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TEST OF DN40 PN40 MATERIAL 316L GASKET AT MINUS 196°C.

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Abstract: The report presents the measurements of the leakage rate from the flange bolted joint gasketed with full metallic gasket operating in – 196 Celsius degree

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1. The basic of the elaboration

The basic of the elaboration of report herein is an official ordering document sent by Pipeotech AS company (called further Orderer) on 02.12.2020 to the Wroclaw University of Science Technology (called further Contractor). The official ordering document was prepared to base on offer No 3 sent by the Contractor to the Orderer on 19.11.2020.

2. Aim and scope of the work

The aim of this work is a leakage test of the metallic gasket DN40 PN40 "DeltaV-Seal" operated in a flange-bolted joint subjected to temperature -196 Celsius degree. The test conditions are specified below:

- Required gaskets: 3 pcs.
- Gasket dimension in line with PN40 DN40 designation,
- Temperature of the test: -196 (liquid nitrogen),
- Testing gas range: from 1bar to 100 bar of helium pressure, (in 10 selected points)
- Leakage measuring method: Helium spectrometer (leakage detector) maximum detecting threshold: 10-12 mbar*I/s.
- The measurements of the gasket's thickness before and after the test.
- Torque instruction provide by the Orderer.

3. Sample for testing

The samples (gaskets) used in the test were delivered by the Pipeotech company. The gaskets were a full metallic one made of stainless steel 316L with dimension DN40 PN40. The picture of the tested gaskets was presented in Figure 1. Each sample was marked by number and by four characteristic points where the gasket's thickness was measured before and after the test.



Fig. 1 Gasket used in the test

4. Measuring of the gasket thickness

Before and after the leakage test the thickness of the each gasket was measured in four position equally distributed along with gasket's circumference.



Fig. 2 Test stand for measuring of the gasket's thickness

This measurement was performed using a micrometer sensor situated in the adjustable stand which was presented in Figure 2. For each circumferential position, three measuring points laying along the gasket's width were done. These three points represented the gasket's ridges which were marked as follows: A- internal ridge, B – middle ridge, C – external ridge. The characteristic points where the gasket's thickness was measured was illustrated in Figure 3.



Fig. 3 The characteristic points for measuring of the gasket's thickness

The measuring data of the gasket's thickness (before and after the test) were collected in table 1.

	Sample No 1							
	Before the test			After the test				
Position	1	2	3	4	1	2	3	4
Ridges	mm	mm	mm	mm	mm	mm	mm	mm
А	3,606	3,589	3,601	3,623	3,470	3,492	3,521	3,451
В	3,590	3,590	3,594	3,622	3,442	3,457	3,479	3.432
С	3.575	3,561	3,582	3,597	3,386	3,316	3,454	3,409
				Sample	e No 2			
		Before	the test		After the test			
Position	1	2	3	4	1	2	3	4
Ridges	mm	mm	mm	mm	mm	mm	mm	mm
А	3,583	3,583	3,599	3,574	3,476	3,454	3,499	3,518
В	3,587	3,597	3,638	3,588	3,376	3,391	3,469	3,491
С	3,573	3,597	3,619	3,593	3,339	3,322	3,374	3,455
			•	Sample	e No 3			
		Before	the test		After the test			
Position	1	2	3	4	1	2	3	4
Ridges	mm	mm	mm	mm	mm	mm	mm	mm
Α	3,629	3,602	3,598	3,589	3,520	3,471	3,464	3,520
В	3,632	3,593	3,583	3,573	3,489	3,417	3,484	3,508
С	3,633	3,572	3,575	3,558	3,476	3,333	3,430	3,436

Table 1 Measuring data of the gasket thickness (before and after the test)

5. Leakage test

5.1 Test rig

Figure 4 presents the schematic chart of the test rig where the gaskets were tested. The main element of the test rig is the flange bolted joint PD40 PN40.



Fig. 4 Schematic chart of the test rig

After the freezing of the joint (in liquid nitrogen) it was placed in a testing chamber - see Figure 5.



Fig. 5 Testing chamber with the gasketed-flange-bolted joint

The two sets of vacuum pumps (connecting to the testing chamber) provide an adequate vacuum level. To keeping the joint temperature as low as possible, the testing chamber was placed in a thermal shield fulfilled with liquid nitrogen. The gasketed joint was pressurized by gaseous helium from a reservoir. Thanks to a vacuum in the chamber the leakage of the helium was detected using a helium spectrometer connected to vacuum pumps. The illustration of the test rig was presented in Figure 6.



Fig. 6 Test rig of the leakage testing

Additional elements of the test rig are leakage detector displayer (showing a present leakage level), helium pressure gauge indicating the pressure in flange joint, helium reducer providing to set an adequate helium pressure, temperature sensor of the flange.

5.2 Mounting procedure

Each gasket was mounted in a flange joint in accordance with the procedures described below:

- The blind flange was mounted in the vise and the upper flange was placed on it in order to check the flanges' parallelism see Figure 7.
- All contact surfaces were cleaned by using a wire brush and a rag with solvent.
- All fasteners (bolts, washers, and nuts) were lubricated using "Molicote G Rapid Plus" – see Figure 8.
- The tested gasket was concentrically placed on the blind flange's contacting surface see Figure 9a.
- The bolts were inserted into flange's holes see Figure 9b.
- The top flange was placed concentrically on the gasket see Figure 9c.
- The nuts were slightly tightened using fingers see Figure 9d.
- Next the tightening procedure using torque wrench was done in line with table 2. The bolts were tightening (in three steps) using a star scheme in sequence bolt 1, bolt 3, bolt 4, and bolt 2 See Figure 10.
- After 10 minutes the nuts were retightened in a clockwise direction using a maximal torque of 96 Nm.



Fig. 7 Checking of the flange alignment / parallelism

Table 2 Required torque values in particular steps

Torque Sequence [Nm]						
Initial Step 20% 40% 80% 100%						
Hand tighten	19	38	76	96		



Fig. 8 Lubrications of the fasteners



Fig. 9 Assembly procedure of the tested gasket in a flange joint



Fig. 10 Bolt tightening sequence

5.3 Test procedure

After the joint was assembled and properly tightened the test procedure was as follows:

• The flange joint was submerged in liquid nitrogen to obtain its temperature -196 C $^\circ$ – see Figure 11.



Fig. 11 Flange joint after immersing in liquid nitrogen

- Then the joint was placed and tightly closed in the testing chamber.
- Next, the vacuum pumps were started up. The pressure in the chamber was approximately 5.10⁻³ mbar.
- When the suitable vacuum was reached the leakage detector was started up.
- Next, the gasketed joint was pressurized by gaseous helium in the range from 10 to 100 bar in 10 bar increments.
- At each pressure point, the leakage of the helium was measured.
- When the temperature of the flange was smaller than -186.2° C (temperature drops at 5%) the joint was immersed in the liquid nitrogen again.
- When the leakage test was completed the flange joint was disassembled and the gasket's thickness was measured as described in section 3.

5.4 Test results

Figure 12 presents the leakage volume flow (mbar·l/s) as a function of the helium pressure (MPa). This table contains the ten measuring points obtained from the flange joint with gasket No 1.



Fig. 12 Leakage volume flow (mbar·l/s) as a function of the helium pressure for gasket No 1 The value of the leakage volume flow for each tested gasket was collected in the Table from 3 to 5.

Measuring point Ambient		Flange's	Helium	Leakage	Remark
	temperature	temperature	pressure	(volume flow)	
-	°C	°C	MPa	mbar·l/s	-
1	22,7	-196,6	1	5,6·10 ⁻⁹	
2	22,7	-194,1	2	3,3·10 ⁻⁹	
3	22,6	-192,2	3	2,4·10 ⁻⁹	
4	22,7	-189,7	4	2,4·10 ⁻⁹	
5	22,8	-186,8	5	2,4·10 ⁻⁹	
6	22,9	-196,7	6	3,1·10 ⁻⁹	Refreezing
7	22,7	-196,3	7	2,9·10 ⁻⁹	
8	22,8	-195,4	8	2,8·10 ⁻⁹	
9	22,6	-194,2	9	3,1·10 ⁻⁹	
10	22,7	-191,3	10	4,1·10 ⁻⁹	

Table 3 The measuring data concerning joint with sample No 1

Table 4 The measuring data concerning joint with sample No 2

Measuring	Ambient	Flange's	Helium	Leakage	Remark
point	temperature	temperature	pressure	(volume flow)	
-	°C	°C	MPa	mbar·l/s	-
1	22,3	-196,5	1	1,6·10 ⁻⁸	
2	22,3	-191,3	2	1,4·10 ⁻⁸	
3	22,4	-186,5	3	1,3·10 ⁻⁸	
4	22,5	-196,7	4	1,4·10 ⁻⁸	Refreezing
5	22,6	-192,2	5	1,1·10 ⁻⁸	
6	22,6	-191,3	6	9,6·10 ⁻⁹	
7	22,7	-188,8	7	9,0·10 ⁻⁹	
8	22,7	-186,5	8	9,3·10 ⁻⁹	
9	22,8	-196,6	9	1,2·10 ⁻⁸	Refreezing
10	22,7	-195,5	10	1,1·10 ⁻⁸	

Table 5 The measuring data concerning joint with sample No 3

	1				
Measuring	Ambient	Flange's	Helium	Leakage	Remark
point	temperature	temperature	pressure	(volume flow)	
-	°C	°C	MPa	mbar·l/s	-
1	22,5	-196,7	1	1,20·10 ⁻⁸	
2	22,7	-192,4	2	1,10·10 ⁻⁸	
3	22,7	-188,1	3	9,50·10 ⁻⁹	
4	22,8	-186,3	4	8,90·10 ⁻⁹	
5	22,8	-196,6	5	9,80·10 ⁻⁹	Refreezing
6	22,7	-194,4	6	7,00·10 ⁻⁹	
7	22,7	-192,2	7	7,10·10 ⁻⁹	
8	22,6	-191,1	8	7,40·10 ⁻⁹	
9	22,7	-188,2	9	7,70·10 ⁻⁹	
10	22,7	-187,3	10	8,70·10 ⁻⁹	

<u>Mass flow</u>

To convert the volume flow obtained directly from leakage detector the equation of state for ideal gas was used:

$$pV = \left(\frac{m}{M}\right)RT\tag{1}$$

where:

p – gas pressure [mbar],

V-gas volume [I],

m – gas mass [g],

M – molecular of the gas [g/mol],

R – the molar mass constant [mbar·l/(mol·K)],

T – gas temperature, [K].

Inserting to equation (1) the volume flow q_L in mbar·l/s units we get:

$$q_m = \frac{q_L \cdot M}{R \cdot T} \tag{2}$$

where:

 q_m – mass flow [g/s].

In equation (2) the number of the molar gas equals R= 83.145 [mbar·l/(mol·K)] and molar of the helium is M = 4 [g/mol]. The temperature of the helium was assumed as an ambient temperature in Kelvin.

Tables from 6 to 8 contain the data of the helium mass flow as a function of the helium pressure.

Helium Flange's Measuring Helium Leakage Leakage (mass flow) point temperature temperature pressure (mass flow) -°C MPa g/s mg/s Κ 295,85 1 -196,7 1 9,11E-13 9,11E-10 2 2 295,85 -192,4 5,37E-13 5,37E-10 295,75 3,90E-13 3,90E-10 3 -188,1 3 4 4 295,85 -186,3 3,90E-13 3,90E-10 5 5 295,95 -196,6 3,90E-13 3,90E-10 6 6 5,04E-10 296,05 -194,4 5,04E-13 7 295,85 -192,2 7 4,72E-13 4,72E-10 8 295,95 -191,1 8 4,55E-13 4,55E-10 9 295,75 -188,2 9 5,04E-13 5,04E-10 6,67E-13 6,67E-10 10 295,85 -187,3 10

Table 6 The measuring data concerning joint with sample No 1

Measuring	Helium	Flange's	Helium	Leakage	Leakage
Wiedsumg	ricium	Tidlige 5	ricitatii	LCurrage	LCukuge
point	temperature	temperature	pressure	(mass flow)	(mass flow)
-	К	°C	MPa	g/s	mg/s
1	295,45	-196,5	1	2,61E-12	2,61E-09
2	295,45	-191,3	2	2,28E-12	2,28E-09
3	295,55	-186,5	3	2,12E-12	2,12E-09
4	295,65	-196,7	4	2,28E-12	2,28E-09
5	295,75	-192,2	5	1,79E-12	1,79E-09
6	295,75	-191,3	6	1,56E-12	1,56E-09
7	295,85	-188,8	7	1,46E-12	1,46E-09
8	295,85	-186,5	8	1,51E-12	1,51E-09
9	295,95	-196,6	9	1,95E-12	1,95E-09
10	295,85	-195,5	10	1,89E-12	1,89E-09

Table 7 The measuring data concerning joint with sample No 2

Table 8 The measuring data concerning joint with sample No 3

Measuring	Helium	Flange's	Helium	Leakage	Leakage
point	temperature	temperature	pressure	(mass flow)	(mass flow)
-	К	°C	MPa	g/s	mg/s
1	295,65	-196,7	1	1,95E-12	1,95E-09
2	295,85	-192,4	2	1,79E-12	1,79E-09
3	295,85	-188,1	3	1,54E-12	1,54E-09
4	295,95	-186,3	4	1,45E-12	1,45E-09
5	295,95	-196,6	5	1,59E-12	1,59E-09
6	295,85	-194,4	6	1,14E-12	1,14E-09
7	295,85	-192,2	7	1,15E-12	1,15E-09
8	295,75	-191,1	8	1,20E-12	1,20E-09
9	295,85	-188,2	9	1,25E-12	1,25E-09
10	295,85	-187,3	10	1,41E-12	1,41E-09

The graphical presentation of the mass flow (leakage) as a function of helium pressure was shown from Figure 13 to Figure 15.



Fig. 13 Mass flow (leakage) as a function of helium pressure for sample No 1



Fig. 14 Mass flow (leakage) as a function of helium pressure for sample No 2



Fig. 15 Mass flow (leakage) as a function of helium pressure for sample No 3