BAM

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# TEST REPORT

on Testing a Nonmetallic Material for Reactivity with Oxygen

| | |
|---|---|
| **Reference Number** | 18026661 E |
| **Our Reference** | 02-3527 |
| **Copy** | 1. copy of 2 copies |
| **Customer** | SGL Carbon GmbH<br>Werner-von-Siemens-Str. 18<br>86405 Meitingen<br>Germany |
| **Date of Request** | June 15, 2018 |
| **Your Reference** | Sigraflex Hochdruck BAM 2018 |
| **Receipt of Signed Contract** | July 5, 2018 |
| **Test Samples** | Gasket material Sigraflex® Hochdruck V20011Z3I, undisclosed batch; |
| **Receipt of Samples** | July 10, 2018 |
| **Test Date** | August 1 to October 26, 2018 |
| **Test Location** | BAM – Division 2.1 „Gases, Gas Plants"; building no. 41 |
| **Test Procedure or Requirement according to**<br><br>(in the current version) | DIN EN 1797 und ISO 21010<br>"Cryogenic Vessels - Gas/Material Compatibility";<br>Annex of code of practice M 034-1 (BGI 617-1)<br>"List of nonmetallic materials compatible with oxygen", by German Social Accident Insurance Institution for the raw materials and chemical industry;<br>TRGS 407 Technical Rules for Hazardous Substances<br>"Tätigkeiten mit Gasen - Gefährdungsbeurteilung"<br>chapter 3 "Informationsermittlung und Gefährdungsbeurteilung" and<br>chapter 4 "Schutzmaßnahmen bei Tätigkeiten mit Gasen" |

All pressures of this report are excess pressures.
This test report consists of page 1 to 10 and annex 1 to 4.



**2015-06** / 2015-09-17

2015-06



TEST REPORT

## 1 Documents and Test Samples

The following documents and samples were submitted to BAM:

1 Test application
Safety-related investigation of the gasket material Sigraflex® Hochdruck V20011Z3I, undisclosed batch, for flanges in gaseous oxygen service at temperatures up to 300 °C and pressures up to 160 bars and for liquid oxygen service

1 Safety Data Sheet Sigraflex® Hochdruck
(6 pages, SGL Carbon GmbH, Revisions-Nr. 1.03, date of issue: January 10, 2017)

15 Disks Sigraflex® Hochdruck V20011Z3I, undisclosed batch
Dimensions:
Diameter 140 mm, Thickness 2 mm
Color: Grey, labeled with "SIGRAFLEX® HOCHDRUCK"


## 2 Applied Test Methods

The gasket material Sigraflex® Hochdruck V20011Z3I, undisclosed batch, is a flat gasket material intended for use in flanges on components for gaseous oxygen service at pressures up to 160 bars and temperatures up to 300 °C and 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. For sealing materials in flange connections, the safety margin between AIT and maximum use temperature is only 50 °C because of the particular mounting situation.

## 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 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

As the gasket material is electrically conductive, the disks were prepared as shown in figure 1. A nonmetallic ignition aid, ignitable by an electrical filament, was placed into the central bore of the disk to help ignite the disk.

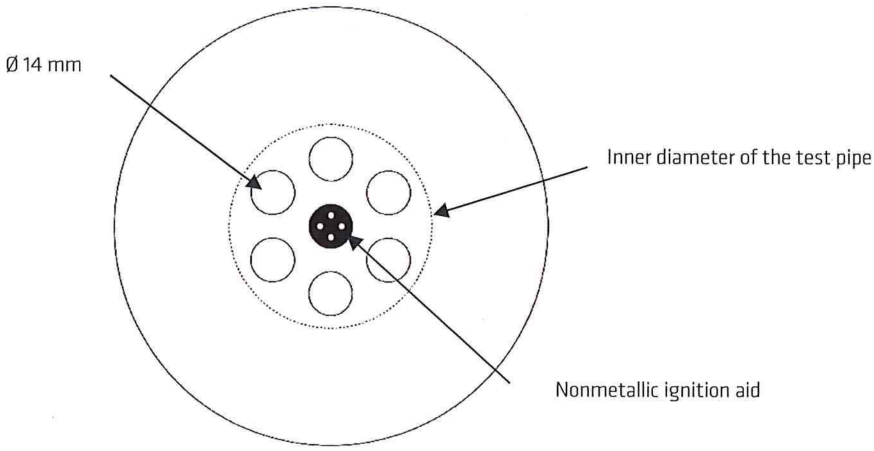


Figure 1: Preparation of the conductive flat gasket material

For all other tests, the graphite was separated from the metal foils and cut into pieces of about 1 mm to 2 mm 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.

The AIT determination was performed at a final oxygen pressure of approximately 160 bars according to the intended use conditions given 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 | 165 | > 500 |
| 2 | 62 | 162 | > 500 |
| 3 | 62 | 166 | > 500 |
| 4 | 62 | 163 | > 500 |
| 5 | 62 | 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 is also the maximum operating temperature of the test apparatus. 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 an elevated temperature, that is 25 °C above the maximum operating temperature. In this case, the test was carried out at a final oxygen pressure of 160 bars and a temperature of 325 °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 | 325 | 160 | -2.0 |

After aging, the test sample was apparently unchanged but had lost 2.0 % 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 under the same conditions as described in chapter 4.1.

| Test No. | Initial Oxygen Pressure $p_I$ [bar] | Final Oxygen Pressure $p_F$ [bar] | AIT [°C] |
|---|---|---|---|
| 1 | 62 | 164 | > 500 |
| 2 | 62 | 163 | > 500 |
| 3 | 62 | 163 | > 500 |
| 4 | 62 | 165 | > 500 |
| 5 | 62 | 163 | > 500 |

In five separate tests, with a mean final oxygen pressure of 163 bars, no ignition of the aged sample could be detected up to temperatures of 500 °C. This temperature is also the maximum operating temperature of the test apparatus. Consequently, the AIT of the aged 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 use conditions given by the customer, the flange test was carried out at a final oxygen pressure of 160 bars and a temperature of 300 °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 | 300 | 160 | All parts of the gasket burn that project into the pipe. The flange faces remain undamaged. The flange connection remains gas-tight. |
| 2 | 300 | 160 | Same behavior of test sample as in test no. 1 |
| 3 | 300 | 160 | Same behavior of test sample as in test no. 1 |
| 4 | 300 | 160 | Same behavior of test sample as in test no. 1 |
| 5 | 300 | 160 | Same behavior of test sample as in test no. 1 |

In five tests at 300 °C and 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 2.1 mm.

### 4.4 Testing for Reactivity with Liquid Oxygen on Mechanical Impact

The test method is described in annex 4.

#### 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 | No reaction in 1 single test |
| 2 | 1.00 | 750 | No reaction in a series of 10 single tests |

At a drop height of 1.00 m (impact energy 750 Nm) no reactions of the sample with liquid oxygen could be detected in a series of 10 single tests.

## 5 Summary of the Test Results

At a final oxygen pressure $p_F$ of 160 bars, the test sample has an autogenous ignition temperature that is greater than 500 °C.

The material is sufficient aging resistant at 325 °C and 160 bars oxygen pressure.

The investigation of the burning behavior of disks of the gasket material in a standard flange showed that at 300 °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 reactions could be detected in a series of 10 single tests at an impact energy of 750 Nm.

## 6 Opinion and Interpretation

The gasket material Sigraflex® Hochdruck V20011Z3I, undisclosed batch, shall be used as a flat gasket material in flanges on components for gaseous and liquid oxygen service.

On basis of the test results, the requirements for sealing materials, described in annex 1 of code of practice M034, annex 2 of code of practice M034-1, Technical Rules for Hazardous Substances TRGS 407 and BAM´s safety philosophy, there are no objections regarding technical safety, to use the nonmetallic flat gasket material Sigraflex® Hochdruck V20011Z3I, undisclosed batch, with a maximum thickness of 2 mm in flange connections made of copper, copper alloys or steel for gaseous oxygen service at following operating 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 nonmetallic flat gasket material Sigraflex® Hochdruck V20011Z3I, 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.

## 7 Comments

This safety-related investigation considers the fact, that rapid oxygen pressure changes - so-called oxygen pressure surges – can be safely excluded in usage.

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

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 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.

**Bundesanstalt für Materialforschung und -prüfung (BAM)**
**12200 Berlin**

November 7, 2018

Division 2.1 "Gases, Gas Plants"

By order

Dr. Thomas Kasch

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