



## TEST REPORT

On Testing a Nonmetallic Material for Reactivity with Gaseous Oxygen

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<b>BAM reference</b>	2-491/2015 E
<b>Copy</b>	1 <sup>st</sup> copy of 2 copies
<b>Customer</b>	FLEXITALLIC LTD Scandinavia Mill; Hunsworth Lane Cleckheaton; West Yorkshire BD19 4LN United Kingdom
<b>Order date</b>	February 17, 2015
<b>Reference</b>	PO315309
<b>Receipt of order</b>	February 20, 2015
<b>Test samples</b>	Flexitallic SIGMA® 511, batch 430440-5; BAM Order-No.: 2.1/52 545
<b>Receipt of samples</b>	February 19, 2015
<b>Test date</b>	July 6 to September 24, 2015
<b>Test location</b>	BAM - Working Group "Safe Handling of Oxygen"; building no. 41, room no. 073
<b>Test procedure according to</b>	ISO 21010:2014 and DIN EN 1797:2002-02 "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, Edition: March 2014; TRGS 407 Technical Rules for Hazardous Substances "Tätigkeiten mit Gasen - Gefährdungsbeurteilung" chapter 3 "Informationsermittlung und Gefährdungsbeurteilung" and chapter 4 "Schutzmaßnahmen bei Tätigkeiten mit Gasen", Edition: June 2013
<b>Safety Related Maximum Operating Conditions</b>	See chapter 4 "Summary and Evaluation"

All pressures of this report are excess pressures.  
This test report consists of page 1 to 5 and annexes 1 to 3

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The German version is legally binding, except an English version is issued exclusively.

## 1 Documents and Test Samples

The following documents and samples were submitted to BAM:

- 1 Test application  
"Testing and evaluating the compatibility of the nonmetallic material Flexitallic SIGMA® 511, batch 430440-5, for use as a gasket in flanged connections in valves, fittings or in other components for gaseous oxygen service at temperatures up to 225 °C and at pressures up to 40 bar"
- 1 Safety Data Sheet  
(5 pages, version PDS 617, issued: 09/14)
- 1 Material Data Sheet  
(1 page, version PDS 124, revision 2)
- 15 Disks Flexitallic SIGMA® 511, batch 430440-5  
Outer-Ø: 140 mm; Thickness: 1.5 mm  
Color: Pink

## 2 Test Methods

To evaluate the compatibility of Flexitallic SIGMA® 511, batch 430440-5, for use as a gasket material in gaseous oxygen service at temperatures up to 225 °C, a determination of the autogenous ignition temperature (AIT), an investigation of the aging resistance and a flange test were carried out.

Tests on ignition sensitivity to gaseous oxygen impacts were not carried out. According to the customer, oxygen pressure impacts in valves and fittings or in other components can be safely excluded in the intended service.

## 3 Results

### 3.1 Autogenous Ignition Temperature (AIT)

Based on the specified maximum operating conditions, the autogenous ignition temperature test was performed at a final oxygen pressure of approximately 40 bar. 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	16	43	487
2	16	43	488
3	16	43	491
4	16	43	490
5	16	43	491

In five tests with an initial oxygen pressure of  $p_i = 16$  bar, an AIT of 489 °C was determined with a standard deviation of  $\pm 2$  °C. The final oxygen pressure  $p_f$  at ignition is 43 bar.

### 3.2 Artificial Aging

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 250 °C and at 40 bar. The test method is described in annex 2.

Results:

Time [h]	Temperature [°C]	Oxygen Pressure [bar]	Mass Change [%]
100	250	40	$\pm 0$

After aging of the test sample at 250 °C and at 40 bar oxygen pressure, the test sample was apparently unchanged. The mass of the test sample did not change.

#### 3.2.1 AIT after Artificial Aging

The same test conditions as in chapter 3.1 were used for determining the autogenous ignition temperature after aging. 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	16	43	494
2	16	43	490
3	16	43	491
4	16	43	491
5	16	43	489

In five tests with an initial oxygen pressure of  $p_i = 16$  bar, an AIT of 491 °C was determined with a standard deviation of  $\pm 2$  °C. The final oxygen pressure  $p_f$  at ignition is 43 bar. This shows that the AIT of the aged sample is unchanged compared to the AIT of the non-aged sample.

### 3.3 Flange Test

Based on the specified maximum operating conditions, the flange test was performed at 40 bar oxygen pressure and at a temperature of 225 °C. The test method is described in annex 3.

## Results:

Test Number	Oxygen Pressure [bar]	Temperature [°C]	Notes
1	40	225	Only those parts of the gasket burn that project into the pipe, the flange connection remains gas-tight
2	40	225	same behavior as in test no. 1
3	40	225	same behavior as in test no. 1
4	40	225	same behavior as in test no. 1
5	40	225	same behavior as in test no. 1

In five tests at 40 bar oxygen pressure and at 225 °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 remains gas-tight.

## 4 Summary and Evaluation

The tests have shown that the autogenous ignition temperature of the gasket material Flexitallic SIGMA® 511, batch 430440-5, is 489 °C at 40 bar oxygen pressure. The standard deviation of the AIT is  $\pm 2$  °C.

At a temperature of 250 °C and an oxygen pressure of 40 bar, the gasket material proved to be sufficient aging resistant. The mass of the test sample did not change.

The tests have shown that the autogenous ignition temperature of the aged gasket material is 491 °C at 40 bar oxygen pressure. The standard deviation of the AIT is  $\pm 2$  °C. This shows that the AIT of the aged sample is unchanged compared to the AIT of the non-aged sample.

Generally, in evaluating nonmetallic materials for oxygen service, a safety margin of 50 °C between AIT and maximum operating temperature is being considered for safety reasons. As the maximum operating temperature is 225 °C, the gasket material Flexitallic SIGMA® 511, batch 430440-5, undisclosed batch, fulfills this criterion.

On basis of the test results and the results of the flange test, there are no objections with regard to technical safety, to use Flexitallic SIGMA® 511, batch 430440-5, as a gasket material with a maximum thickness of 1.5 mm in flange connections made of copper, copper alloys or steel at following conditions if oxygen pressure impacts can be safely excluded in the intended service:

Maximum Temperature [°C]	Maximum Oxygen Pressure [bar]
225	40

This applies to flat faced flanges, male/female flanges, and flanges with tongue and groove.

This evaluation does not cover the use of the gasket material Flexitallic SIGMA® 511, batch 430440-5, for liquid oxygen service. For this case, a particular test for reactivity with liquid oxygen needs to be carried out.

## 5 Comments

This evaluation is based exclusively on the test results of batch 430440-5 of Flexitallic SIGMA® 511.

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.

### **Bundesanstalt für Materialforschung und -prüfung (BAM)**

12200 Berlin

October 7, 2015

Division 2.1  
"Gases, Gas Plants"

by order



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Dr. Thomas Kasch

Distribution list:           1<sup>st</sup>   copy: FLEXITALLIC LTD  
                                      2<sup>nd</sup>   copy: BAM - Division 2.1 "Gases, Gas Plants"

Enclosures



## 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<sup>3</sup> 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 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.