BAM
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TEST REPORT

on Testing a Nonmetallic Material for Reactivity with Oxygen

Reference Number	17042533 E
Our Reference	02-3446
Copy	1. copy of 2 copies
Customer	Beldam Crossley Ltd. Lostock Lane Industrial Estate Bolton BL6 9AS United Kingdom
Date of Request	August 4, 2017
Your Reference	RD4640
Receipt of Signed Contract	September 6, 2017
Test Samples	S.3313 unsintered PTFE thread seal tape, Batch RD4640-1;
Receipt of Samples	September 6, 2017
Test Date	September 7 to November 21, 2017
Test Location	BAM – Division 2.1 „Gases, Gas Plants"; building no. 41
Test Procedure or Requirement according to (in the current version)	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 9 and annex 1 to 4.

1 Documents and Test Samples

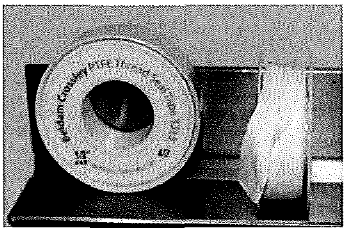
The following documents and samples were submitted to BAM:

1 Test application
Safety-related investigation of the nonmetallic material S.3313, batch RD4640-1, for use as a thread seal tape in gaseous oxygen service at temperatures up to 90 °C and for use in liquid oxygen service.

1 Safety Data Sheet S.3313 unsintered PTFE Tape
(3 Pages, Beldam Crossley Limited, date of issue: 08.02.2017)

1 Material Data Sheet S.3313, Product Data Sheet Number 104
(1 Page, Beldam Crossley Limited, date of issue: 03.10.2013)

1m PTFE thread seal tape, Batch RD4640-1
color: white



2 Applied Test Methods

The product S.3313, batch RD4640-1, is a thread seal tape that shall be used in gaseous oxygen service at temperatures up to 90 °C and for use in liquid oxygen.

The following test methods were applied:

2.1 Testing for Ignition Sensitivity to Gaseous Oxygen Impacts

This test method is required if rapid oxygen pressure changes – so-called oxygen pressure impacts - on the material cannot be safely excluded in usage.

2.2 Determination of the Autogenous Ignition Temperature (AIT) in High Pressure Oxygen

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

The 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. For sealing materials, the safety margin between AIT and maximum use temperature is 100 °C.

2.3 Testing of 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 the aging processes.

2.4 Testing for Reactivity with Liquid Oxygen on Mechanical Impact

Generally, this test method is required if direct contact of the material with liquid oxygen and mechanical impacts cannot be safely excluded in usage.

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 Testing for Ignition Sensitivity to Gaseous Oxygen Impacts

The test method is described in annex 1. Based on the specified use conditions by the customer, the test was performed at 60 °C and 90 °C.

4.1.1 Assessment Criterion

According to DIN EN 1797 „Cryogenic Vessels - Gas/Material Compatibility" and to ISO 21010 „Cryogenic Vessels - Gas/Material Compatibility" the criterion for a reaction of the sample to gaseous oxygen impacts is a temperature rise of at least 20 °C.

If the sample exhibits a change of color, or of consistency after testing, this is also considered as a positive reaction by BAM for safety reasons, even if there is no temperature rise detectable of at least 20 °C.

4.1.2 Results

In each of the test series, the initial oxygen pressure p_I was at ambient pressure.

Sample Temperature t_a [°C]	Final Oxygen Pressure p_F [bar]	Reaction
60	30	no reaction*
60	40	Ignition on 1. impact
60	30	no reaction*
90	30	no reaction*
90	30	no reaction*

* within a series of five consecutive impacts

In two separate tests, each consisting of a series of five consecutive impacts, no reactions of the sample with oxygen could be observed at following conditions:

Sample Temperature t_a [°C]	Final Oxygen Pressure p_F [bar]
60	30
90	30

4.2 Determination of the Autogenous Ignition Temperature (AIT) in High Pressure Oxygen

The test method is described in annex 2.

Based on the test results of the oxygen impact testing at 60 °C, the AIT determination was performed at a final oxygen pressure of approximately 30 bar.

4.2.1 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.

4.2.2 Results

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	12	31	443
2	12	31	439
3	12	31	444
4	12	31	433
5	12	31	439

In five separate tests, the following mean AIT could be determined.

Mean Final Oxygen Pressure p_F [bar]	Mean AIT [°C]	Standard Deviation [°C]
31	440	± 4

4.3 Testing of the Aging Behavior in High Pressure Oxygen

The test method is described in annex 3.

In general, artificial aging is carried out at the maximum use pressure and an elevated temperature, that is 25 °C above the maximum operating temperature. In this case, the test was carried out at a final oxygen pressure of 30 bar and a temperature of 115 °C.

4.3.1 Assessment Criterions

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 sample 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.

4.3.2 Results

4.3.2.1 Change in Mass or Physical Appearance

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	115	30	0,0

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

4.3.2.2 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.

Test No.	Initial Oxygen Pressure p_I [bar]	Final Oxygen Pressure p_F [bar]	AIT [°C]
1	12	31	445
2	12	31	443
3	12	30	429
4	12	30	433
5	12	30	443

In five separate tests, the following mean AIT could be determined:

Mean Final Oxygen Pressure p_F [bar]	Mean AIT [°C]	Standard Deviation [°C]
30	439	± 7

4.4 Testing for Reactivity with Liquid Oxygen on Mechanical Impact

The test method is described in annex 4.

4.4.1 Assessment Criterion

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

4.4.2 Results

Test Series No.	Drop Height [m]	Impact Energy [Nm]	Behavior on Mechanical Impact
1	1.00	750	reaction on 2. impact
2	0.83	625	reaction on 8. impact
3	0.67	500	no reaction*

* within a series of ten single tests

At a drop height of 1.00 m and 0.83 m (impact energies 750 Nm and 625 Nm) reactions of the sample with liquid oxygen could be regularly detected - to some extent under the formation of sparks.

At a drop height of 0.67 m (impact energy 500 Nm) no reaction of the sample with liquid oxygen could be detected in a series of ten single tests.

5 Summary of the Test Results

In two separate tests, each consisting of a series of five consecutive impacts, no reactions of the sample with oxygen could be observed at 60 °C and a final pressure of 30 bar.

In another two separate tests, each consisting of a series of five consecutive impacts, no reactions of the sample with oxygen could be observed at 90 °C and a final pressure of 30 bar.

At a final oxygen pressure p_F of 30 bar, the test sample has an autogenous ignition temperature of 440 °C with a standard deviation of ± 4 °C.

The material proved to be aging resistant at 115 °C and 30 bar oxygen pressure.

Testing of the material for reactivity to mechanical impacts in liquid oxygen showed that reactions occurred at impact energies of 750 Nm and 625 Nm. No reactions occurred at an impact energy of 500 Nm.

6 Opinion and Interpretation

It is intended to use the unsintered PTFE thread seal tape S.3313, batch RD4640-1, for gaseous oxygen service at temperatures up to 90 °C.

On basis of the test results, the requirements for sealing materials, described in annex 1 to attachment 2 of code of practice M034, annex 2 of code of practice M034-1, Technical Rules for Hazardous Substances TRGS 407 and BAM´s safety philosophy, there are no objections regarding technical safety, to use the unsintered PTFE thread seal tape S.3313, batch RD4640-1, for gaseous oxygen service at following use conditions:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
90	30

Based on the test results and according to BAM' s standard "Testing for Reactivity with Liquid Oxygen on Mechanical Impact", there are also no objections with regard to technical safety to use the unsintered PTFE thread seal tape S.3313, batch RD4640-1, 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.

7 Comments

This safety-related investigation considers the fact, that neither rapid oxygen pressure changes - so-called oxygen pressure surges –, nor direct contact of the material with liquid oxygen and mechanical impacts can be safely excluded in usage.

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

Our experience shows, that the safety characteristics of a product may vary from batch to batch. Therefore, today, we recommend batch testing of products, that are included for oxygen service. In this context, we would like to mention our paper from September 2009: "The Importance of Quality Assurance and Batch Testing on Nonmetallic Materials Used for Oxygen Service", Journal of ASTM International, Vol. 8th; Paper ID JAI102309. This publication can be purchased at www.astm.org.

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 be used for gaseous and for 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.

Bundesanstalt für Materialforschung und -prüfung (BAM)
12200 Berlin

December 8, 2017

Division 2.1 "Gases, Gas Plants"

By order

Dr. Thomas Kasch

Distribution list:	1. copy:	Beldam Crossley Ltd.
	2. copy:	BAM - Division 2.1 "Gases, Gas Plants"

Annex 1

Testing for Ignition Sensitivity to Gaseous Oxygen Impacts

Approximately 0.2 g to 0.5 g of the pasty or divided solid sample is placed into a heatable steel tube, 15 cm^3 in volume. In case of liquids to be tested, ceramic fibre, soaked with the sample, is used. The sample tube is connected by a 750 mm long pipe (internal diameter 14 mm) and a pneumatically operated quick opening valve to a high-pressure oxygen accumulator.

A heater allows to set the sample tube to the test temperature t_a After the tube and pipe are at test pressure p_I, the quick opening valve is opened and preheated oxygen of 60 °C and of pressure p_F flows abruptly into the pipe and tube. In this way, the oxygen in the tube and in the pipe is almost adiabatically compressed from pressure p_I to p_F in 17.5 ms ±2.5 ms (according to DIN EN 1797 and ISO 21010) and heated. If there is a reaction of the sample with oxygen, indicated by a steep temperature rise in the tube, further tests with a new sample are performed at a lower pressure ratio p_F/p_I. If, however, no reaction of the sample with oxygen can be detected after a waiting period of 30 seconds, the tube is de-pressurized and the test is repeated (up to four times) until a reaction takes place. This means, each test series consists of a maximum of five single tests with the same material under the same conditions. If no reaction can be observed, even after the fifth single test of a test series, testing is continued with new samples at greater pressure ratios p_F/p_I, until finally that pressure ratio is determined, at which no reaction can be observed within a test series of five single tests.If the repetition of that test series with a new sample shows the same result, the test can be finished or continued at a different test temperature t_a.

Annex 2

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 3

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.