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Bundesanstalt für Materialforschung und -prüfung

Report

on Testing of a Gasket Material for Gaseous Oxygen Service

Reference Number	II-611/2009 E
Copy	1. Copy of 2 Copies

1 Application

Customer	Rich. Klinger Dichtungstechnik GmbH & Co. KG Am Kanal 8 - 10 2352 Gumpoldskirchen AUSTRIA
Order Date	March 6, 2009
Receipt of Order	March 10, 2009
Test Samples	Gasket material KLINGER®Quantum for use in flanged connections in gaseous oxygen piping, in valves and fittings or other components for gaseous oxygen service at temperatures up to 90 °C and oxygen pressures up to 160 bar and for liquid oxygen service; BAM-Order No. II.1/ 49 565
Receipt of Samples	March 9, 2009
Test Date	March 31, 2009 to July 9, 2009
Test Location	BAM-Working Group "Safe Handling of Oxygen"; building no. 41, room no. 073
Test procedure according to	DIN EN 1797: 2002-02 „Cryogenic Vessels - Gas/Material Compatibility" 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 to 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 report consists of page 1 to 5 and annex 1 to 4.

TEST REPORT

2 Documents

The following documents and samples were submitted to BAM:

1 Application
1 Material information
14 Discs of KLINGER®Quantum
Diameter: 140 mm, thickness: 2 mm
colour: beige

3 Test Methods and Results

3.1 Autogenous Ignition Temperature (AIT)

The test method is described in annex 1.

Results:

Test No.	Initial oxygen pressure p_a [bar]	Final oxygen pressure p_e [bar]	AIT [°C]
1	121	166	133
2	121	164	125
3	121	162	120
4	121	162	122
5	121	161	118

In five tests with an initial oxygen pressure of p_a = 121 bar, an AIT of 124 °C was determined with a standard deviation of ± 6 °C. The final oxygen pressure p_e at ignition is approximately 163 bar.

3.2 Artificial Aging

The test method is described in annex 2.

Results:

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	90	160	+ 0,8

After aging of the gasket material KLINGER®Quantum at 90 °C and 160 bar oxygen pressure, the material was slightly brittle and the color was brownish. The test sample gained 0,8 % in mass.

3.2.1 AIT after Artificial Aging

The test method is described in annex 1.

Results:

Test No.	Initial oxygen pressure p_a [bar]	Final oxygen pressure p_e [bar]	AIT [°C]
1	121	175	155
2	121	176	156
3	121	173	147
4	121	173	147
5	121	180	165

In five tests with an initial oxygen pressure of p_a = 121 bar, an AIT of 154 °C was determined with a standard deviation of ± 7 °C for the aged gasket material. The final oxygen pressure p_e at ignition is approximately 175 bar.

This shows, that the AIT of the aged sample was slightly higher compared to the AIT of the non-aged sample.

3.3 Flange Test

The test method is described in annex 3.

Results:

Number of tests	Oxygen pressure [bar]	Temperature [°C]	Remarks
1	160	65	Only those parts of the gasket burn that project into the pipe.
2	160	65	Same behavior as in test no. 1.
3	160	65	Same behavior as in test no. 1.
4	160	65	Same behavior as in test no. 1.
5	160	65	Same behavior as in test no. 1.

At 160 bar oxygen pressure and 65 °C only those parts of the gasket material KLINGER®Quantum 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.

3.4 Reactivity with Liquid Oxygen on Mechanical Impact

The test method is described in annex 4.

Results:

Test No.	Drop Height [m]	Impact Energy [Nm]	Reaction
1	0,67	500	Violent ignition on 1. impact
2	0,33	250	Violent ignition on 1. impact
3	0,17	125	Violent ignition on 1. impact

At drop heights of 0,67 m, 0,33 m, and 0,17m (impact energy 500 Nm, 250 Nm, and 125 Nm), violent reactions of the material KLINGER®Quantum with liquid oxygen could be detected in all tests.

4 Evaluation

The tests have shown that the autogenous ignition temperature of the flat gasket KLINGER®Quantum is (124 ± 6) °C at 163 bar oxygen pressure.

After aging at a temperature of 90 °C and an oxygen pressure of 160 bar, the material proved to be insufficient aging resistant. The discoloration and the increase in autogenous ignition temperature after aging up to 154 °C have no influence on the oxygen compatibility.

However, because of the slight brittleness of the material after aging KLINGER®Quantum can only be used for gaseous oxygen service, if dynamic stresses on flange connections can safely be excluded.

On basis of the above-mentioned criterion and the test results and the results of the flange testing, there are no objections with regard to technical safety to use the gasket KLINGER®Quantum in flange connections made of copper, copper alloys or steel at following conditions:

Maximum Temperature	Maximum Oxygen Pressure
65 °C	160 bar

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 2, the gasket KLINGER®Quantum is not suitable for liquid oxygen service.

5 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.

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12200 Berlin, July 31, 2009

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Copies:
1. Copy: Rich. Klinger Dichtungstechnik GmbH & Co. KG
2. Copy: BAM – Working Group "Safe Handling of Oxygen"

- Working Group "Safe Handling of Oxygen" -

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.

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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.

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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.