BAM
Bundesanstalt für
Materialforschung
und -prüfung

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TEST REPORT

on Testing a Nonmetallic Material for Reactivity with Oxygen

Reference Number	16012984 E
Copy	1. copy of 2 copies
Customer	Garlock GmbH Falkenweg 1 41468 Neuss Germany
Date of Request	March 15, 2016
Receipt of Signed Contract and the Extension of the Order	April 27, 2016 June 29, 2016
Test Samples	Sealing Material GYLON BIO-PRO® PLUS (GYLON®3522/ 3527), undisclosed batch; BAM Order-No.: 2.1/53 108
Receipt of Samples	March 15, 2016
Test Date	April 28 to July 7, 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"

TEST REPORT

All pressures of this report are excess pressures.
This test report consists of page 1 to 8 and annexes 1 to 3.

1 Documents and Test Samples

The following documents and samples were submitted to BAM:

1 Test application

„Testing and evaluating the sealing material GYLON® Style 3522/3527, undisclosed batch, for use as GYLON BIO-PRO® PLUS™ gaskets in stainless steel clamp pipe couplings (e. g. TRI-CLAMP Couplings) for gaseous oxygen service at 55 bar and temperatures up to 260 °C and for liquid oxygen service."

Note:

GYLON® Style 3522 denotes the raw material;

GYLON® Style 3527 is the designation of the molded material.

GYLON BIO-PRO® PLUS is the trademarked name of the product

Fig. 1: Example of TRI-CLAMP-Coupling

1 Safety Data Sheet GYLON® Style 3522
(7 pages, date of issue: 19/10/2006)

1 Material Data Sheet Garlock CLEAR GYLON® Style 3522
(1 page)

15 Discs GYLON® Style 3522/3527

Dimension: Outer diameter Ø: 140 mm, Thickness: 1,6 mm;
Color: White, translucent

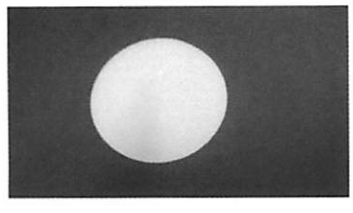

Abb. 2: Disc GYLON® Style 3522/3527

2 Applied Test Methods for Evaluating the Technical Safety

The gaskets GYLON BIO-PRO® PLUS are intended for use in stainless steel clamp pipe couplings (e. g. TRI-CLAMP Couplings) for gaseous oxygen service at 55 bar and temperatures up to 260 °C and for liquid oxygen service.

Tests for 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. According to the information by the customer, the designs of the couplings and of the gaskets prevent the sealing material from extending into the bore in the intended service.

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 process.

2.3 Reactivity with Liquid Oxygen on Mechanical Impact

The compatibility of the material with liquid oxygen was tested by its reactivity with liquid oxygen on mechanical impact.

3 Preparation of Samples

Prior to testing, the material was cut into pieces of approximately 1 mm^3 up to 2 mm^3.

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 test application the autogenous ignition temperature test was performed at a final oxygen pressure of approximately 55 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	22	57	467
2	22	58	470
3	22	57	467
4	22	58	471
5	22	59	487

Five tests resulted in the following mean AIT and its corresponding standard deviation:

Mean Final Oxygen Pressure p_F [bar]	Mean AIT [°C]	Standard Deviation [°C]
58	472	± 8

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 55 bar and at 285 °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	285	55	± 0.0

After aging, the mass of the test sample did not change and the test sample was apparently unchanged.

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

The test method is described in annex 2. The AIT test of the aged material was performed under the same conditions as described in chapter 4.2 of the non-aged material.

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	22	56	454
2	22	56	455
3	22	56	454
4	22	56	450
5	22	56	449

Five tests resulted in the following mean AIT and its corresponding standard deviation:

Mean Final Oxygen Pressure p_F [bar]	Mean AIT [°C]	Standard Deviation [°C]
56	452	± 3

At a final oxygen pressure p_F of 56 bar, the autogenous ignition temperature of the aged material is 452 °C with a standard deviation of ± 3 °C. This shows that the AIT of the aged sample is lower compared to the AIT of the non-aged sample.

4.3 Reactivity Testing with Liquid Oxygen on Mechanical Impact

The test method is described in annex 3.

4.3.1 Assessment Criterion

According to the BAM-Standard "Testing for Reactivity with Liquid Oxygen on Mechanical Impact", a nonmetallic material is not compatible with liquid oxygen, if reactions occur at a drop height of 0.17 m (impact energy 125 Nm) or less.

4.3.2 Results

Test No.	Drop Height [m]	Impact Energy [Nm]	Reaction
1	0,33	250	no reaction
2	0,50	375	no reaction
3	0,67	500	no reaction
4	0,83	625	no reaction
5	1,00	750	no reaction
6	1,00	750	no reaction
7	1,00	750	no reaction
8	1,00	750	no reaction
9	1,00	750	no reaction
10	1,00	750	no reaction
11	1,00	750	no reaction
12	1,00	750	no reaction
13	1,00	750	no reaction
14	1,00	750	no reaction

At a drop height of 1.00 m (impact energy 750 Nm), in ten separate tests, no reaction of the sample with liquid oxygen could be detected.

5 Summary and Evaluation

It is intended to use the product as a gasket in stainless steel clamp pipe couplings (e. g. TRI-CLAMP Couplings) for gaseous oxygen service and for liquid oxygen service.

Based on the information by the customer that rapid oxygen pressure changes - so-called oxygen pressure surges – and extensions of the gasket material into the bore of the pipes can be safely excluded, tests for ignition sensitivity to gaseous oxygen impacts and the flange test were not carried out.

At a final oxygen pressure p_F of 58 bar, the autogenous ignition temperature of the material is 472 °C with a standard deviation of ± 8 °C.

At a temperature of 285 °C and an oxygen pressure of 55 bar, the material proved to be aging resistant. After aging, the mass of the test sample did not change and the test sample was apparently unchanged. The tests have shown that the autogenous ignition temperature of the aged material is 452 °C at 56 bar oxygen pressure. The standard deviation of the AIT is ± 3 °C. Regarding technical safety, the decrease in AIT down to 452 °C can be neglected as the maximum use temperature is 260 °C.

Based on the test results and the pre-condition that any oxygen pressure impacts and an extending of the sealing material into the bore of the pipes can be safely excluded, there are no objections with regard to technical safety, to use the gaskets GYLON BIO-PRO® PLUS (GYLON® Style 3522/3527), undisclosed batch, for stainless steel clamp pipe couplings (e. g. TRI-CLAMP Couplings) for gaseous oxygen service at following operating conditions:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
260	55

Based on the test results, there are also no objections with regard to technical safety to use GYLON BIO-PRO® PLUS (GYLON® Style 3522/3527), undisclosed batch, as a gasket in stainless steel clamp pipe couplings (e. g. TRI-CLAMP Couplings) 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 material.

6 Comments

This safety evaluation considers the facts, that on the one hand rapid oxygen pressure changes - so-called oxygen pressure surges – and an extending of the sealing material into the bore of the pipes can be safely excluded. On the other hand, direct contact of the material with liquid oxygen and mechanical impacts cannot be safely excluded in usage

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

July 22, 2016

Division 2.1 "Gases, Gas Plants"

By order

Dipl.-Ing. Peter Hartwig

Distribution list: 1. copy: Garlock 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.

Annex 3

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.