BAM
Bundesanstalt für
Materialforschung
und -prüfung

# Report
on Testing a Gasket Material for Reactivity with Oxygen

TEST REPORT

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| **Reference Number** | II-662/2008 E |
| **Copy** | 1. Copy of 2 Copies |

## 1 Application

| | |
|---|---|
| **Customer** | F.M.I. S.p.A.<br>Via Taranto 10/12<br>25036 S. Pancrazio di Palazzolo S/Oglio (BS)<br>Italien |
| **Order Date** | March 3, 2008 |
| **Receipt of Order** | March 14, 2008 |
| **Test Samples** | Gasket UNIGRAPH 500 for use in flanged connections in piping, valves and fittings, and components for gaseous oxygen at 100 bar and for liquid oxygen service.<br>BAM-Order No. II.1/49 199 |
| **Receipt of Samples** | March 13, 2008 |
| **Test Date** | June 25, 2008 to August 29, 2008 |
| **Test Location** | BAM - Working Group "Safe Handling of Oxygen";<br>building no. 41, room no. 073 |
| **Test Procedure According to** | DIN EN 1797: 2002-02<br>„Cryogenic Vessels - Gas/Material Compatibility"<br>Annex of pamphlet M 034-1 (BGI 617-1)<br>„Liste der nichtmetallischen Materialien die von der Bundesanstalt für Materialforschung und –prüfung (BAM) zum Einsatz in Anlageteilen für Sauerstoff als geeignet befunden worden sind.",<br>to pamphlet M 034 „Sauerstoff" (BGI 617)<br>Berufsgenossenschaft der chemischen Industrie<br>Edition: October 2007;<br>according chapter 3.17 „Gleitmittel und Dichtwerkstoffe" to rule BGR 500 „Betreiben von Arbeitsmitteln" part 2, chapter 2.32 „Betreiben von Sauerstoffanlagen", Edition: April 2008. |

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

## 2 Documents and Test Samples

The following documents and samples were submitted to BAM:

1 Test Application
1 Material Safety Data Sheet
27 Gaskets of UNIGRAPH 500;
asbestos-free graphite sheet material covered with a special polymer coating on both sides
Diameter 140 mm; thickness 2,2 mm
Colour: grey

## 3 Test Methods and Results

Both faces of the gasket are covered by a special polymer coating that can easily be removed. Therefore, the graphite and also the special polymer coating has to undergo the autogenous ignition temperature test and the artificial aging test separately.

### 3.1 Autogenous Ignition Temperature (AIT)

#### 3.1.1 Graphite Material

The test method is described in annex 1.

Results:

| Test No. | Oxygen Pressure $p_a$ [bar] | Oxygen Pressure $p_e$ [bar] | AIT [°C] |
|---|---|---|---|
| 1 | 39 | 103 | > 500 |
| 2 | 39 | 104 | > 500 |
| 3 | 39 | 104 | > 500 |
| 4 | 39 | 104 | > 500 |
| 5 | 39 | 103 | > 500 |

Up to temperatures of 500 °C, no ignition of the material could be detected in five tests with oxygen pressures of $p_a$ = 39 bar. The oxygen pressure $p_e$ was approximately 104 bar.

#### 3.1.2 Special Polymer Coating

The test method is described in annex 1.

Results:

| Test No. | Oxygen Pressure $p_a$ [bar] | Oxygen Pressure $p_e$ [bar] | AIT [°C] |
|---|---|---|---|
| 1 | 76 | 106 | 143 |
| 2 | 76 | 109 | 149 |
| 3 | 76 | 106 | 143 |
| 4 | 76 | 110 | 157 |
| 5 | 76 | 111 | 156 |

In five tests with an oxygen pressure of $p_a$ = 76 bar, an AIT of 150 °C was determined with a standard deviation of ± 7 °C. The oxygen pressure $p_e$ at ignition is approximately 109 bar.

## 3.2 Artificial Aging

### 3.2.1 Graphite Material

The test method is described in annex 2.

Results:

| Time [h] | Temperature [°C] | Oxygen Pressure [bar] | Mass Change [%] |
|---|---|---|---|
| 100 | 118 | 100 | 0 |

After aging of the graphite material at 100 bar oxygen pressure and a temperature of 118 °C, the material was apparently unchanged. The mass of the test sample did not change.

### 3.2.2 Special Polymer Coating

The test method is described in annex 2.

Results:

| Time [h] | Temperature [°C] | Oxygen Pressure [bar] | Mass Change [%] |
|---|---|---|---|
| 100 | 118 | 100 | + 1 |

After aging of the special polymer coating at 100 bar oxygen pressure and a temperature of 118 °C, the material was apparently unchanged. The mass of the test sample increased 1 % in mass.

### 3.2.3 AIT after Artificial Aging

#### 3.2.3.1 Graphite Material

The test method is described in annex 1.

Results:

| Number of Tests | Oxygen Pressure $p_a$ [bar] | Oxygen Pressure $p_e$ [bar] | AIT [°C] |
|---|---|---|---|
| 1 | 39 | 103 | > 500 |
| 2 | 39 | 103 | > 500 |
| 3 | 39 | 103 | > 500 |
| 4 | 39 | 104 | > 500 |
| 5 | 39 | 104 | > 500 |

Up to temperatures of 500 °C, no ignition of the material could be detected in five tests with oxygen pressures of $p_a$ = 39 bar. The oxygen pressure $p_e$ was approximately 103 bar.

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.

#### 3.2.3.2 Special Polymer Coating

The test method is described in annex 1.

Results:

| Test No. | Oxygen Pressure $p_a$ [bar] | Oxygen Pressure $p_e$ [bar] | AIT [°C] |
|---|---|---|---|
| 1 | 76 | 105 | 135 |
| 2 | 76 | 109 | 148 |
| 3 | 76 | 103 | 124 |
| 4 | 76 | 107 | 140 |
| 5 | 76 | 107 | 139 |

In five tests with an oxygen pressure of $p_a$ = 76 bar, an AIT of 137 °C was determined with a standard deviation of ± 9 °C. The oxygen pressure $p_e$ at ignition is approximately 106 bar.

This shows, that the AIT of the aged sample is significant lower than the AIT of the non-aged sample within the precision of measurement.

### 3.3 Flange Test

The gasket UNIGRAPH 500 was tested completely.

The test method is described in annex 3.

Results:

| Number of Tests | Oxygen Pressure [bar] | Temperature [°C] | Notes |
|---|---|---|---|
| 1 | 100 | 90 | Only those parts of the special polymer coating burn that project into the pipe. The graphite material was not ignited. |
| 2 | 100 | 90 | same behavior as in test no. 1 |
| 3 | 100 | 90 | same behavior as in test no. 1 |
| 4 | 100 | 90 | same behavior as in test no. 1 |
| 5 | 100 | 90 | same behavior as in test no. 1 |
| 6 | 100 | 90 | About 30 seconds after ignition, the oxygen is pressed out of the flange and the gasket is radial destroyed. There were no signs of ignition. |
| 7 | 100 | 90 | same behavior as in test no. 1 |

In five tests at 100 bar oxygen pressure and 90 °C, only those parts of special polymer coating burn that project into the pipe; the fire is neither transmitted to the graphite material and steel nor does the gasket burn between the flanges. The flange remains gas-tight.

## 3.4 Reactivity with Liquid Oxygen on Mechanical Impact

The gasket UNIGRAPH 500 was tested completely.

The test method is described in annex 4.

Results:

| Test No. | Drop Heights [m] | Impact Energy [Nm] | Reaction |
|---|---|---|---|
| 1 | 0,67 | 500 | no reaction |
| 2 | 0,83 | 625 | no reaction |
| 3 | 1,0 | 750 | no reaction |
| 4 | 1,0 | 750 | no reaction |
| 5 | 1,0 | 750 | no reaction |
| 6 | 1,0 | 750 | no reaction |
| 7 | 1,0 | 750 | no reaction |
| 8 | 1,0 | 750 | no reaction |
| 9 | 1,0 | 750 | no reaction |
| 10 | 1,0 | 750 | no reaction |
| 11 | 1,0 | 750 | no reaction |
| 12 | 1,0 | 750 | no reaction |

At drop heights of 1,0m (impact energy 750 Nm), in ten separate tests, no reaction of the material with liquid oxygen could be detected.

## 4 Evaluation

The tests have shown that the autogenous ignition temperature of the gasket material UNIGRAPH 500 is 150 °C at 109 bar oxygen pressure. The standard deviation is ± 7 °C.

At a temperature of 118 °C and an oxygen pressure of 100 bar, the material proved to be sufficient aging resistant. The sample increased 1 % in mass. The autogenous ignition temperature of the aged gasket material UNIGRAPH 500 is 137 °C at 106 bar oxygen pressure. The standard deviation is ± 9 °C.

On basis of these test results and the results of the flange testing, there are no objections with regard to technical safety to use the gasket UNIGRAPH 500 in flange connections made of copper, copper alloys or steel at following conditions:

| Maximum Temperature | Maximum Oxygen Pressure |
|---|---|
| up to 70 °C | up to 100 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, there are no objections with regard to technical safety to use the UNIGRAPH 500 in components and apparatuses 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 gasket material.

## 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 be used for gaseous oxygen service and liquid 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, September 23, 2008**

**Division II.1**
**"Gases, Gas Plants"**

Dr. Chr. Binder
Head of Working Group

**Working Group**
**"Safe Handling of Oxygen"**

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Copies:
1. Copy: F.M.I. S.p.A.
2. Copy: BAM – 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.

**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.