

Report

on the Check Test of a Gasket Material for Gaseous Oxygen Service

Reference Number

II-1093/2009 IV E

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Customer

DONIT TESNIT d.o.o.
Technical Services

Cesta komandanta Staneta 38

1215 MEDVODE SLOWENIEN

Order Date

April 23, 2009

Receipt of Order

May 8, 2009

Test Samples

Gasket material GRAFILIT SP for use in flanged connections in piping, valves and fittings or other components for gaseous oxygen service up to 130 bar

and at temperatures up to 200 °C. BAM-Order No. II.1/49 626

Receipt of Samples

May 8, 2009

Test Date

September 22, 2009

Test Location

BAM Working Group "Safe Handling of Oxygen"; building no. 41, room no. 073 and no. 120

Test Procedure According to Annex of pamphlet M 034-1 (BGI 617-1)

"Liste der nichtmetallischen Materialien die von der Bundesanstalt für Materialforschung und –prüfung (BAM) zum Einsatz in Anlageteilen für Sauerstoff als geeignet

befunden worden sind.",

to pamphlet M 034 "Sauerstoff" (BGI 617) Berufsgenossenschaft der chemischen Industrie

Edition: October 2008;

according chapter 3.17 "Gleitmittel und Dichtwerkstoffe" to rule BGR 500 "Betreiben von Arbeitsmitteln" part 2, chapter 2.32 "Betreiben von Sauerstoffanlagen",

Edition: September 2008.

All pressures of this report are excess pressures. This test report consists of page 1 to 4 and Annex 1 to 3.

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In case a German version of the test report is available, exclusively the German version is binding.





















1 Documents and Test Samples

The following documents and samples were submitted to BAM:

- 1 Test Application
- 1 Safety Data Sheet
- 1 Material Data Sheet
- 15 Disks GRAFILIT SP, perforated metal sheet, both sides coated with graphite

Diameter 140 mm; Thickness 2 mm

With Imprint on one side: Tesnit GRAFILIT SP

Colour: Grey

2 Test Methods

The material GRAFILIT SP was already tested and evaluated as a gasket for use in flanged connections in piping, valves and fittings or other components for gaseous oxygen service under reference number II.1-375/98 IV, in 1998. In this case, only a check test has been performed.

3 Results

3.1 Autogenous Ignition Temperature (AIT)

The test method is described in annex 1.

Results:

Test No.	Oxygen Pressure p _a [bar]	Oxygen Pressure p _e [bar]	AIT [°C]
1	50	133	> 500
2	50	133	> 500
3	50	133	> 500
4	50	134	> 500
5	50	132	> 500

Up to temperatures of 500 °C, no ignition of the material could be detected in five tests with oxygen pressures of $p_a = 50$ bar. The oxygen pressure p_e was approximately 133 bar.

3.2 Artificial Aging

The test method is described in annex 2.

Results:

Time	Temperature	Oxygen Pressure	Mass Change
[h]	[°C]	[bar]	[%]
100	225	130	- 0,2

After aging of GRAFILIT SP at 130 bar oxygen pressure and at a temperature of 225 °C, the material was apparently unchanged. The sample lost 0,2 % in mass.

3.3 Flange Test

The test method is described in annex 3.

Results:

Number of tests	Oxygen pressure [bar]	Temperature [°C]	Notes
1	130	200	Only those parts of the gasket burn that project into the pipe. The connection remained gas-tight.
2	130	200	same behavior as in test no. 1
3	130	200	same behavior as in test no. 1
4	130	200	same behavior as in test no. 1
5	130	200	same behavior as in test no. 1

In five tests at 130 bar oxygen pressure and 200 °C only those parts of the gasket GRAFILIT SP burn that project into the pipe; the fire is neither transmitted to the steel nor does the gasket burn between the flanges. The flange connection remained gas-tight.

4 Evaluation

The product GRAFILIT SP was already tested and evaluated as a sealing material for use in flanged connections in piping, valves and fittings or other components for gaseous oxygen service under reference number II.1-375/98 IV, in 1998.

At a temperature of 225 $^{\circ}$ C and an oxygen pressure of 130 bar, the material proved to be sufficient aging resistant. The mass of the test sample lost 0,2 $^{\circ}$ C.

On basis of the former test results and the results of the check test, there are no objections with regard to technical safety to use the gasket GRAFILIT SP in flanged connections made of copper, copper alloys or steel at following conditions:

Maximum Temperature	Maximum Oxygen Pressure
200 °C	130 bar

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

This report does not cover the use of the gasket GRAFILIT SP for liquid oxygen service. A particular test for reactivity with liquid oxygen needs to be carried out to evaluate the compatibility of the gasket GRAFILIT SP with liquid oxygen.

Comments

The test results refer exclusively to the tested material.

Products that have been tested by us, and which are on the market, shall be marked according to our evaluation in the BAM test report. A label on a product saying that a BAM test has been performed and (or) citing our reference number, only, is not tolerable. The use of the product and its safe operating conditions must also be given.

It shall be clear that the product may only be used for gaseous 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.

> **BAM Federal Institute for Materials Research and Testing** 12200 Berlin, March 4, 2010

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Dr./Chr. Binder

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"Safe Handling of Oxygen"

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Annex 1

<u>Determination of the Autogenous Ignition Temperature in High Pressure Oxygen</u>

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 cm³ in volume) with a chrome/nickel lining. Liquid samples are applied onto ceramic fiber.

The autoclave is pressurized to the desired pressure p_a 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 pressure. The oxygen pressure on ignition p_e is calculated.

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

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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.