are actually a little more complicated, but not much. There are two types of sorption by NEG pumps: reversible and non-reversible. Hydrogen is the one gas that is sorbed reversibly (this also includes the isotopes of hydrogen). Carbon, nitrogen, and oxygen form stable chemical compounds with the NEG alloy and are sorbed irreversibly.

There are three parts of sorption of gases by a NEG pump. These parts are dissociation, surface sorption, and bulk diffusion. All sorption by a getter is of atoms, not molecules. So the NEG pump removes water vapor from a vacuum chamber by reacting with oxygen and hydrogen, not with the water molecule. At room temperature, all species go through the dissocia-

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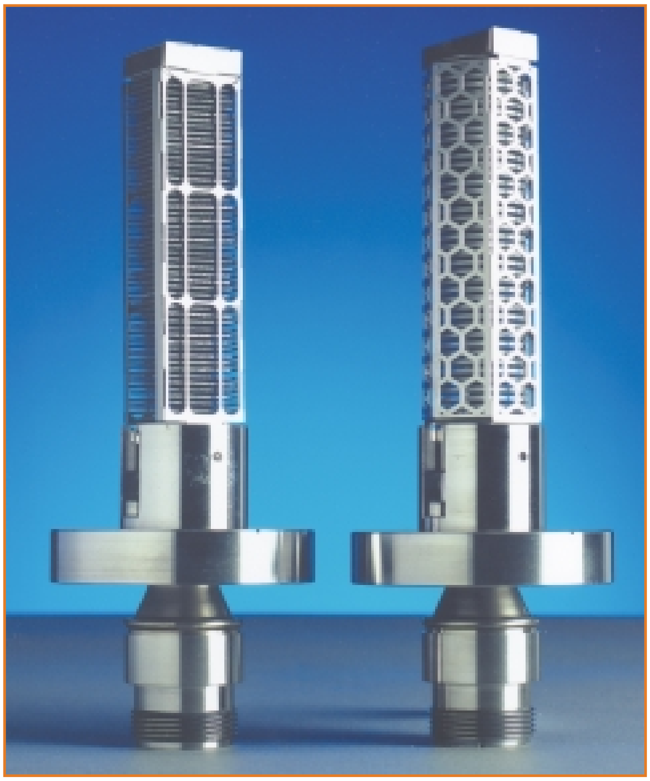
tion and surface sorption. Hydrogen diffuses into the bulk of the getter at room temperature, but carbon, nitrogen, and oxygen do not.

The closest example of "pumping" by phase change is a cryo pump. The cryo pump has no moving parts (at least none that come into contact with the gas in the chamber). It "pumps" by freezing gases to its cold arrays. It turns gases into solids by making them cold enough that they freeze out of the gas phase – so to speak. With the getter pump, gases become solids, but they go through the chemical processes described below rather than freezing. One important practical difference between a NEG pump and a cryo pump is that the NEG does not release the gases it has sorbed if it loses power.

Let us deal with the irreversibly sorbed species first. As mentioned above, these elements form stable chemical compounds with the getter metal. At room temperature, which is the usual operation temperature for NEG pumps, this happens on the surface of the getter material. Again, the gas molecules arrive on the surface of the getter, are cracked into their component atoms, and react with the getter metal. They form extremely low-vapor-pressure solid compounds. When the surface is completely coated with irreversibly sorbed gases, it is "passivated." This is the condition of a new NEG pump cartridge. The presence of the passivation layer makes it possible to handle the NEG material in air without the NEG material completely reacting with gas species in the atmosphere. As an example of irreversible sorption, let us look at oxygen. Oxygen, either from a compound such as carbon monoxide or as an oxygen molecule, lands on an activated getter surface. The molecule splits into component elements, and the oxygen atoms react with the getter to form either titanium-oxide or zirconium-oxide, both of which are extremely stable, low-vapor-pressure solids. So the oxygen is still in the chamber, but as a solid, not as a gas. At room temperature, the irreversibly sorbed gases will remain on the surface of the NEG alloy.

Hydrogen sorption is quite different. Hydrogen molecules that land on the surface of the getter material split into two hydrogen atoms and diffuse into the bulk of the getter, forming a solid solution. This happens when the concentration of hydrogen in the getter material is at low to moderate levels. At these levels, the hydrogen does not react with the getter metal to form a new compound as the irreversibly sorbed gases do. It is distributed nearly uniformly throughout the bulk of the getter alloy. At higher levels of concentration, the hydrogen begins to form a hydride with the getter material. Because this changes the crystal lattice, which ultimately results in the getter material embrittling and crumbling, it is recommended that the getter pump be regenerated before these higher concentrations of hydrogen are reached. This condition is not difficult to avoid, but more will be discussed about that later. (A safe maximum quantity of hydrogen in a NEG alloy is in the range of 20 Torr-liters of hydrogen per gram of getter material.) But what about hydrogen sorption being reversible? As was mentioned above, hydrogen forms a solid solution in the getter material. The solubility of hydrogen is inversely proportional to the temperature of the getter material. So, to remove hydrogen from the NEG pump, heat it up. More will be said on that later, as well.

Let us sum up what a NEG pump is and how it is used. A NEG pump usually consists of a flange, heater, and cartridge. The heater and flange are usually combined in one unit, and the cartridge is installed onto that unit. When the cartridge is new or when it has been exposed to atmosphere, the getter material has a passivation layer on its surface. The passivation layer allows the getter material to be handled safely in air. This layer is primarily composed of a thin, very dense layer of carbon and oxygen. The passivation layer is a barrier to further reaction of the getter material with active gases. Familiar examples of materials that are protected from ongoing reaction with gases in the environment are stainless steel and titanium. Stainless steel and titanium are actually very reactive metals, but they form a very dense passivating oxide layer on their surfaces, which prevents



Photograph of the two SAES Getters CapaciTorr pump models that use a 2 1/2" Conflat flange. The B-200 cartridge uses St 185 (Ti-V) getter alloy and the D-400 cartridge uses St 172 (Zr-V-Fe) getter alloy. CapaciTorr pumps use porous sintered getter elements in the cartridges further reaction. Once the getter pump is installed onto a vacuum system and the system is pumped to a suitable pressure, the pump is heated to achieve "activation." Here, the term "suitable pressure" means at least 10^{-4} Torr, but a lower pressure, such as 10^{-6} Torr or better, is preferable.

Activation/Reactivation versus Regeneration

Activation (and also reactivation) is the removal of the passivation layer from the surface by providing sufficient energy in the form of heat to allow the sorbed species on the surface to diffuse into the bulk of the getter material. This results in a clean surface of the getter cartridge, where sorption can take place. Activation is the first cleaning of the surface on a new cartridge; reactivation is any subsequent activation process. There is another issue to consider. The first activation/reactivation process after a pump has been brought to vacuum varies from subsequent processes without breaking vacuum. The difference is that in the first activation/reactivation process there are physisorbed gases on the NEG cartridge, which will be released when the cartridge starts to heat up. In subsequent reactivations without breaking vacuum, these species will not be present.

The difference between activation/reactivation and regeneration is that in activation/reactivation the gases that are diffused into the bulk do not go away–they build up in the getter alloy. Regeneration is the removal of hydrogen and a renewal of the getter's sorption capacity. Remember that I said that to remove hydrogen from the getter material you need to heat the getter? That is the basis for regeneration. It goes back to the inverse relationship between temperature and hydrogen solubility in the getter alloy. If, when the NEG pump is being heated for reactivation, there is another pump on the system removing the hydrogen that is released, the capacity of the getter for hydrogen is renewed. Thus, hydrogen capacity can be renewed again and again, if needed. Without a pump to remove the hydrogen from the chamber, it will be resorbed by the NEG pump when it cools. Activation/reactivation and regeneration both occur when the NEG cartridge is heated. If there is a backing pump operating on a chamber when the pump is heated, both processes can occur simultaneously. In many cases, regeneration is not necessary.

NEG pumps have very large capacities for hydrogen, so unless it is present in quantities sufficient to cause a problem such as the NEG material embrittling, this issue can be safely ignored. The NEG pump can be heated to provide hydrogen pressure, if required. The maximum hydrogen capacity for a NEG pump is defined by the embrittlement limit, approximately 20 to 30 Torr-liters of hydrogen per gram of getter material, depending on the alloy. So for a NEG pump with a cartridge containing 600 grams of getter alloy, 12,000 to 18,000 Torr-liters of hydrogen can be safely pumped, depending on the embrittlement limit for the alloy involved. Because hydrogen diffuses into the bulk of the getter at room temperature, the capacity is much higher than the room-temperature capacity for gases like CO. The CO capacity at room temperature for that same pump is 6 Torr-liters. After subsequent activations, the total CO capacity is 5,400 Torr-liters.

There is a strong relationship between the concentration of hydrogen in the getter, the temperature, and the hydrogen pressure in the chamber. The Sieverts' Law equation is very useful in finding predicting behavior of the system if two variables are known:

$$Log P = A + 2 \log q - \frac{B}{T} \qquad (1)$$

where:
q = concentration of hydrogen in getter in Torr-liters/g
P = equilibrium pressure, in Torr
T = the getter temperature, in K.

For those who need to regenerate NEG pumps, the regeneration formula follows:

$$t = \frac{M}{F}\left(\frac{1}{q_f} - \frac{1}{q_i}\right) 10^{-\left(A - \frac{B}{T}\right)} \qquad (2)$$

where:
t = regeneration time, in seconds
M = mass of the getter material, in grams
F = pumping speed of the backing pump for hydrogen, in liters/s
q_f = final hydrogen concentration, in Torr-liter/g
q_i = initial hydrogen concentration, in Torr-liter/g
A = 4.536 for St 185
B = 5719 for St 185
T = the regeneration temperature, in K.

Let us take a regeneration example of a pump with 600 grams of St 185 alloy that has a level of five Torr-liters of hydrogen per gram of NEG alloy. If we heat it to 500°C for 45 minutes (standard activation conditions for this alloy), with a backing pump with a pumping speed for hydrogen of 200 liters per second, the hydrogen level is reduced to a level of less than one Torr-liter per gram.

I should mention that St 185 is one of the NEG alloys used in SAES Getters pumps. It is an alloy of titanium and vanadium. Other SAES' pumps use St 101, St 172, and St 707. These are all zirconium-based alloys. "A" and "B" in the above equations are thermodynamic constants that are unique to each getter alloy. This leads into a discussion of the different NEG alloys used in SAES' pumps. **Table 1** shows the various alloys and some of their characteristics.

Table 1. Getter alloys and their characteristics

Getter Name	Composition	Form	Activation Conditions	Pump Type
St 707	Zr-V-Fe	Mechanically Bonded Strip	450°C for 45 min.	GP series
St 172	Zr-V-Fe	Sintered Disk	450°C for 45 min.	CapaciTorr series
St 185	Ti-V	Sintered Blade	500°C for 45 min.	CapaciTorr series

From left to right: GP 200 MK5, CapaciTorr B-200, CapaciTorr D 400, InsiTorr, WaferGetter, CapaciTorr B-1300.

A Little About Capacity

Capacity is a word that is part of NEG parlance, but not usually an issue with vacuum pumps (except for cryo pumps). Again, cryo pumps are the closest example of a pump where capacity is an important concept. Because both NEG pumps and cryo pumps are capture pumps, capacity is an important issue. For NEG pumps there are three capacities of concern. There is the safe capacity of hydrogen, which has been discussed. For the gases that are sorbed non-reversibly, there is room-temperature capacity and total capacity. Room-temperature capacity and total capacity relate to carbon, nitrogen, and oxygen. At first glance, it would be natural to think that the capacity is when the surface of the pump is completely filled up. The problem with that definition is that the pumping speed can be below what the system needs to maintain the necessary vacuum. So a NEG pump at room temperature has a room-temperature capacity that is based on some minimum acceptable pumping speed. In the example above, the CO capacity for room temperature was based on a minimum pumping speed of 50 liters per second. The initial CO pumping speed after activation for that pump is 1,000 liters per second. So in this case, the capacity is based on a drop to 5% of the initial pumping speed, but this varies with each application.

The question of how long a cartridge will last is often raised. This is straightforward in terms of quantity of gas sorbed, but not in terms of time. There are two aspects to consider here. The first is room-temperature capacity. The second is overall capacity. If the gas load is known, both can be calculated. If not, watching the system for a pressure increase is necessary.

The gas load can be calculated to a reasonable approximation using the pumping speed of the pumping already on the chamber and the base pressure of the chamber. RGA data can make this even more precise. With this information, reasonable estimates of reactivation frequency and expected cartridge life can be made. A cartridge should last at least 40 to 50 cycles, where cycle means one pump down, activation (sorption at room temperature), and vent to atmosphere. Operating the cartridge at elevated temperature will decrease the number of cycles before end of life.

Venting the chamber when the cartridge is at elevated temperature can either shorten or end cartridge life, depending on the temperature of the cartridge. If the cartridge is at a moderately high temperature when vented, it will sorb a higher than normal quantity of gas in forming the passivation layer. If the cartridge is at activation temperature at time of venting, it will react until its capacity is completely consumed. This situation is easy to avoid, and is an extremely rare event.

No Pump Is an Island

NEG pumps are nearly always used in combination with another type of pump. There is no single, ideal pump for UHV. Other pumps are generally deficient in pumping hydrogen. The NEG pump excels in pumping hydrogen. It is excellent at pumping water and pumps CO and CO_2 well. It is very poor at pumping hydrocarbons at room temperature, and it will not pump noble gases at all. The combination of a NEG pump with another pump allows the two pumps to contribute their strengths and for each to compensate for the deficiencies of the other. A

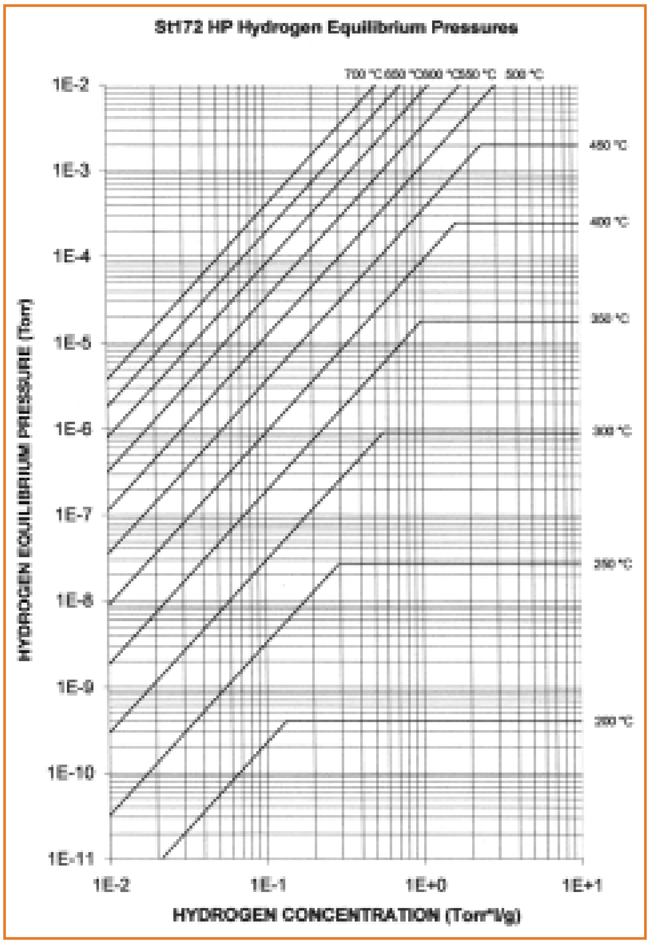


Figure 1. Hydrogen pressure-concentration-temperature relationships for St 172.

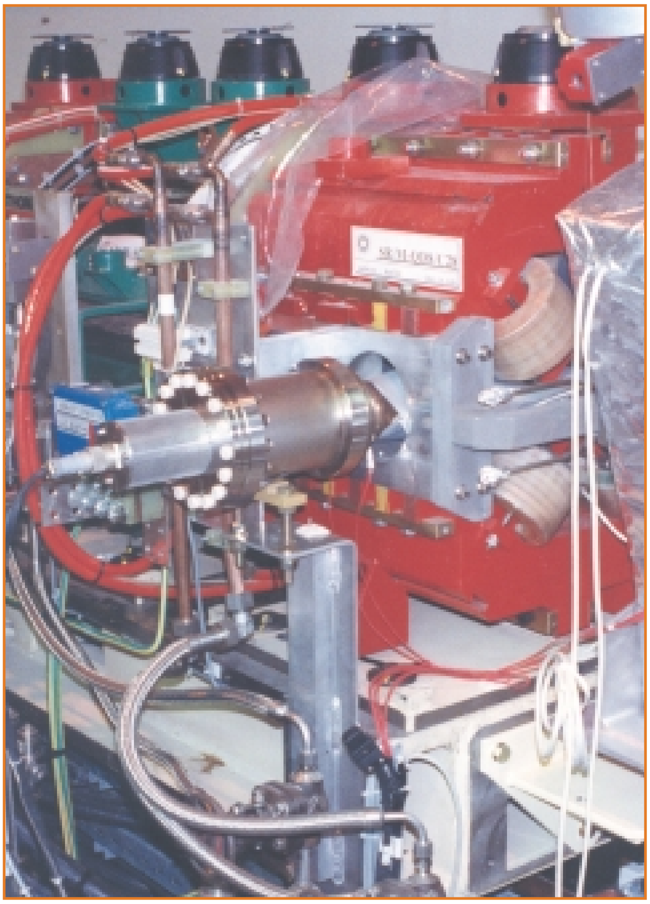
very typical combination is a NEG pump used with an ion pump. The NEG pump usually has a larger appetite for hydrogen than an ion pump, and an ion pump can compensate for the absence of noble gas and methane pumping on the part of the NEG. The situation is similar for NEG pumps in combination with cryo pumps and turbo pumps.

Use of the Pump

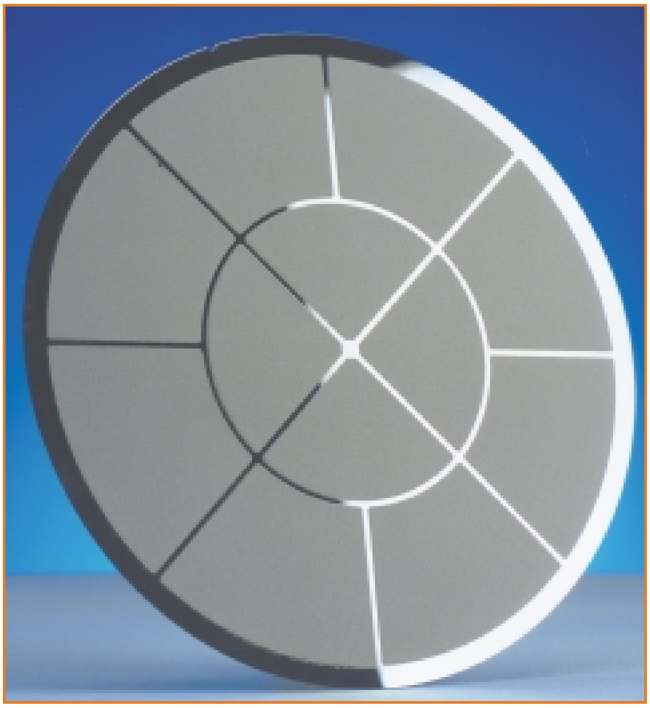
As mentioned above, NEG pumps are shipped with a passivation layer on the getter alloy, and need to be activated to be ready for use. For SAES' pumps with St 707 or St 172 cartridges, 45 minutes activation at 450°C is sufficient for activation. For SAES' pumps with St 185 cartridges, 45 minutes at 500°C is required. NEG pumps used on UHV systems typically need very infrequent reactivation due to the small gas loads. If the gas load is small enough that the pump will need infrequent reactivation, it is used at room temperature. NEGs are most commonly used in UHV applications. This is an area where the characteristics of getters make them an excellent choice to improve the base pressure.

In high-gas-load situations, operating the NEG pump at higher temperature can help keep the diffusion process functioning, reducing the need for pump reactivation. The issue that can arise when operating the pump at elevated temperatures is that of hydrogen pressure. Remember that hydrogen is less soluble in the NEG at high temperature, so there is a trade-off between diffusivity of the non-reversible species into the bulk and equilibrium pressure of hydrogen. As the temperature of the NEG pump increases, so will the hydrogen pressure in the system. The graph in **Figure 1** shows the hydrogen pressure-concentration-temperature relationships for St 172.

In the case of a pump using St 172, 200°C to 300°C is the recommended temperature range of operation to allow for diffusion of sorbed species into the bulk. Calculating the Sieverts' Law equation for 200°C and a concentration of 5 Torr-liters hydrogen per gram of getter material results in a pressure of 5.4 x 10^{-7} Torr. This is a respectable vacuum, but certainly not UHV. If the hydrogen concentration is held to less than 1 Torr-liter per gram, the pressure drops to 2.2 x 10^{-8} Torr at the same temperature. If the NEG pump cartridge is a CapaciTorr D 400 with 45 grams of St 172, this means that to keep the pressure down to the low 10^{-8} Torr range at 200°C, about 45 Torr liters of hydrogen can be sorbed. At 2 x 10^{-8} Torr, sorbing at 200 liters per second, in a 1-liter chamber it would take several months before a regeneration of hydrogen is needed. These examples give equilibrium values of hydrogen pressure for a static system (no other pumping on the chamber). If another kind of vacuum pump is operating on the chamber, the hydrogen pressure could decrease from the values given, depending on the ability of that pump to remove hydrogen from the chamber.



A GP series cartridge pump being used in a particle-accelerator application at the ESRF facility in Grenoble, France.



WaferGetter is NEG material coated and sintered onto a silicon wafer. It is used to decrease pump down time after PM.

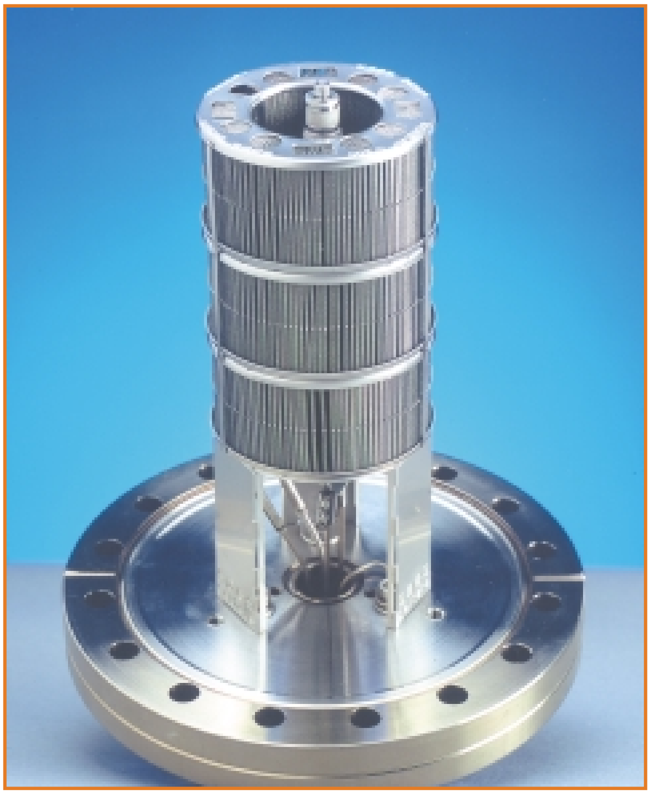
Applications of NEG Pumps

The traditional use for NEG pumps is in UHV systems, including particle accelerators. In a UHV application, the NEG pump can bring the pressure down significantly because the primary species without a NEG is usually hydrogen. Many UHV systems are rarely vented, making them ideal applications for NEG pumping. In particle accelerators, the NEG elements can be specially configured for the needs of the application. Another UHV application for NEG pumps is that of surface science, where a clean environment is critical to an accurate result. NEG pumps are used in processing sealed devices, such as X-ray tubes. The excellent hydrogen pumping by NEG pumps well suits them for keeping this gas out of the tubes.

The fact that NEG pumps do not pump inert gases makes them ideal as a pump for an in-situ pump (InsiTorr) for many physical vapor deposition (PVD) applications. SAES Getters has developed InsiTorr pumps to fit in the deposition chambers of cluster tools. These pumps can help the chamber to reach base pressure much sooner than without the pump. In addition, they do not pump the argon used during the process. They remove impurities from the argon, but they do not remove the argon itself. The reduction of impurities in the argon can result in better films, as well. This application is a departure from traditional applications for NEG pumps, but it is a good fit. The NEG pump can shorten the pump down after preventative maintenance by several hours. The NEG pump can remain in a metal sputter-deposition chamber and help maintain a pure environment, even during the process. There also is a variation on this concept called WaferGetter, which is removed from a chamber before deposition or that chamber's process. Because the WaferGetter is removed from a chamber before the process is performed, it can be used in chambers where the process results in a harsh environment, including CVD, reactive sputtering, or etch. WaferGetter is used to shorten the time to reach process qualification.

Once activated, NEG pumps operate without power. In places like California, where continuous power is not reliable, this could be a valuable feature. In this era of rolling blackouts, NEG pumps can ensure vacuum during a time when power is down. We have sold a pump to a California customer who installed it onto a system for just that purpose. How many more are used for protection against blackouts, we do not know. When all else shuts down, the NEG pump will keep on sorbing gases. In cases where a breach of vacuum would be catastrophic, a NEG pump could be attached to a chamber in an appendage, separated by a valve that would open when power goes off. The NEG pump could be powered to a low temperature to keep its surface clean, and in the case of a blackout would have a high capacity for the time until power was restored.

NEG pumps are lightweight and use very little or no power during operation, which makes them appropriate for portable and remote applications. There are a number of companies making remote repeaters for wireless phone transmissions. These repeaters require a vacuum, which is in many cases maintained by a small NEG pump. The Inficon HAPSITE, a portable gas chromatograph/mass spectrometer (GC/MS) instrument, uses a small NEG pump as its primary pumping when used in the field.



The MK5 series of SAES Getters GP cartridge pumps. GP pumps incorporate a cartridge that uses St 707 getter material mechanically bonded onto a strip, which is then slotted and pleated.

The InsiTorr pump inside a deposition chamber of a PVD cluster tool. The InsiTorr helps shorten pump down after PM and keeps the atmosphere clean during the deposition process.

Silver-Bullet Syndrome

When considering the addition of a NEG pump to a system, some people tend to think of the NEG as a "silver bullet," a magic fix for the problems of that system. NEG pumps can really make a positive difference in a system, but sound vacuum principles cannot be ignored. An example is choosing a NEG pump based on a convenient flange size without considering its pumping speed. Another problem is putting the NEG pump where there is a flange, rather than putting it in the area where better vacuum is needed. In some systems that are made of long tubes rather than a single chamber, the NEG pump will not do much if situated far from the critical area. This sounds obvious, but there are many times that such obvious aspects are ignored.

Conclusions

NEG pumps provide clean, non-contaminating pumping for a variety of applications. They are staples in UHV applications due to their superior pumping characteristics for hydrogen. They pump other active gases, including water vapor, very well. They are lightweight and compact for their pumping capabilities. They can operate on little or no power, making them appropriate for many remote applications, and also where power is intermittent. They have been shown to be effective in situ pumps, helping to shorten pump down in many applications. They can be used in some sputtering applications for maintaining the purity of the process gas. While their traditional home has been UHV and particle accelerator applications, they have found a home in other applications where weight, power consumption, process time, or purity of the chamber atmosphere is critical.

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