eJSSNT Review Paper

e-Journal of Surface Science and Nanotechnology **22**, 287–295 (2024)

Water in Vacuum Systems: Problems and Solutions

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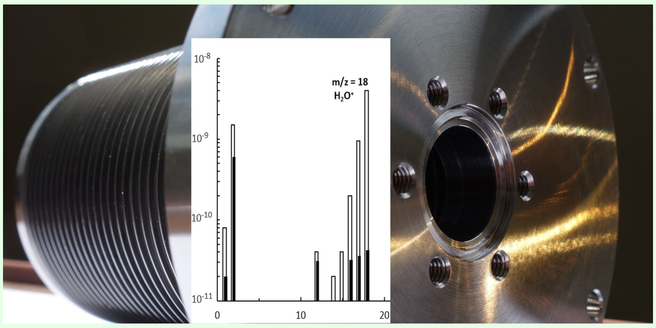
Received: 4 December, 2023; Accepted: 20 August, 2024; J-STAGE Advance Publication: 19 September, 2024; Published: 19 September, 2024



For many processes in vacuum, the presence of water is both undesirable and unavoidable. This review offers a qualitative look at a broad spectrum of water related issues and their solutions in vacuum technology. First, a systematic overview outlines the most important technical innovations and publications from the last decades. Relevant properties of surfaces, materials and other contributors of water molecules are also discussed. Second, the review offers practical advice for a multitude of applications ranging from compact lab ultrahigh vacuum devices, production machines like coaters and etchers, to large-scale facilities for space simulation and extreme ultraviolet lithography. The goal of this article is to provide a comprehensive tutorial on effective water handling for users, designers and process engineers in basic research and industry.

Keywords *Water outgassing; Contamination; Permeation; Vacuum technology; Residual gas*

I. INTRODUCTION

Why does water play such an important role in vacuum technology? Several reasons exist, here are the two most prominent ones. First, water binds to technical surfaces in a very inconvenient way: neither does it desorb quickly, nor does it remain bound forever. In many cases the water molecules dominate the residual gas of the vacuum system due to continuous outgassing processes. Second, water is almost everywhere and always present: in the cleaning bath, in the air, in the cooling line, etc. The problems with water in vacuum systems are extensively discussed in books [1, 2], other texts [3–13] and review publications from the last decades [14–16].

The main source of water is air, which “donates” its moisture to the surfaces of an open vacuum system. Under normal conditions and relative humidity 40%, one cubic meter of air contains about 9 g of water. This amount would be enough to cover 10 m^2 of inner surfaces of a chamber with thousands (!) of monolayers of water. In this sense, any venting with air is a very problematic step, as previously dry surfaces will become filled with water molecules.

Parts made of plastic can store water in their volume, which will slowly leak into the vacuum. In addition, water permeates from the outside through the components of polymer materials, for example elastomer seals.

A process gas can also carry water; H_2O is at the top of the list of impurities in many technical gases.

II. WATER DURING EVACUATION

Most water molecules present in the volume of a vacuum chamber at the start of pumping are usually unproblematic (see Section IX for rare exceptions such as aerosol formation) and are evacuated relatively quickly along with nitrogen, oxygen and other volatile molecules. Water molecules attached to other water molecules are only weakly bound to surfaces and will be also soon removed. Depending on the humidity of the venting gases, duration of exposure, temperature and affinity of the parts to water, several, even up to some hundred monolayers of water can adhere to the surfaces [3, 11, 13, 17, 18] and be pumped up in the first seconds and minutes.

In contrast, desorption of last monolayers of water is a slow process. This is related to the binding energy of water molecules on surfaces. Quoting Ref. 2: “Unfortunately for vacuum technology”, the desorption energy of water on stainless steel and aluminum is in the range of 80–104 kJ mol^{-1}.

The molecules with much lower desorption energy (below

$71\,\mathrm{kJ\,mol^{-1}}$) are quickly gone, the molecules with desorption energy above $105\,\mathrm{kJ\,mol^{-1}}$ hardly desorb at all. In addition, a water molecule that has just been desorbed is very likely to hit a surface again and be adsorbed there. The re-adsorption of water is thus part of the problem. The "unfortunate" binding energies of the water molecules on technical surfaces are the reason, why the residual gas of a typical, non-baked vacuum chamber amounts to 80–90% water. At constant temperature and pumping speed, in an all-metal system, the outgassing rate, and thus the pressure, changes roughly inversely proportional to time, $p \propto t^{-1}$. Outgassing is accelerated by higher temperature, plasma, friction, and the supply of other types of energy; see the Section VII with *in situ* cleaning for more details.

III. WATER AND SURFACE

When examined under a microscope, the surfaces of metal components are far from homogeneous and smooth, even mirror-polished ones. Components of stainless steel and aluminum have a more or less thick oxide layer that can contain a lot of water molecules in its microcracks, pores and other defects and evolve H_2O into the vacuum for a very long time. Water emission of stainless steel after 1 h of pumping at room temperature is in the range of $10^{-9}\,\mathrm{mbar\,L\,cm^{-2}\,s^{-1}}$. The usual aluminum oxide layers are somewhat more spongy, therefore the aluminum components outgas an order of magnitude more water after venting than stainless-steel parts [19–21].

After decades of research, there is a broad consensus on how to effectively reduce outgassing of metal parts: a clean and dense oxide layer on a smooth, preferably mechanically or electrochemically polished surface is a favorable universal solution. One of the last steps for stainless-steel parts is the so-called passivation in a bath of mild nitric or citric acid. During the process, which is not only common in vacuum technology, the "natural" layer of chromium oxide Cr_2O_3 becomes even denser and more protective. Some vacuum parts are coated with various other chemically neutral films, for example gold plating. Parts made of normal steel are often nickel-plated, which, among other things, greatly reduces the absorption of water and consequently outgassing. In general, a lower partial pressure of water can be achieved by improving surface finishing.

Not every good coating helps against water in vacuum. Aluminum surfaces treated by anodizing represent an extreme sponge, made of countless thin parallel aluminum oxide tubes thousands of nanometers high, see Figure 1. Such coatings protect parts of vacuum valves in etching processes, for example. But the water absorption and outgassing of the anodized parts is several orders of magnitude higher, making them fundamentally unsuitable for vacuum systems that are often exposed to water vapor.

Similar problems are known with coaters whose internals get suboptimal, porous coatings. With each subsequent batch, they become thicker and outgas more. For less water in the residual gas, the processes and vacuum parts should be designed to minimize the growth of the unnecessary, spongy layers.

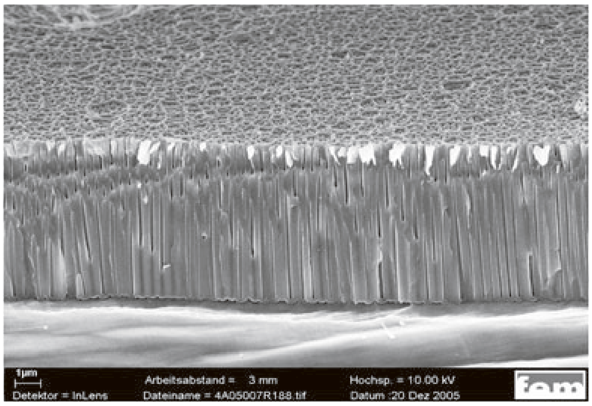


Figure 1: SEM image of typical anodized surface. The film may protect the aluminum part very well from various external influences, but its porous structure makes it inappropriate for vacuum technology. Picture: fem Research Institute Precious Metals.

Glass and quartz parts with their "naturally" smooth surface exhibit low outgassing of water, comparable with metals like aluminum. A high-performance pure ceramic without pores, e.g., of 99.6% Al_2O_3 is a good choice too.

IV. WATER AND POLYMERS

Two phenomena provide higher water outgassing of polymeric parts in vacuum; first, water can be dissolved in these materials, and second, they have much higher permeability compared to metals and let water through from outside [22–26].

Viton™ (The Chemours Company FC, LLC), polytetrafluoroethylene (PTFE), and other polymers commonly used in vacuum technology for sealings can store larger amounts of water in their volume, up to 1% of their own weight! The outgassing rate of dissolved water from plastic parts decreases even more slowly than for metals, approximately proportional to $t^{-0.5}$ [6, 27, 28]. The specific outgassing of polymeric parts after ten hours of evacuation is typically in the range of $10^{-7}\,\mathrm{mbar\,L\,cm^{-2}\,s^{-1}}$, i.e., about three orders of magnitude higher than for stainless steel.

Water diffuses slowly in polymers, so both the intake and outgassing of water can last for several days or even weeks. These times are also characteristic for permeation of water through seals and hoses made of plastic. The "long breath" of polymers is shown very impressively on the example of Viton seals in Ref. 29, see Figure 2. The diffusion of water molecules dissolved in the O-ring (KF40, approximately 5 mm thick) is determining the outgassing during the first few days, compare curves B and C. After 50–100 h, permeation through the seal becomes more and more noticeable. Only after about 40 days do Viton seals "forget" their prehistory, and outgassing is driven only by permeation. Dissolved helium gets out in a few minutes from such a seal [1]. Water would take a month or more!

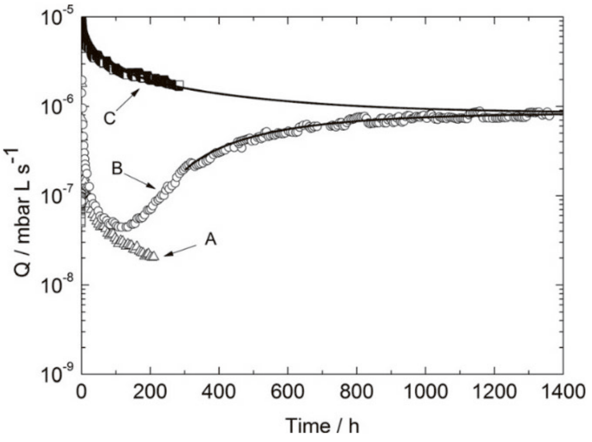


Figure 2: Gas flow versus time. Total leakage rate of H_2O, N_2, O_2, Ar, and CO_2 in a vacuum system over about 2 months at room temperature. Content of H_2O was 90% or more. The solid curves represent simulated trends, the measured points show outgassing of the system with: (A) aluminum gasket, (B) degassed Viton O-ring, and (C) Viton O-ring saturated with humid air. The figure is reproduced from Figure 2 in Ref. 29 [M. Sefa and J. Setina, J. Vac. Sci. Technol. A **35**, 041603 (2017)]. Copyright (2017) by The American Vacuum Society.

It is mainly the water molecules that pass through the elastomer seals from the air [23, 25, 29–32]. The relatively inexpensive Viton seals are among the better ones, their permeability is comparatively low.

Water and other gases diffuse faster through the polymeric materials at higher temperatures [2, 33]. Thus, permeation of water through Viton increases by a factor of about 4–5 at 80–100°C. To drive out the gases dissolved in a polymeric part, it should be heated in a vacuum for a few days, depending on its thickness. A 5–10 mm thick Viton O-ring needs about 2–3 days at 100–150°C in vacuum. Immediately after this treatment, it would not outgas much more water than a metal gasket, see curve B in Figure 2.

The problem of permeability of elastomer O-rings can be avoided entirely by replacing them with metal seals, see Figure 3. These can be edged seals made of soft metal, e.g., pure aluminum or copper. Another variant of metal gaskets are elastic parts with a C-shaped profile, so-called C-rings, usually made of Inconel™ (The Special Metals); they often have a shell of aluminum. With special fine versions of such gaskets, even glass windows can be sealed. Metal seals are much more expensive, require a higher sealing force, can be used only once, and have higher demands on the quality of the sealing surfaces. Despite the disadvantages, some rubber-sealed chambers have been retrofitted with metal gaskets for clean processes.

Where a metal seal is not an option, for example on a chamber door, double O-rings with pumped interspace [25, 30] can help. The pumping stage for the differentially pumped seals does not have to be of high performance; a pressure below 1 mbar is already sufficient to reduce permeation from the air by a factor of 1000.

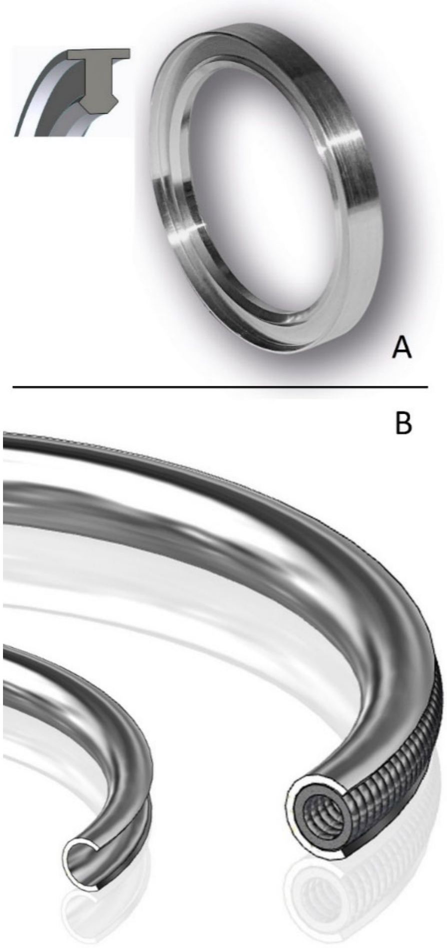


Figure 3: Metal seals replacing polymer O-rings. (A) Edged metal gasket, readily available off-shelf for standard vacuum connections DIN 28403/ISO 2861 and DIN 28404/ISO 1609. Courtesy of Schwer fittings GmbH. (B) Metal C-rings, often energized with a helical spring. Courtesy of GFD-Gesellschaft für Dichtungstechnik mbH.

Outgassing properties of some high temperature polymer materials like polyether ether ketone (PEEK) and Kapton™ (Dupont Electronics) have been recently examined in Ref. 27, including measurements at room temperature, 100 and 200°C. After 10 h evacuation at room temperature, water emission of these materials is high, in the range of 10^{-7} mbar L cm^{-2} s^{-1}, falling approximately proportional to $t^{-0.5}$. Thin Kapton foils ($\leq$0.05 mm) provide an exception, their outgassing rate of water is comparable to stainless steel.

Thus, in-vacuum insulators of choice are glass or tight clean ceramic. Alternatively, the thickness of polymer parts could be reduced to a minimum.

V. EFFECTS OF WATER ON PROCESSES AND COMPONENTS

Water can cause a wide variety of problems in a vacuum system; a complete list would go beyond the scope of a brief review. Let's name three of the most troublesome ones. First, failures of mechanical backing pumps can occur when their moving parts are damaged by ice crystals. Second, when operating with aggressive gases, for example in semiconductor manufacturing etching equipment, water contamination should be kept at minimum, otherwise surfaces can corrode much faster [34]. Third, water often interferes with coating processes, and its presence can greatly affect coating growth and properties [35, 36].

One of the few positive effects of water in a vacuum system is that parts slide much better on each other without lubrication and without special layers as long as they are covered with a water film. The "natural" water layer also prevents seizing or cold welding of the surfaces. We usually do not notice the lubricating effect of the adsorbed water molecules; it is simply present under normal conditions. However, when this lubricating layer is missing, the rubbing parts can suddenly malfunction, in a clean vacuum system as well as in a mechanism that is permanently purged with a dry gas [37, 38].

Feeding water to a process is not widely used in vacuum technology and sounds paradoxical, but it can be a reasonable solution for some applications, e.g., with temperature sensitive components: sometimes it is not the water itself that disturbs, but the fact that its partial pressure is not reproducible or changes with time. A defined, slightly higher partial pressure of water can help to keep its impact stable and to control the process better. For this purpose, water can be supplied from a tank via a small nozzle; anyway, such a controlled gas flow of water is technically much easier to accomplish than the tedious elimination of water molecules.

VI. REDUCING WATER INTAKE PRIOR TO THE PROCESS

One of the most common and effective measures against water is loading and unloading a chamber via a load lock. While this device increases the price and dimensions of a vacuum system, it speeds up processes tremendously. Load-locks enable to maintain best vacuum conditions without interruptions for substrate exchange. They find application in machines for wafer processing, electron microscopes, ultrahigh vacuum (UHV) surface analysis instruments and many others. Cycle times can be reduced from days and weeks to minutes by using a load-lock. Typically, load-lock is designed as an additional vacuum chamber, roughly as large as the parts being loaded and unloaded, see Figure 4.

Another variant of a load lock is a gas-purged container, e.g., a glove box, which is docked to or encloses the vacuum chamber (see Figure 5). Such load locks are usually inexpensive because they do not have to be vacuum-proof and do not require vacuum pumps. When using a gas-filled load lock, the vacuum chamber must be filled with atmospheric pressure too and then pumped down again. If the venting of the chamber and the other operations are carried out with a clean dry gas [18], only very few water molecules will enter the vacuum system.

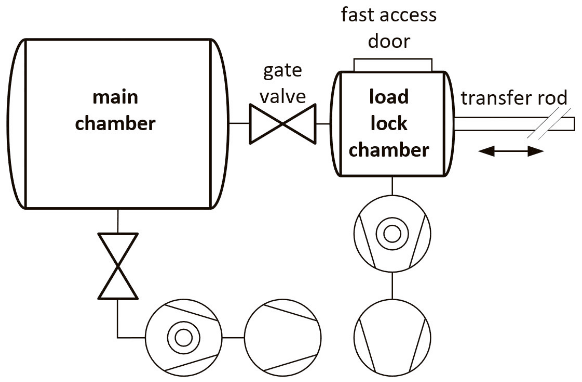


Figure 4: A typical lab configuration with a vacuum load lock. For the safe handling of 4" wafers, the gate valve and the fast entry access port have inner diameter 6". With a 200 L s^{-1} turbomolecular pump on the load-lock, transfer of the wafer into the main chamber can start below 10^{-6} mbar after 1/2 h of pumping.

Figure 5: A section of a metal organic chemical vapor deposition system. The glove box on the left protects the reactor chamber from air during service, the glove box on the right is used to load and unload the substrates. With permission from AIXTRON SE.

In the absence of a gas-purged or vacuum load lock, the entering of water into a vacuum chamber during servicing, loading and unloading cannot be avoided, but can be somewhat diminished by the following measures.

First, the chamber should be vented with dry gas and its slight overpressure maintained until pump-down is started. This way, the diffusion of air moisture will be mitigated by counterflow. Turbulence and other causes of an inflow of air should be avoided, and the opening should be as small as possible. Secondly, the interior of the chamber should be as warm as possible before and during venting. This reduces adsorption of water on the vacuum parts and prevents condensation. Third, the duration of venting should be as short as possible. For metal parts, it makes virtually no difference whether the chamber is open for three hours or three days, because the surfaces will be covered with about a monolayer of water after only one hour or faster, even if partial pressure of water is well below 1 mbar. However, it makes a big difference whether the exposure to moisture is limited to a few minutes or even seconds [4]. Though the elastomers such as Viton still do take up water after days, but here, too, the minutes at the beginning of the venting count much more [31].

Last but not least, the water content in the air should be low. The parts that will be brought into the vacuum should also be as dry as possible. Especially for the machines with polymer seals and porous coatings, the water intake and outgassing correlates with the humidity of the air during loading [11]. Thus, it may be worthwhile to limit the humidity of the ambient air to about 40%, which is still sufficient from the point of view of health protection in the workplace. Breathing out into the chamber should also be kept to a minimum, as the air we exhale contains double or even triple the amount of water.

VII. REMOVING WATER MOLECULES BEFORE THE PROCESS – *IN SITU* CLEANING

Bake-out is the common *in situ* cleaning method in ultra-high vacuum technique [2]. In this process, the vented chamber is first evacuated, then heated and then cooled down again, see Figure 6. In all-metal systems, water can be practically completely removed with bakeout temperatures up to 450°C. Bakeout at 150–200°C is sufficient for radical reduction of outgassing of water [39]. After 24 h at 150°C, the main component of the residual gas in a stainless-steel chamber is usually no longer water but hydrogen; the partial pressure of water is then 2–3 orders of magnitude lower than in an unheated system, see Figure 10. At these temperatures, the elastomer seals of valves and doors are not subjected to excessive stress. The max. permissible thermal load on turbomolecular pumps or other components can be a good reason for heating only up to 100–120°C. But even a bakeout at 60–80°C noticeably reduces the partial pressure of water.

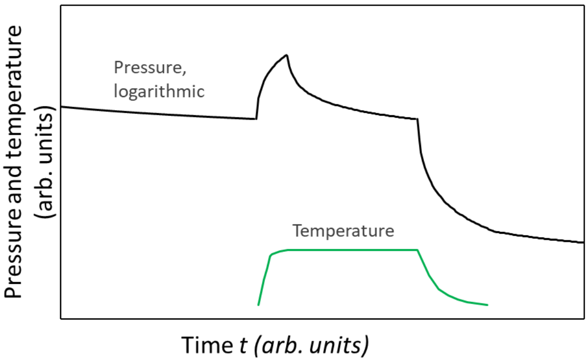


Figure 6: Schematic pressure and temperature curves during bake-out. Because water dominates the residual gas after a venting, the pressure curve is also characteristic for partial pressure of water. In the phases of constant temperature before and during heating, the pressure falls with time in about $p \propto t^{-1}$, and much more slowly thereafter.

A bakeout is often started first in the fine or high vacuum range so that the high vacuum pump can continue to be used when the pressure rises during heating. Typically, bakeout lasts 12–48 h, but sometimes a week. It is useful in this time to also turn on the in-vacuum components that will be heated during normal operation - filaments, heating elements and others.

The relatively long time required for a bake-out is rewarded by the effect that the water will be removed more thoroughly than with the other *in situ* cleaning methods. The elevated temperatures not only provide greater desorption and less re-adsorption, but also have a depth effect: they accelerate diffusion from plastics and from the porous parts and layers.

Another method, cleaning with plasma, can also accelerate desorption of water and is much faster than bake out. In the simplest case, a glow discharge (Figure 7) will be used. Typically, plasma is ignited in argon or helium, often with the addition of oxygen, which improves the passivating oxide layers on technical surfaces. The pressure is in the range of a few hundredths of a mbar. The water molecules on the surfaces are removed very quickly and effectively with plasma; due to the high energy of the plasma particles, 10–30 min are often enough. In the process, the plasma almost does not heat up the internal components. However, compared with bake-out, the pores and other “hidden places” will not be cleaned as successfully.

For all *in situ* cleaning methods, the vacuum system should be heated or cleaned as uniformly as possible. Otherwise, the water molecules will be re-adsorbed at the colder or hidden spots and continue to outgas from there. Therefore, locally limited cleaning methods such as exposure to ultraviolet light, ions, etc. are less effective and are worthwhile only in certain cases.

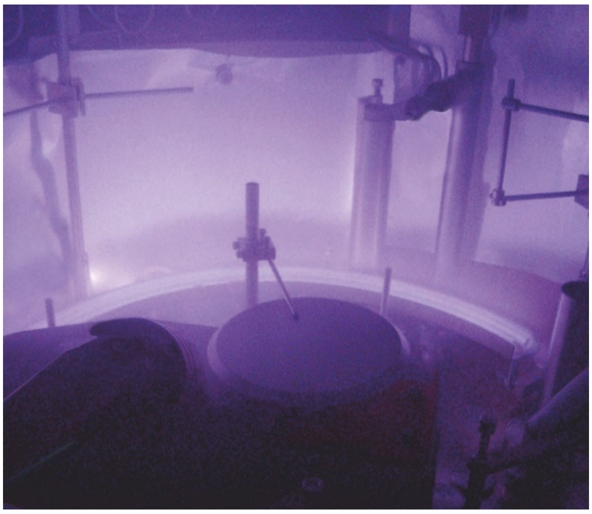


Figure 7: Glow discharge in a coater. The long bright arc in the lower half is the glow rod. Courtesy of Marquis Automatisierungstechnik GmbH.

In some applications, neither bakeout nor plasma cleaning are possible or useful. In these cases, cyclic purging (flushing) with dry gases can help [9, 16, 40, 41]. This method is very gentle and has been used for decades in semiconductor manufacturing to remove all kinds of contamination, especially against water in geometries with labyrinths like in gas supply subsystems or extremely narrow volumes of display panels of 40 cm × 40 cm × 75 µm. Typically, dry, possibly warm nitrogen is let in and pumped out in dozens of cycles, while the pressure changes repeatedly every minute between 50 and 0.1 mbar. The cleaning effect is that the gas molecules replace the adsorbed water through the countless impacts. The energy input and thus the probability of desorption is rather low compared to the other *in situ* cleaning processes. But instead, the desorbed water molecules will be ripped off by the force of the viscous flow from the remotest corners and have low probability of re-adsorption. Fortunately, various types of backing pumps are very effective in this pressure range, allowing short cycle times and fast gas flows. Such a cleaning takes about one hour, for larger volumes and smaller pumps several hours.

Of course, the above *in situ* cleaning methods are often combined to achieve better results. The methods help not only against water, but also against other contaminants, especially organic ones.

VIII. REDUCING THE AMOUNT OF WATER MOLECULES DURING PROCESSING

Cryopumps are a powerful tool to reduce the number of disturbing water molecules. Modern refrigerator cryopumps, available as catalog goods, trap practically every incoming water molecule. Therefore, the pumps have a very high pumping speed especially for water. Meissner traps [42], a subtype of cryopumps, are even more helpful against water. They are usually placed directly inside the vacuum chamber, and their pumping speed is not limited by the conductance of a pump port and valves. This is the most effective way to break the long chain of multiple desorption and re-adsorption and reduce partial pressure of water by several orders of magnitude. With one square meter of surface area, a Meissner trap can capture up to 150000 $\mathrm{L\,s^{-1}}$ of water, see Figure 8. Originally, Meissner traps were cooled with liquid nitrogen to about 77 K. Such solutions, sometimes as double-walled shielding with complex geometry, are still used, e.g., for molecular beam epitaxy.

In the last 30–40 years more and more Meissner traps are used in combination with refrigerators. In this case, traps are formed from a tube, usually copper or even stainless steel, see Figure 9. Modern refrigeration units bring the traps to operating temperatures below −130°C within a few minutes and back to room temperature similarly quickly. At moderate vacuum, the pumping speed is roughly proportional to the surface area of the trap, compare the blue and black curves of Figure 8. To reach the partial pressure of water in the UHV range, the trap must be cooled down to −140°C and lower temperature, see the green and black curves and the Table 1. Due to gigantic pumping speed for water and the short times for cooling and heating up, the traps are attractive for many processes. The Meissner traps help especially in coating machines, where they enable greatly shortened cycle times and more stable results. The adsorbed water molecules protect the surfaces of traps from coating.

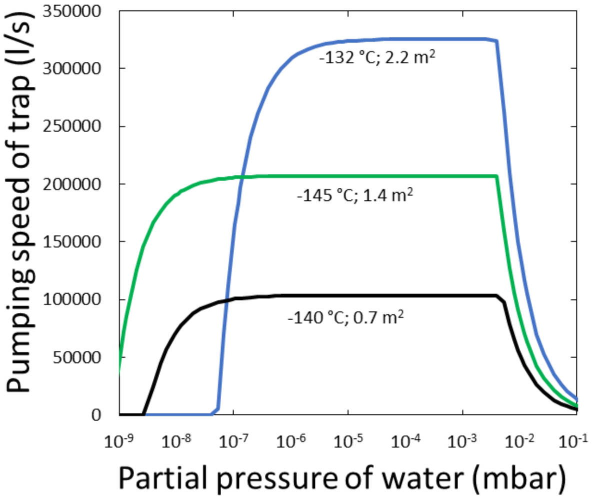


Figure 8: Pumping speed of cold traps, calculated for water vapor. Legends include temperature and area of the cold trap. Data kindly provided by Edwards Vacuum LLC.

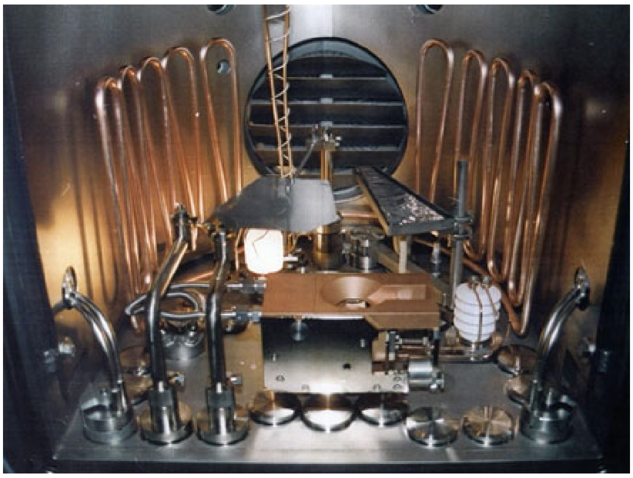


Figure 9: Meissner trap (copper tube meander) in a box coater. Courtesy of Edwards Vacuum LLC.

Table 1: Selected values of saturated water vapor pressure.

Temperature (°C)	Temperature (K)	Pressure (mbar)	Comment
−143	130.16	$<2 \times 10^{-10}$	UHV
−100	173.16	$<2 \times 10^{-5}$	HV
0	273.16	~6	triple point
19	292.16	22	~ room temp.
100	373.16	~1000	boiling point

If gases are let in during a process, they could bring parasitic water with them. Even at room temperature and a partial pressure of water in the range of 10^{-9} mbar, the amount of water on a surface can still increase [11]. This means, for completely dry processes, all gases must also be extremely dry. For example, water content of a few ppm could be troublesome at a process pressure of 0.01 mbar. Either the gas source (cylinder, pressure reducer, filter, etc.) may already contain too much water, or the components of the gas inlet system may allow too much moisture from the atmosphere to pass through due to permeation of plastic hoses, rubber seals, and the like. The less gas flows through the piping, the higher its humidity will rise as a result of permeation. The problem is known, for example, in the turbomolecular pumps, when vented with nitrogen or dry air only from time to time.

The amount of water from the gas supply system can be reduced by avoiding polymer components, permanently purging of gas lines and/or drying the gases directly prior to the vacuum chamber.

IX. PREVENTING AEROSOL FORMATION

If moist air is evacuated quickly, it can cool down enough to form a water aerosol. This interesting phenomenon can only occur under very specific conditions; first of all, it requires fast pumps. In addition, for the gas expansion to be adiabatic, i.e., practically without heat exchange with the in-vacuum components, the volume must be relatively large and free of metal parts. Such a situation arises, for example, in a nearly empty cubic chamber 10 m^3 being evacuated with 25000 $m^3\,h^{-1}$. The conditions for fog formation are well researched and can be described by the so-called Zhao factor [1, 9, 16].

The impact of water aerosol can be critical for sensitive surfaces. The problem: droplets leave marks and particles, which can disturb optical properties, coating adhesion, nanostructures, etc. Why condensed water leads to increased particle contamination on surfaces is not self-evident; after all, the droplets could evaporate without a trace. Mostly, however, the droplets and particles benefit each other: the fog droplets form around particles in the air and bring them to the surfaces; otherwise, the particles would simply be blown away when pumped off [43]. In Ref. 44, another possible explanation for contamination in an application is presented: The droplets may not only consist of pure water, but contain traces of sulfuric acid.

Measures against the formation of aerosols can be listed quickly. Firstly, pumping down can be slowed down; it is sufficient to throttle the pumping speed only in the critical range, i.e., at pressures above 600 mbar. Alternatively or additionally, the moist air could be replaced by a dry gas before pumping down. However, some process time would be wasted for both variants. Another measure, without any loss of time, is given in Ref. 45; metal parts are placed close to the critical surface. This increases the thermal transfer between the humid gas and the surrounding internals, and the gas cools down less. Another possibility is to add a gas with high thermal conductivity, e.g., hydrogen or helium, in a spatially and temporally limited manner. Such a well-dosed, directed feeding of a warm, dry, good heat-conducting gas near the important surfaces (comparable to minimally invasive surgery) could be a pragmatic solution that does not significantly increase process times.

X. PREPARING PARTS FOR A LEAK TEST

Water freezes in a vacuum, so leaks can be sealed by ice and remain undetected during a helium leak test [1]. Because of this, the vacuum parts should not be immersed in water before a helium leak test, or, it is essential to dry them after a water pressure test.

For the same reason, if a cooling circuit inside the vacuum system should be examined with a helium leak test, it must be dried before the test starts. First, liquid can be removed simply with compressed air. The most effective way to completely remove water residue from all gaps and corners of a cooling circuit is to evacuate it with a vacuum pump. At room temperature, for example, this can be accomplished by a diaphragm pump with an ultimate pressure below 10 mbar. To prevent water from blocking or even damaging the pump, it should be repeatedly flushed with ballast gas, as it is common in vacuum drying systems. The pressure in a labyrinthine cooling circuit may remain high for hours or days, roughly equal to the saturation vapor pressure of water at a given temperature, but then falls abruptly as soon as the last drop of water has evaporated. Now the cooling circuit can be vented and the helium leak test can begin.

XI. DETECTING THE PARTIAL PRESSURE OF WATER

The quantity of water molecules moving in the volume of a vacuum chamber can be observed using a mass spectrometer. From this, conclusions can be drawn about the behavior and disturbing effect of water in various phases of the process. The so-called residual gas analyzers (RGA), the most common, inexpensive type of mass spectrometer [1, 2], are recommended for the task. Much more important than features of the RGA, and usually more costly than the instrument itself, is careful integration of the analyzer head into the vacuum system. Ideally, the RGA should be operated continuously, preferably in its own permanently heated and pumped, small UHV chamber, connected to the process chamber via a gate valve. In this way, many measurement artifacts can be avoided and even the smallest amounts of H_2O can be reliably measured. The maximal operating pressure of a typical RGA is ~10^{-5} mbar because the mean free path length of the ions must be comparable to or greater than the filter length of ~0.1 m. If the processes run at a higher pressure, a differential pump stage will be used, the gate valve will be replaced by a set of smaller valves and flow

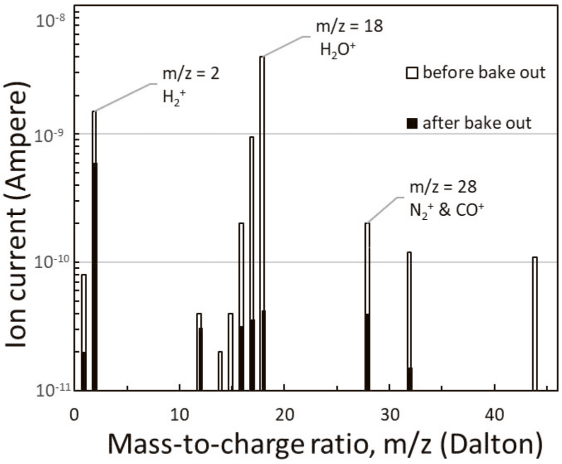


Figure 10: Typical mass spectra of a chamber before and after bake-out at 120°C. After the procedure, the total pressure (corresponding to the sum of all peaks) decreased by an order of magnitude, while the partial pressure of water (peaks at *m*/*z* 18 and 17) fell by a factor of 100.

Figure 11: Analyzer head of an RGA. Its main components are an ion source, a mass filter, and an ion current detector (not shown). The ion source is built on the white ceramic terminal. Behind the terminal, there are four rods of the quadrupole mass filter between two supporting shafts. Courtesy of SRS Stanford Research Systems.

restrictors.

The main reason for continuous operation in a separate heated chamber is that ion source of every RGA uses very hot cathode, see two thin wires around the grid (anode) in Figure 11. The filament emits at >1300°C not only electrons for ionization of the residual gas molecules, but heat radiation too. Thus, in the first 12–24 h, an RGA would measure mainly outgassing of its own ion source and other surfaces being gradually warmed up by the filament.

Often interpretation of mass spectra can be very complicated, but a carefully operated RGA under well-defined conditions will provide helpful information about the behavior of water at different stages of the process.

XII. CONCLUSIONS

Over more than 100 years of vacuum technology, the issue of water has been probably the main source of worries. Minutes and hours after venting and evacuation, water content in residual gas is 80% or more. Thus, many processes will be started rather in water vapor, which should therefore be considered as an unavoidable process gas and treated accordingly.

Until today, published data on some aspects of the subject remains relatively inconclusive. For example, permeation of various polymers should be evaluated more systematically over a broader temperature range. It would also be useful to quantify porosity of commercial technical ceramics, high quality alumina ceramics, machinable glass-ceramic and others.

Furthermore, a systematic study on water outgassing versus temperature and duration of bake-out would be really helpful for many applications.

Acknowledgments

The author would like to thank for support and useful discussions Hin-Yiu Anthony Chung, Gennady Fedosenko and Tais Gorkhover.

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Published by The Japan Society of Vacuum and Surface Science