Report

on Testing a Nonmetallic Material for Reactivity with Oxygen

Bundesanstalt für Materialforschung und -prüfung

Reference Number	2-1432/2014 E
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Customer	SGL Carbon GmbH Werner-von-Siemens-Str. 18 86405 Meitingen Germany
Order Date	June 10, 2014, and order extension of July 15, 2014
Reference	Delivery No.: 31015513
Receipt of Order	June 12 and July 15, 2014
Test Samples	Sigraflex® Folie Typ E, undisclosed batch, for use as a sealing material in piping, valves and fittings or other components at temperatures up to 250 °C and for use as a gasket material for gaseous oxygen service at temperatures up to 250 °C and 130 bar and for liquid oxygen service; BAM Order-No.: 2.1/52 150
Receipt of Samples	June 11, 2014
Test Date	July 2 to September 5, 2014
Test Location	BAM - Working Group "Safe Handling of Oxygen"; building no. 41, room no. 073 and no. 120
Test Procedure or Requirement According to	DIN EN 1797: 2002-02 "Cryogenic Vessels - Gas/Material Compatibility" ISO 21010: 2004-07 "Cryogenic Vessels - Gas/Material Compatibility" Annex of pamphlet M 034-1 (BGI 617-1) "List of nonmetallic materials compatible with oxygen by BAM Federal Institute for Material Research and Testing.", by Berufsgenossenschaft Rohstoffe und chemische Industrie, Edition: September 2011 TRGS 407 Technical Rules for Hazardous Substances "Tätigkeiten mit Gasen - Gefährdungsbeurteilung" chapter 3 "Informationsermittlung und Gefährdungsbeurteilung" and chapter 4 "Schutzmaßnahmen bei Tätigkeiten mit Gasen" Edition: June 2013

All pressures of this report are excess pressures.
This test report consists of page 1 to 7 and annex 1 to 5.

1 Documents and Test Samples

The following documents and samples were submitted to BAM:

1 Test Application
1 Material Data Sheet (2 pages, date of issue: 03/2011)
1 Safety Data Sheet
(7 pages, revision no. 1.02, date of issue: April 24, 2014)
15 Disks Sigraflex® Folie Typ E, undisclosed batch
Outer-Ø: 140 mm; Thickness: 0.5 mm
Color: Grey

2 Test Methods

To evaluate the compatibility of Sigraflex® Folie Typ E, undisclosed batch, for use as a sealing material in piping, valves and fittings or other components for gaseous oxygen service at temperatures up to 250 °C, the ignition sensitivity to gaseous oxygen impacts at 60 °C, at 200°C and at 250 °C, a determination of the autogenous ignition temperature (AIT) and an investigation of the aging resistance in high pressure oxygen were carried out.

To evaluate the compatibility of Sigraflex® Folie Typ E, undisclosed batch, for use as a gasket material for gaseous oxygen service at temperatures up to 250 °C and 130 bar, in addition a flange test at 250 °C and 130 bar was carried out.

The compatibility of Sigraflex® Folie Typ E, undisclosed batch, with liquid oxygen was tested by its reactivity with liquid oxygen on mechanical impact.

3 Results

3.1 Ignition Sensitivity to Gaseous Oxygen Impacts

The test method is described in annex 1.

Results:

Sample Temperature t_a [°C]	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	Reaction on Impact
60	1	450	no reaction*
60	1	450	ignition on 1. impact
60	1	440	ignition on 4. impact
60	1	430	ignition on 2. impact
60	1	420	no reaction*
60	1	420	ignition on 1. impact

* within a series of five consecutive impacts

Sample Temperature t_a [°C]	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	Reaction on Impact
60	1	410	no reaction*
60	1	410	ignition on 1. impact
60	1	400	no reaction*
60	1	400	ignition on 1. impact
60	1	390	no reaction*
60	1	390	ignition on 1. impact
60	1	380	ignition on 3. impact
60	1	370	no reaction*
60	1	370	ignition on 1. impact
60	1	360	no reaction*
60	1	360	ignition on 1. impact
60	1	350	ignition on 1. impact
60	1	340	ignition on 2. impact
60	1	320	no reaction*
60	1	320	ignition on 1. impact
60	1	300	no reaction*
60	1	300	ignition on 1. impact
60	1	280	no reaction*
60	1	280	ignition on 1. impact
60	1	270	no reaction*
60	1	270	no reaction*
200	1	270	no reaction*
200	1	270	no reaction*
250	1	270	no reaction*
250	1	270	ignition on 1. impact
250	1	250	no reaction*
250	1	260	ignition on 4. impact
250	1	250	no reaction*

* within a series of five consecutive impacts

In two separate tests, each consisting of a series of five consecutive impacts, no reactions of Sigraflex® Folie Typ E, undisclosed batch, with oxygen could be observed at a final oxygen pressure p_F of 270 bar and at temperatures of 60 °C and 200 °C.

In two separate tests, each consisting of a series of five consecutive impacts, no reactions of Sigraflex® Folie Typ E, undisclosed batch, with oxygen could be observed at a final oxygen pressure p_F of 250 bar and at a temperature of 250 °C.

3.2 Autogenous Ignition Temperature (AIT)

As the maximum operating pressure of Sigraflex® Folie Typ E, undisclosed batch, for use as a gasket material is 130 bar and in accordance with the costumer, the test was performed at a final oxygen pressure of approximately 130 bar. The test method is described in annex 2.

Results:

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	50	134	> 500
2	50	133	> 500
3	50	134	> 500
4	50	134	> 500
5	50	133	> 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 133 bar.

3.3 Artificial Aging

The test method is described in annex 3.

Results:

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	275	130	± 0

After aging of the test sample at 275 °C and 130 bar oxygen pressure, the test sample was apparently unchanged. The mass of the test sample did not change.

3.3.1 AIT after Artificial Aging

The test method is described in annex 2.

Results:

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	50	131	> 500
2	50	133	> 500
3	50	133	> 500
4	50	134	> 500
5	50	131	> 500

Up to temperatures of 500 °C, no ignition of the aged sample could be detected in five tests with initial oxygen pressures of p_I = 50 bar. The final oxygen pressure p_F was approximately 132 bar. This shows, that, as the non-aged sample, also the aged sample did not ignite at temperatures up to 500 °C.

3.4 Flange Test

According to the above-mentioned operating conditions of Sigraflex® Folie Typ E, undisclosed batch, for use as a gasket material the flange test was performed at 130 bar oxygen pressure and 250 °C. The test method is described in annex 4.

Results:

Test No.	Oxygen Pressure [bar]	Temperature [°C]	Notes
1	130	250	Only those parts of the gasket burn that project into the pipe. The flange remains gas-tight.
2	130	250	Same behavior as in test no. 1
3	130	250	Same behavior as in test no. 1
4	130	250	Same behavior as in test no. 1
5	130	250	Same behavior as in test no. 1

In five tests at 130 bar oxygen pressure and 250 °C, 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.

3.5 Reactivity with Liquid Oxygen on Mechanical Impact

In general, 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. The test method is described in annex 5.

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

4 Summary and Evaluation

Up to temperatures of 500 °C, no ignition of the nonmetallic material Sigraflex® Folie Typ E, undisclosed batch, could be detected in five tests with final oxygen pressures of approximately p_F = 133 bar.

At a temperature of 275 °C and an oxygen pressure of 130 bar, the material proved to be aging resistant. The mass of the test sample did not change.

Up to temperatures of 500 °C, no ignition of the aged material could be detected in five tests with final oxygen pressures of approximately p_F = 132 bar. This shows, that, as the non-aged sample, also the aged sample did not ignite at temperatures up to 500 °C.

Generally, in evaluating nonmetallic materials for oxygen service, a safety margin of 100 °C between AIT and maximum operating temperature is being considered for safety reasons. As the maximum operating temperature is 250 °C, Sigraflex® Folie Typ E, undisclosed batch, fulfills this criterion.

According to DIN EN 1797: 2002-02 „Cryogenic Vessels - Gas/Material Compatibility" and to ISO 21010: 2004-07 „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.

On basis of the above-mentioned criterion and the test results, there are no objections with regard to technical safety, to use Sigraflex® Folie Typ E, undisclosed batch, as a sealing material in piping, valves and fittings, or other components for gaseous oxygen service at following operating conditions:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
60	270
> 60 to 250	130

On basis of the test results and the results of the flange test, there are also no abjections with regard to technical safety, to use Sigraflex® Folie Typ E, undisclosed batch, as a gasket material with a maximum thickness of 0.5 mm in flange connections made of copper, copper alloys or steel at following conditions:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
60	130

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

According to the BAM-Standard "Testing for Reactivity with Liquid Oxygen on Mechanical Impact", described in annex 5, there are also no objections with regard to technical safety to use Sigraflex® Folie Typ E, undisclosed batch, in valves and fittings or other components 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.

5 Comments

The test results refer exclusively to the tested batch of Sigraflex® Folie Typ E, undisclosed 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.

For safety reason, it is not justifiable to use our BAM reference number without additional information about the purpose of use and the maximum operating conditions.

It shall be clear that the product may only be used for gaseous oxygen service and liquid oxygen service. The maximum safe oxygen pressure of the product and its maximum operating temperature as well as other restrictions in use shall be given.

BAM Federal Institute for Materials Research and Testing
12200 Berlin, September 15, 2014

Division 2.1
"Gases, Gas Plants"

On behalf of

Dr. Thomas Kasch

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