Determination of the gasket characteristics at cryogenic temperatures

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So far, the knowledge about gasket characteristics in cryogenic conditions is still limited. The determination of gasket factors according to EN13555 would give the possibility to calculate flange connections according to EN1591-1. In this paper the first investigations to determine EN13555 test data at cryogenic temperatures are presented. The tests were done at a newly developed test machine at temperatures as low as -150 °C. In the tests high performance PTFE gaskets showed superior results in comparison to elastomer bonded fibre gaskets. With the results some of in field applications experienced challenges and failures of non-asbestos fiber gaskets at cryogenic temperatures can be explained.

1 Introduction

The European standard EN 13555 is widely used to generate gasket factors to calculate flange connections according EN 1591-1 and to evaluate the performance of sealing material. The EN 13555 includes tests for tightness, creep relaxation, Q_{smax} and E-Modulus of gasket materials. All those tests are performed at room or elevated temperature. The testing and evaluation at cryogenic conditions is not part of EN 13555, yet.

A wide range of applications require gaskets which can operate at cryogenic temperature. LNG, LPG or LOX are some of the common media. An extension of EN 13555 to gasket factors at cryogenic temperature would provide a basis for calculations. Cryogenic temperatures significantly influence the characteristics of the sealing materials and their sealing performance. It is known that at -40 °C binders in elastomer bonded sheet gasket materials are getting brittle and pipe vibrations could lead to sudden blow outs.

This paper presents a study of potential additional low temperature EN 13555 tests including tightness, creep relaxation, Q_{smax} and E-Modulus. The tests were done at a newly developed test machine at temperatures as low as -150 °C. The first investigations were performed with different types of high performance PTFE gaskets which are currently used in cryogenic applications in comparison to non asbestos fiber gaskets.

2 Subject of investigations

Checking the suitability of different flat gaskets in cryogenic applications.

Three different gasket materials, cut out of sheets, were tested for this:

- Structured PTFE with hollow glass microspheres (TF 1570)
- 100 % expanded PTFE (ePTFE 24 SH)
- Compressed non asbestos fiber gasket (CNAF)

3 Objective

The aim of the investigation is to determine the following gasket parameters based on the test procedures of the European test standard DIN EN 13555:2014-07, which are required for the calculation according to DIN EN 1591-1:2014-04:

- Maximum permissible gasket stress Q_{smax} (-50, -100, -150 °C),
- Modulus of elasticity EG (-50, -100, -150 °C),
- Creep relaxation factor PQR (-50, -100, -150 °C),
- Compression set Δe_{GC} (-50, -100, -150 °C),
- Minimum gasket stress at assembly $Q_{min(L)}$ (40 bar/ -150 °C) and
- Minimum gasket stress at operating condition $Q_{smin(L)}$ (40 bar/ -150 °C).

4 Test samples

The tested gaskets have the standard dimension DN40/PN40 with an outside diameter of 92 mm and an inside diameter of 49 mm. The gasket thickness is 3 mm for structured PTFE (TF) and 2 mm for expanded PTFE (ePTFE) and fiber gasket (NA).

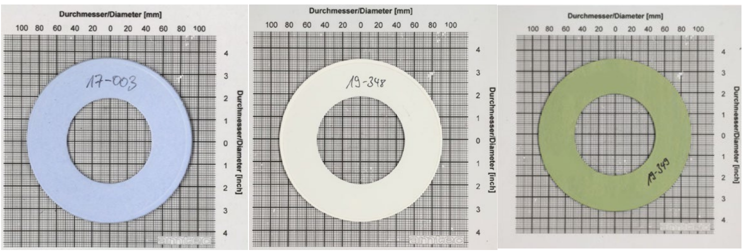
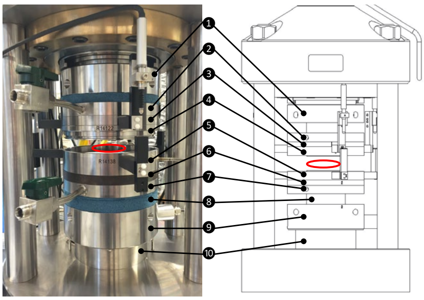


Figure 1: Pictures of test samples TF, ePTFE and NA

5 Test equipment

The tests were carried out on the following equipment (Figure 2 and Figure 3) in the laboratory of AMTEC Messtechnischer Service GmbH.

Figure 2: Equipment for cryogenic tests



Nr.:	Components
1 and 9	Temperature control plate
2 and 7	Cooling plates
3 and 6	Heating plates
4 and 5	Test plates
8	Isolation
10	Pressure measurement sensor

Figure 3: Details of test equipment

6 Test procedure

6.1 Compression test

In this compression test, the gasket is cooled down to the test temperature at a low gasket stress before the test gasket is then subjected to increasingly higher loads in loading and unloading cycles until the gasket fails.

The result in this test is used to determine the maximum permissible gasket stress at operating conditions $Q_{smax(T)}$ for the gasket.

The modulus of elasticity EG of the gasket material can be determined from the compression curve at alternating between loading and unloading the gasket with defined stresses.

6.2 Creep relaxation test

In the creep relaxation test, the gasket is pressed to the initial gasket stress before the test gasket is cooled to the test temperature. The temperature is then kept constant for 4 hours and the relaxation behavior of the seal is determined. For this purpose, after setting the initial gasket stress, the stiffness of a flange connection is simulated by regulating the gasket stress in a displacement-controlled manner.

The ratio of the residual gasket stress at the end of the test to the initial gasket stress results in the creep relaxation factor PQR, which is given as a function of the initial gasket stress, the test temperature and the system stiffness. The associated settling amount of the gasket thickness is referred to as Δe_{GC}.

6.3 Leakage test

In the first two leakage tests, the gasket is loaded to an initial stress of 10 MPa and then cooled to a test temperature of -150 °C. The gasket is then loaded in several steps up to 80 MPa and relieved again after each load step, with the leakage rate being determined for each defined load step.

In a further leakage test, the gasket is loaded to a predefined assembly gasket stress of 30 MPa before the test item is cooled to a test temperature of -150 °C. The gasket is then unloaded and loaded again in several steps, with the leakage rate determination for each load step. In addition to the loads of 30 and 40 MPa, unloading curves are also drawn up. The smallest load level is 5 MPa.

The test medium for these leakage tests is helium and the test pressure is 40 bar.

The required minimum gasket stresses $Q_{min(L)}$ and $Q_{smin(L)}$ can be determined from the leakage curve for the various tightness classes L, depending on the previously applied initial gasket stress QA.

7 Results and discussion

Table 1: List of all tests carried out

Dichtungstyp	***durchgeführte Versuche***	
	Prüfung	***Temperatur** [°C]*
TF 3 mm	Leckage 40 bar	-150
TF 3 mm	Leckage 40 bar	-150
TF 3 mm	Leckage 40 bar	-150
TF 3 mm	E-Modul	-50
TF 3 mm	E-Modul	-100
TF 3 mm	E-Modul	-150
TF 3 mm	E-Modul	-150
TF 3 mm	PQR 30 MPa	-50
TF 3 mm	PQR 30 MPa	-100
TF 3 mm	PQR 30 MPa	-100
TF 3 mm	PQR 30 MPa	-150
TF 3 mm	PQR 30 MPa	-150
TF 3 mm	PQR 30 MPa	-150
ePTFE 2 mm	E-Modul	-100
ePTFE 2 mm	E-Modul	-150
ePTFE 2 mm	PQR 30 MPa	-150
NA 2 mm	E-Modul	-150
NA 2 mm	PQR 30 MPa	-100
NA 2 mm	PQR 30 MPa	-150

- Maximum permissible gasket stress Q_{smax} (-50, -100, -150 °C) at operating temperature,
- Modulus of elasticity EG (-50, -100, -150 °C), which describes the deformation behaviour of the gasket,
- Creep relaxation factor PQR, which describes the decrease in gasket stress due to creep of the gasket under load and temperature and the amount of compression Δe_{GC},
- Required minimum gasket stress $Q_{min(L)}$ for tightness class L,
- Required minimum gasket stress $Q_{smin(L)}$ for tightness class L depending on the initial gasket stress QA.

7.1 Compression test

The tests were carried out at -50, -100 and -150 °C. Based on the test standard, intermediate reliefs were also recorded to determine the elasticity of the gasket.

The gasket parameters Q_{smax} and EG determined from these curves are tabulated.

The compression test at -50 °C shows a continuous increase in the elastic modulus. The TF gasket shows stronger deformations at the gasket stress levels above 60 MPa.

At -100 °C, a similar behavior only occurs in the last step above 80 MPa. The gasket shows no signs of damage. The modulus of elasticity increases steadily as a function of the gasket stress.

In the compression curves at -150 °C, no gasket damage can be identified up to a load of 100 MPa. The course of the modulus of elasticity at -150 °C increases continuously with increasing gasket stress up to 100 MPa in all tests. The lower the temperature the bigger the curve slope which shows a stiffer material with higher E-Modulus.

The maximum tested gasket stress was set at 100 MPa at all temperatures. No damage to the test specimens was found in the tests carried out.

A good reproducibility of the double tests can be seen.

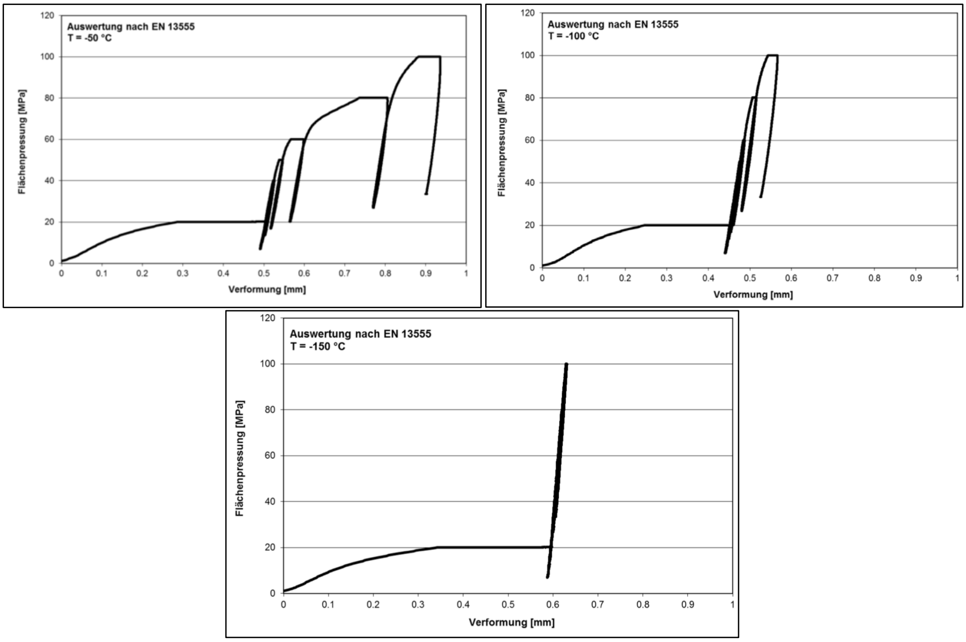


Figure 4: Compression test at -50, -100 and -150 °C of TF

Figure 4 shows the different compression curves for -50, -100 and -150 °C. The lower the temperature, the less the seal deforms as the load increases.

Maximal zulässige Flächenpressung Q_{smax} [MPa]

T [°C]	-50	-100	-150	-150
Q_{smax} [MPa]	> 100	> 100	> 100	> 100
Test #	17-023	17-002	17-041	17-350

Modulus of Elasticity E_G [MPa]

T [°C] / Q [MPa]	-50		-100		-150		-150	
	E_G [MPa]	e_G [mm]	E_G [MPa]	e_G [mm]	E_G [MPa]	e_G [mm]	E_G [MPa]	e_G [mm]
0		3.040		3.110		3.080		3.070
1		2.943		2.989		2.992		2.978
20	2307	2.439	3122	2.538	3651	2.396	2668	2.393
30	2604	2.429	3173	2.529	4621	2.392	4886	2.391
40	2692	2.417	3373	2.520	5002	2.388	5507	2.387
50	2669	2.397	3536	2.511	5649	2.385	5597	2.383
60	2658	2.344	3568	2.501	5788	2.380	5850	2.379
80	3167	2.138	3699	2.474	6181	2.371	6304	2.371
100	3788	2.008	3888	2.422	6262	2.361	6074	2.362
Test #	17-023		17-002		17-041		17-350	

Figure 5: Q_{smax} and EG Values at -50, -100 und -150 °C of TF

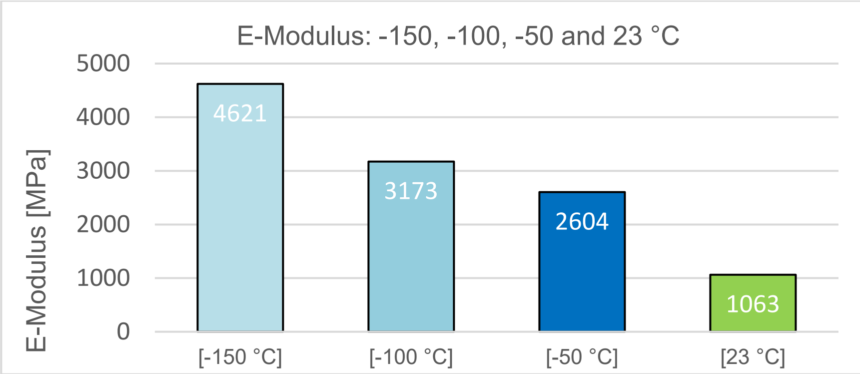


Figure 6: E-Modulus comparison of PTFE at 30 MPa and different Temperatures

Figure 5 shows the Q_{smax} and EG values tabulated. Figure 6 compares the EG values at 30 MPa and the different temperatures. As the temperature decreases, the modulus of elasticity increases at the same time. The modulus of elasticity at RT is 1063 MPa and at -150 °C the modulus of elasticity is already 4621 MPa.

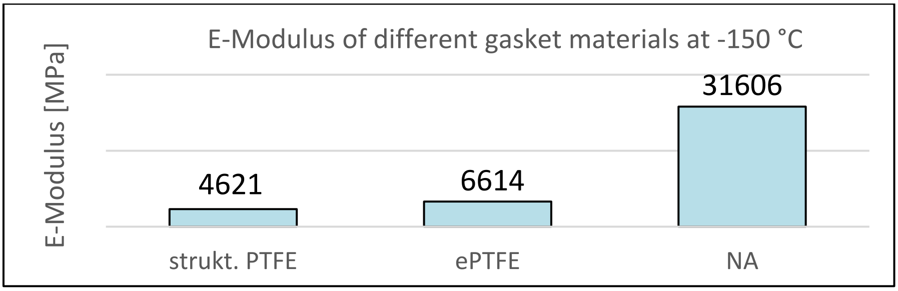


Figure 7: E-Modulus comparison of different materials at -150 °C

Figure 7 compares the E-modulus of TF with ePTFE and NA. At 6614 MPa, ePTFE is slightly higher than the TF, which is also caused by the thinner initial thickness of the ePTFE gasket. The higher E-Modulus is also related to the thinner chosen ePTFE gasket (2 mm). The fiber gasket, which is also 2 mm thick, has a significant higher E-Modulus of 31606 MPa.

7.2 Creep relaxation test

To determine the creep relaxation factor PQR, the relationship between residual and initial gasket stress QR/QI is defined based on EN 13555.

Creep relaxation tests were carried out at different temperatures and with a stiffness of 500 kN/mm. The initial gasket stress was set at 30 MPa.

The temperatures were -50, -100 and -150 °C.

A loss of gasket stress occurs, in the tests carried out under temperature. The creep relaxation factor PQR for TF was tested in a range from 0.64 to 0.77, for NA at 0.91 to 0.93 and for ePTFE at 0.89.

The amount of compression set was always measured with an initial gasket stress of 30 MPa. The maximum compression set Δe_{GC} of 91 µm was determined for the TF and 31 µm for the ePTFE with a temperature of -150 °C and for the fiber seals of 27 µm with -100 °C. Only at NA, the compression set became smaller as lower temperature.

A good reproducibility of the double tests under cryogenic temperature can be seen.

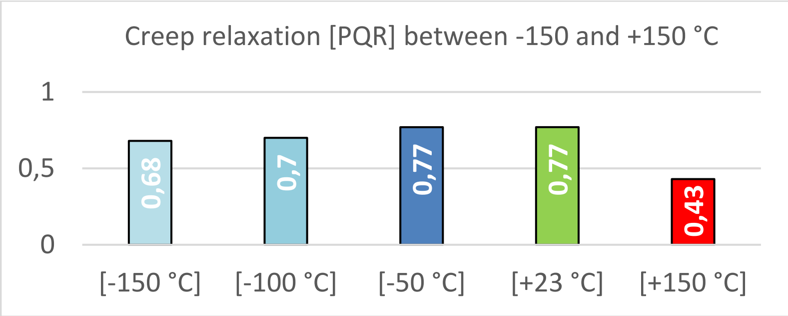


Figure 8: PQR comparison of TF at different temperature

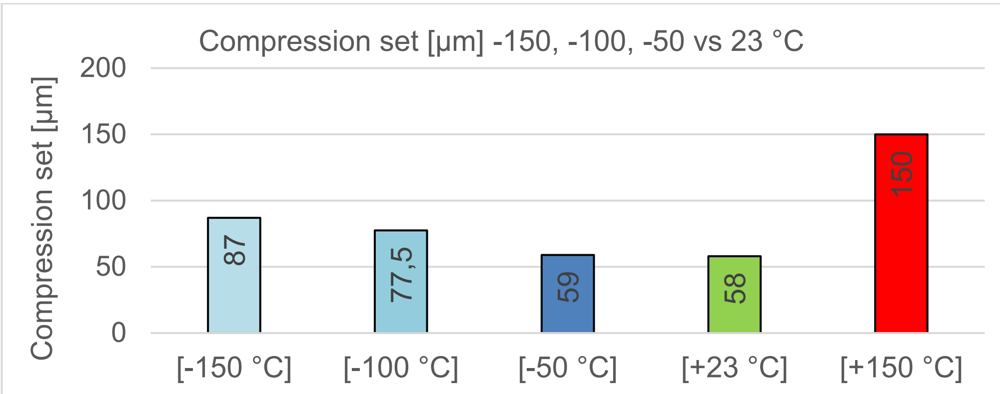


Figure 9: Compression set comparison of TF at different temperature

In Figure 8 the compression set Δe_{GC} and in Figure 9 the PQR values are compared at temperature between -150 and +150 °C. At +150 °C compared to RT, as expected, the amount of settlement increases and the PQR decreases as the gasket softens. However, as the gasket cools, it becomes harder and stiffer, as shown in Figure 6, with the effect that the compression set increases and the PQR decrease. Temperature expansion also seems to be responsible for this effect. Figure 10 illustrates the shrinkage of PTFE as it cools down to -150 °C.

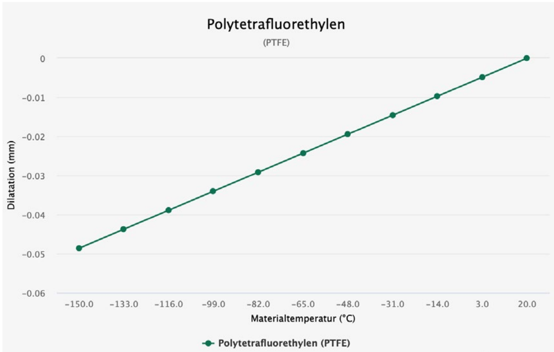


Figure 10: Temperature effect at a 2 mm thick PTFE gasket

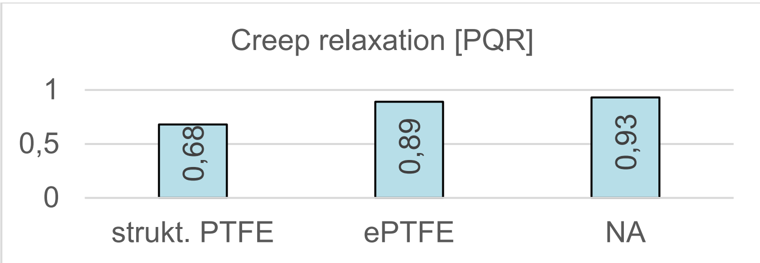


Figure 11: Creep relaxation comparison for different materials at -150 °C

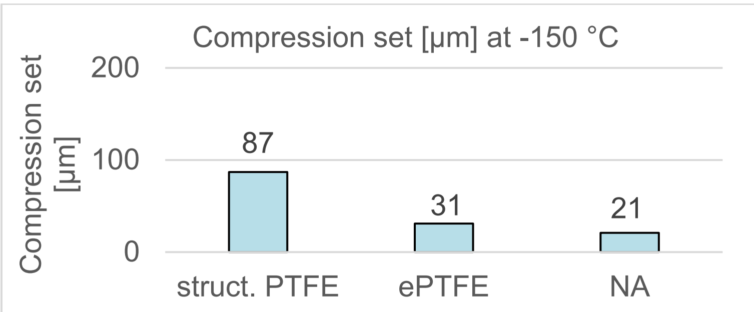


Figure 12: Compression set comparison for different materials at -150 °C

Figure 11 compares the PQR and Figure 12 the compression set of TF with ePTFE and NA. The different gasket thicknesses of TF, ePTFE and NA must be taken into account. The lower the PTFE gasket thickness, the higher the PQR value. Furthermore, high-quality ePTFE gaskets have better mechanical parameters.

These properties result in a higher PQR and lower compression set for ePTFE versus TF. The fiber gasket showed the highest PQR value and the smallest amount of compression. The fiber gasket is so hard at -150 °C that no further deformation of the gasket occurs. The temperature expansion does not appear to have a significant effect. If elastomer or elastomer based gaskets are suitable at cryogenic conditions in terms of safety should be checked in additional tests.

7.3 Leakage test

The sealing behavior of the flat gaskets was examined at an internal pressure of 40 bar with helium and a test temperature of -150 °C. A differential pressure measuring device was used as the measuring system.

In these tests, the tightness class L0.001 was already achieved at a surface pressure of 30 MPa. If the seal is further stressed to 40 MPa, the leakage rate continues to drop.

The unloading steps were carried out up to a gasket stress of 5 MPa and show only a slight increase in the leakage rate.

A good reproducibility of the results can be seen in the leakage tests.

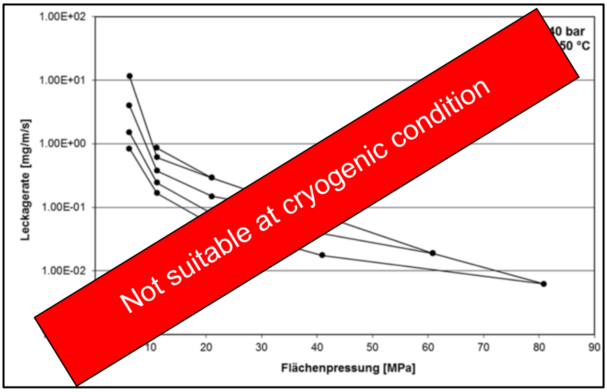


Figure 13: Leakage test procedure according EN 13555 at -150 °C (Test starts with an iniatial gasket stress of 10 MPa)

In Figure 13, the leakage was tested as described in EN 13555. Here the first load was selected at 10 MPa and then the temperature is lowered to -150 °C. The following load levels were only applied to the already cryogenic seal. Since the gasket does not significantly deform or adapt at this temperature, there is an atypically high leakage rate for the TF gasket measured. The results show that this test procedure is not suitable at cryogenic temperatures. The test procedure was therefore modified as follows.

In this further leakage test, instead of an initial gasket stress of 10 MPa, an initial gasket stress of 30 MPa was selected, which is applied at room temperature. This higher initial gasket stress was chosen according to a typical applied gasket stress during installation in the system.

The following leakage result, fits to the typical leakage performance of this gasket material, as shown in Figure 14.

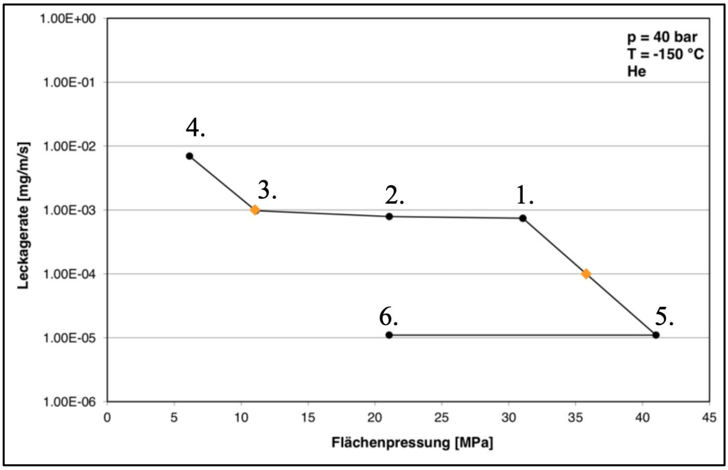


Figure 14: Leakage curve at -150 °C and initial gasket stress of 30 MPa

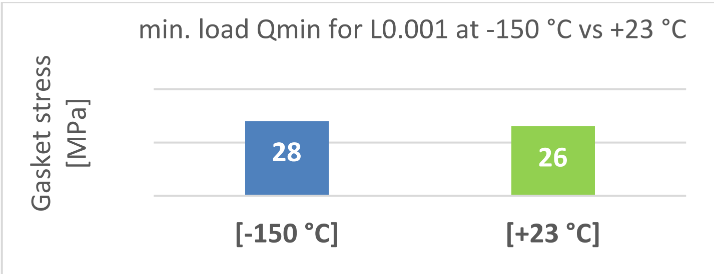


Figure 15: Leakage comparison at room temperature and -150 °C

Figure 15 shows the necessary gasket stress to achieve tightness class L0.001 at room temperature and -150 °C.

Unfortunately, there are no further results for the other sealing materials available so that comparisons can´t be made.

8 Summary and Conclusion

The results clearly show that tests based on EN 13555 are possible up to -150 °C. The test results are repeatable and documented with double tests.

The first EN 1591-1 calculations have already been carried out with good results using the available gasket parameters for TF.

8.1 E-Modulus and Q_{smax}

The tests to determine the maximum gasket stress have so far only been carried out on TF up to 100 MPa. No damage was found on the gaskets. The maximum gasket stress on the TF material is therefore >100 MPa.

The lower the temperature, the higher the modulus of elasticity. As it is well known, plastics become stiffer, harder and more brittle as the temperature decreases. However, this effect has not yet been examined in detail here. Fiber gaskets in particular requires further investigations for safety reasons due to their significantly higher E-modulus. We classify the measured modulus of elasticity as alarmingly high. The glass transition temperature of elastomers is usually around -40 °C. Below this temperature, elastomers become hard and brittle, like glass.

8.2 Creep relaxation “PQR” and compression set “Δe_{GC}”

The PQR value of the TF gasket falls with decreasing temperature from 0.77 (RT) to an average of 0.68 (-150 °C) and at -150 °C roughly corresponds to the PQR value at elevated temperature of +80 °C.

If you compare the creep relaxation of ePTFE and NA gaskets with characteristic values (room temperature) published on GasketData.org or ESAData.org, you can also see smaller PQR values at cryogenic temperature. With ePTFE and NA, this effect is smaller than with TF.

The compression set behaves accordingly. The compression set increase at the TF gaskets from RT to -150 °C. The tested TF gaskets are 3 mm and the ePTFE and NA gaskets are only 2 mm. This is one of the reasons why the PQR values of ePTFE and NA gaskets are bigger and the compression sets are smaller compared to TF. A direct comparison of the materials is therefore limited.

8.3 Leakage performance

For reasons of cost and time, the leakage behavior has so far only been carried out for TF gaskets.

In the first two leakage measurements with the test procedure according to EN 13555, relatively high leakage values were measured, which are atypical for this sealing material.

After detailed analysis, it was determined that the typical loading and unloading steps are not appropriate for testing at cryogenic conditions. The test procedure was therefore modified.

The gasket is pressed at RT to the defined installation gasket stress before it is cooled to -150 °C. Only the first load level is carried out at ambient temperature and all other gasket stress levels at test temperature. At cryogenic test temperature, the first gasket stress should be selected according to the installation load in order to be able to measure the full sealing performance at further load levels. In the modified test procedure, the first gasket stress level was therefore increased from 10 MPa to 30 MPa. As expected, the tightness class of L0.001 was reached below 30 MPa. Tightness class L0.001 is expected to be reached at 28 MPa. Comparable values are obtained with the results at RT.

8.4 Further investigations

This work is intended to show that it is possible to determine gasket parameters under cryogenic conditions based on EN 13555. The generation of extensive gasket parameters is becoming more and more important in order to be able to calculate flange connections according to EN 1591-1. Including the determination of characteristic values at cryogenic temperatures in EN 13555 could be a useful addition.

Based on the results, a temperature value of -150 °C appears to be sufficient.

However, more extensive research needs to be done.

In order to better understand the behavior of gaskets in cryogenic conditions, the following questions should be investigated in the next step:

- What is the influence of different gasket thicknesses?
- What is the leakage behavior of other sealing materials?
- Can the tests be carried out reliably at even lower temperatures?

It should also be investigated how brittle gaskets in pipelines behave at these temperatures. Additional forces and vibrations should also be taken into account.

9 Nomenclature

Variable	***Description***	***Unit***
Qmax	Maximum Permissible Gasket Stress	[N/mm²]
$Q_{\mathrm{min\,(L)}}$	Minimum Gasket Stress for Tightness Class L at Loading	[N/mm²]
$Q_{\mathrm{Smin\,(L)}}$	Minimum Gasket Stress for Tightness Class L at Unloading	[N/mm²]
L	Tightness Class at Defined Pressure and Medium	[mg/m/s]
RT	Room Temperature	[K]
PQR	Creep Relaxation Factor	[-]
Δe_{GC}	Compression Set	[µm]
EG	E-Modulus	[N/mm2]
QR	Residual Gasket Stress	[N/mm2]
QI or QA	Initial Gasket Stress	[N/mm2]

10 References

[1] EN 13555: 2014-07: Flanges and their Joints - Gasket parameters and test procedures relevant to the design rules for gasketed circular flange connections

[2] DIN EN 1591-1:2014-04: Flanges and their joints - Design rules for gasketed circular flange connections – Part 1: Calculation

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