BAM
Bundesanstalt für
Materialforschung
und -prüfung

TEST REPORT

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

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BAM reference	2-113/2015 I E
Copy	1st copy of 2 copies
Customer	FMI S.p.A. Via Consolare 41/43 25030, Zocco di Erbusco (BS), Italy
Order date	January 7, 2015
Reference	PO 15/00318
Receipt of order	February 10, 2015
Test samples	Flexigraf FGS4 - F4RGS400160, batch 13/000002; BAM Order-No.: 2.1/52 470
Receipt of samples	January 9, 2015
Test date	February 12 to October 16, 2015
Test location	BAM - Working Group "Safe Handling of Oxygen"; building no. 41, room no. 073
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 6 and annexes 1 to 4

TEST REPORT

1 Documents and Test Samples

The following documents and samples were submitted to BAM:

1 Test application
"Testing and evaluating the compatibility of the gasket material Flexigraf FGS4 - F4RGS400160, batch 13/000002, for use in flanged connections for gaseous oxygen service at temperatures up to 250 °C and at pressures up to 150 bar as well as for liquid oxygen service"

1 Safety Data Sheet
(5 pages, version 01 / 2007, issued: February 23, 2007)

15 Disks Flexigraf FGS4 - F4RGS400160, batch 13/000002
Outer-Ø: 140 mm; Thickness: 2 mm
Color: Grey

2 Test Methods

To evaluate the compatibility of Flexigraf FGS4 - F4RGS400160, batch 13/000002, for use as a gasket material in gaseous oxygen service at temperatures up to 250 °C and at pressures up to 150 bar, a determination of the autogenous ignition temperature (AIT), an investigation of the aging resistance and a flange test were carried out.

Tests on ignition sensitivity to gaseous oxygen impacts were not carried out. According to the customer, oxygen pressure impacts in valves and fittings or in other components can be safely excluded in the intended service.

The compatibility of the material with liquid oxygen was tested by its reactivity with liquid oxygen on mechanical impact.

3 Results

3.1 Autogenous Ignition Temperature (AIT)

Based on the specified maximum operating conditions, the autogenous ignition temperature test was performed at a final oxygen pressure of approximately 150 bar. The test method is described in annex 1.

Results:

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	58	153	> 500
2	58	153	> 500
3	58	154	> 500
4	58	155	> 500
5	58	156	> 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 = 58 bar. The final oxygen pressure p_F was approximately 154 bar.

3.2 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 275 °C and at 150 bar. The test method is described in annex 2.

Results:

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	275	150	- 0.3

After aging of the test sample at 275 °C and at 150 bar oxygen pressure, the test sample was apparently unchanged. The sample lost 0.3 % in mass.

3.2.1 AIT after Artificial Aging

The same test conditions as in chapter 3.1 were used for determining the autogenous ignition temperature after aging. The test method is described in annex 1.

Results:

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	58	152	> 500
2	58	155	> 500
3	58	153	> 500
4	58	153	> 500
5	58	154	> 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 = 58 bar. The final oxygen pressure p_F was approximately 153 bar. This shows that, as the non-aged sample, also the aged sample did not ignite at temperatures up to 500 °C.

3.3 Flange Test

Based on the specified maximum operating conditions, flange testing was performed at 150 bar oxygen pressure and at a temperature of 250 °C. The test method is described in annex 3.

Results:

Test Number	Oxygen Pressure [bar]	Temperature [°C]	Notes
1	150	250	Only those parts of the gasket burn that project into the pipe, the flange connection remains gas-tight
2	150	250	same behavior as in test no. 1
3	150	250	same behavior as in test no. 1
4	150	250	same behavior as in test no. 1
5	150	250	same behavior as in test no. 1

In five tests at 150 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.

3.3 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 4.

Results:

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

Test No.	Drop Height [m]	Impact Energy [Nm]	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
19	0.67	500	no reaction

No reaction of the test sample with liquid oxygen could be detected at drop heights of 0.67 m (impact energy 500 Nm), in ten separate tests.

4 Summary and Evaluation

Up to temperatures of 500 °C, no ignition of the test sample could be detected in five tests with initial oxygen pressures of p_I = 58 bar. The final oxygen pressure p_F is 154 bar.

At a temperature of 275 °C and an oxygen pressure of 150 bar, the gasket material Flexigraf FGS4 - F4RGS400160, batch 13/000002, proved to be sufficient aging resistant. The sample lost 0.3 % in mass.

Up to temperatures of 500 °C, no ignition of the aged test sample could be detected in five tests with initial oxygen pressures of p_I = 58 bar. The final oxygen pressure p_F is 153 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, the gasket material Flexigraf FGS4 - F4RGS400160, batch 13/000002, fulfills this criterion.

On basis of the test results, there are no abjections with regard to technical safety, to use the gasket material Flexigraf FGS4 - F4RGS400160, batch 13/000002, with a maximum thickness of 2 mm in gaseous oxygen service in flange connections made of copper, copper alloys or steel at following conditions if oxygen pressure impacts can be safely excluded in the intended service:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
250	150

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 4, there are also no objections with regard to technical safety to use the gasket material Flexigraf FGS4 - F4RGS400160, batch 13/000002, 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 test results of batch 13/000002 of Flexigraf FGS4 - F4RGS400160.

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 oxygen service and for 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

October 22, 2015

Division 2.1
"Gases, Gas Plants"

by order

Dr. Thomas Kasch

Distribution list: 1st copy: FMI S.p.A
2nd copy: BAM - Division 2.1 "Gases, Gas Plants"

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Annex 1

Determination of the Autogenous Ignition Temperature in High Pressure Oxygen

A mass of approximately 0.1 g to 0.5 g of the pasty or of the divided solid sample is placed into an autoclave (34 cm^3 in volume) with a chrome/nickel lining. Liquid samples are applied onto ceramic fiber.

The autoclave is pressurized to the desired initial pressure p_I at the beginning of the test A low-frequency heater inductively heats the autoclave in an almost linear way at a rate of 110 K/min. The temperature is monitored by means of a thermocouple at the position of the sample.

The pressure in the autoclave is measured by means of a pressure transducer. Pressure and temperature are recorded. During the test, as the temperature increases, the oxygen pressure increases within the autoclave. The ignition of the sample can be recognized by a sudden rise in temperature and the final pressure p_F.

It is important to know the oxygen pressure p_F, as the autogenous ignition temperature of a material is a function of pressure. It may decrease as the oxygen pressure increases.

Annex 2

Testing for Aging Resistance in High Pressure Oxygen

A sample with known mass is exposed to high-pressure oxygen at elevated temperature in an autoclave for 100 hours. The temperature, at which the sample is aged, is at least 100 °C lower than the autogenous ignition temperature of the sample.

This test shows whether the sample gradually reacts with oxygen or whether it undergoes other visible changes. If there is no change in appearance, in mass, and in the autogenous ignition temperature of the material, it is considered aging resistant.

Annex 3

Testing of Gaskets for Flanges in Oxygen Steel Pipings

The test apparatus mainly consists of two DN 65 PN 160 steel pipes, each approximately 2 m in length, with corresponding standard flanges welded to each pipe.

Both pipes are sealed using the gasket to be tested. In case of a gasket disk its inner diameter is chosen in such a way that it projects into the pipe. If a gasket tape is under test, both ends of the tape are allowed to project into the pipe. The test apparatus is then pressurized with oxygen up to the desired test pressure. The flange is heated by heating sleeves to the test temperature, at least 50 K lower than the ignition temperature of the gasket. An electrical filament ignites that part of the gasket projecting into the pipe. If the gasket is electrically conductive, such as spiral seals or graphite foils, a nonconductive primer capsule of organic material (PTFE, rubber) is used which acts on the seal.

The gasket's behavior after ignition is important for its evaluation. If the seal burns with such a hot flame that the fire is transmitted to the steel of the flange (in most case the test apparatus is destroyed), the seal is considered unsuitable from the beginning. If only those parts of the seal burn that project into the pipe and the fire is not transmitted to the flanges and if the seal does not burn between the flanges there are no objections with regard to technical safety to use the seal under the conditions tested. Such a positive result is to confirm in four additional tests. If, however, the flanged connection becomes un-tight during a test, e. g., because of softening or burning of the seal, 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.

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Annex 4

Testing for Reactivity with Liquid Oxygen on Mechanical Impact

Approximately 0.5 g of the liquid or divided sample 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 over the sample which is then exposed to the mechanical impact of a plummet (mass = 76.5 kg). The drop height of the plummet can be varied. A steel anvil with a chrome/nickel steel plate supports the sample cup. The anvil, having a mass eight times of the plummet, is supported by four damping elements mounted on the steel frame of the test apparatus that rests on a concrete base.

A reaction of the sample with liquid oxygen is usually indicated by a flame and a more or less strong noise of an explosion. The impact energy, at which no reaction occurs, is determined in varying the drop height of the plummet. This result shall be confirmed in a series of ten consecutive tests under the same conditions. The tests are finished, if reactions can be observed at impact energies of 125 Nm or less (equivalent to a drop height of the plummet of 0.17 m or less). In this case, with regard to technical safety, the material is not suitable for liquid oxygen service.