Report

on Testing a Gasket Material for Reactivity with Oxygen

BAM
Bundesanstalt für Materialforschung und -prüfung

Reference Number	2-2501/2014 I E
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Customer	Klinger AG Egliswil Webereistraße 1 5704 Egliswil Switzerland
Order Date	September 29, 2014
Reference	René Baumgartner
Receipt of Order	October 17, 2014
Test Samples	Gasket material Klinger Graphit Laminat SLS150, undisclosed batch, for use in flanged connections in piping, valves and fittings or other components for gaseous oxygen service at pressures up to 130 bar and at temperatures up to 200 °C, and for liquid oxygen service; BAM Order-No.: 2.1/52 350
Receipt of Samples	October 10, 2014
Test Date	November 27, 2014, to June 19, 2015
Test Location	BAM - Working Group "Safe Handling of Oxygen"; building no. 41, room no. 073
Test Procedure or Requirement According to	DIN EN 1797: 2002-02 "Cryogenic Vessels - Gas/Material Compatibility" ISO 21010: 2014 "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: 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

All pressures of this report are excess pressures.
This test report consists of page 1 to 6 and annex 1 to 4.

TEST REPORT

1 Documents and Test Samples

The following documents and samples were submitted to BAM:

1 Test Application

1 Safety Data Sheet
(5 pages, date of issue: December 2, 2008)

1 Material Data Sheet
(1 page, date of issue: 11.2013)

15 Disks Klinger Graphit Laminat SLS150, undisclosed batch
Metal sheet coated with nonmetallic sealing material
Outer-Ø: 140 mm; Thickness: 1.5 mm
Color of the nonmetallic sealing material: Grey

2 Test Methods

To evaluate the compatibility of the gasket material Graphit Laminat SLS150, undisclosed batch, for use in flanged connections in piping, valves and fittings or other components for gaseous oxygen service at pressures up to 130 bar and at temperatures up to 200 °C, a determination of the autogenous ignition temperature (AIT), an investigation of the aging resistance and a flange test in high pressure oxygen were carried out.

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

Prior to testing, the nonmetallic sealing material was removed from the metal sheet and was used to perform the tests.

3 Results

3.1 Autogenous Ignition Temperature (AIT)

According to the maximum operating conditions, the determination of the autogenous ignition temperature test was performed at a final oxygen pressure of approximately 130 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	50	134	> 500
2	50	134	> 500
3	50	135	> 500
4	50	136	> 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 134 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 of the material. In this case, the aging test was carried out at 130 bar and at 225 °C. The test method is described in annex 2.

Results:

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	225	130	- 0.3

After aging at 225 °C and 130 bar oxygen pressure, the test sample was apparently unchanged. The sample lost 0.3 % in mass. In general, a nonmetallic material is insufficient aging resistant, if there is a change in mass of more than 2 %.

3.2.1 AIT after Artificial 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	50	134	> 500
2	50	136	> 500
3	50	134	> 500
4	50	135	> 500
5	50	133	> 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 134 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

According to the given maximum operating conditions of Graphit Laminat SLS150, undisclosed batch, for use as a gasket material, the flange test was performed at 130 bar oxygen pressure and at 200 °C. The test method is described in annex 3.

Results:

Test No.	Oxygen Pressure [bar]	Temperature [°C]	Notes
1	130	200	Only those parts of the gasket burn that project into the pipe. The flange remains 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 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.

3.4 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	no reaction
3	1.00	750	no reaction
4	1.00	750	no reaction
5	1.00	750	no reaction
6	1.00	750	severe
7	0.83	625	no reaction
8	0.83	625	no reaction
9	0.83	625	no reaction
10	0.83	625	no reaction
11	0.83	625	no reaction
12	0.83	625	no reaction
13	0.83	625	severe
14	0.67	500	no reaction
15	0.67	500	no reaction
16	0.67	500	no reaction
17	0.67	500	severe
18	0.50	375	no reaction
19	0.50	375	no reaction
20	0.50	375	no reaction
21	0.50	375	no reaction
22	0.50	375	no reaction
23	0.50	375	no reaction
24	0.50	375	no reaction
25	0.50	375	no reaction
26	0.50	375	no reaction
27	0.50	375	no reaction

At drop heights of 0.50 m (impact energy 375 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 gasket material Klinger Graphit Laminat SLS150, undisclosed batch, could be detected in five tests with final oxygen pressures of p_F = 134 bar.

At a temperature of 225 °C and an oxygen pressure of 130 bar, the material proved to be aging resistant. The test sample lost 0.3 % in mass.

Up to temperatures of 500 °C, no ignition of the aged material could be detected in five tests with final oxygen pressures of p_F = 134 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 200 °C, the nonmetallic gasket material Klinger Graphit Laminat SLS150, undisclosed batch, fulfills this criterion.

On basis of the above-mentioned criterion and the test results, there are no objections with regard to technical safety, to use the gasket material Klinger Graphit Laminat SLS150, undisclosed batch, with a maximum thickness of 1.5 mm in flanged connections in piping, valves and fittings or other components for gaseous oxygen service at following operating conditions:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
200	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 4, there are also no objections with regard to technical safety to use Klinger Graphit Laminat SLS150, 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 Klinger Graphit Laminat SLS150.

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. 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. 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 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, July 14, 2015

Division 2.1
"Gases, Gas Plants"

On behalf of

Dr. Thomas Kasch

Copies: 1. Copy: Klinger AG Egliswil
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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 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.

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.

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.