Bundesanstalt für Materialforschung und -prüfung

TEST REPORT

BAM reference	22028874-E a
Copy	1st copy of 2
Customer	SGL Carbon GmbH Werner-von-Siemens-Str. 18 86405 Meitingen Germany
Date of Request	August 18, 2022
Your Reference	Rainer Zeuss
Receipt of Signed Contract	August 31, 2022
Test Samples	Graphit-based gasket material SIGRAFLEX®UNIVERSAL V20010C2I, batch 2201060201
Receipt of Samples	August 31, 2022
Test Date	August 31 to December 14, 2022
Test Location	Division 2.1 "Safety of Energy Carriers"; building no. 41
Test Procedure or Requirement according to (in the current version)	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 annex 1 to 4.

TEST REPORT

1 Documents and Test Samples

The following documents and samples were submitted to BAM:

1	Test application Safety-related investigation on Graphit-based gasket material SIGRAFLEX®UNIVERSAL V20010C2I, batch 2201060201, for use in gaseous oxygen service at temperatures up to 250 °C and at pressures up to 160 bars as well as for use in liquid oxygen service
1	Safety Data Sheet SIGRAFLEX®UNIVERSAL (6 pages, SGL Carbon GmbH, Version Nr.: 1.04, date of issue: January 11, 2021)
1	Completely filled Customer Master Data Sheet (CMDS) (July 8, 2022)
15	Disks of the Graphit-based gasket material SIGRAFLEX®UNIVERSAL V20010C2I, batch 2201060201, on a metallic carrier labeled with Sigraflex in blue letters Dimensions: outer-ø 140 mm, thickness 2 mm Color: Grey

2 Applied Test Methods

The Graphit-based gasket material SIGRAFLEX®UNIVERSAL V20010C2I, batch 2201060201, shall be used for gaseous oxygen service at temperatures up to 250 °C and at pressures up to 160 bars as well as for liquid oxygen service.

The following test methods were applied:

2.1 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 100 °C below this AIT.

2.2 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.

2.3 Testing of Gaskets for Flanges in High Pressure Oxygen

This test simulates the faulty installation of a gasket in a flange connection where the sealing material projects into the inner diameter of the pipe. This test investigates the fire behavior of the gasket material in a standard flange after artificial ignition. It shows whether the fire of the disk is transferred to the metal of the flange connection or if the flange connection becomes leaky.

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

To test the conductive gasket material, the disks were prepared as shown in figure 1.

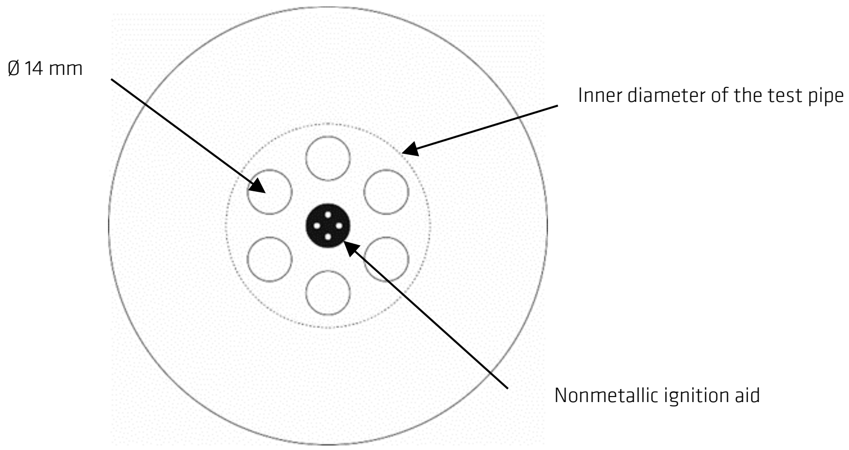

Figure 1: Preparation of the conductive flat gasket material

For all other tests, the material was removed from the carrier and was cut into parts of ca. 1 mm to 2 mm in edge length.

4 Tests

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

The test method is described in annex 1.

Based on customers information on the intended maximum use pressure, the autogenous ignition temperature test was performed at a final oxygen pressure of approximately 160 bars.

4.1.1 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.

4.1.2 Results

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

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

4.2 Testing for Aging Resistance in High Pressure Oxygen

The test method is described in annex 2.

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. Based on customers information on the intended maximum use pressure, the autogenous ignition temperature test was performed at a final oxygen pressure of approximately 160 bars and at a temperature of 275 °C.

4.2.1 Assessment Criterions

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.

4.2.2 Results

4.2.2.1 Testing for Change in Mass or Physical Appearance

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	275	160	- 1.0

After aging, the test sample was apparently unchanged and lost 1.0 % in mass.

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

The test method is described in annex 1. The AIT test of the aged material was performed at same conditions as described in chapter 4.1.

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

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

4.3 Testing of Gaskets for Flanges in High Pressure Oxygen

The test method is described in annex 3.

According to the use conditions given by the customer, the flange test was carried out at a final oxygen pressure of 160 bars and at a temperature of 250 °C.

4.3.1 Assessment Criterion

If after artificial ignition only those parts of the gasket burn that project into the pipe and the fire is not transmitted to the flanges, and if the gasket does not burn between the flange faces and the flange connection is still gas tight, there are no objections regarding technical safety to use the gasket under the conditions tested. Such a positive result has to be confirmed in four additional tests.

If, however, the gasket burns between the flange faces or the flange connection becomes un-tight, the gasket material has not passed the test. In this case, the test may be continued at a lower temperature or oxygen pressure after consultation with the customer.

4.3.2 Results

Test Number	Temperature [°C]	Oxygen Pressure [bar]	Notes
1	250	160	All parts of the gasket burn that project into the pipe. The flange faces remain undamaged. The flange connection remains gas tight.
2	250	160	Same behavior of test sample as in test no. 1
3	250	160	Same behavior of test sample as in test no. 1
4	250	160	Same behavior of test sample as in test no. 1
5	250	160	Same behavior of test sample as in test no. 1

In five tests at 250 °C and at a final oxygen pressure of 160 bars, only those parts of the disk burn that project into the pipe. In all tests, the fire is neither transmitted to the steel nor does the sample burn between the flange faces. The flange remains gas tight. After testing, the samples exhibit a thickness of 1.9 mm.

4.4 Testing for Reactivity with Liquid Oxygen on Mechanical Impact

The test method is described in annex 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	1.00	750	Reaction on 2nd single test
2	0.83	625	Reaction on 4th single test
3	0.67	500	Reaction on 7th single test
4	0.50	375	No reaction in a series of 10 single tests

At a drop height of 0.50 m (impact energy 375 Nm) no reaction of the sample with liquid oxygen could be detected in a series of 10 single tests.

5 Summary of the Test Results

At a mean final oxygen pressure p_F of 164 bars, the test sample has an autogenous ignition temperature that is greater than 500 °C.

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

The investigation of the burning behavior of disks of the gasket material in a standard flange showed that at 250 °C and an oxygen pressure of 160 bars only those parts of the sample burn that project into the pipe. The sample does not burn between the flange faces. In all cases the flange connection remained gas tight.

Testing of the material for reactivity to mechanical impacts in liquid oxygen showed that no reaction could be detected in a series of 10 single tests at an impact energy of 375 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 extended uncertainty is 0.8K (according to the calibration protocol from January 7, 2021) for the temperature measuring system, and the uncertainty is 0.3 bars (according to the calibration protocol from January 7, 2021) for the used pressure measuring system.

For the test in chapter 4.2, the extended uncertainty is 1 K (according to the calibration protocol from January 27, 2021) for the temperature measuring system, and the uncertainty is 0.2 bars (according to the calibration protocol from January 27, 2021) for the used pressure measuring system.

For the test in chapter 4.3, the extended uncertainty is 2.8 K (according to the calibration protocol from September 5, 2022) for the temperature measuring system, and the uncertainty is 0.7 bars (according to the calibration protocol from September 8, 2022) 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

The Graphit-based gasket material SIGRAFLEX®UNIVERSAL V20010C2I, batch 2201060201, shall be used for gaseous oxygen service at temperatures up to 250 °C and at pressures up to 160 bars as well as for use 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 Graphit-based gasket material SIGRAFLEX®UNIVERSAL V20010C2I, batch 2201060201, for gaseous oxygen service at following use conditions only:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
250	160

Based on the test results and according to BAM' s standard "Testing for Reactivity with Liquid Oxygen on Mechanical Impact", there are also no objections with regard to technical safety to use the Graphit-based gasket material SIGRAFLEX®UNIVERSAL V20010C2I, batch 2201060201, in liquid oxygen service. In this case, a limitation to a particular pressure range is not necessary as compression of liquid oxygen causes no significant change in concentration and therefore has no considerable influence on the reactivity of the material.

Bundesanstalt für Materialforschung und -prüfung

9 Comments

This safety-related investigation considers the fact, that rapid oxygen pressure changes - so-called oxygen pressure surges – can be safely excluded in usage, and that direct contact of the material with liquid oxygen and mechanicals impacts cannot be safely excluded in usage. In addition, the safety related investigation considers the fact, that the material shall be used in gaseous oxygen service at temperatures greater than 60 °C.

The content of the test report refers exclusively to the test sample of the material SIGRAFLEX®UNIVERSAL V20010C2I, batch 2201060201.

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.

The product may be used 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.

Bundesanstalt für Materialforschung und -prüfung (BAM)
12200 Berlin

January 23, 2023

Division 2.1 "Safety of Energy Carriers"

by order

Dr. Thomas Kasch Study Director	Dr. Kai Holtappels Head of Division

This document was created electronically and is valid without a signature.

Distribution list: 1st copy: SGL Carbon GmbH
2nd copy: BAM - Division 2.1

Annex 1

Determination of the Autogenous Ignition Temperature of Nonmetallic Materials in High Pressure Oxygen (V 2023-01)

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 cm^3. 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 bars, and the maximum test temperature is 500 °C.

Annex 2

Testing for Aging Resistance in High Pressure Oxygen (V 2023-01)

A sample with known mass is exposed to high-pressure oxygen at elevated temperature in an autoclave for 100 hours. The test temperature is usually 25 ° C above the operating temperature.

This test shows whether the sample gradually reacts with oxygen and / or other noticeable changes occur. Criteria for material resistance to oxygen under the respective test conditions are - taking into account the tolerances specified in the test reports - the retention of the external properties of the sample, the comparison of the sample mass and the autogenous ignition temperature of the material before and after aging.
The maximum test conditions of the test facility are 250 bars and 325 °C.

Annex 3

Testing Nonmetallic Gaskets for Flanges in Oxygen Steel Pipings (V 2023-01)

The test facility mainly consists of two DN 65 PN 160 steel pipes, each 2 m in length, with corresponding standard flanges welded to each pipe. The customer provides the nonmetallic gasket material in form of disks. Using this disk, the standard flanges are flanged gas tight.

The test facility is heated to the intended maximum use temperature and pressurized to the intended maximum use pressure with oxygen. Thereafter, the part of the nonmetallic gasket material is ignited, which projects into the pipe.

The nonmetallic gasket material passes the test, if only that part of the gasket burns that projects in to the pipe, the material does not burn between the flange faces more than 2 mm, and the flange connection is still gas tight. The test is finished, if there is no reaction of the material detected in five single tests.

If a reaction occurs and after consultation the customer, testing the nonmetallic gasket material can be continued for use at lower operating conditions.

For this test, the maximum test pressure is 160 bars, and the maximum test temperature is 300 °C.

Annex 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.