Sealability of PTFE gaskets in cryogenic and gaseous hydrogen environments

This article examines the technical tightness of modified PTFE gaskets in cryogenic and high-temperature hydrogen environments, presenting test results that demonstrate their exceptional performance and suitability for various applications in the valve industry.

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Hydrogen plays a crucial role in both the energy transition and future technology. As hydrogen applications become more prevalent, there is an increasing demand for reliable sealing solutions that can withstand various hydrogen environments.

In Germany, hydrogen falls under the scope of the Technical Instructions on Air Quality Control (TA-Luft), which sets limit values for substances that are harmful to humans or the environment. In accordance with these regulations, sealing materials for hydrogen applications must meet stringent requirements. These materials must provide mathematical proof of technical tightness as outlined in VDI 2290, using calculations based on EN 1591-1 for round flange connections. This necessitates determining the EN 13555 characteristics of gasket materials, focusing on factors such as creep at elevated temperatures and maximum tolerated surface pressure.

Modified, calendared third-generation PTFE gasket materials possess the necessary EN 13555 characteristics and are already widely used with media subject to TA-Luft regulations. These materials are suitable for applications involving emissions or substances that are harmful to humans or the environment, adhering to the specific limit values established in the TA-Luft.

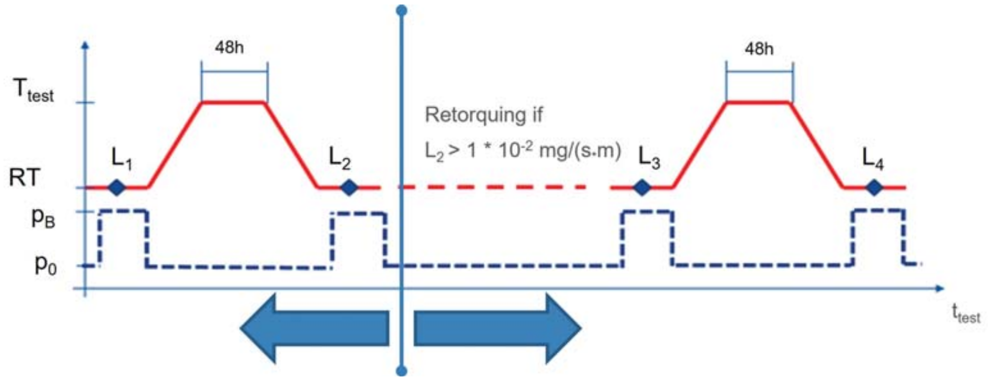
Research questions

This study aims to investigate the performance of modified, calendered third-generation PTFE gaskets under various hydrogen conditions. The following questions were addressed:

1. How do these gaskets change in terms of hardness, tear strength, elongation at break and density when exposed to cryogenic hydrogen?
2. How do these gaskets change in terms of hardness, tear strength, elongation at break and density when exposed to gaseous hydrogen at 150°C?
3. Do modified, calendered third-generation PTFE gaskets achieve the same level of technical tightness in a hydrogen environment as they do in tests conducted with helium?
4. How do these gaskets perform in terms of sealability:
    a) When manufactured from a single piece?
    b) When manufactured in segments or welded?

Additionally, the study examined the overall sealability of these gaskets under both cryogenic (-196°C) and high-temperature (up to 200°C) hydrogen conditions.

Test procedure



T_{test}	Test temperature [°C]
t_{test}	Test timeline
L_1	Determination of leakage at room temperature after installation (RT)
L_2	Determination of leakage after temperature ageing
L_3	Determination of the leakage after retightening the connection
L_4	Determination of the leakage with retightening and after the 2nd temperature cycle
p_0	Ambient pressure
p_B	Test pressure / operating pressure

Figure 1: Component test according to VDI2290 for technical tightness

About the author

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Technical tightness of PTFE gaskets with microscopic glass bubble and barium modification

Third-generation PTFE materials have demonstrated suitability for use in both cryogenic and high-temperature hydrogen environments without sustaining damage, as detailed in a previously published Valve World article (May 2024).

To investigate the achievable tightness classes, GAIST, a spin-off of Münster University of Applied Sciences, conducted comprehensive tests. These tests measured the achievable technical tightness in two key areas:

1. The cryogenic range, after ageing at -196°C
2. The gaseous range, with ageing at up to 200°C

The tests were performed using a setup for component testing according to VDI 2290 (pending approval), as shown in Figure 1. This rigorous testing methodology aimed to provide a thorough assessment of the gaskets' performance under extreme conditions.

Cryogenic sampling

Both images clearly show the effects of cryogenic storage before and after testing.

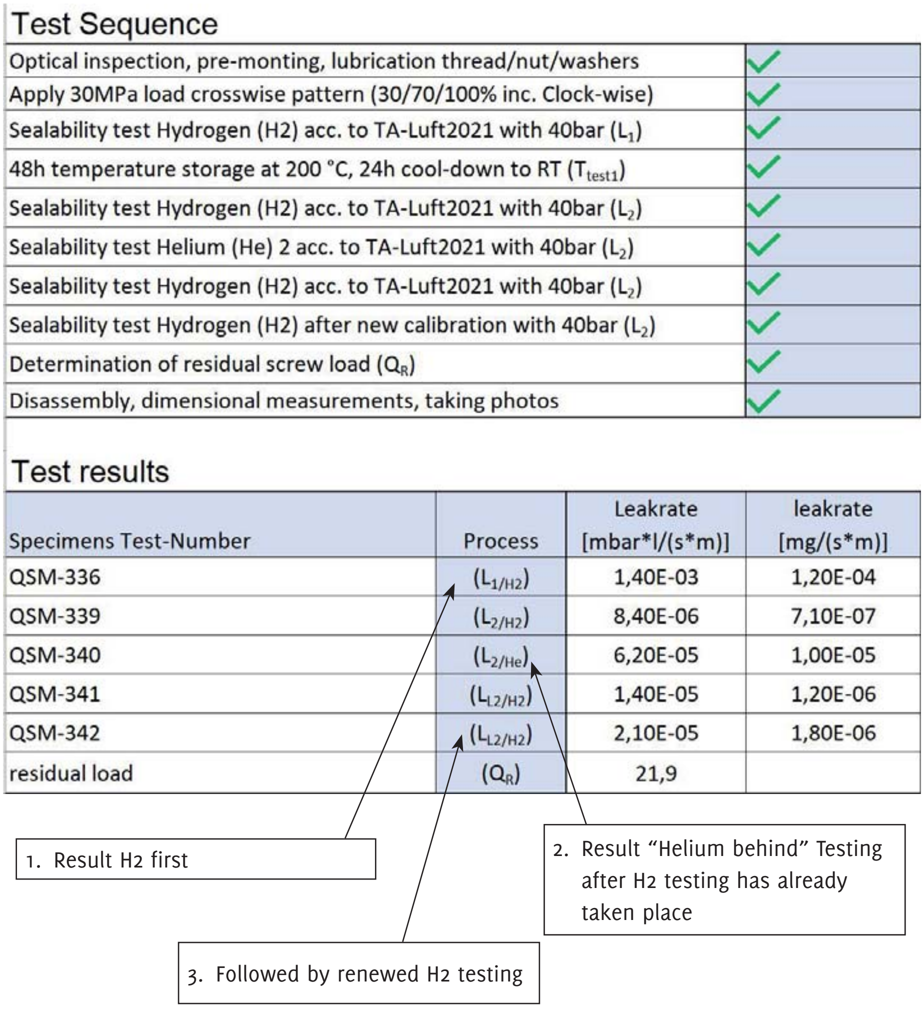
Figure 2: Deep-freeze test chamber with vacuum connection

Figure 3: Deep-freeze test flange

Results and technical tightness

The testing procedure involved two stages to compare the performance of the gaskets with different test gases. Initially, leakage was determined using helium in the first mounting of the flanges. In a subsequent test, the gas was switched to hydrogen, and leakage was measured at 1 bar.

To address potential concerns about helium molecules potentially affecting the leakage paths for hydrogen molecules, the procedure was also conducted in reverse order, starting with hydrogen



Test Sequence

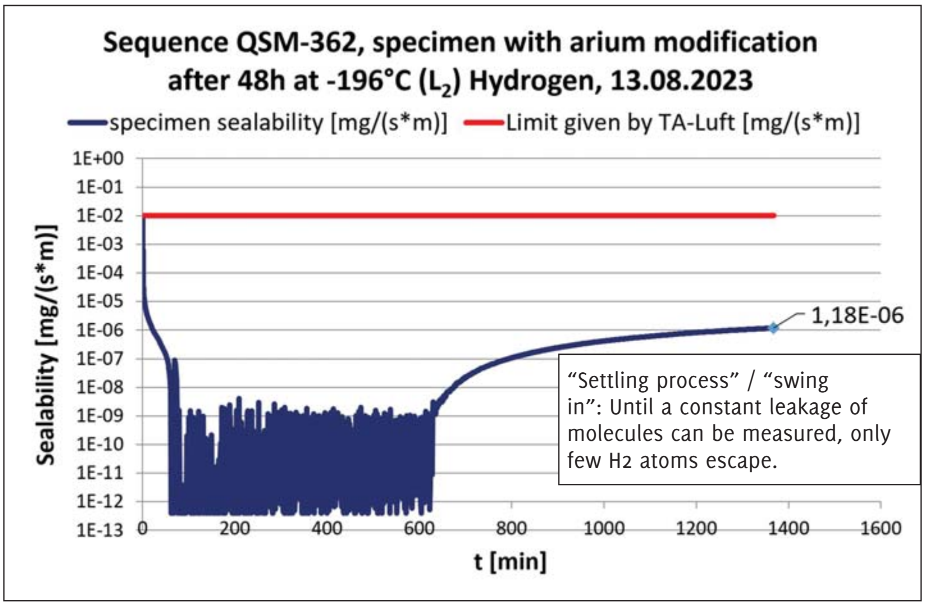
Optical inspection, pre-monting, lubrication thread/nut/washers	✓
Apply 30MPa load crosswise pattern (30/70/100% inc. Clock-wise)	✓
Sealability test Hydrogen (H2) acc. to TA-Luft2021 with 40bar (L_1)	✓
48h temperature storage at 200 °C, 24h cool-down to RT (T_{test1})	✓
Sealability test Hydrogen (H2) acc. to TA-Luft2021 with 40bar (L_2)	✓
Sealability test Helium (He) 2 acc. to TA-Luft2021 with 40bar (L_2)	✓
Sealability test Hydrogen (H2) acc. to TA-Luft2021 with 40bar (L_2)	✓
Sealability test Hydrogen (H2) after new calibration with 40bar (L_2)	✓
Determination of residual screw load (Q_R)	✓
Disassembly, dimensional measurements, taking photos	✓

Test results

Specimens Test-Number	Process	Leakrate [mbar*l/(s*m)]	leakrate [mg/(s*m)]
QSM-336	$(L_{1/H2})$	1,40E-03	1,20E-04
QSM-339	$(L_{2/H2})$	8,40E-06	7,10E-07
QSM-340	$(L_{2/He})$	6,20E-05	1,00E-05
QSM-341	$(L_{L2/H2})$	1,40E-05	1,20E-06
QSM-342	$(L_{L2/H2})$	2,10E-05	1,80E-06
residual load	(Q_R)	21,9	

1. Result H2 first
2. Result "Helium behind" Testing after H2 testing has already taken place
3. Followed by renewed H2 testing

Figure 4: Test procedure and results



Sequence QSM-362, specimen with arium modification after 48h at -196°C (L_2) Hydrogen, 13.08.2023

"Settling process" / "swing in": Until a constant leakage of molecules can be measured, only few H2 atoms escape.

Figure 5: Result with barium modified PTFE gasket

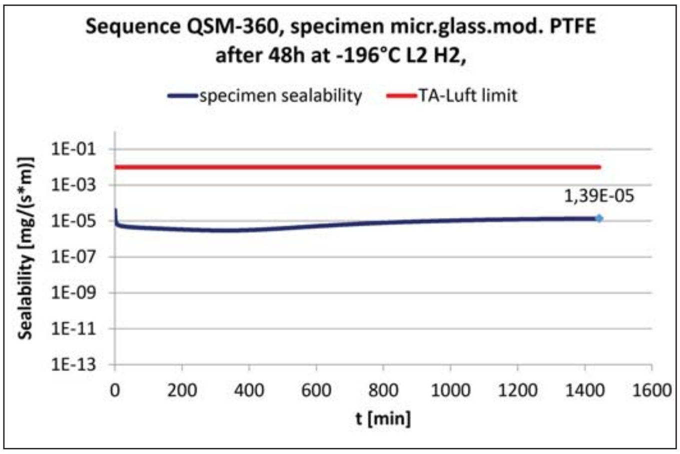


Figure 6: Result microscopic glass bubble modified PTFE gasket

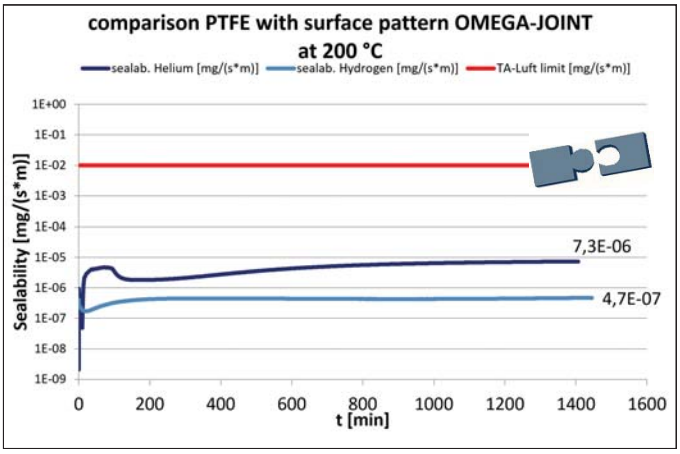


Figure 7: Multi-button

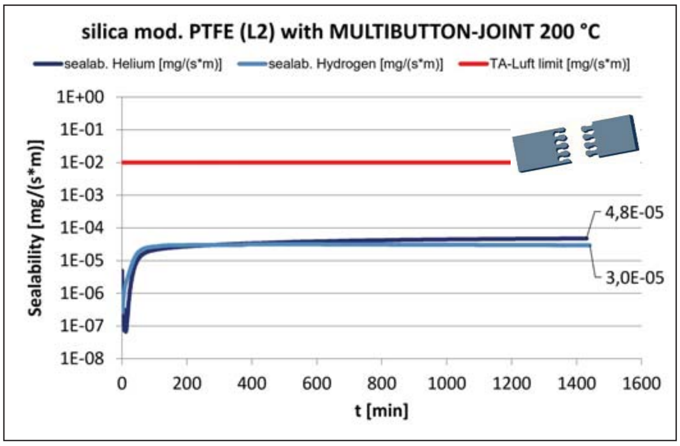


Figure 8: Omega button

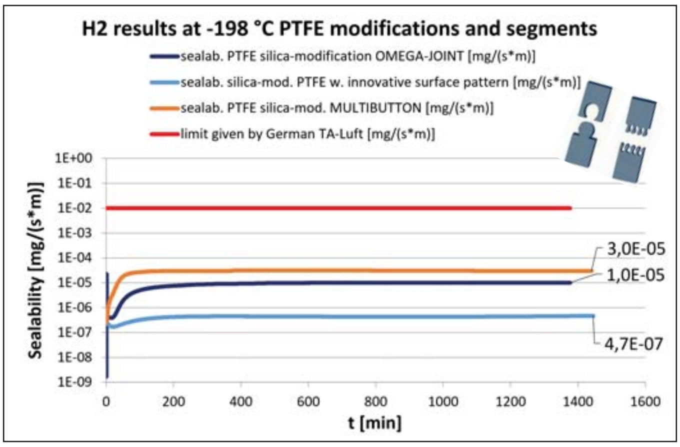


Figure 9: Comparison of the density classes achieved after temperature ageing at -196°C

followed by helium. The results fell within the mass spectrometer's margin of deviation, indicating that the sequence of test gases does not significantly affect the resulting tightness class.

Figure 4 presents an example of a test sequence with documented results. This particular test was conducted at 40 bar test pressure with a 30 MPa initial load on the gasket during assembly. The gasket tested in this sequence was a silica-modified PTFE gasket.

The following graphs illustrate the results of leakage testing after cryogenic ageing with hydrogen at -196°C:

Sampling results and technical tightness

The testing program was comprehensive, encompassing not only discrete cut gaskets but also 'Omega connections' and 'Multi-button connections'. Figures 7 and 8 illustrate the achievable tightness classes for these segmented gaskets, demonstrating that high-quality sealing performance can also be achieved with segmented PTFE materials.

All the values summarised above and in this report were obtained after assembly in a real flange setup with a surface load of 30 MPa in each case.

The values measured after storage in a cryogenic environment at -196°C are well below the leakage limit required by TA-Luft. The best performance in this comparison was achieved by a modified PTFE gasket with a patented surface texture, performing two orders of magnitude better (i.e., 100 times better) than gaskets without the innovative surface pattern.

Excellent leak tightness can be achieved not only with gaskets made from a single piece. Segmented gaskets also achieve low leakage classes in both gaseous and cryogenic hydrogen environments. As expected, the residual surface load in the cryogenic range was higher than in the gaseous range at +200°C, and all samples were rated as good.

Summary and conclusion

- The results from testing by BAM Berlin (Federal Institute for Materials Research and Testing), presented in the May 2024 issue of Valve World Magazine, demonstrate that modified, calendered PTFE gaskets exhibit little or no change in mechanical characteristics under cryogenic and gaseous hydrogen environments.
- Leakage tests with hydrogen, compared to helium tests performed with various modifications of PTFE gaskets, consistently exceed the required tightness class of 1.0x10^-2 mg/(s*m) under both cryogenic and gaseous conditions at an assembly load of 30 MPa.
- The 'worst' results achieved in the cryogenic range, at 1.39x10^-5 mg/(s*m), are already 1000 times better than the TA-Luft requirements and three orders of magnitude better in the gaseous state.
- The results obtained from helium testing can be used to evaluate the technical tightness of modified, calendered PTFE sealing materials with regard to air pollution legislation requirements such as the German TA-Luft.

Corresponding test reports and certificates in accordance with the new TA-Luft for all results and tests presented in this article are available upon request from the author. ■