

Design and Fabrication of UHV Chambers: Best Practices and Pitfalls on the Road to Ultra-high Vacuum

What is the impact of material selection, surface finish, and design execution in ultra-high vacuum? What pumping power is actually needed for the application? And why can it be difficult to achieve good ultimate pressure?



Ultra-high vacuum (UHV) by definition begins at an absolute pressure of 10^{-7} mbar, which means that surface outgassing becomes critical to ultimate pressure at this pressure range. The flow in the UHV is molecular, the mean free path length is more than 1 km. If the pressure continues to drop to 10^{-12} mbar, the free path length grows to 10,000 km. The remaining particles left in the vacuum chamber now experience interactions only with the vessel walls, but not, or almost not, with each other. In this range, the materials and the surfaces of the chamber become massively more important. So what do you need to consider when designing, manufacturing and operating chambers and components for this pressure range?



UHV chamber

Criteria for material selection

As a start, you need high gas tightness for the chamber wall, as well as low intrinsic vapor pressure and low content of foreign gases. If this cannot be avoided, the material should at least outgas rapidly so that any troublesome residual gases can be pumped out quickly. In UHV, the chamber volume is not an important factor, at most it acts as a buffer during the pressure rise after the pump is switched off or pushed off. In this case, the residual gases come from the surfaces and volume of the vessel walls or installations.

Strength and corrosion resistance are further criteria. Since the sealing surfaces must not deform at a pressure difference of 1 bar, a sufficiently strong material is required.

Corrosion resistance must also be ensured under difficult conditions such as bakeout in atmosphere or with chemically active process gases. It is therefore important to test the materials for their resistance. Good stability during temperature changes and adapted expansion behavior are needed to ensure that the chamber is and remains tight. Materials for flanges and gaskets must be matched.

Stainless steel and copper have similar coefficients of thermal expansion and are therefore a good combination. Stainless steel and aluminum are only a limited match because after temperatures of over 150 °C, the flange connections are often no longer tight when they cool down.

Properties are followed by handling and availability of the materials, because they should be able to be processed with reasonable affordable effort, and must of course be available.

Due to the low demand in UHV technology, there is no own material development and one has to work with what is already available. Austenitic stainless steel is particularly suitable for UHV applications.

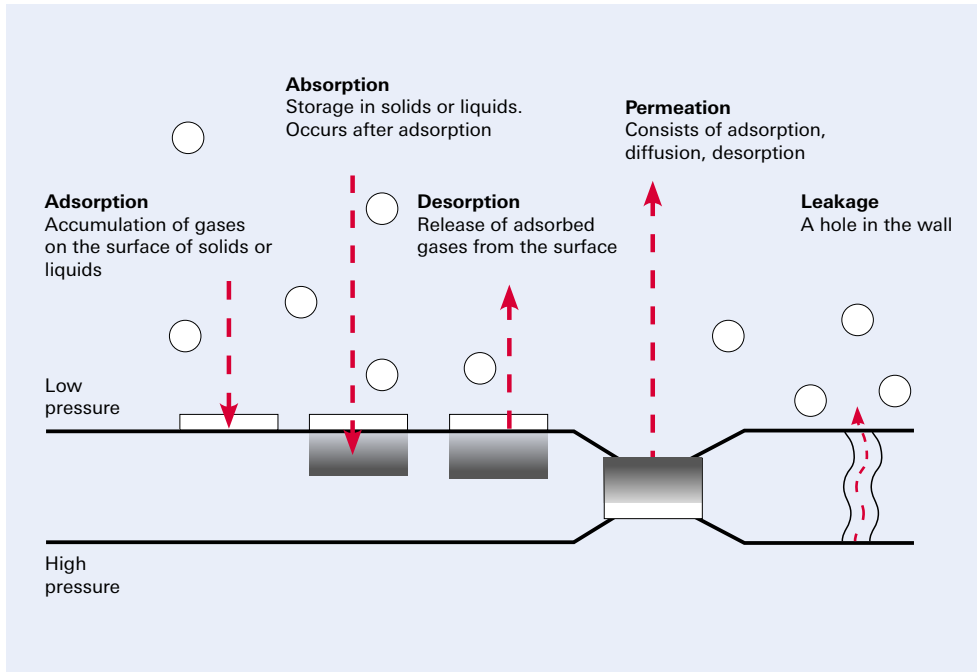


Figure 1: Interactions between the surfaces of a vacuum chamber and the surrounding gas.

Effects in ultra-high vacuum

The following terms describe the effects that happen at surfaces in UHV:

1. Adsorption: Gas deposits on the surface of solids or liquids, such as particles sticking to the chamber wall.
2. Absorption: Gas trapped in solids or liquids. Absorption often follows adsorption. Particles previously attached only to the surface are now embedded in the chamber wall.
3. Desorption: Release of adsorbed gas into the environment. The particles retained by the first two effects detach from the chamber wall again.
4. Permeation: Transport of gas through a liquid or solid.
Permeation = adsorption + diffusion + desorption.

Pressurization, adsorption and absorption are not problems because the particles are held and do not disturb the vacuum. Both effects happen on all surfaces that are in contact with atmosphere, as well with each ventilation.

Desorption is the main opponent on the way to a good ultimate pressure. This is because particles attached to the outside of the chamber diffuse through the chamber wall during permeation, increasing desorption into the vacuum chamber.

Definition of working pressure

Some basics about the requirements for the materials have now been described. Next, the desired working pressure p_{Work} must be determined in order to proceed with the construction of a vacuum chamber.

The backing pump, up to a maximum of 10^{-3} mbar pumps out the volume after which a high or ultra-high vacuum pump with a suitable pumping capacity provides the working pressure. For this purpose, it is necessary to calculate or at least estimate the gas loads resulting from desorption, permeation, leakage and process gases.

$$p_{\text{Work}} = p_{\text{End}} + + \frac{Q_{\text{Desorption}}}{S_{\text{eff}}} + \frac{Q_{\text{Permeation}}}{S_{\text{eff}}} + \frac{Q_{\text{Leakage}}}{S_{\text{eff}}} + \frac{Q_{\text{Process}}}{S_{\text{eff}}}$$

In the following it will be explained how the different gas loads Q_i can be influenced. They are opposed by the effective suction power S_{eff} of the pump, which depends on the gas, the pressure and the type of installation.

- p_{End} : The final pressure of the pump, depending on the pump selection.
- S_{eff} : The effective pumping speed, depending on gas, pressure and type of installation.
- $Q_{\text{Desorption}}$: The gas load due to desorption. It depends on the selection of materials and surface, including cleanliness. Desorption is accelerated by bakeout.
- $Q_{\text{Permeation}}$: Permeation is also dependent on material selection as well as chamber dimensions.
- Q_{Leakage} : Leakage is prevented by proper manufacturing technology.
- Q_{Process} : Process gases and intrinsic vapor pressures, depending on the application.



Multi-stage Roots pump ACP, cubic chamber and vacuum valve.



ISO-KF components

Desorption

Over time, the gas load decreases due to desorption from metal surfaces. It is assumed that the decrease is linear from a time $t > t_0$.

t_0 is assumed to be one hour.

Thus, the desorption flow can be described as follows:

$$Q_{\text{DesM}} \approx q_{\text{DesM}} \cdot A_{\text{M}} \cdot \frac{t_0}{t},$$

wobei

Q_{DesM} : Desorption flow of metal [$\text{Pa} \cdot \text{m}^3/\text{s}$],

q_{DesM} : Desorption flow density (area specific) of metal [$\text{Pa} \cdot \text{m}^3/\text{s} \cdot \text{m}^2$],

A_{M} : Metal area [m^2] from which gas molecules dissolve, and

t : Time [s] that the surface is under vacuum.

In UHV, desorption of metals has less importance than that of plastics. Gaskets, in particular, release the gases dissolved in them. After a longer period of time, desorption from plastics may therefore predominate over that from metal surfaces. The relatively small surface area of gaskets, is outweighed by the slower decrease in desorption rate over time. It is assumed that the temporal decrease occurs with the square root of time.

Thus, the gas accumulation by plastic surfaces can be described as follows:

$$Q_{\text{DesK}} \approx q_{\text{DesK}} \cdot A_{\text{K}} \cdot \sqrt{\frac{t_0}{t}},$$

mit

Q_{DesK} : Desorption flow of plastic [$\text{Pa} \cdot \text{m}^3/\text{s}$],

q_{DesK} : Desorption flow density (area specific) of plastic [$\text{Pa} \cdot \text{m}^3/\text{s} \cdot \text{m}^2$],

A_{K} : Area of plastic [m^2] from which gas molecules detach, and

t : Time [s] that the surface is under vacuum.

However, only particles that have energy $E_{\text{kin}} > E_{\text{Des}}$ desorb.

Desorption flow density: $j_{\text{Des}} \propto e^{-\left(\frac{E_{\text{Des}}}{k_{\text{B}} \cdot T}\right)}$

Increasing the temperature, meaning baking out the chamber, is the easiest way to provide this energy.

The specific properties of the surface (such as production, further processing, cleaning) have a large influence on the area-related gas emission q_{Des} .

A variation by a factor of 10 or more requires a 10-fold increase in pumping power.

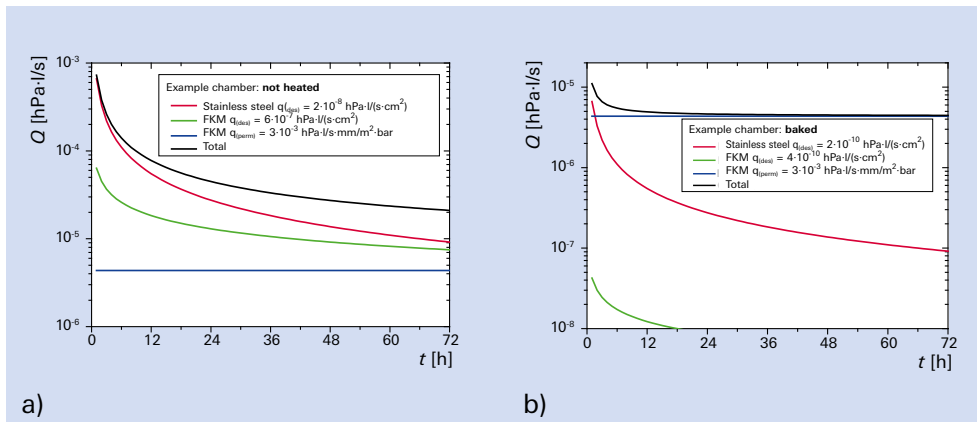


Figure 2: Desorption & permeation – example calculation, chamber (a) not heated and (b) baked out.

The calculation of a typical example chamber illustrates this:

1. Stainless steel cylindrical main body
 - a) Ø 600 mm, length 1,000 mm area approx. 25,000 cm²
 - b) Various flange outlets (metal-sealed) result in an additional area of approx. 8,000 cm² total stainless steel area: 33,000 cm².
2. Main flange with fluorocarbon rubber (FKM) O-ring
 - a) Ø 640 mm, cord thickness 7 mm, groove depth 5.25 mm FKM area to vacuum: approx. 106 cm²
 - b) Width (pressed): approx. 7.3 mm
3. Installation of a polytetrafluoroethylene (PTFE) plate 50 · 50 · 10 mm³
 - a) PTFE area: 70 cm².

You could alternatively take a metal gasket for the main flange. However, since this can only be used once and must be replaced after each opening, each opening would cause costs of approx. 1,000 €. In addition, there would be the costs for the flange with sealing edge when building the chamber. These additional costs are only worthwhile in special cases and are therefore not considered in our example calculation.

A gas load due to fittings is represented by the PTFE plate. This is a rather small area with a high desorption rate.

The graph in Figure 2 illustrates the calculation of desorption: A clean recipient surface is assumed. The desorption of the surface depends on the cleanliness and may well be five times larger as $1 \cdot 10^{-7}$ hPa · l/s. Figure 2 shows that an effective pumping speed of 1,000 l/s results in a pressure p_{72h} of $1.8 \cdot 10^{-8}$ hPa after three days.

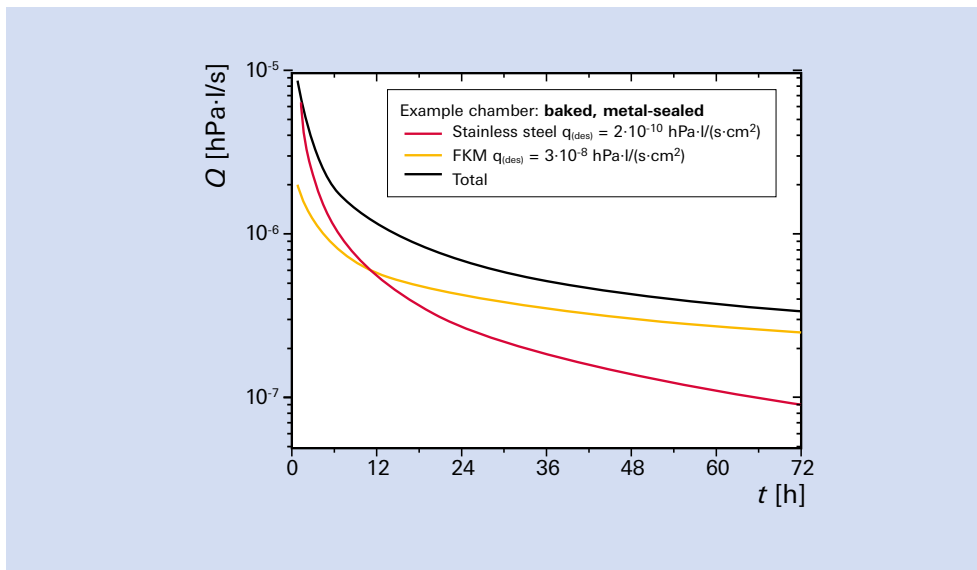


Figure 3: Desorption with fittings

Permeation

Permeation is the gas permeability of the material or its permeation conductivity.

$$Q_{\text{Perm}} \approx q_{\text{Perm}} \cdot \frac{A}{d} \cdot \Delta p$$

where

- Q_{Perm} Permeation flow [$\text{Pa} \cdot \text{m}^3/\text{s}$],
- q_{Perm} permeation constant (material specific) [m^2/s],
- A Area [m^2] which is under vacuum,
- d wall thickness, path length [m] to be covered by the molecules, and
- Δp pressure difference.

Thus, permeation is independent of time and constantly provides an increase in the final pressure. Its dependence on the pressure difference can be used to reduce it. So-called differential pumping, meaning a double seal with interstage pumping, reduces Δp by three to four orders of magnitude from 1 bar to 1 to 10 mbar. This also results in a reduction of the permeation flow by a factor of 100 or 1000. However, permeation also depends on temperature and on certain combinations of medium and material. Some materials are more permeable to certain media than to others, such as fused silica to helium. Elastomers, for example, have the comparatively highest permeation, which is also strongly temperature-dependent. Their permeation conductivity increases a hundredfold for a temperature increase of 100 K. However, temperatures of more than 150 °C are not possible with elastomers, as the O-rings then become oval.

As a matter of principle, do not confuse permeation at very low pressures ($<10^{-8}$ hPa) with true leakage.

Permeation is now added to our example calculation of desorption from Figure 2:

Permeation as a time-independent constant appears in Figure 3 as a line parallel to the time axis.

With the same pump as before, a pressure p_{72h} of $2 \cdot 10^{-8}$ hPa is reached after three days.

The result of the same calculation for a heated chamber is shown in Figure 4: The desorption of the surface is a hundred times smaller than before thanks to the bakeout, and permeation is the dominant effect.

The final pressure after three days is $p_{72h} = 3.5 \cdot 10^{-9}$ hPa.

Leakage

Possible leakages are found in the course of local or integral leak detection.

In both cases, the vacuum chamber is under vacuum (vacuum method).

Leak rate of local leak detection (vacuum method): 10^{-10} Pa · m³/s \approx 0.1 μ m capillary diameter.

Leak rate of integral leak detection (vacuum method): 10^{-9} Pa · m³/s \approx 0.3 μ m capillary diameter.

At the start of the leak test, the remaining gas loads should be in the range of 10^{-7} Pa · m³/s. Occasionally, values of 10^{-10} or 10^{-11} Pa · m³/s are required.

However, these demands are academic, because they do not provide any new findings, despite the significantly increased time required. Experience has shown: If no leak is found at 10^{-7} Pa · m³/s, none will be found at 10^{-10} or 10^{-11} Pa · m³/s.

When leak testing a system, it is important to note that no indication is not the same as no leak, because the pumping speed for the test gas helium and the vacuum in the chamber affect the signal. If there is a leak, gas will flow into the chamber, disperse and, with relatively low probability, hit the connection to the leak detector. In addition, the signal can be obscured if the process is too fast.

The time constant $\tau = \frac{V}{S_{\text{eff}}}$ must be taken into account.

Example 1:

$S_{\text{eff(He)}}$ of the leak detector: 1 l/s

Chamber volume: 315 l

→ $\tau = 315$ s

Leak detector indication can only be expected after at least five minutes.

Example 2:

Improved pumping speed with a turbo pump $S_{\text{eff(He)}}$ of the leak detector:

1,300 l/s

Chamber volume: 315 l

→ $\tau = 0,24$ s

A reaction of the leak detector can be expected within one second.

It is generally recommended to familiarize yourself with the test equipment.

This can easily be done by means of a test leak.

Fittings and intrinsic vapor pressure

Fittings represent additional desorbing surfaces. This effect can be mitigated by using materials with low outgassing rates, working cleanly and outgassing the parts beforehand.

The intrinsic vapor pressure plays an important role with oils and greases. Since it limits the final vacuum pressure, it should be sufficiently low. This effect is also present with metals. At temperatures above 100 °C, for example, the partial pressure of zinc in brass increases massively, so that use in this area is not recommended.

During construction, poorly ventable volumes (virtual leaks) should already be eliminated. A favorable selection and positioning of the pump can improve the situation if high gas loads cannot be avoided, for example because gases are introduced, materials with high outgassing rates have to be used or surfaces need to be sputtered.

Example calculation for desorption with fittings

To better represent the graphs in Figure 3, the example chamber is now completely metal-sealed. The graph for the elastomer seal of the main flange is omitted, but the graph for the desorption of the PTFE plate appears in yellow.

A final pressure p_{72h} of $3.5 \cdot 10^{-10}$ hPa is reached after three days.

The actual design of a UHV chamber

There is no mandatory set of rules for vacuum chambers, quite unlike pressure vessels such as gas cylinders or tank cars. However, the responsibility to manufacture a safe product remains. The design is based on experience according to "good engineering practice". Accordingly, the design responsibility rests with the person who designed the chamber. A contract manufacturer is seen as an extended workbench.

For the design of vacuum chambers, reference can be made to related rules and standards, such as the AD 2000 for pressure vessels. The calculation of pressure vessels (data sheet B0), cylindrical shells (data sheet B6) or disc bottoms (data sheet B4) is often used.

Calculations can also be performed using the finite element method (FEM). In this case, however, it must be taken into account that the pressure ratios are reversed, especially in the case of symmetrical components and pipes. In vacuum chambers, the pressure acts from the outside, which is why elastic buckling and plastic deformations occur.

In addition, all operating conditions of the vacuum chamber must be taken into account. For example, increased temperatures during bakeout can cause a decrease in the strength of the material. There are also additional loads, such as feet on the chamber, that can place lateral loads on the main tube.

Machinery directive and risk assessment

No vacuum chamber gets a CE mark, why is that? A component must be within the scope of an EU directive that provides for a CE mark in order to receive a CE mark. Since a vacuum chamber is not a machine, this does not apply to a vacuum chamber. So there is no declaration of conformity, no CE marking and no declaration of incorporation.

However, if components are mounted to the chamber that are machines, this changes. Product liability then lies with the person placing the product on the market, i.e. possibly the operator. The overall system is subject to a risk assessment in accordance with EN ISO 12100, which follows the three-stage doctrine for safe design. The requirements are (in descending priority): First, hazards must be eliminated by design. If there are no hazardous points, there is no risk. If hazards cannot be avoided, technical protective measures must be taken, such as enclosing the hazardous area. If this cannot be implemented either, the user must be made aware of the hazard in the operating instructions.

Welding

Welding is the core process in vacuum vessel fabrication. Inclusions of volume are avoided by performing the welds from the inside. If necessary, there are support welds that are not continuous from the outside. If the welds were continuous on the inside and outside, virtual leaks could occur. Under these conditions, a leak in the inner weld could not be found, and it would be very difficult to pump out the volume trapped between the welds. For example, if it is not possible to weld from the inside due to space limitations, only the outside is welded. These welds are made completely through-welded so that no narrow gaps remain on the vacuum side.



Due to strong local heating, welding distortion occurs. Experienced welders therefore determine the sequence of the seams to be welded in advance so that distortion is kept to a minimum. Functional surfaces such as sealing surfaces generally require additional rework.

Qualifications can be used to determine whether a company has the necessary experience in welding. Welding and procedure qualification tests provide an insight into the capabilities of the company performing the work.

Tungsten inert gas (TIG) welding, laser welding and electron beam welding, are the most common welding processes for vacuum welds.

Welding causes a microstructure difference in the weld and the mechanical, magnetic as well as chemical properties change in this area. Particular attention must be paid to the magnetic properties. Rapid cooling causes martensitic transformation, which increases the relative magnetic permeability μ_r . This particularly affects applications where magnetic fields are used, such as in particle accelerators. The interaction of the chamber material and the external magnetic field must be so small that it is hardly perceptible.



Welding of a vacuum component

Cleaning for UHV

Finally, the vacuum chamber must be cleaned to achieve cleanliness suitable for UHV. However, the groundwork for this is laid during chamber fabrication by using clean materials and avoiding contamination with, for example, mild steel. Scale layers formed during forging are completely removed. Lubricants used for the machines are water-soluble and are not allowed to dry on. As part of the finishing process, tarnish from welding is removed by brushing, grinding (with silicon carbide SiC), glass bead steeling, pickling or e-polishing. Only clean tools are used in this process. Finally, the contaminants are completely removed in a cleaning bath (e.g. ultrasonic) and any last residues of cleaning agents are removed by thorough rinsing with fully demineralized or deionized water.

The highest degree of cleanliness is achieved by vacuum annealing, also called low hydrogen annealing or demagnetization annealing. In this process, austenitic stainless steel is annealed at 950 – 1,050 °C in a vacuum of $p < 10^{-4}$ hPa. During cooling in a nitrogen atmosphere, the temperature range of 900 – 550 °C must be passed through within half an hour.

However, sufficiently high strength of the cutting edges of the flanges is a prerequisite for annealing, so they should be made of 1.4429 ESU, a high-purity steel obtained by the electro-slag remelting process, instead of 1.4307.

Your Success. Our Passion.

We give our best every day for you –
worldwide!

Are you looking for an
optimal vacuum solution?
Please contact us:

Pfeiffer Vacuum GmbH
Germany
T +49 6441 802-0

**Pfeiffer Vacuum Components
& Solutions GmbH**
Germany
T +49 551 99963-0



Errors excepted. All data subject to change without prior notice. PI 0537 PEN (March 2022/0)



www.pfeiffer-vacuum.com

PFEIFFER  **VACUUM**