



TEST REPORT

on Testing a Nonmetallic Material for Reactivity with Oxygen

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Customer	SGL Carbon GmbH Werner-von-Siemens-Str. 18 86405 Meitingen Germany
Date of Request	July 19, 2016
Reference	GMS-EG HOCHDRUCK-PRO
Receipt of Signed Contract	August 9, 2016
Test Samples	Sigraflex Hochdruck Pro V20011Z3I-P, Thickness 2 mm, undisclosed batch; BAM Auftrags-Nr.: 2.1/53 239
Receipt of Samples	August 29, 2016
Test Date	September 7, 2016 to February 3, 2017
Test Location	BAM – Division 2.1 „Gases, Gas Plants“; building no. 41
Test Procedure or Requirement According to	DIN EN 1797 und ISO 21010 “Cryogenic Vessels - Gas/Material Compatibility“; Annex of code of practice M 034-1 (BGI 617-1)
(in the current version at test time)	“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“

All pressures of this report are excess pressures.
This test report consists of page 1 to 8 and annex 1 to 4.

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

2015-06 / 2015-09-17

1 Documents and Test Samples

The following documents and samples were submitted to BAM:

- 1 Test application
„Testing and evaluating the sealing material of the gasket Sigraflex Hochdruck Pro V20011Z3I-P, Thickness 2 mm, undisclosed batch, for gaseous oxygen service at 160 bar and 300 °C and for liquid oxygen service.“
- 1 Safety Data Sheet Sigraflex Hochdruck
(OB_MEI2045002, 5 Pages, date of issue January 01, 2016)
- 1 Material Data Sheet
Sigraflex Hochdruck Pro TDS HOCHDRUCK_PRO_Sheet_DE.01
(4 Pages, date of issue 08.2016)
- 18 Sigraflex Hochdruck Pro V20011Z3I-P, Thickness 2 mm, undisclosed batch,
Dimension: Outer Diameter Ø 140 mm, Thickness 2 mm
Color of the sealing material: Grey, purple printed

2 Applied Test Methods for Evaluating the Technical Safety

Tests on ignition sensitivity to gaseous oxygen impacts were not carried out because oxygen pressure impacts can be safely excluded in the intended service according to the information by the customer.

The nonmetallic material is intended for use as a sealing material in gaskets for gaseous oxygen service at 160 bar and temperatures up to 300 °C and for liquid oxygen service.

2.1 Determination of the Autogenous Ignition Temperature in High Pressure Oxygen

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

The autogenous ignition temperature (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 the Aging Behavior 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 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 inside 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 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 Preparation of Samples

To test the conductive gasket material five discs with a diameter of 140 mm were cut from the samples. The discs were prepared as shown in figure 1.

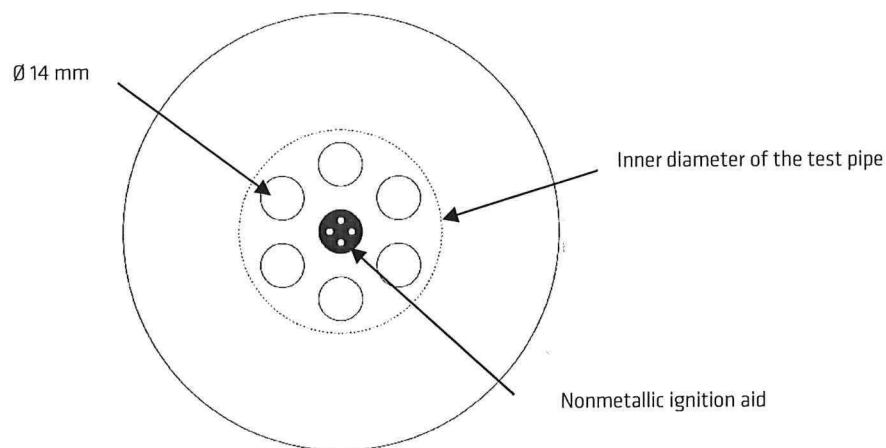


Figure 1: Preparation of the conductive material

For the other tests, only the nonmetallic material of the gasket was used and cut into pieces of approximately 1 mm^3 up to 2 mm^3 .

4 Tests

4.1 Determination of the Autogenous Ignition Temperature in High Pressure Oxygen

The test method is described in annex 1. It was performed with a final oxygen pressure of approximately 160 bar according to the requirement by the customer.

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.	Initial Oxygen Pressure p_i [bar]	Final Oxygen Pressure p_f [bar]	AIT [°C]
1	62	167	> 500
2	62	168	> 500
3	62	166	> 500
4	62	166	> 500
5	62	167	> 500

Up to temperatures of 500 °C, no ignition of the sample could be detected in five tests with initial oxygen pressures of $p_i = 50$ bar. The final oxygen pressure p_f was approximately 167 bar.

This test can only be performed up to 500 °C. This equals the maximum working temperature of the test equipment.

4.2 Aging Behavior

The test method is described in annex 2. In general, the aging test is carried out at the maximum operating pressure and at an elevated temperature, which is 25 °C above the maximum operating temperature. In this case, the aging test was carried out at 160 bar and at 325 °C.

4.2.1 Assessment Criterion

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 samples 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 Change of Mass or Physical Appearance

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	325	160	- 4

After aging, the test sample was apparently unchanged and lost 4 % 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 under the same conditions as described in chapter 4.1 of the non-aged material.

Test No.	Initial Oxygen Pressure p_i [bar]	Final Oxygen Pressure p_f [bar]	AIT [°C]
1	62	166	> 500
2	62	166	> 500
3	62	166	> 500
4	62	166	> 500
5	62	167	> 500

Up to temperatures of 500 °C, no ignition of the sample could be detected in five tests with initial oxygen pressures of $p_i = 62$ bar. The final oxygen pressure p_f was approximately 166 bar. This shows that, also the aged sample does not ignite at temperatures up to 500 °C.

This test can only be performed up to 500 °C. This equals the maximum working temperature of the test equipment.

4.3 Testing of Gaskets for Flanges in High Pressure Oxygen

The test method is described in annex 3. Based on the specified use conditions by the customer the flange test was performed at a final oxygen pressure of approximately 160 bar and 300 °C.

4.4.1 Assessment Criterion

If 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 flanges and the flange connection is still gas tight there are no objections with regard to 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 flange connection becomes un-tight during a test, e. g., because of softening or burning of the gasket, the test has to be continued at a lower temperature and oxygen pressure until a positive test result is reached in five tests, as mentioned above.

4.4.2 Results

Test Number	Temperature [°C]	Oxygen Pressure [bar]	Notes
1	300	160	Only those parts of the gasket burn that project into the pipe. The flange connection remains gas-tight.
2	300	160	same behavior as in test no. 1
3	300	160	same behavior as in test no. 1
4	300	160	same behavior as in test no. 1
5	300	160	same behavior as in test no. 1

In five tests at 300 °C and 160 bar oxygen pressure, only those parts of the gasket burn that project into the pipe. The fire is neither transmitted to the steel nor does the gasket burn between the flanges. The flange remains gas-tight. After the tests the sample had a thickness of approximately 1.9 mm in the area of the sealing face.

4.3 Reactivity Testing with Liquid Oxygen on Mechanical Impact

The test method is described in annex 4.

4.3.1 Assessment Criterion

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

4.3.2 Results

Test No.	Drop Height [m]	Impact Energy [Nm]	Reaction
1	0.83	625	no reaction
2	1.00	750	no reaction
3	1.00	750	no reaction
4	1.00	750	no reaction
5	1.00	750	no reaction
6	1.00	750	no reaction
7	1.00	750	no reaction
8	1.00	750	no reaction
9	1.00	750	no reaction
10	1.00	750	no reaction
11	1.00	750	no reaction

At a drop height of 1.00 m (impact energy 750 Nm), in ten separate tests, no reaction of the sample with liquid oxygen could be detected.

5 Summary and Evaluation

It is intended to use the product Sigraflex Hochdruck Pro V20011Z3I-P, Thickness 2 mm, undisclosed batch, as gasket in components for gaseous oxygen service and liquid oxygen service.

At a temperature of 325 °C and an oxygen pressure of 160 bar, the sealing material proved to be insufficient aging resistant. Please note, that the insufficient aging behavior can limit the practical use of the gasket.

On basis of the test results and the note on the above-mentioned aging behavior, there are no objections with regard to technical safety to use the mentioned gasket with a maximum thickness of 2 mm in flange connections made of copper, copper alloys or steel at following conditions:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
300	160

This applies to flat face flanges, male/female flanges, and flanges with tongue and groove.

Based on the test results, there are also no objections with regard to technical safety to use the gasket Sigraflex Hochdruck Pro V20011Z3I-P, Thickness 2 mm, undisclosed batch, for 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.

6 Comments

This safety evaluation considers the fact, that rapid oxygen pressure changes - so-called oxygen pressure surges - can be excluded. However, direct contact of the material with liquid oxygen and mechanical impacts cannot be safely excluded in usage

This evaluation is based exclusively on the results of the tested sample of a particular batch.

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 for gaseous and 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.

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12200 Berlin**

February 17, 2017

Division 2.1 "Gases, Gas Plants"

By order



Dipl.-Ing. Peter Hartwig

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