



TEST REPORT

on Testing a Nonmetallic Material for Oxygen Compatibility

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|------------------------------------|---|
| BAM reference | 24009109-E_mod.1 |
| Copy | 1 st copy of 2 copies |
| Customer | SGL Carbon GmbH Werner-von-Siemens-Str. 18 86405 Meitingen Germany |
| Order date | April 8, 2024 |
| Reference | PO-Nr.: 450525221 / 04.04.2024 |
| Receipt of order | April 8, 2024 |
| Test samples | Sigraflex® Folie Typ E, batch 2403135002 |
| Receipt of samples | April 12, 2024 |
| Test date | April 12, 2024, to December 6, 2024 |
| Test location | BAM, Division 2.1 "Safety of Energy Carriers"; building no. 41; Unter den Eichen 87, 12205 Berlin, Germany |
| Test procedure according to | DIN EN 1797 und ISO 21010 "Cryogenic Vessels - Gas/Material Compatibility"; Annex of code of practice M 034-1 (BGI 617-1) "List of nonmetallic materials compatible with oxygen", by German Social Accident Insurance Institution for the raw materials and chemical industry; TRGS 407 Technical Rules for Hazardous Substances "Tätigkeiten mit Gasen - Gefährdungsbeurteilung" chapter 3 "Informationsermittlung und Gefährdungsbeurteilung" and chapter 4 "Schutzmaßnahmen bei Tätigkeiten mit Gasen" |

This test report consists of page 1 to 9 and enclosures 1 to 4.

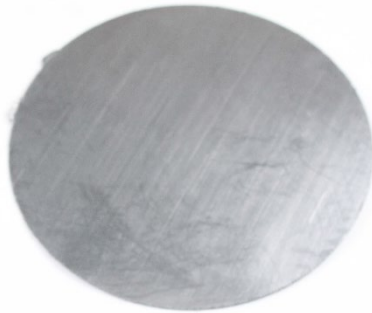
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The German version is legally binding, except an English version is issued exclusively.

1 Documents and Test Samples

The following documents and samples were submitted to BAM:

- 1 Test application
Safety-related investigation on the Graphite-based material Sigraflex® Folie Typ E, batch 2403135002, for use as a sealing material in spiral wound gaskets, grooved metal gaskets and corrugated gaskets, and for use as a sealing material in gaseous oxygen service at temperatures up to 250 °C and at pressures up to 160 bar, and in liquid oxygen service
- 1 Sicherheitsdatenblatt (Safety Data Sheet) Sigraflex® Folie Type...
(8 pages, Fa. SGL Carbon GmbH, Version 2.2, date of issue: May 09, 2023)
- 1 Customer Master Data Sheet (CMDS) (March 12, 2024)
- 25 Disks Graphite-based material Sigraflex® Folie Typ E, batch 2403135002
Dimensions: Ø 140 mm, Thickness 0.5 mm
Color: Grey



2 Applied Test Methods

On the one hand, the Graphite-based sealing material Sigraflex® Folie Typ E, batch 2403135002, shall be used in spiral wound gaskets, grooved metal gaskets and corrugated gaskets. On the other hand, it shall be used as an ordinary sealing material. In both cases, it shall be used in gaseous oxygen service at temperatures up to 250 °C, and at 160 bar oxygen pressure, and in liquid oxygen service.

As the material shall not be used as a flat gasket material in flanged connections, the flange test was not carried out.

The following test methods were applied:

2.1 Testing for Ignition Sensitivity to Gaseous Oxygen Impacts

Generally, this test method is required if rapid oxygen pressure changes, so-called gaseous oxygen impacts, on the ordinary sealing material cannot be safely excluded in usage.

2.2 Determination of the Autogenous Ignition Temperature (AIT) in High Pressure Oxygen

Usually, this test method is required if the material is for service temperatures greater than 60 °C.

The AIT is a safety characteristic and indicates the temperature at which the material shows self-ignition in the presence of oxygen without an additional ignition source. Therefore, it is relevant for the maximum use temperature, that is generally set 50 °C below this AIT for gasket materials and 100 °C below this AIT for sealing materials.

2.3 Testing for Aging Resistance in High Pressure Oxygen

This test is necessary whenever a material is intended for service at higher temperatures than 60 °C. It simulates the use of a material in practice and helps analyze whether ignition temperature or properties of the material change due to the aging processes.

2.4 Testing for Reactivity with Liquid Oxygen on Mechanical Impact

Generally, this test method is required if direct contact of the material with liquid oxygen and mechanical impacts cannot be safely excluded in usage.

3 Sampling

The material sample used for the investigation was provided by the customer.

3.1 Preparation of Samples

For testing the “Test for Ignition Sensitivity to Gaseous Oxygen Impacts” and “Determination of the Autogenous Ignition Temperature (AIT) in High Pressure Oxygen”, the Graphite-based material Sigraflex® Folie Typ E, batch 2403135002, was cut into parts of ca. 1 mm to 2 mm in edge length and was used in this form.

For testing the “Aging Resistance in High Pressure Oxygen”, the Graphite-based material Sigraflex® Folie Typ E, batch 2403135002, was cut into parts of ca. 40 mm to 20 mm in edge length and was used in this form.

4 Tests

4.1 Testing for Ignition Sensitivity to Gaseous Oxygen Impacts

The test method is described in enclosure 1.

Based on the specified intended maximum temperature by the customer, the test was performed at 60 °C, at 200 °C, and at 250 °C.

4.1.1 Assessment Criterion

According to DIN EN 1797 “Cryogenic Vessels - Gas/Material Compatibility” and to ISO 21010 “Cryogenic Vessels - Gas/Material Compatibility” the criterion for a reaction of the sample to gaseous oxygen impacts is a temperature rise of at least 20 °C.

If the sample exhibits a change in color, or in consistency after testing, this is also considered as a positive reaction by BAM for safety reasons, even if there is no temperature rise detectable of at least 20 °C.

4.1.2 Results

In each of the test series, the initial oxygen pressure p_i was at ambient pressure.

| Sample Temperature t_i [°C] | Final Oxygen Pressure p_F [bar] | Reaction |
|----------------------------------|--------------------------------------|----------|
| 60 | 360 | no* |
| 60 | 400 | no* |
| 60 | 420 | no* |
| 60 | 450 | no* |
| 60 | 450 | no* |
| 200 | 450 | no* |
| 200 | 450 | no* |
| 250 | 450 | no* |
| 250 | 450 | no* |

* within a series of five consecutive impacts

In two separate test series, each consisting of a series of five consecutive impacts, no reaction of the Graphite-based material Sigraflex® Folie Typ E, batch 2403135002, with oxygen could be observed at following conditions:

| Sample Temperature t_i [°C] | Final Oxygen Pressure p_F [bar] |
|----------------------------------|--------------------------------------|
| 60 | 450 |
| 200 | 450 |
| 250 | 450 |

4.2 Determination of the Autogenous Ignition Temperature (AIT) in High Pressure Oxygen

The test method is described in enclosure 2.

Based on the intended maximum use pressure, mentioned by the customer, this test was performed at a final oxygen pressure of approximately 160 bar.

4.2.1 Assessment Criterion

The criterion for a reaction of the sample with oxygen is a distinct increase in pressure and a more or less steep increase in temperature.

4.2.2 Results

| Test No. | Final Oxygen Pressure p_F [bar] | AIT [°C] |
|----------|--------------------------------------|-------------|
| 1 | 164 | >500 |
| 2 | 163 | >500 |
| 3 | 162 | >500 |
| 4 | 165 | >500 |
| 5 | 162 | >500 |

In five separate tests, with a mean final oxygen pressure of 163 bar, no ignition of the sample could be detected up to temperatures of 500 °C. This temperature equals the maximum operating temperature of the test apparatus. Consequently, the AIT of the sample is higher than 500 °C.

4.3 Testing for Aging Resistance in High Pressure Oxygen

The test method is described in enclosure 3.

In general, artificial aging is carried out at the maximum use pressure and at an elevated temperature, that is 25 °C above the maximum operating temperature. Consequently, the test temperature was 275 °C. Based on the intended maximum use pressure for use as a gasket material, mentioned by the customer, this test was performed at a final oxygen pressure of approximately 160 bar.

4.3.1 Assessment Criteria

There are three criteria for evaluating the aging behavior:

If there is a change in mass $\Delta m \leq 1\%$, the sample is aging resistant, in case of $\Delta m > 1\%$ and $\Delta m \leq 2\%$, the sample is sufficient aging resistant, and in case of $\Delta m > 2\%$, the sample is insufficient aging resistant.

Changes in color, consistency, shape or surface texture of the sample or gas releases from the sample that can be detected after testing will be also considered by BAM.

The AIT of the aged sample is compared to the AIT of the non-aged sample. If there is a distinct deviation between both AITs, the lower value is considered for safety reasons.

4.3.2 Results

4.3.2.1 Testing for Change in Mass or Physical Appearance

| Time [h] | Temperature [°C] | Oxygen Pressure [bar] | Mass Change [%] |
|----------|------------------|-----------------------|-----------------|
| 100 | 275 | 160 | - 0.2 |

After aging, the test sample was apparently unchanged. The sample decreased 0.2 % in mass.

4.3.2.2 Determination of the AIT of the Aged Material in High Pressure Oxygen

The test method is described in enclosure 2. The AIT test of the aged material was performed at same conditions as described in chapter 4.2.

| Test No. | Final Oxygen Pressure p_F [bar] | AIT [°C] |
|----------|-----------------------------------|----------|
| 1 | 163 | >500 |
| 2 | 164 | >500 |
| 3 | 163 | >500 |
| 4 | 165 | >500 |
| 5 | 164 | >500 |

In five separate tests, with a mean final oxygen pressure of 164 bar, no ignition of the sample could be detected up to temperatures of 500 °C. This temperature equals the maximum operating temperature of the test apparatus. Consequently, the AIT of the sample is higher than 500 °C.

4.4 Testing for Reactivity with Liquid Oxygen on Mechanical Impact

The test method is described in enclosure 5.

4.4.1 Assessment Criterion

According to the BAM-Standard "Testing for Reactivity with Liquid Oxygen on Mechanical Impact", a nonmetallic material is not suitable for liquid oxygen service if reactions occur with liquid oxygen at a drop height of 0.17 m (impact energy 125 Nm) or less.

4.4.2 Results

| Test Series No. | Drop Height [m] | Impact Energy [Nm] | Behavior on Mechanical Impact |
|-----------------|-----------------|--------------------|---|
| 1 | 0.83 | 625 | reaction in 1 st single test |
| 2 | 0.67 | 500 | reaction in 5 th single test |
| 3 | 0.50 | 375 | reaction in 3 rd single test |
| 4 | 0.33 | 250 | reaction in 4 th single test |
| 5 | 0.17 | 125 | no reaction |

At a drop height of 0.17 m (impact energy of 125 Nm), no reaction of the test sample with the liquid oxygen could be detected in a series of 10 single tests.

5 Summary of the Test Results

In two separate tests, each consisting of a series of five consecutive impacts, no reaction of the sample with oxygen could be observed at final pressures of 450 bar at temperatures of 60 °C, of 200 °C, and of 250 °C.

At a mean final oxygen pressure p_F of 163 bar, the test sample has an autogenous ignition temperature that is greater than 500 °C.

The material proved to be aging resistant at 275 °C and at 160 bar oxygen pressure.

Testing of the sample for reactivity to mechanical impacts in liquid oxygen showed that no reaction was detected at an impact energy of 125 Nm.

6 Measurement uncertainty

The tests are carried out in accordance with the standards or guidelines indicated on the cover sheet of this report. Thereafter, the temperature measurement should have a maximum deviation of ± 2 K and the pressure measurement should have a maximum deviation of ± 2 bar.

For the test in chapter 4.1, the uncertainty is 1.7 K (according to the calibration protocol from January 17, 2024) for the temperature measuring system, and the uncertainty is 0.16 bar (according to the calibration protocol from December 7, 2023) for the used pressure measuring system.

For the test in chapter 4.2, the uncertainty is 0.7 K (according to the calibration protocol from May 13, 2024) for the temperature measuring system, and the uncertainty is 0.3 bar (according to the calibration protocol from January 25, 2023) for the used pressure measuring system.

For the test in chapter 4.3, the uncertainty is 1.0 K (according to the calibration protocol from May 23, 2024) for the temperature measuring system, and the uncertainty is 0.3 bar (according to the calibration protocol from February 13, 2023) for the used pressure measuring system.

The measurement uncertainty in determining the height of fall during the test in Chapter 4.4 is estimated to be ± 0.01 m. Experience has shown that this measurement uncertainty has no influence on the test result.

7 Statements of conformity

The tests are carried out in accordance with the standards or guidelines, stated on the cover sheet of this report. Deviating or supplementary test criteria are described in the respective subchapter "Assessment Criterion" in Chapter 4 "Tests".

8 Opinion and Interpretation

On the one hand, the Graphite-based sealing material Sigraflex® Folie Typ E, batch 2403135002, shall be used in spiral wound gaskets, grooved metal gaskets and corrugated gaskets, on the other hand, it shall be used as an ordinary sealing material in gaseous oxygen service at temperatures up to 250 °C, and at 160 bar oxygen pressure, and in liquid oxygen service.

On basis of the test results, the requirements for sealing materials, described in the code of practice M034, annex 2 of code of practice M034-1, Technical Rules for Hazardous Substances TRGS 407 and based on the assessment criteria described in this test report, there are no objections regarding technical safety, to use the Graphite-based material Sigraflex® Folie Typ E, batch 2403135002, as a sealing material in spiral wound gaskets, grooved metal gaskets and corrugated gaskets for gaseous oxygen service at following use conditions only:

| Maximum Temperature [°C] | Maximum Oxygen Pressure [bar] |
|-----------------------------|----------------------------------|
| up to 250 | 160 |

On basis of the test results, the requirements for sealing materials, described in the code of practice M034, annex 2 of code of practice M034-1, Technical Rules for Hazardous Substances TRGS 407 and based on the assessment criteria described in this test report, there are no objections regarding technical safety, to use the Graphite-based material Sigraflex® Folie Typ E, batch 2403135002, as an ordinary sealing material for gaseous oxygen service at following use conditions only:

| Maximum Temperature [°C] | Maximum Oxygen Pressure [bar] |
|-----------------------------|----------------------------------|
| 60 | 450 |
| >60 up to 250 | 160 |

Based on the test results and according to BAM' s standard "Testing for Reactivity with Liquid Oxygen on Mechanical Impact", there are no objections with regard to technical safety to use the Graphite-based sealing material Sigraflex® Folie Typ E, batch 2403135002, in liquid oxygen service. As liquid oxygen is largely incompressible, a change in pressure has no significant effect on the ignition of the test material. A limitation to a certain pressure range is therefore not necessary.

The content of the test report refers exclusively to the test sample of Graphite-based sealing material Sigraflex® Folie Typ E, batch 2403135002.

The product may be used as a sealing material in spiral wound gaskets, grooved metal gaskets and corrugated gaskets, and as an ordinary sealing material in gaseous and in liquid oxygen service. The maximum safe oxygen pressure of the product and its maximum use temperature as well as other restrictions in use shall be given.

9 Comments

This safety-related investigation of the material for use as an ordinary sealing material considers the fact, that rapid oxygen pressure changes - so-called oxygen pressure surges - cannot be safely excluded in usage.

The safety-related investigation of the material for use as a sealing material in spiral wound gaskets, grooved metal gaskets and corrugated gaskets, considers the fact, that rapid oxygen pressure changes - so-called oxygen pressure surges - can be safely excluded in usage.

In addition, the safety related investigation considers the fact, that the sealing material shall be used in gaseous oxygen service at temperatures greater than 60 °C, and that direct contact of the material with liquid oxygen and mechanical impacts cannot be safely excluded in usage.

Our experience shows that the safety characteristics of a product may vary from batch to batch. Therefore, today, we recommend batch testing of products, that are included for oxygen service. In this context, we would like to mention our paper from September 2009: "The Importance of Quality Assurance and Batch Testing on Nonmetallic Materials Used for Oxygen Service", Journal of ASTM International, Vol. 8th; Paper ID JAI102309. This publication can be purchased at www.astm.org.

Products on the market that contain a reference to BAM testing shall be marked accordingly. It shall be evident that only a sample of a batch has been tested and evaluated for oxygen compatibility. The reference shall not produce a presumption of conformity that monitoring of the production on a regular basis is being performed by BAM.

Bundesanstalt für Materialforschung und -prüfung (BAM)

12200 Berlin

January 20, 2025

Division 2.1 "Safety of Energy Carriers"

by order

Dr. Thomas Kasch
Study Director

Dr. Kai Holtappels
Head of Division

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Enclosure 1

Testing Nonmetallic Materials for Ignition Sensitivity to Gaseous Oxygen Impacts (V 2024-01)

0.2 g to 0.5 g of the paste-like, of the divided solid, or with ceramic fibres mixed liquid material is placed into a steel tube with a volume of 15 cm³. The heatable steel tube is connected to a high-pressure oxygen accumulator using a 750 mm long pipe (internal diameter 14 mm) and a quick opening valve.

After heating the tube to the test temperature of 60 °C minimum, the quick opening valve is opened. Preheated oxygen of 60 °C at final pressure p_F flows abruptly into the pipe and tube. In this case, the oxygen in the tube and in the pipe is heated and almost adiabatically compressed from initial pressure p_I to final pressure p_F in 17.5 ms \pm 2.5 ms (according to DIN EN 1797 and ISO 21010). If there is a reaction of the sample with oxygen, further tests are performed at a lower final pressure p_F . If there is no reaction of the sample with oxygen, further tests are performed at a higher final pressure p_F . The test is finished, if there is no reaction of the material detected in two test series each consisting of five single tests.

For this test, the maximum test pressure is 450 bar, and the maximum test temperature is 300 °C.

Enclosure 2

Determination of the Autogenous Ignition Temperature of Nonmetallic Materials in High Pressure Oxygen (V 2024-01)

0.2 g to 0.5 g of the paste-like, of the divided solid, or with ceramic fibres mixed liquid material is placed into an autoclave with a volume of 34 cm³. The autoclave is pressurized to the initial pressure p_i and inductive heated. The temperature increases in an almost linear way at a rate of 110 K/min.

Pressure and temperature are recorded by a PC-system. As the temperature increases, the oxygen pressure in the autoclave increases continuously. The ignition of the material is recognized by a sudden rise in temperature and a more or less rise of the pressure.

In this way, the ignition temperature is determined at a specific final oxygen pressure p_f . In principle, the ignition temperature of a material depends on the pressure. The ignition temperature decreases with increasing final oxygen pressure.

For this test, the maximum test pressure is 250 bar, and the maximum test temperature is 500 °C.

Enclosure 3

Testing for Aging Resistance of nonmetallic materials in High Pressure Oxygen (V 2024-01)

A sample of the solid material is exposed to compressed oxygen and elevated temperature in an autoclave for 100 hours. The sample mass is determined before the test.

This test is intended to simulate the use of the material in practice and to show whether the material properties such as color, consistency, surface texture or the ignition temperature of the material change as a result due to aging processes.

For this test, the maximum test pressure is 250 bar, and the maximum test temperature is 325 °C.

Enclosure 4

Testing for Reactivity with Liquid Oxygen on Mechanical Impact (V 2023-01)

Approximately 0.5 g of the paste-like, of the divided solid, or with ceramic fibres mixed liquid material is placed into a sample cup (height = 10 mm; diameter = 30 mm), made of 0.01 mm copper foil. Liquid oxygen is poured into the cup which is then exposed to the mechanical impact of a plummet (mass = 76.5 kg).

A reaction of the material is indicated by a flame and a more or less strong noise of an explosion. The impact energy, at which no reaction occurs, is determined by varying the drop height of the plummet. This result shall be confirmed in a series of ten consecutive tests at same conditions, and then this test is finished.

Test is stopped, if a reaction is observed at an impact energy of 125 Nm, this equals a drop height of the plummet of 0.17 m. In this case, with regard to technical safety, the material is not suitable for use in liquid oxygen.