Checking coating systems for leak tightness

After installing a new system, or maintaining an existing one, leaks occur frequently. This is often due to joining errors, or forgotten defective seals. Correct handling of helium leak detectors is crucial for finding these leaks and controlling equipment for tightness. The more is known about the physical phenomena underlying the leak test as well as the optimization possibilities during testing, the easier it is to use helium leak detectors in practice. This also leads to more reliable measurement results.

The following overview provides practical information on the correct handling of helium leak detectors and the successful implementation of leak tests on vacuum systems.

What needs be considered when connecting the leak detector to a coating system?

Leaks that occur after start-up or maintenance of vacuum systems are often very large. However, commercially available helium leak detectors can no longer be used from a certain leak size on. Their maximum working pressure is usually between about 6 and 25 mbar. If there are large leaks, this pressure may not be reached during evacuation. Figure 1 shows a Si3N4 (silicon nitride) coating system. After maintenance, only a pressure of 80 mbar is reached during evacuation. One way to reduce the inlet pressure of the leak detector used is to use a needle metering valve.

However, this is accompanied by an extension of the response time and inevitably requires the use of an auxiliary pump. Instead of throttling a vacuum leak detector, the Pfeiffer Vacuum ASM 340 leak detector can be used to create a qualitative massive leak detection mode, which helps to localize the present leak.

Process pump to support the leak detector

Optimally, the leak detector should be connected to the fore-vacuum line of a vacuum system as shown in Figure 1 and 2. To protect the leak detector, which is designed for use in clean environments, from severe thermal stress due to the compression heat generated during pump down, an additional process pump may also be used. It is insensitive to the thermal stress and also pumps off all outgassings, vapors and any stirred-up particles.

The use of an additional process pump can increase the availability of the leak detector and significantly extend its maintenance intervals. This results in considerably reduced operating costs.

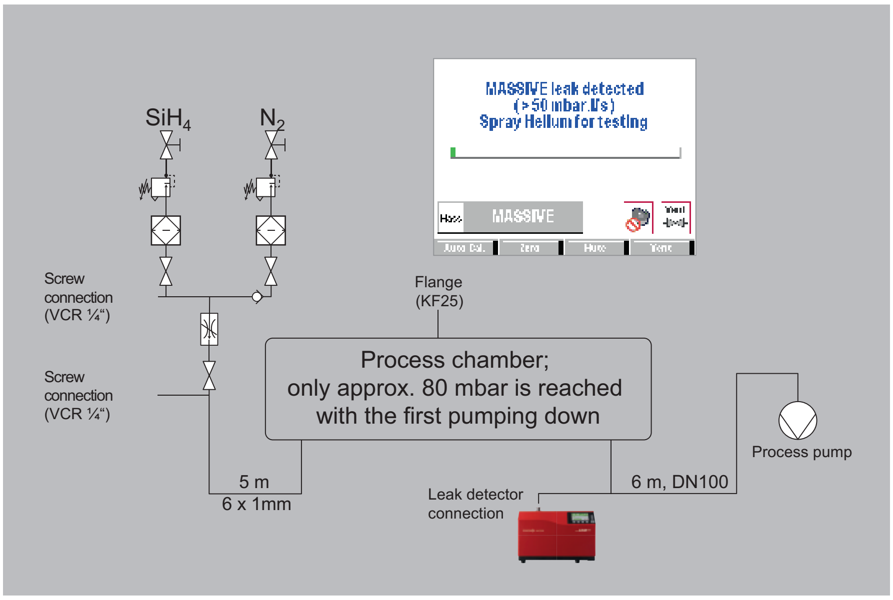

Figure 1: Integrating the helium leak detector in a coating system

Once the pressure has reached a correspondingly low level during the process of pumping down, the leak detector is able to keep up the vacuum. The more powerful the backing pump in the leak detector is dimensioned, the more outgassings the leak detector can still evacuate and the larger the inner surfaces of the evacuated container can be.

If a stable operating pressure in the working pressure range of the leak detector is reached, the process pump is obsolete and can be switched off, as now 100 % of the tracer gas will reach the leak detector. In order to shorten the response time of the leak detector again significantly, a turbopump can be used between the chamber and the leak detector. It acts like a booster and shortens the response time of the leak detector based on its additional pumping speed.

A test leak flanged onto the process chamber makes the development of the leakage rate signal traceable over time. In addition, it is also possible to determine which parts of the helium escaping from the test leak are evacuated by the process pump and which are detected by the leak detector – the so-called partial flow factor can be measured directly.

Here, different flow conditions affect the gas dynamics due to different pressures, which in turn affects the partial flow ratio. For this reason, measurements should be made near the test pressure. Simply estimating the partial flow factor is not sufficient, since the process pump and leak detector have different pumping speeds and compression ratios in different pressure ranges.

Time constant

The time constant of a vacuum system is represented as follows:

τ = Time constant until 63% of the maximum signal intensity [s] is reached

S_{eff} = Effective pumping speed for the tracer gas[l/s]

V = Volume of the specimen [l]

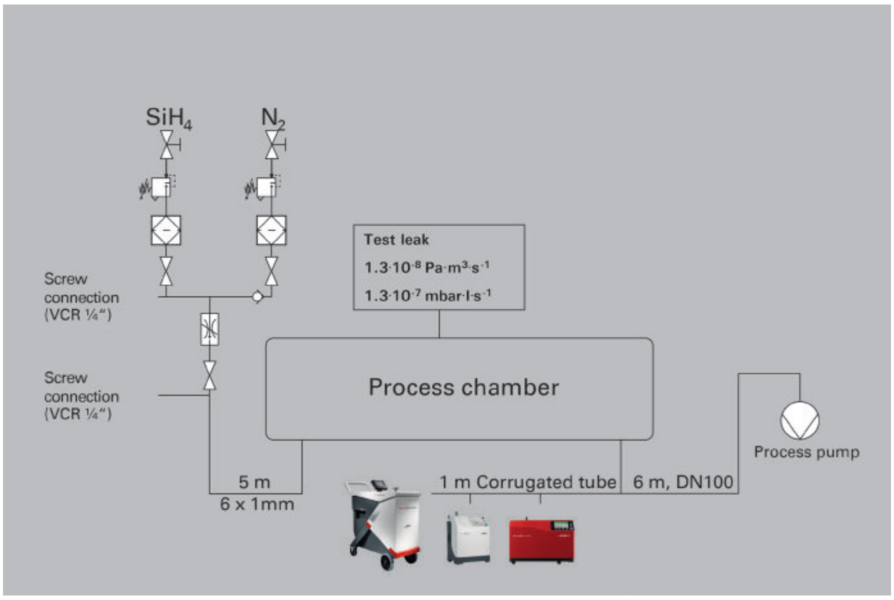

Figure 2: Integrating different Pfeiffer Vacuum leak detectors (ASM 390, ASM 310, ASM 340) in the system

On the one hand, the time constant of a vacuum system depends on the volume of the system and, on the other hand, on the helium pumping speed of the pumping system.

Figure 3 shows the correlation for the time constant, which applies to the time it takes to reach 63 % of the maximum signal intensity. In addition, definitions are available for time scales at 3τ at a signal level of 95 % or 5τ at a signal intensity of 99 %.

Gas supply systems

If the test leak is placed in the gas supply line, the response time is extended by the flow resistance of the narrow gas line and other installation elements. This can be measured with the help of the leak detector.

In our sample system shown in Figure 4, the signal from the leak detector was fluctuating and inconsistent. It did not stabilize even after a long wait. This was due to the control loops of the mass flow controller, which regulated the system control independently of the leak detection.

As can be seen in Figure 5, the test leak was moved again as a result. This made it possible to separate the influence of the mass flow controller and the gas supply line between the MFC and the system.

Conductivities and flow resistances

The response time of the leak detector could be shortened and the signal stabilized after a component, which limited high conductivities, had been removed. The flow resistance prevailing in the gas line resulted in the fact that it took four minutes to reproduce the leakage rate previously measured without conductivity limitations. In a quantitative measurement, exactly the same signal intensities were measured in the case of the test leak that had been moved as with the test leak directly connected to the process chamber (Figure 5).

Important aspects of leak testing mass flow controllers

In order to be able to test the mass flow controller separately, the leak detector was

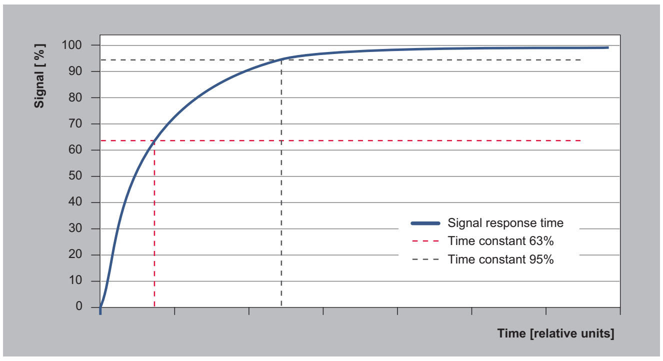

Figure 3: Signal curve on the leak detector with permanent exposure of the leak to helium (idealized)

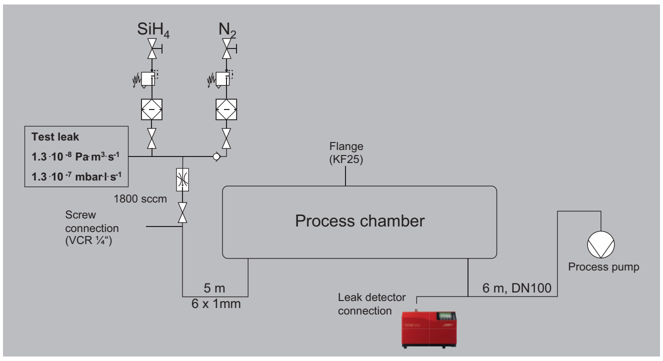

Figure 4: Integrating the leak detector for testing gas supply system

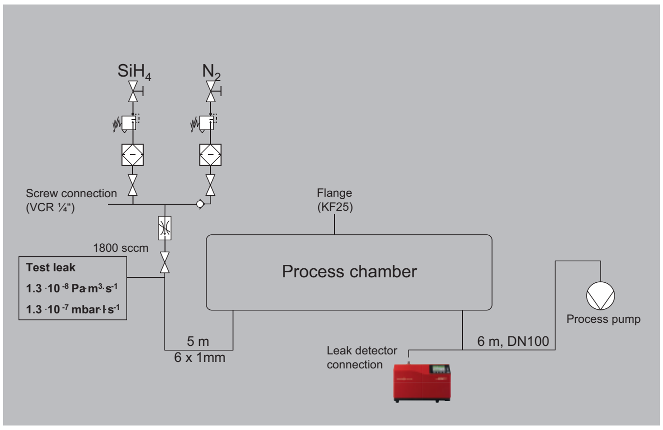

Figure 5: Setup of a gas supply system without a component with high conductivity limiting

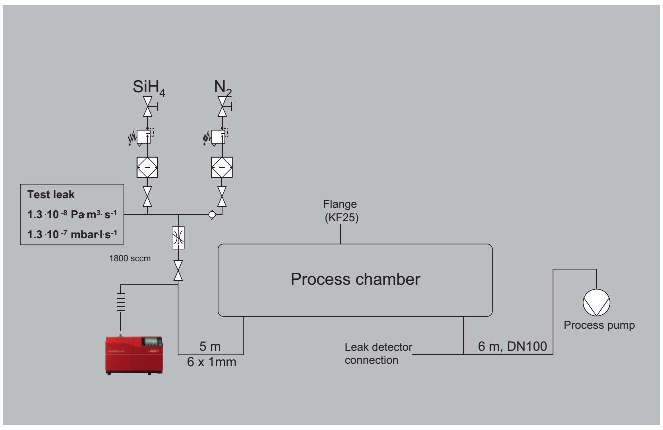

Figure 6: Integrating the leak detector in the system for a leak test of mass flow controllers

connected to the gas supply line directly behind the MFC. In this case, the response times were under one second. Here, the leak detector benefited from the change in the partial flow ratio between the leak detector and the process pump: It was able to measure 100% of the gas flow discharged from the test leak. The reason for this was the high flow resistance of the gas supply line, whose internal diameter was only 4mm. In comparison, the line to the leak detector was a DN 25 line. This process is shown in Figure 6.

Flow resistances of ultra-pure gas supplies

In vacuum technology, flow resistances are also known as conductance. Figures 7 and 8 show conductivity simulations of gas supply lines as a function of the tubing length with dimensions of 6x1mm, which are typical for these lines. The outer diameter is 6mm and the wall thickness is 1mm, which results in an inner diameter of 4mm.

At high pressures, there is a laminar-viscous flow. There is a linear correlation between the flow resistance and the mean pressure of the line. At pressure ratios below about 0.1 mbar, molecular flow is present. In this range, the flow resistance no longer depends on the prevailing pressure conditions, but only on the geometry of the line.

Transitional flow, also known as Knudsen flow, is present in the pressure range between approximately 5 and 0.1 mbar. Gas specific effects may also occur in this range. In the molecular flow range, the conductivity for helium with a 20m long line is only 0.001 l/s. This results in a significant delay in the response time.

In addition, the signal can be stretched in time. If the leak is not exposed to helium, for at least the same amount of time as the system is in the tested range, the signal, even at its maximum indicated intensity, will give a much smaller value than the actual size of the leak.

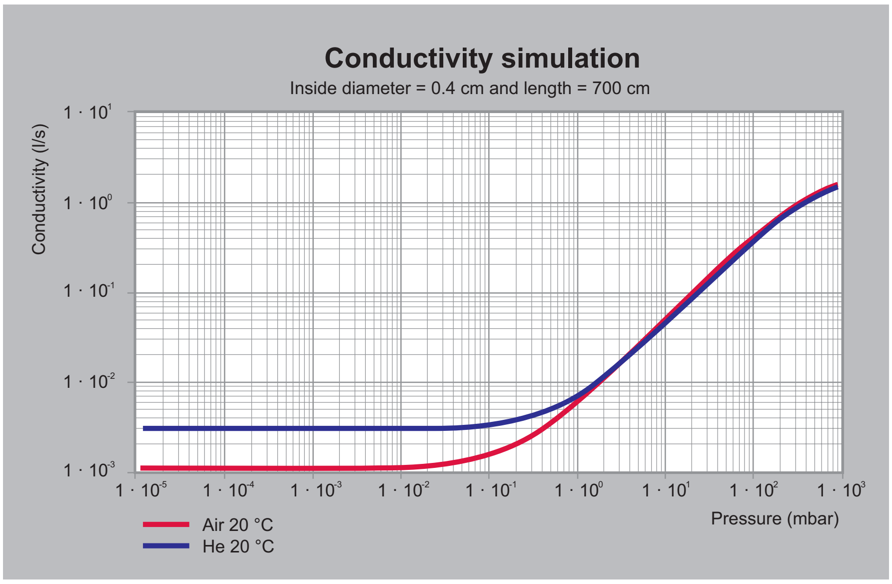

Figure 7: Conductivity vs. pressure at a line diameter of 0.4cm and a line length of 700cm

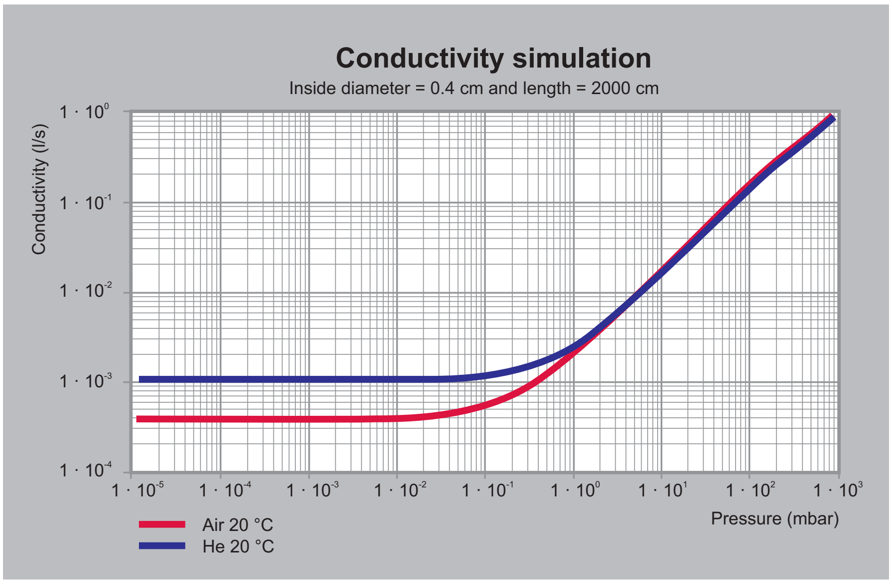

Figure 8: Conductivity vs. pressure at a line diameter of 0.4cm. Line length 2,000cm

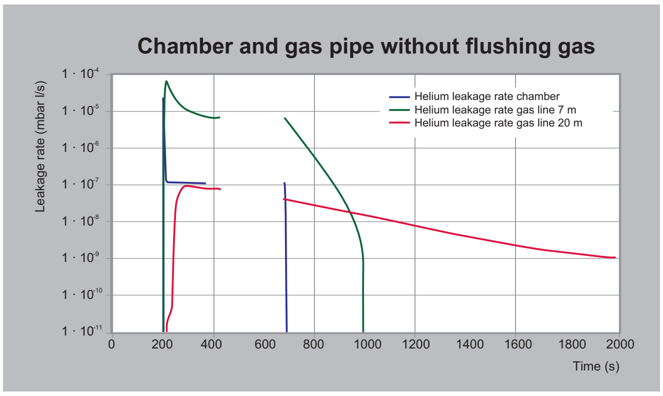

Figure 9: 1.5l chamber and gas line without carrier gas

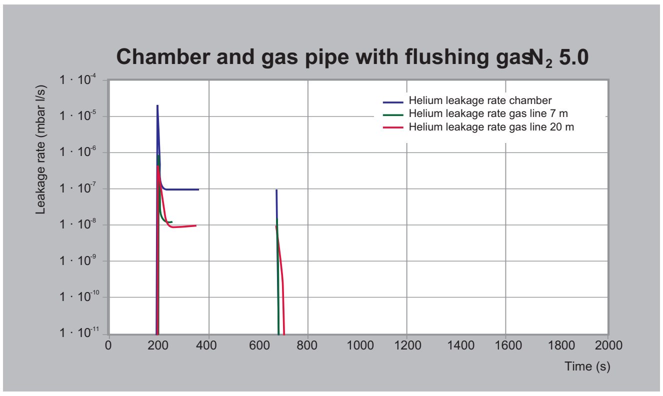

Figure 10: Chamber and gas pipe with carrier gas N_2, purity 5.0

Leak detection with and without tracer gas on components that are limited in conductivity

Figures 9 and 10 show the time behavior during a leak test of gas supply lines 6 x 1mm with a length of 7m and 20m. In Figure 9, the blue curve represents the example of an evacuated chamber with a volume of 1.5l, to which, after reaching a specified background signal, a test leak with a reservoir with a leakage rate Q=$1{\cdot}10^{-7}$ mbar·l/s, has been flanged and opened.

The sudden release of helium, which has accumulated between the permeation element of the test leak and the shut-off valve, causes a short overshoot. However, the display of the nominal test leak value then stabilizes quickly. After closing the test leak, the signal also subsides quickly.

The green curve shows the response behavior of a modified body. A 7m long gas supply line (6 x 1mm) is now looped in between the container described above and the leak detector. The flow resistance of the line leads to a significant extension of the settling behavior and decay time of a test leak with a leak rate Q of 5 - 10^{-6} mbar-l/s (5 - 10^{-7} Pa m^3/s).

The red curve shows the response time with a gas supply line of 20m (6 x 1mm). Here, with the same test setup, the helium is not completely evacuated even after more than half an hour of pumping down time. Therefore, the nominal value of the test leak is not achieved.

The example in Figure 10 shows the behavior in an identical experimental setup, if the lines are not evacuated, but the almost helium free gas nitrogen 5.0 flows. The most sensitive measuring range should be achieved on the leak detector with simultaneous maximum test pressure.

Here, minimum detection limits should be combined with the best possible flow dynamics, i.e. the shortest possible response time. The pressure threshold for reaching the most sensitive measuring range is device specific and is at 0,5 mbar for the ASM 340 leak detector used. Accordingly, the gas flow rate is selected: the in-let pressure is about 12 mbar at the inlet of the line. If a carrier gas is used for leak detection, the response and decay times are much shorter. However, this is accompanied by a loss of intensity of about a decade.

To accurately determine the response time and partial flow factor, such measurements must be calibrated using test leaks. Test leaks without a reservoir or test leaks specially designed for installation in gas supply lines are best suited for this purpose.

The use of carrier gas methods has now also found its way into international standardization and is described in DIN EN ISO 20485: Non-destructive testing - leak testing - test gas methods: ISO 20485:2017; German version EN ISO 20485:2018.

Performing sniffing leak detection on gas supply lines

One of the most important requirements for leak testing is measurement under real conditions: The pressure to which the test object is subjected during the test must correspond to that in actual use. Here, the gas lines are under overpressure. Vacuum testing is the only test procedure that achieves the mostly required sensitivity for ultra-pure media supplies in the range of $1{\cdot}10^{-9}$ mbar·l/s to $1{\cdot}10^{-10}$ mbar·l/s. The sniffing test that corresponds to the correct pressure direction is shown in Figure 11.

Pumping down and pressure change flushing influence the signal recovery behavior

The signal recovery behavior in a process chamber with 700l volume is shown in Figure 12. Here, the decay of the helium signal is displayed within four minutes of detecting a leak and then pumping it down. In the four-minute period, only 50% of the signal will be lost. However, a signal degradation of several decades is achieved when three pressure change flushes are carried out, i.e. if after a short evacuation to a pressure in the mbar range, it is ventilated to a pressure of several hundred mbar and then pumped again.

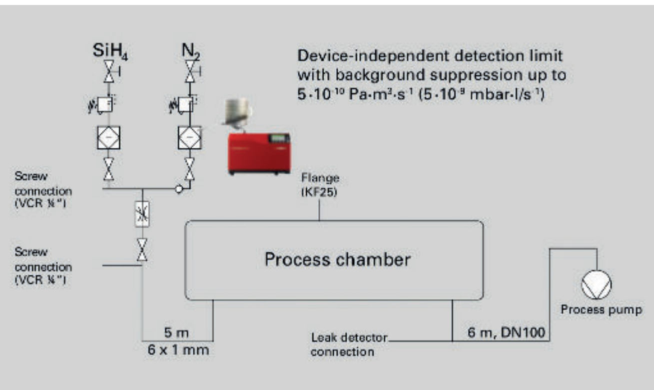

Figure 11: Sniffing the gas supply lines

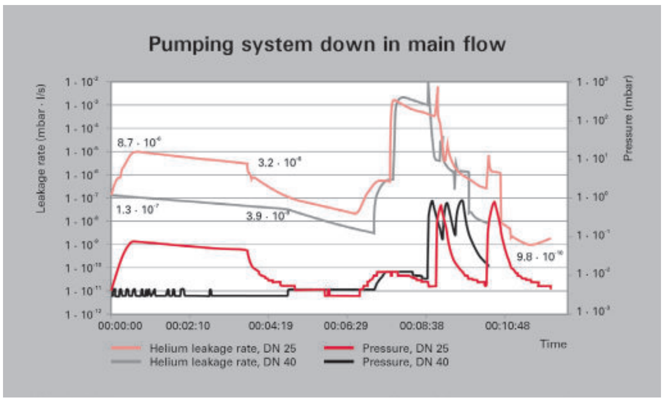

Figure 12: Signal recovery behavior of the leak detector in main flow operation

To prevent the system from being accidentally ventilated by the leak detector, the automatic ventilation function of the leak detector must be switched off by the end of a test cycle. It is also recommended to use gases with very low helium content for ventilation. This ensures an effective helium decomposition during the test process and a low signal background. If the gas also has a low residual moisture content, these purging processes also have a positive effect on covering the chamber walls with moisture, thus shortening the pump-down time during repeated evacuation.

What to do in the case of an intense signal?

If the helium leak detector shows a strong signal, it should be noted that regeneration takes place more quickly at a higher pumping speed. In addition, it is possible that the decay behavior of the leak detector may take a long time after detecting a high leak rate.

High leak rates are referred to as values of > $1{\cdot}10^{-4}$ mbar l/s ($1{\cdot}10^{-5}$ Pa m^3/s). The leak detector can be protected against the entry of large quantities of helium by setting a limit value in form of a maximum value for the detection of leak rates. If a very intensive signal is detected, the leak detector aborts the test.

This can considerably reduce the time required for pumping out during the next test. It should be noted here that the leak detector vent valve must be set 'Off', otherwise the chamber will be ventilated through the leak detector. The volume of the test object connected to the leak detector has a significant effect on the recovery behavior of the signal.

This recovery time of the leak detector as well as that of the entire system can be shortened by carrying out so called pressure change flushing with a helium-free gas. The maximum inlet pressure permitted for the leak detector must be taken into account. Alternatively, an overpressure safety valve may be used. In addition, the vent valve must be set to 'Off'.

Figure 13: Pfeiffer Vacuum Classic coating system

Leak detector	He-Pumping speed [l/s]	Line	He-Leckage rate [mbar.l/s]	Partial flow ratio
ASM 310	1,0	DN 25	$4{,}4 \cdot 10^{-09}$	1:30
ASM 340	2,5	DN 25	$1{,}2 \cdot 10^{-08}$	1:11
ASM 390	10,0	DN 40	Not measured	
ASM 392	25,0	DN 40	Not measured	

Table 1: Dependence of the measured leakage rate on partial flow ratio, helium pumping speed and line cross-section

Dependence of the leakage rates on the type of leak detector used

Of course, the size of a leak and thus the leakage rate does not depend on the leak detector used. However, the pumping speed of the used leak detector in relation to the pumping speed of a pump operated in parallel influences the displayed value of the leakage rate via the partial flow ratio. The overview in Table 1 shows the influence of partial flow ratio, helium pumping speed and line cross-section on the leak rate measured. The following configuration of the system is assumed:

- Chamber volume: 700l
- Process pump: Dry pump with a pumping speed of 1800 m^3/h

ASM 310, ASM 340, ASM 390

- Size of the test leak: $1.3 \cdot 10^{-8}$ Pa m³/s ($1.3 \cdot 10^{-7}$ mbar·l/s)
- Measurement is carried out in partial flow
- Length of the corrugated tube: 1m

If objects with a large volume are to be checked for leaks, or there are large leaks or high desorption rates are to be expected when testing large surfaces, the combined use of the leak detector with an auxiliary pump is recommended. When used in partial flow operation, only a small portion of the tracer gas that has entered through the leak reaches the leak detector.

This means:

$$y = \frac{S_L}{S_L + S_T} = \frac{Q_L}{Q_L + Q_T}$$

y = Partial flow ratio

(Proportion of the gas reaching the leak detector) SL = He pumping speed of leak detector [l/s]

S_T = He pumping speed of auxiliary pump [l/s]

Q_L = Gas flow to the leak detector [mbar · l/s]

Q_T = Gas flow to the auxiliary pump [mbar · l/s]

From the observations made, there are consequences for practical application: The response times of the leak detector are very short with values under one second. The partial flow ratio and the reproducibility improve as the helium pumping speed increases.

These two factors can be further improved by expanding the cross-section of the corrugated tube as a connection between the leak detector and the test object from 25mm to 40mm. This is particularly important for high performance leak detectors, whose high helium suction capacity cannot otherwise be used effectively. As a rule of thumb for the connection between the system and the leak detector, the cross-section of the inlet flange of the leak detector should not be smaller than the cross-section of the leak detector.

Avoiding mistakes when handling the leak detector

Operating errors or incorrect handling of the leak detector may result in falsified measurement results or incorrect measurements. Therefore, it is important to clean the chamber from outgassing as much as possible before the leak test begins. Pressure change flushes with inert, dry gases and, under certain circumstances, also baking out the system are suitable for this purpose.

It is also important that leaks can be clearly identified. For this purpose, an outlet pressure adapted to the particular application is necessary, in which the residual helium content and so also the background leakage rate are significantly below the leakage rates to be detected.

In addition, for trouble free and reliable measurements, auxiliary pumps should be used and all measurements should be recorded by means of internal or external storage media. Plus, it is recommended not to move the leak detector directly after a measurement and to observe the tracking indicator of the device. In coating systems, leak detection should be carried out in partial flow.

Leak testing on a process chamber should be carried out from the test connection on the gas panel. At the auxiliary pump near the final vacuum, leakages can be measured using the backflowing tracer gas, i.e. even if the leak is upstream of the leak detector connection. If a gas panel test is planned, measurements must be carried out at partial flow directly on the gas panel.

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