

Report

on Testing a Sealing Material for Reactivity with Oxygen

BAM reference II-1734/2004 E

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1 Application

Customer Rich. Klinger
Dichtungstechnik GmbH & Co KG
Am Kanal 8 - 10
2352 GUMPOLDSKIRCHEN
AUSTRIA

Order Date May 6, 2004

Reference MÜ

Receipt of Order May 11, 2004

Test Samples Gasket KLINGERSIL® C-4500 for use in flanged connections in oxygen piping at temperatures greater than 60 °C and for liquid oxygen;
BAM Order No. II.1/47 421

Receipt of Samples May 10, 2004

Test Date May 28 to July 1, 2004

Test Location BAM-Laboratory II.13; building no. 41, room no. 073

Test Procedures According to Regulation BGV B 7 „Oxygen“ of the „Berufsgenossenschaft der chemischen Industrie“. Test methods according to the annex of the pamphlet „Liste der nichtmetallischen Materialien die von der Bundesanstalt für Materialforschung und -prüfung (BAM) zum Einsatz in Anlageteilen für Sauerstoff als geeignet befunden worden sind.“ (Edition: 31. August 2003) of BGV B 7.

This test report consists of page 1 to 4 and annex 1 to 4.

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

TESTREPORT

2 Documents and Test Samples

The following documents and samples were submitted:

- 1 Application for testing,
- 1 Product data sheet and
- 10 disks of KLINGERSIL® C-4500 (thickness: ca. 2 mm; diameter: 140 mm), color: one side tinged with anthracite, the other side anthracite with imprint „KLINGERSIL® C-4500“

3 Test Procedure and Test Results

3.1 Autogenous Ignition Temperature (AIT)

The test method is described in annex 1.

Results:

In five tests with an oxygen pressure of $p_a = 112$ bar, an AIT of 164 °C was determined with a standard deviation of ± 3 °C. The oxygen pressure p_a at ignition is approximately 166 bar.

3.2 Artificial Aging

The test method is described in annex 2.

Results:

After aging of KLINGERSIL® C-4500 at 160 bar oxygen pressure and 110 °C, the material was very brittle. The sample gained 5,8 % in mass.

The AIT of the aged sample at 170 bar oxygen pressure was 176 °C with a standard deviation of ± 6 °C. The test showed that the AIT of the aged sample was slightly greater than the AIT of the non-aged sample.

3.3 Flange Test

The test method is described in annex 3.

Results:

The samples of KLINGERSIL® C-4500 were tested at 160 bar oxygen pressure and 85 °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

remained gas-tight. Thereupon, the test was repeated four times at 160 bar and 85 °C. The same result was obtained as before.

3.4 Reactivity with Liquid Oxygen on Mechanical Impact

The test method is described in annex 4.

Results:

At a drop height of 0,17 m (Impact energy = 125 Nm) a very violent reaction of the material with liquid oxygen could be observed already in the first test.

4 Evaluation

The tests have shown that the autogenous ignition temperature of the material is $(164 \pm 3)^\circ\text{C}$ at an oxygen pressure of 166 bar.

At a temperature of 110 °C and an oxygen pressure of 160 bar, the material proved not to be sufficient aging resistant. As a result of the aging test, the material was brittle. Therefore, the gasket KLINGERSIL® C-4500 is suitable only for use in flanges that are not dynamically stressed. Furthermore, the unfavourable aging behaviour may reduce the gasket's usability.

Regarding technical safety, the flat gasket KLINGERSIL® C-4500 may be used in flange connections made of copper, copper alloys or steel at oxygen pressures up to 160 bar and temperatures up to 85 °C, if the above-mentioned requirement is met. This applies to flat faced flanges, male and female flanges, and flanges with tongue and groove.

As a result of the test with liquid oxygen the gasket KLINGERSIL® C-4500 is not suitable for use in plants or installations for liquid oxygen.

5 Note

This report expires at once, if the composition of the tested material is changed. This report expires on July 31, 2014, at the latest. A prolongation beyond this date is possible, if the manufacturer confirms in writing that the material has not changed since this evaluation.

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.

Federal Institute for Materials Research and Testing (BAM)
12200 Berlin, July 28, 2004

Subdivision II.1
"Gases, Gas Plants"



Dr. Chr. Binder
Head of Laboratory

Laboratory II.13
"Equipment for Gases, Oxygen"



Dipl.-Ing. K. Arit
Engineer in Charge

Copies:

1. Copy: Rich. Klinger Dichtungstechnik GmbH & Co KG
2. Copy: BAM – Laboratory II.13, Dr. Binder

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 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_o is calculated.

It is important to know the oxygen pressure p_o , 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.