Report

on Testing a Gasket Material for Reactivity with Oxygen

Reference Number	2-1244/2012 V E
Copy	1. Copy of 2 Copies
Customer	Frenzelit-Werke GmbH Frankenhammer 7 95460 Bad Berneck Germany
Order Date	April 24, 2012
Reference	EMP / BWI
Receipt of Order	May 16, 2012
Test Samples	Gasket material novaflon® 300 for use in flanged connections in piping, valves and fittings or other components for gaseous oxygen service and for liquid oxygen service. BAM-Order No. 2.1/51 093
Receipt of Samples	May 15, 2012
Test Date	July 25, 2012 to November 27, 2012
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: 2004-07 „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: September 2011; Rule BGR 500 "Betreiben von Arbeitsmitteln" part 2, chapter 2.32 "Betreiben von Sauerstoffanlagen", paragraph 3.17 "Lubricants and sealing materials", Edition: April 2008.

All pressures of this report are excess pressures.
This test report consists of page 1 to 5 and annex 1 to 4.

1 Documents and Test Samples

The following documents and samples were submitted to BAM:

1 Test Application

"Evaluate the compatibility of the gasket material novaflon® 300 for use in flanged connections in piping, valves and fittings or other components for gaseous oxygen service at temperatures greater than 250 °C and 83 bar and for liquid oxygen service.

15 Disks of the gasket material novaflon® 300

Diameter 140 mm; Thickness 3 mm

With Imprint: novaflon® 300

Colour: Brown

2 Test Methods

To evaluate the compatibility of the gasket material novaflon® 300 for use in flanged connections in piping, valves and fittings or other components for gaseous oxygen service, a determination of the autogenous ignition temperature (AIT), an investigation of the aging resistance in high pressure oxygen with an re-investigation of the aged material, and the "Flange Test" were carried out at an oxygen pressure of 83 bar.

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)

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	34	88	469
2	34	86	460
3	34	88	466
4	34	87	463
5	34	85	457

In five tests with an initial oxygen pressure of p_I = 34 bar, an AIT of 463 °C was determined with a standard deviation of ± 5 °C. The oxygen pressure p_F at ignition is approximately 87 bar.

3.2 Artificial Aging

The test method is described in annex 2.

Results:

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	275	83	0

After aging of the test sample at 83 bar oxygen pressure and 275 °C the test sample was apparently unchanged. The mass of the test sample did not change.

3.2.1 AIT after Artificial Aging

The test method is described in annex 1.

Results:

Number of Tests	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	34	85	465
2	34	86	468
3	34	86	464
4	34	87	465
5	34	86	462

In five tests with an initial oxygen pressure of p_I = 34 bar, an AIT of 465 °C was determined with a standard deviation of ± 2 °C. The final oxygen pressure p_F at ignition is approximately 86 bar.

3.3 Flange Test

The test method is described in annex 3.

Results:

Number of Tests	Temperature [°C]	Oxygen Pressure [bar]	Notes
1	250	83	About 3 minutes after ignition, the gasket is pressed out of the flange and is destroyed.
2	250	83	same behavior as in test no. 1
3	250	60	The gasket burns 5 mm between the flanges. The flange connection remains gas-tight.
4	250	50	Only those parts of the gasket burn that project into the pipe. The flange connection remains gas-tight.

Number of Tests	Temperature [°C]	Oxygen Pressure [bar]	Notes
5	250	50	same behavior as in test no. 4
6	250	50	same behavior as in test no. 4
7	250	50	same behavior as in test no. 4
8	250	50	same behavior as in test no. 4

In five tests at 50 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 connection remains gas-tight.

3.4 Reactivity with Liquid Oxygen on Mechanical Impact

The test method is described in annex 4.

Results:

Test No.	Drop Heights [m]	Impact Energy [Nm]	Reaction
1	0.50	375	reaction
2	0.17	125	reaction

At drop heights of 0.50 m and 0.17 m (impact energy 375 Nm and 125 Nm), severe reactions of the sample with liquid oxygen occurred.

4 Summary and Evaluation

The product novaflon® 300 had already been tested and evaluated in 2007 as a gasket material for use in valves and fittings or other components for gaseous oxygen service under reference numbers Tgb.-Nr.: II-1253/2007.

The tests have shown that the autogenous ignition temperature of the gasket material novaflon® 300 is 463 °C at 87 bar oxygen pressure. The standard deviation of the AIT is ± 5 °C.

At a temperature of 250 °C and an oxygen pressure of 83 bar, the gasket material novaflon® 300 proved to be sufficient aging resistant. The mass of the sample did not change.

The tests have shown that the autogenous ignition temperature of the aged gasket material novaflon® 300 is 465 °C at 86 bar oxygen pressure. The standard deviation of the AIT is ± 2 °C.

This shows, that the AIT of the aged sample is unchanged compared to the AIT of the non-aged sample within the precision of measurement.

On basis of the result of the flange test, there are no objections with regard to technical safety to use the gasket material novaflon® 300 in flange connections made of copper, copper alloys or steel at following conditions:

Maximum Temperature	Maximum Oxygen Pressure
250 °C	50 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 4, gasket material novaflon® 300 is not suitable for liquid oxygen service.

5 Comments

The test results refer exclusively to the batch of the tested material.

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. 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, April 8, 2013

Division 2.1
"Gases, Gas Plants"

on behalf of

Dipl.-Ing. P. Hartwig
Study Director "Safe Handling of Oxygen"

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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 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.