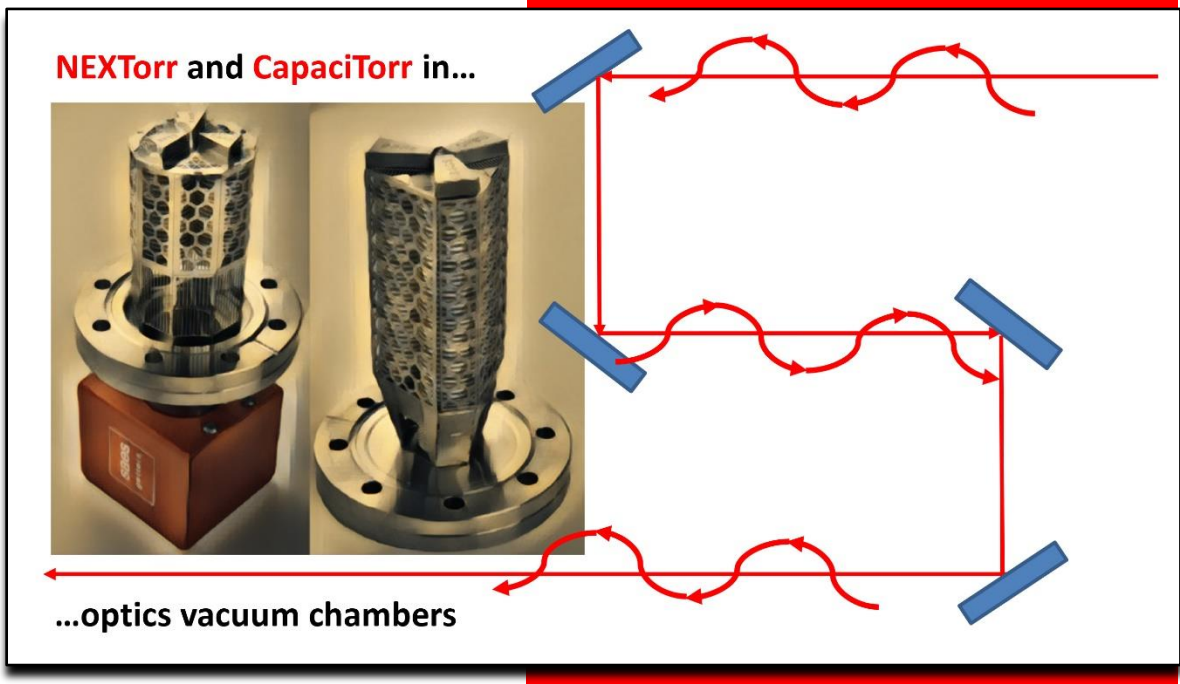
White Paper:

ZAO® Based Non Evaporable Getter Pumps in Optics Vacuum Chambers



SAES Group S.p.A
Viale Italia 77
20020 - Linate (MI) - Italy
www.saesgroup.com

S. Wulfsberg, E. Maccallini,

A. Cadoppi, D. Nicolosi, M. Urbano,

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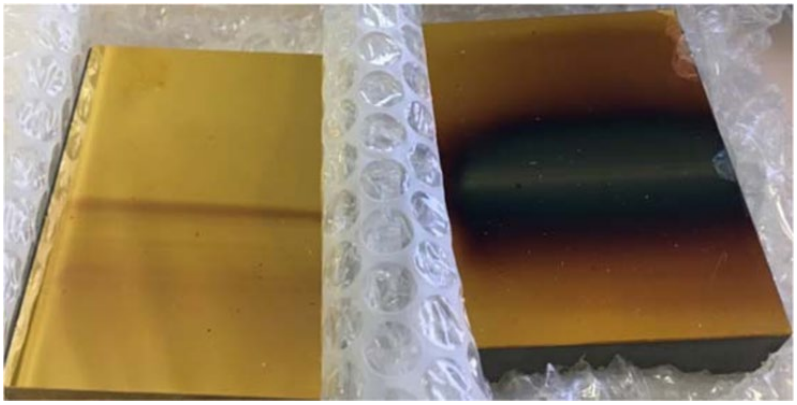
Carbon Contamination in Vacuum Chambers

Vacuum engineers and scientists have long known that even if a sample material is initially clean and handled with ultra-high vacuum (UHV) standards, a carbon contamination layer will deposit and grow on the material's surface after placing it in a high vacuum (HV) or UHV chamber. This condition is also true for the optics vacuum chambers found in particle accelerators and synchrotron beamlines. For the optics vacuum chambers, this situation is worsened by X-ray irradiation, which can result in a one to two orders of magnitude pressure increase and high yield of carbon contaminants [1]. The effects of carbon contamination on the X-ray optics can significantly reduce the X-ray transmission downstream to the experimental stations, and as next-generation synchrotrons usher in X-ray brightness increases of two to three orders of magnitude, it is critical to minimize these losses from carbon contamination.

Multiple studies have shown that carbon contamination develops on X-ray optics and reduces the transmission of photons near the carbon K-edge, around 285 eV, as well as at higher energies around 1000 eV [1-4]. As early as the 1980s, this carbon contamination layer was shown to cause intensity modulations in X-ray absorption spectra that closely resembled those above the carbon K edge in bulk crystalline graphite [3]. These results suggested the formation of graphitic carbon contamination even under UHV conditions. Carbon contamination is not only experimentally detected; it is also visually evident after a few months to a year of beamline operation. It will usually appear as a black line where the X-rays strike the optics. See figure 1 (courtesy of Brookhaven National Laboratory).

Figure 1. Carbon contamination on mirrors at Beamline at NSLS-II at Brookhaven National Laboratory. Courtesy of Rob Todd, NSLS-II, BNL, 2019



As the research into this problem progresses, and the body of literature grows larger, scientists and engineers have identified several sources of carbon contamination. The following are two common and significant sources:

- Residual gases such as CO and CO_2
- Hydrocarbons and other carbon species absorbed on the components housed in the optics chamber, including the optics themselves

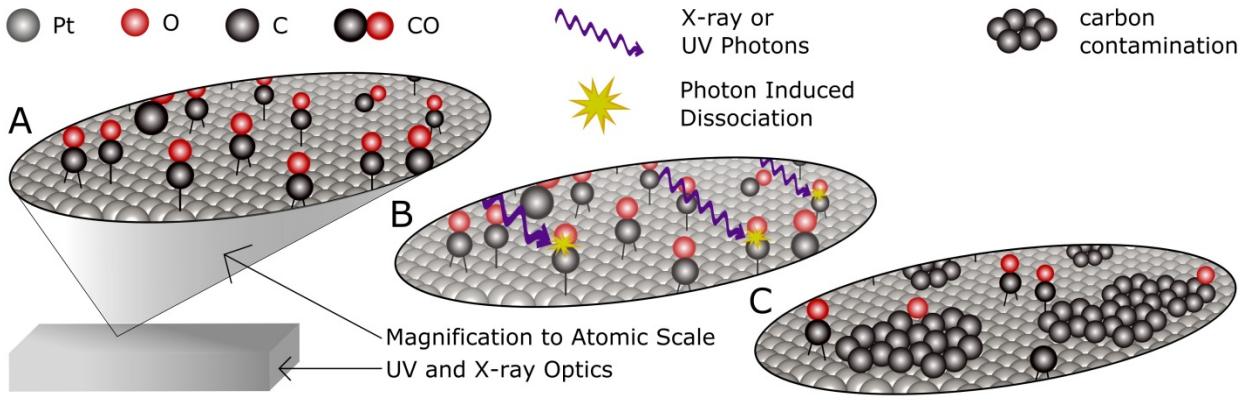
Residual Gases

In the UHV optics chambers found in synchrotron beamlines, pressures ranging from 1 to 5 x 10^{-10} Torr [1,3,5,6] are typically achieved and maintained. For example, the optics chamber's base pressure on the TEMPO beamline at SOLEIL in France has a pressure of 3.5 x 10^{-10} Torr and consists mostly of H_2, CO, H_2O, and CO_2. As X-rays irradiate the chamber during beam operation, the pressure increases about two orders of magnitude to 4.0 x 10^{-8} Torr, with CO and CO_2 accounting for the majority of background gas species [1]. At LURE laboratory in France, researchers found that a photon dose of 3 x10^{21} photons/m resulted in the desorption of approximately 1 Torr l/m of H_2, CH_4, CO and CO_2 from aluminum, 10^{-1} Torr l/m from copper, and 5 x 10^{-2} Torr l/m from stainless steel [7]. These studies detail the extent of background gases in X-ray optics chambers and reveal the significance of this issue.

Further examining the issue of residual gases, such as CO, in X-ray optics chambers, Risterucci et al. used high-resolution XPS to study the mechanism of carbon contamination on the Pt (111) surface by CO during x-ray irradiation under conditions similar to those found in beamline optics chambers during operation [4]. The Pt surface was chosen due to the extensive past research on the oxidation of CO on Pt, and because it is common to coat mirror surfaces with metals such as Pt in order to increase reflectivity and preserve the

incident X-ray photon flux. Risterucci et al. found that after CO exposure, CO chemisorbed as Pt-CO species in one-fold (atop) and two-fold (bridged) coordination sites on the Pt surface. Step A in figure 2 graphically illustrates these Pt-CO chemisorbed species adsorption sites. In Risterucci's study, the Pt-CO surface species was identified by peaks in X-ray photoelectron spectra. After 5 hours of X-ray irradiation with a flux similar to those present during beamline operation, the Pt-CO chemisorbed surface species decreased to approximately 10% of its original value, and a new peak appeared in the spectra corresponding to the presence of newly formed C-C surface species. This result suggests the occurrence of photon-induced dissociation of chemisorbed CO (the Pt-CO species) to produce a carbon contamination layer. The photon-induced dissociation is also consistent with the appearance of the black band observed on the optics surface where X-rays strike and not on the other parts. These chemical surface species are shown graphically in Figure 2, as identified by Risterucci et al.

Figure 2. Steps in carbon contamination formation from residual $CO_{(g)}$. step A (left) shows the chemisorbed CO, step B (center) shows X-ray irradiation/photon-induced dissociation, and step C (right) shows the resulting carbon contamination layer. It should be noted that there will be other mechanistic steps when going from B to C that have not been discussed or illustrated. This graphic was produced by SAES Group.



Hydrocarbons and Other Carbon Species Adsorbed to the Chamber

For the second source, carbon species are found to be adsorbed on and dissolved in materials commonly used in vacuum applications and X-ray optics chambers, despite the best efforts of proper vacuum preparation and treatment. The literature shows that carbon-containing residues were thermally desorbed at 400 °C from 304 stainless steel and alloy 5052 aluminum, despite being cleaned and prepared with standard vacuum procedures [5]. Considering these two materials are commonly used in the construction of optics chambers,

this study reveals the potential for carbon contamination.

With the large number of components in optics chambers, the presence of residual hydrocarbons is inevitable. Multiple studies have shown the extent of carbon contamination in optics chambers, and that its growth rate increases monotonically with the residual hydrocarbon pressure [8,9,10]. Given these findings, it is critical to design a robust pumping system utilizing state of the art pumping technologies.

Pumping Systems in Optics Chambers

Many methods have been devised and employed to deal with this problem [11,12,3]. The most traditional approach and the first line of defense is the use of vacuum pumps capable of sorbing the outgassed carbon species.

Several requirements that the pumping systems in optics vacuum chambers should meet are the following:

- Minimization of particle generation
- Minimization of heat irradiation during operation
- High pumping speeds for carbon gas species

While non-evaporable getter (NEG) pumps have gained popularity over the past several decades in many accelerator and synchrotron applications, with the arrival of the new ZAO® getter material, they can now meet the requirements needed for optics vacuum chambers.

Minimization of Particle Generation by the ZAO® NEG Material

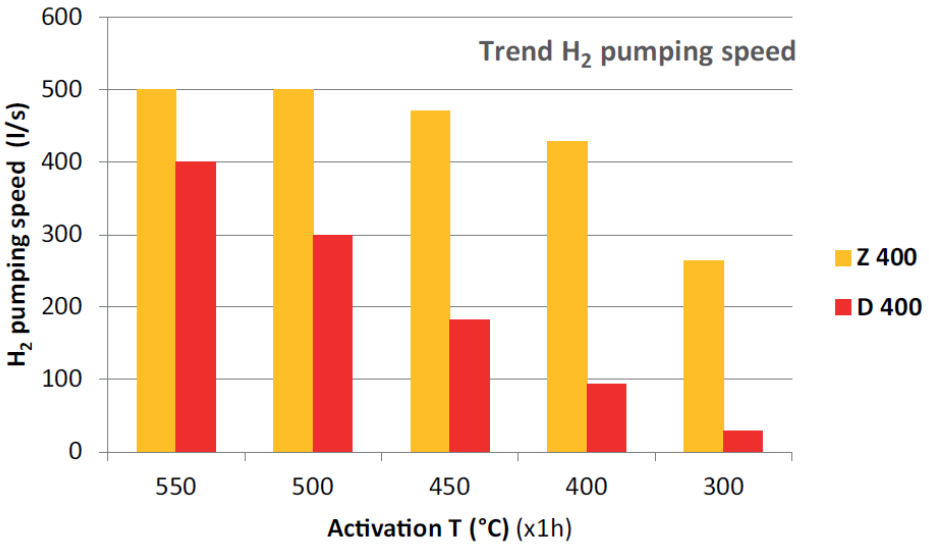
The ZAO® getter material, designed, developed, and manufactured by SAES Getters, meets the low-particle requirements of optics chambers. This has been validated over the last several years, as particle sensitive applications such as photocathode transportation [13], electron microscopes [14], OLED deposition chambers [15], and cold trap systems [16] have successfully employed SAES NEG pumps. Furthermore, the SAES getter materials have undergone stringent particle-counting and functional tests by multiple accelerator facilities. Jefferson Lab recently performed particle-counting tests on NEG pumps featuring the ZAO® getter material and found the pumps meet the rigorous low-particle requirements of their high-gradient SRF cavity applications [17].

Minimization of Heat Irradiation by ZAO® Based Pumps

The ZAO® sintered NEG alloy is capable of a higher degree of activation at lower temperatures. This is a significant development, and it allows for the activation of the ZAO® NEG pumps at lower temperatures than those needed for St172 NEG alloys. Figure 3, below, reports the degree of activation at different

temperatures for the ZAO® vs. the St172 for NEG pumps of the same size. An activation for several hours at 400° C is sufficient for the ZAO® NEG material, and the required power is 40-200 W (approximately 3.5 amps), depending on the NEG pump's size and model. While it is always good to consume less energy in general, the lower required power also results in decreased thermal irradiation and dispersion, which minimizes the pump's effects on the temperature and thermal properties of the optics.

Figure 3. Degree of Activation after 1 hour at different temperatures for the CapaciTorr D400 (St172) and the CapaciTorr Z400

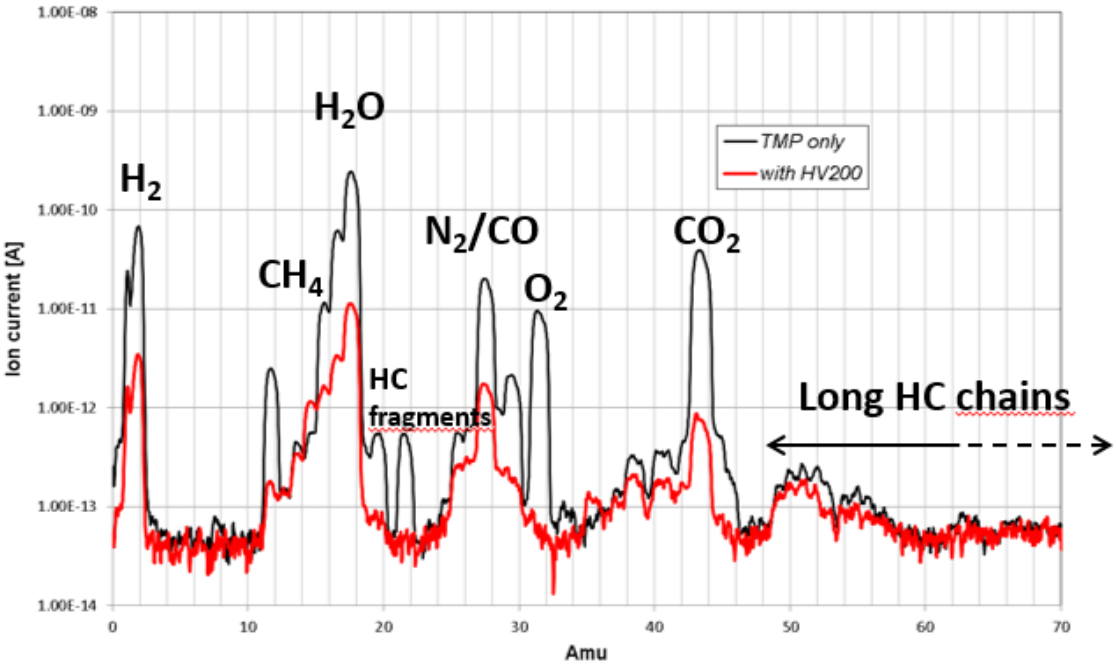


The High Pumping Speed of the ZAO Based NEG Pumps for Carbon Gas Species

Another essential feature of the ZAO® NEG pumps is its pumping speed for carbon-containing gases, including hydrocarbon species. Figure 4 illustrates this by comparing two RGA spectra: one obtained using only a turbomolecular pump (TMP) and one obtained using a TMP in combination with a ZAO® NEG pump. The spectra were acquired in a vacuum system with large amounts of carbon species, as evidenced by the peaks in figure 4. For this type of application, the ZAO® NEG pump operates at approximately 200° C. At this temperature, the ZAO® getter alloy promotes the cracking of hydrocarbon molecules and will sorb carbon, nitrogen, and oxygen species into its bulk. Other getter alloys can also work at 200° C but will release significant amounts of hydrogen gas, which can negatively affect the vacuum pressure and conditions. The ZAO NEG alloy does not have this problem. SAES specifically designed and engineered the ZAO NEG alloy to exhibit a hydrogen gas equilibrium pressure one to two orders of magnitude lower than

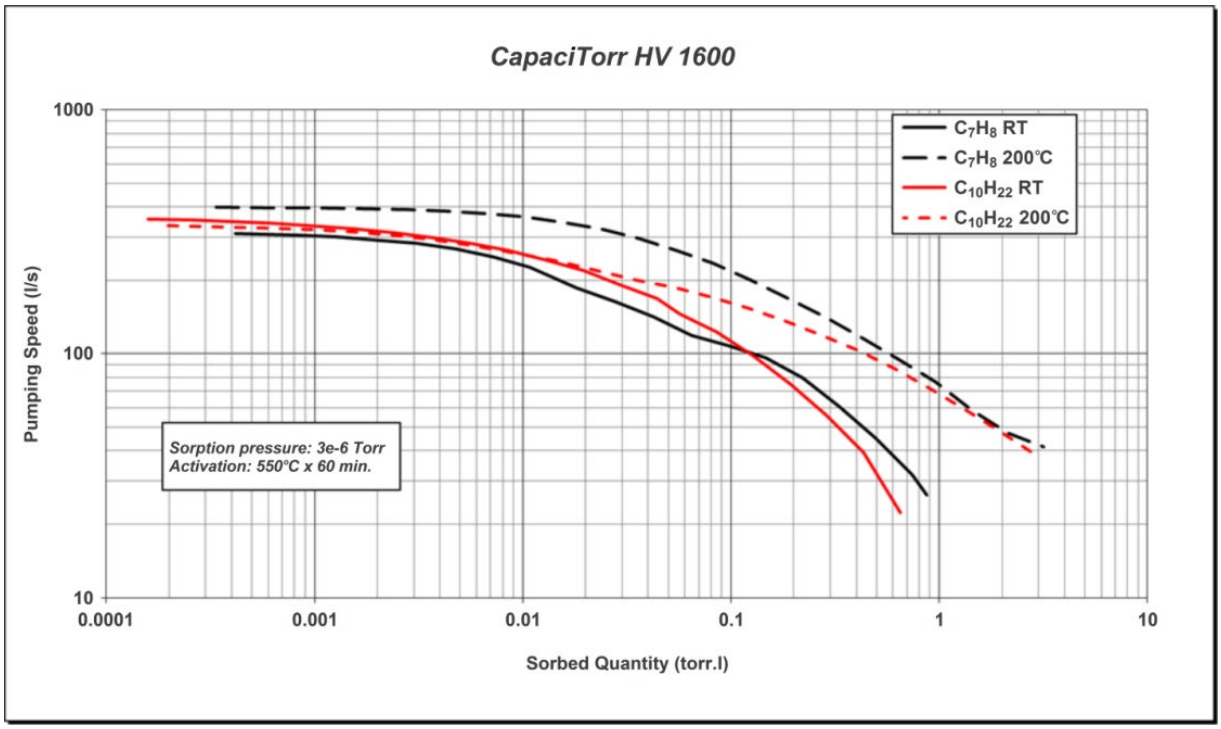
standard NEG alloys, which allows the ZAO to circumvent this issue gracefully. This is a unique and unmatched feature of the ZAO® sintered NEG alloy.

Figure 4. Comparision of two RGA spectra: one obtained using only a TMP (200 l/s) and one obtained using a TMP (70 l/s) in combination with a ZAO® NEG pump (CapaciTorr HV200).



To further investigate and document the ZAO's pumping capabilities, the CapaciTorr HV 1600, featuring the ZAO NEG alloy, was tested for its ability to pump toluene and n-decane. These molecules are representative of two relevant classes of hydrocarbons (i.e. aromatic and long-chain linear hydrocarbons) and demonstrate the ability of the ZAO based pumps to sorb similar molecules belonging to such classes. While operating at room temperature, the CapaciTorr HV 1600 can pump 200 and 250 l/s of toluene and n-decane, respectively. Operating at 200 C, the CapaciTorr HV 1600 can pump 400 l/s of toluene. Figure 5 illustrates this data. Moreover, the CapaciTorr HV 1600 is available on a CF150/8" flange, only weighs approximately 6.5 kg, and comes in a small package. All these characteristics are ideal for the tight spaces that house optics chambers.

Figure 5. Pumping speed vs. capacity of CapaciTorr HV1600 NEG pump for Toluene and Decane. The measurements were collected at RT and 200°C.



Additional Features of ZAO Based Pumps

Another important feature of the ZAO-based pumps is their ability to undergo activation at a much higher pressure relative to conventional getter alloys. In fact, NEG pumps based on the ZAO getter alloy can start their activation at 1 x 10^{-3} Torr or lower. This stems from the alloy's intrinsically robust and durable nature.

SAES Pump Portfolio for Optics Chambers

Compared to conventional pumping technology used in optics chambers, SAES NEG pumps can deliver high pumping speeds in much smaller dimensional packages and significantly lower weights. These characteristics allow for two powerful yet differing approaches:

1) The installation of one large pump delivering one to four thousand l/s pumping speeds
2) The distribution of multiple smaller NEG pumps around the vacuum chamber

In the first approach, a large NEG pump, such as the CapaciTorr Z 3500 (or CapaciTorr HV 1600), featuring the ZAO® getter material, can be installed. These NEG pumps will provide pumping speeds up 3900 l/s (1700 l/s) H_2 and

1400 l/s (1000 l/s) CO. It is available on a CF150/8" flange and only weighs approximately 6.5 kg; a significant contrast to the larger and heavier vacuum pumps typically used in optics chambers. Figure 6, below, shows the CapaciTorr Z 3500.

The second approach uses multiple smaller pumps but maximizes their conduction and speed through spatial distribution around the chamber. These NEG pumps will typically have speeds of 200 to 500 l/s. This approach can be very effective given the conductance limitations imposed by the many components installed in optics chambers. Furthermore, the SAES NEXTorr pump integrates the NEG and ion technologies into one pump model, allowing for the distribution of the pumping speed of non-getterable gases by its compact ion pump component. Figure 7 presents the NEXTorr Z 300.

Figure 6. The CapaciTorr Z 3500 on a CF150/8" flange

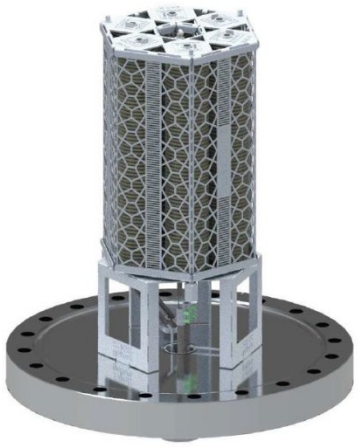
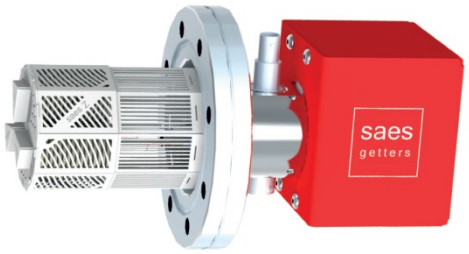


Figure 7. The NEXTorr Z 300 on a CF63/4.5" flange



Traditionally, SAES NEG pumps have found success in UHV and XHV applications. However, by the development of the ZAO getter alloy, they can reach beyond these pressure ranges. With the new ZAO getter material, SAES NEG pumps can operate in the HV pressure range as well. SAES now possesses an extensive portfolio of vacuum pumps that spans many applications from high vacuum chambers to electron storage rings to XHV experimental end stations.

The SAES pump portfolio consists of the following:

NEXTorr Series

The NEXTorr Series optimally integrates the NEG and SIP technologies into one pump through a patented design that maximizes the NEG pump's speed. Within the NEXTorr pump family, there are the HV and Z series, which are described as follows:

NEXTorr HV Series

- Pressure Range: $1x10^{-7}$ Torr to XHV.
- NEG Material: ZAO that has been engineered to maximize capacity.
- Sizes:

Pump	Pumping Speed, l/s		Flange size	Weight, kg (lbs)
	H_2	CO*		
NEXTorr HV 100	80	40	CF35/2.75"	2.2 (4.9)
NEXTorr HV 200	155	90	CF35/2.75"	2.3 (5.1)
NEXTorr HV 300	300	190	CF63/4.5"	4.5 (9.9)

**These speeds are based off the measured speeds for CO_2*

NEXTorr Z Series

- Pressure Range: $1x10^{-9}$ Torr to XHV.
- NEG Material: ZAO that has been engineered to maximize speed.
- Sizes:

Pump	Pumping Speed, l/s		Flange size	Weight, kg (lbs)
	H_2	CO		
NEXTorr Z 100	150	60	CF35/2.75"	2.2 (4.6)
NEXTorr Z 200	290	130	CF35/2.75"	2.3 (5.1)
NEXTorr Z 300	400	190	CF63/4.50"	4.4 (9.7)
NEXTorr Z 1000	1150	500	CF100/6.00"	7.1 (15.6)

CapaciTorr Series

The CapaciTorr series are stand-alone NEG pumps. These pumps provide large pumping speeds in compact designs for the getterable gases. When supplemented by technologies capable of pumping non-getterable gases, the CapaciTorr Series pumps create a powerful pumping system. Within the CapaciTorr pump family, there are the HV and Z series. These two series are described as follows:

CapaciTorr HV Series

- Pressure Range: $1x10^{-7}$ Torr to XHV.
- NEG Material: ZAO engineered to maximize capacity.
- Sizes:

Pump	Pumping Speed, l/s		Flange size	Weight, kg (lbs)
	H_2	CO*		
CapaciTorr HV 200	105	25	CF35/2.75"	0.7 (1.5)
	210	65	CF63/4.5"	
CapaciTorr HV 1600	1700	620	CF150/8.00"	6.5 (14.3)
CapaciTorr HV 2100	2100	880	CF200/10.0"	6.9 (15.2)

**These speeds are based off the measured speeds for CO_2*

CapaciTorr Z Series

- Pressure Range: $1x10^{-9}$ Torr to XHV.
- NEG Material: ZAO engineered to maximize speed.
- Sizes:

Pump	Pumping Speed, l/s		Flange size	Weight, kg (lbs)
	H_2	CO		
CapaciTorr Z 100	150	65	CF35/2.75"	0.4 (0.9)
CapaciTorr Z 200	290	130	CF35/2.75"	0.5 (1.1)
CapaciTorr Z 400	500	210	CF63/4.50"	0.7 (1.5)
CapaciTorr Z 1000	1250	550	CF63/4.50"	1.6 (3.5)
			CF100/6.00"	2.1 (4.6)
CapaciTorr Z 3500	3900	1400	CF150/8.00"	6.5 (14.3)
			CF200/10.0"	7.5 (16.5)

Conclusions

As the vacuum community knows too well, carbon contamination happens. It happens in surface science UHV chambers in universities all around the world, it happens in experimental end-stations at synchrotron beamlines, and it happens in the optic chambers of the beamlines themselves. To say the least, it is quite frustrating, if not detrimental to operations. There was a time, when carbon contamination was a show stopper; when mirrors had to be removed, then cleaned, and sometimes even replaced. Fortunately, clever mitigation methods and new pumping technologies now reduce this problem and decrease operational downtime.

The SAES state of the art ZAO NEG pumps can bolster pumping system designs by supplying hundreds and even thousands of l/s of pumping speed, significantly reducing the carbon residual contamination precursor gases. Compared to other approaches like traditional pumps (e.g. TMP and ion pump) and oxygen treatments, ZAO NEG pumps are significantly more compact and less invasive, respectively. The new ZAO getter alloy minimizes weight and space requirements of the pumping system, reduces particle generation to levels acceptable for SRF and photocathode application, minimizes heat dissipation during activation, and it is a vibration-free pumping system. Additionally, the ZAO getter alloy expands the NEG pump's applications to the HV pressure range as well. These properties make the SAES NEG pumps, featuring the ZAO getter alloy, a valuable addition to any vacuum chamber, especially optics chambers or any other chambers that suffer from carbon contamination.

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