BAM
Bundesanstalt für
Materialforschung
und -prüfung

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TEST REPORT

on Testing a Nonmetallic Material for Reactivity with Oxygen

Reference Number	16032228 E
Copy	1. copy of 2 copies
Customer	amtec advanced measurements Messtechnischer Service GmbH Hoher Steg 13 74348 Lauffen
Date of Request	July 6, 2016
Receipt of Signed Contract	July 29, 2016
Test Samples	Spiral Wound Gasket Spira Power undisclosed batch, BAM Order-No.: 2.1/53 230
Receipt of Samples	July 20, 2016
Test Date	September 2 to November 2, 2016
Test Location	BAM – Division 2.1 „Gases, Gas Plants"; building no. 41, room 073
Test Procedure or Requirement According to (in the current version at test time)	DIN EN 1797 und ISO 21010 "Cryogenic Vessels - Gas/Material Compatibility"; Annex of code of practice M 034-1 (BGI 617-1) "List of nonmetallic materials compatible with oxygen", by German Social Accident Insurance Institution for the raw materials and chemical industry; TRGS 407 Technical Rules for Hazardous Substances "Tätigkeiten mit Gasen - Gefährdungsbeurteilung" chapter 3 "Informationsermittlung und Gefährdungsbeurteilung" and chapter 4 "Schutzmaßnahmen bei Tätigkeiten mit Gasen"

All pressures of this report are excess pressures.
This test report consists of page 1 to 6 and annexes 1 to 2.

TEST REPORT

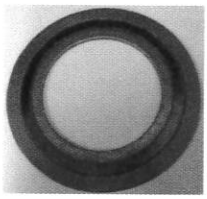
1 Documents and Test Samples

The following documents and samples were submitted to BAM:

1 Test application
„Testing and evaluating the graphite based filler material, undisclosed batch, for spiral wound gaskets Spira Power in gaseous oxygen service at pressures up to 35 bar and at temperatures up to 300 °C"

1 Material safety data sheet for graphite-based filler material
GRAPHITE PRODUCTS
(6 pages, Reviewed on December 20, 2012)

5 Spiral wound gaskets Spira Power,
undisclosed batch,
Outer-Ø: 150 mm, Inner-Ø: 82 mm, Thickness: 5 mm
Color of the filler material: Grey



2 Applied Test Methods for Evaluating the Technical Safety

The filler material shall be used for spiral wound gaskets Spira Power in gaseous oxygen service at pressures up to 35 bar and at temperatures up to 300 °C.

Tests on ignition sensitivity to gaseous oxygen impacts were not carried out because oxygen pressure impacts can be safely excluded in the intended service according to the information by the customer.

Flange tests were not carried out. Based on the intended service as a filler material for spiral wound gaskets, it can be safely excluded that the filler material projects into the pipe.

The following test methods were applied:

2.1 Determination of the Autogenous Ignition Temperature in High Pressure Oxygen

Usually, this test method is required if the material is for service at temperatures greater than 60 °C.

The autogenous ignition temperature (AIT) is a safety characteristic and indicates the temperature at which the material shows self-ignition in the presence of oxygen without an additional ignition source. Therefore, it is relevant for the maximum use temperature that is generally set 100 °C below this AIT.

2.2 Testing the Aging Behavior in High Pressure Oxygen

This test is necessary whenever a material is intended for service at higher temperatures than 60 °C. It simulates the use of a material in practice and helps analyze whether ignition temperature or properties of the material change due to aging processes.

3 Preparation of Samples

Prior to testing, the filler material was scraped off the metallic carrier material.

4 Tests

4.1 Determination of the Autogenous Ignition Temperature in High Pressure Oxygen

The test method is described in annex 1. Based on the specified use conditions by the costumer, the test was performed at a final oxygen pressure of approximately 35 °bar.

4.1.1 Assessment Criterion

The criterion for a reaction of the sample with oxygen is a distinct increase in pressure and a more or less steep increase in temperature.

4.1.2 Results

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	13	36	> 500
2	13	36	> 500
3	13	36	> 500
4	13	36	> 500
5	13	36	> 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 = 13 bar. The final oxygen pressure p_F was 36 bar.

This test can only be performed up to 500 °C. This equals the maximum working temperature of the test equipment.

4.2 Aging Behavior

The test method is described in annex 2. 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. In this case, the aging test was carried out at 35 bar and at 325 °C.

4.2.1 Assessment Criterion

There are three criteria for evaluating the aging behavior:

If there is a change in mass $\Delta m \leq 1$ %, the sample is aging resistant, in case of $\Delta m > 1$ % and $\Delta m \leq 2$ %, the sample is sufficient aging resistant, and in case of $\Delta m > 2$ %, the sample is insufficient aging resistant.

Changes in color, consistency, shape or surface texture of the samples or gas releases from the sample that can be detected after testing will be also considered by BAM.

The AIT of the aged sample is compared to the AIT of the non-aged sample. If there is a distinct deviation between both AITs, the lower value is considered for safety reasons.

4.2.2 Results

4.2.2.1 Change of Mass or Physical Appearance

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	325	35	- 0.8

After aging, the test sample was apparently unchanged and lost 0.8 % in mass.

4.2.2.2 Determination of the AIT of the Aged Material in High Pressure Oxygen

The test method is described in annex 1. The AIT test of the aged material was performed at same conditions as described in chapter 4.1 of the non-aged material.

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	13	36	> 500
2	13	36	> 500
3	13	36	> 500
4	13	36	> 500
5	13	36	> 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 = 13 bar. The final oxygen pressure p_F was 36 bar. This shows that, as the non-aged sample, also the aged sample did not ignite at temperatures up to 500 °C.

This test can only be performed up to 500 °C. This equals the maximum working temperature of the test equipment.

5 Summary and Evaluation

It is intended to use the non-metallic material as a filler material in spiral wound gaskets for gaseous oxygen service.

In determining the AIT, up to temperatures of 500 °C, no ignition of the sample could be detected in five tests with final oxygen pressures p_F = 36 bar.

At a temperature of 325 °C and an oxygen pressure of 35 bar, the filler material proved to be aging resistant.

Based on the test results, there are no objections with regard to technical safety, to use the graphite based filler material, undisclosed batch, for Spiral Wound Gaskets Spira Power in gaseous oxygen service at following operating conditions:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
300	35

This evaluation does not cover the use of the graphite based filler material, undisclosed batch, for liquid oxygen service. For this case, a particular test for reactivity with liquid oxygen needs to be carried out.

6 Comments

This safety evaluation considers the fact, that rapid oxygen pressure changes - so-called oxygen pressure surges - as well as projecting of the non-metallic material into the pipe can be safely excluded in the intended service.

This evaluation is based exclusively on the results of the tested sample of a particular batch.

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.

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.

Bundesanstalt für Materialforschung und -prüfung (BAM)
12200 Berlin

November 23, 2016

Division 2.1 "Gases, Gas Plants"

By order

Dipl.-Ing. Peter Hartwig

Distribution list: 1. copy: advanced measurements Messtechnischer Service GmbH
2. copy: BAM - Division 2.1 "Gases, Gas Plants"

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 initial pressure p_I 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 the final pressure p_F.

It is important to know the oxygen pressure p_F, 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.