BAM

Bundesanstalt für
Materialforschung
und -prüfung

TEST REPORT

On Testing a Nonmetallic Material for Reactivity with Gaseous and Liquid Oxygen

12200 Berlin, Germany
P: +49 30 8104-0
F: +49 30 8104-7 2222

BAM reference	15019178 II E
Copy	1st copy of 2 copies
Customer	SGL Carbon GmbH Werner-von-Siemens-Straße 20 86405 Meitingen Germany
Order date	March 23, 2015
Reference	Order No.: 112-10-45827456
Receipt of order	April 1, 2015
Test samples	Sigraflex® APX, undisclosed batch; BAM Order-No.: 2.1/52 578
Receipt of samples	April 2, 2015
Test date	April 16 to December 4, 2015
Test location	BAM - Working Group "Safe Handling of Oxygen"; building no. 41, room no. 073 and no. 120
Test procedure according to	ISO 21010:2014 and DIN EN 1797:2002-02 "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, Edition: March 2014; 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
Safety Related Maximum Operating Conditions	See chapter 4 "Summary and Evaluation"

All pressures of this report are excess pressures.
This test report consists of page 1 to 8 and annexes 1 to 5.

1 Documents and Test Samples

The following documents and samples were submitted to BAM:

1 Test application
"Testing and evaluating the compatibility of the nonmetallic material Sigraflex® APX, undisclosed batch, for use as a sealing material in components and flanged connections for gaseous oxygen service at temperatures up to 300 °C and at pressures up to 160 bar as well as for use in liquid oxygen service"

1 Safety Data Sheet
(5 pages, version 1.0.1, date of issue: July 25, 2014)

20 Disks of graphite based Sigraflex® APX, undisclosed batch
Outer-Ø: 140 mm; Thickness: 0.5 mm
Color: Grey

2 Test Methods

To evaluate the compatibility of the nonmetallic material Sigraflex® APX, undisclosed batch, for use as a sealing material gaseous oxygen service at temperatures up to 300 °C and at pressures up to 160 bar, testing of ignition sensitivity to gaseous oxygen impacts at 60 °C, 250 °C and 300 °C, a determination of the autogenous ignition temperature (AIT), an investigation of the aging resistance, and a flange test were carried out.

The compatibility of the material 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	150	no reaction*
60	1	180	no reaction*
60	1	250	no reaction*
60	1	300	ignition on 1. impact
60	1	290	ignition on 4. impact
60	1	280	no reaction*
60	1	280	no reaction*
250	1	280	no reaction*
250	1	280	no reaction*
300	1	280	no reaction*
300	1	280	no reaction*

* within a series of five consecutive impacts

In two separate tests, each consisting of a series of five consecutive impacts, no reaction of the test sample with oxygen could be detected at a final oxygen pressure p_F of 280 bar and at temperatures of 60 °C, 250 °C and 300 °C.

3.2 Autogenous Ignition Temperature (AIT)

Based on the specified maximum operating pressure of the nonmetallic material, the autogenous ignition temperature test was performed at a final oxygen pressure of approximately 160 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	62	165	> 500
2	62	165	> 500
3	62	165	> 500
4	62	165	> 500
5	62	164	> 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 165 bar. The autogenous ignition temperature in high pressure oxygen can only be determined up to 500 °C. This equals the maximum operating temperature of the test equipment.

3.3 Artificial Aging

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. The test method is described in annex 3.

Results:

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	325	160	- 1.2

After aging of the test sample at 325 °C and at 160 bar oxygen pressure, the test sample was apparently unchanged. The sample lost 1.2 % in mass.

3.3.1 AIT after Artificial Aging

The same test conditions as in chapter 3.2 were used for determining the autogenous ignition temperature after 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	62	166	> 500
2	62	164	> 500
3	62	166	> 500
4	62	165	> 500
5	62	161	> 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 = 62 bar. The final oxygen pressure p_F was approximately 164 bar. This shows that, as the non-aged sample, also the aged sample did not ignite at temperatures up to 500 °C. The autogenous ignition temperature in high pressure oxygen can only be determined up to 500 °C. This equals the maximum operating temperature of the test equipment.

3.4 Flange Test

Based on the specified maximum operating conditions, flange testing was performed at 160 bar oxygen pressure and at a temperature of 300 °C. The test method is described in annex 4.

Results:

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

In five tests at 160 bar oxygen pressure and 300 °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	1.00	750	no reaction
2	1.00	750	no reaction
3	1.00	750	no reaction
4	1.00	750	severe
5	0.83	625	no reaction
6	0.83	625	no reaction
7	0.83	625	no reaction
8	0.83	625	severe
9	0.67	500	no reaction
10	0.67	500	no reaction
11	0.67	500	no reaction
12	0.67	500	no reaction
13	0.67	500	no reaction
14	0.67	500	no reaction
15	0.67	500	no reaction
16	0.67	500	no reaction
17	0.67	500	no reaction
18	0.67	500	no reaction

At a drop height of 0.67 m (impact energy 500 Nm), in ten separate tests, no reaction of the nonmetallic material with liquid oxygen could be detected.

4 Summary and Evaluation

No autogenous ignition temperature (AIT) could be determined up to 500 °C as no ignition of Sigraflex® APX, undisclosed batch, could be detected in five tests with initial oxygen pressures of p_I = 62 bar and a final oxygen pressure of p_F = 165 bar. The AIT in high pressure oxygen can only be determined up to 500 °C. That equals the maximum operating temperature of the test equipment.

At a temperature of 325 °C and an oxygen pressure of 160 bar, the material proved to be sufficient aging resistant. The sample was apparently unchanged and lost 1.2 % in mass.

No AIT of the aged material, would be determined up to 500 °C, as no ignition could be detected in five tests with initial oxygen pressures of p_I = 62 bar and a final oxygen pressure of p_F = 164 bar. This shows that, as the non-aged sample, also the aged sample did not ignite at temperatures up to 500 °C.

For safety reasons a safety margin of 100 °C between AIT and maximum operating temperature is being considered in evaluating nonmetallic materials for oxygen service. As the maximum operating temperature of the nonmetallic material is 300 °C, Sigraflex® APX, undisclosed batch, fulfills this criterion.

According to DIN EN 1797: 2002-02 „Cryogenic Vessels - Gas/Material Compatibility" and to ISO 21010: 2014 „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 abjections with regard to technical safety, to use Sigraflex® APX, undisclosed batch, as a sealing material in gaseous oxygen service at following conditions:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
60	280
> 60 up to 300	160

In addition and based on the results of the flange test, there are no abjections with regard to technical safety, to use Sigraflex® APX, undisclosed batch, as a gasket material with a maximum thickness of 0.5 mm in gaseous oxygen service 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 faces 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® APX, undisclosed batch, 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.

5 Comments

This evaluation is based exclusively on the results of the tested batch of the nonmetallic material Sigraflex® APX.

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.

It shall be clear that the product may only 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

January 7, 2016

Division 2.1
"Gases, Gas Plants"

By order

Dr. Thomas Kasch

Distribution list:	1st	copy: SGL Carbon GmbH
	2nd	copy: BAM - Division 2.1 "Gases, Gas Plants"