Research Article

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Testing of the gaskets at liquid nitrogen and ambient temperature

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Abstract: The paper presents the results of the leakage measurements of bolted flanged joints operating at temperature of liquid nitrogen (77K) and ambient temperature. Three types of static gasket were tested. The results were presented in the form of a helium leakage rate as a function of its pressure. The measured data was discussed and compared with the leakage results obtained at the ambient temperature.

Keywords: gasket, nitrogen, leakage, cryogenic

1 Introduction

The cryogenic market is currently the most dynamically developing energy sector in Europe. An example can be huge cryogenic installations built in recent years, such as CERN, ITER as well as currently implemented ESS project (European Spallation Source) or the first Polish LNG terminal built in 2015. Its regasification capacity is 5 billion cubic meters per year and it is planned to increase to 7.5 billion cubic meters. As a result, it will cover nearly 50% of Poland's natural gas demand. Moreover, the LNG is used as an alternative fuel to propulsion of engines of the ships. The gasket used in such severe environments should be certified in line with requirements e.g. in [1–3]. One of the main design and operating problems of this type of installation is the maintenance of an appropriate level of tightness of the pipeline system, which includes hundreds of flanged joint connections. Problems related to the sealing of low-temperature media are discussed in [4–10]. The issues included in those papers mainly focused

on the troubles of the thermal contractions of the metal gaskets utilized in liquid oxygen pipeline systems e.g. in thermal cryogenic reactor [8] or distribution fuel systems of the rockets engines [9]. So far, in the cryogenics installations were equipped only with the gaskets made of metal, such as indium, copper, nickel or aluminum. The paper [8] presents the results of tightness testing of a bolted-flange joint with the gasket made of the above-mentioned materials. The model of the thermo reactor with the gasket was placed in a vacuum chamber and cyclically subjected to a variable temperature in the range of 293 K to 11 K. In the test used liquid helium under pressure ranging of 2 to 31 bar and then cooled down the flange joint in a cyclic way starting from 293 K up to 11 K. The leakage was measured by the vacuum method using helium detection. The gaskets made of indium, copper, nickel, aluminum and stainless steel 304 were tested. In all cases, the leakage of the helium was smaller than $1 \cdot 10^{-9} \text{ cm}^3/\text{sec}$ it means that all gaskets materials have a very good sealing properties. The drawback of this kind of materials is their high relative price. The alternatives can be the gaskets made of nonmetallic materials made of expanded graphite, PTFE or elastomeric-fibers sheet. Revision of the literature connected with the nonmetallic gaskets used in cryogenic temperatures claimed that there is no sufficient data about of their sealing properties and thermal behavior. The most interesting data were found in technical report [10] describes the leakage level of the bolted flange joint equipped with the elastomeric-fibers gasket cyclically subjected to temperature range of 293 K to 77 K. The results confirmed that the elastomeric-fibers gasket met general requirements of permissible emission in accordance with [11]. Nevertheless, the assessment of the leakage was a very superficial, since the bubble measuring method used in this test was insufficient accurate. The paper [12] presents the research results of the composite gasket made of PTFE and epoxy resin as well as the gasket made of elastomeric-asbestos fibers. The flange joint with the particular gaskets was placed in the vacuum chamber end cooled down to the temperature 77 K using liquid nitrogen. The results were not give the full background about of the leakage behavior of those materials, since the pre-

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sented data included only the one load case of internal pressure 40 bar. Based on literature review it was found that the data of the behavior of the nonmetallic gaskets materials as well as semi-metallic gaskets used in cryogenic temperatures (especially in liquid nitrogen) are inadequate and should be enlarged.

2 Aim and scope of the work

The aim of this work was to examine the influence of the liquid nitrogen temperature (77 K) on the leakage level of the gaskets used in the bolted flanged joint. Based on the literature review [4–12], it was found that there are no proper methods that would allow to accurately analyze the leakage rate and assess the behavior of the nonmetallic material used as a gaskets subjected to cryogenics conditions. In [13] the proposal of the test rig and the method of conducting the test procedure were proposed. Nonetheless, the bubbling method used in this test seems to be not practical and allows only to captures the leakage of a relatively high value. To more accurate leakage measure, the helium detection method is a better solution [14]. For this purpose, the authors designed and built a test rig, which was previously numerically analyzed in order to determine the heat flux as well as to determine the distribution of the stress and deformation state in the particular components. Three types of gaskets were tested. Two semi-metallic gaskets: the former was kammprofile gasket layered with expanded graphite and the latter was kammprofile gasket layered with PTFE. The third kind of gasket was the soft gasket made of expanded graphite. The experiment was conducted at four values of the gaseous helium pressure: 2, 4, 6 and 8 bar. The initial contact stress on the gasket surface was generated indirectly by the tension of the bolts using a torque wrench. The torque value was chosen in such a way that the resulting force from four bolts caused approximately 50 MPa of the contact pressure acting on the gasket surface.

3 Concept of the experimental rig

Figure 1 shows the scheme of the experimental test rig. The main element was a bolted flanged joint assigned as DN40 PN40 with the gasket.

In order to assure a proper thermal insulation (limitation of the heat flux from the environment) and to accurate collection of the helium leakage, the flange joint was placed in a tightly closed chamber made of stainless

Figure 1: Scheme of the experimental test rig for assessing the leakage of flanged joint operating at cryogenic temperature.

steel 1.4301. Before placing the joint in the chamber, it was cooled down (by immersion in liquid nitrogen) to -196°C (77 K). Then by means of the vacuum pump, air was pumped out of the chamber. Subsequently, helium pressure was supplied to the inside of the flange joint via a copper capillary. The chamber construction together with the flanged joint was presented in Figure 2.

Figure 2: Construction of the test chamber and flanged joint.

4 Numerical analysis of the experimental rig

In order to assess the reliability of the test rig the whole construction was subjected to numerical analysis by means of finite element method. The main goal was to determine the heat flux and the temperature distribution in the particular elements of the test rig. In addition, the numerical analysis allowed to determine the maximum stress and displacement resulting from mechanical and thermal loads.

4.1 The numerical model

Due to the fact that geometrical model of the test rig had a cyclic symmetry, the axisymmetric model was used into computation. Figure 3 presents an axisymmetric model of the test rig and its division onto finite elements. Figure 4 presents the thermal as well as mechanical boundary conditions. Thermo-structural coupled analyzed in the steady state was used. The boundary condition of that model were following:

- In the first step the temperature of 77 K (liquid nitrogen) was applied to the outer surfaces of the flange joint to simulate its cooled down.
- The external surfaces of the chamber were conditioned as a natural convection - 5 W/m^2 .
- In the second step the mechanical boundary conditions were applied in the form of temperature distribution obtained from the first step,
- vacuum pressure inside the chamber was applied,
- maximum internal pressure in the flange joint was applied,

Figure 3: Numerical model a) axisymmetric model of the test rig, b) finite element mesh in the area of the flanged joint and the part of the chamber.

Figure 4: The boundary conditions a) thermal boundary conditions, b) mechanical boundary conditions.

- gravity resulting from the mass of the particular elements was applied.

4.2 The results of numerical analysis

Figure 5 presents the temperature distribution and the heat flux in individual elements of the test rig. It can be seen that the minimum temperature occurred in the upper part of the chamber (connection of the flange with the cover) did not fall below 0°C . Therefore, it was decided to use a rubber O-ring to seal the chamber-cover connection.

Figure 6a presents the distribution of the equivalent stress in a whole structure. The maximum stress was 116 MPa and occurred at the junction of the bottom of the chamber with the cylindrical part. This value is at acceptable level.

Figure 6b presents a map of total deformation. The maximum displacement occurred in the bottom of the cylinder and it not exceeded 1 mm. The above numerical results provide that proposal construction of the test rig will be able to withstand the applied load.

5 The experiment

Above presented construction of the test rig was used to examine the leakage of the gasketed bolted flange joint subjected to temperature 77 K as well as ambient temperature. Three types of gaskets were tested: kammprofile gaskets layered with expanded graphite, kammprofile gaskets layered with PTFE and soft-material gasket made of expanded graphite. In the first step the internal and external diameter as well as the thickness of the particular gaskets were measured. Then the gasket was installed between flanges and bolted. The tension in the bolts was induced by an indirect method, using a torque wrench with the appropriate torque setting. The torque value was set in such a way that the tension force of the four bolts generated a contact pressure on the gasket equals 50 MPa. Then the joint was cooled down by submerging it in liquid nitrogen. Cooling time was determined based on the thermal stabilization of the system. In the moment when the bubbles of the nitrogen vapors disappeared it meant that the temperature of the flange joint 77 K was achieved. Then the joint was put in the chamber and air was pumped out of it. In result the vacuum of $4 \cdot 10^{-3}$ bar was obtained. In the next step the tested joint was loaded with helium pressure in the range of 2 to 8 bar. After reaching the target value of the given pressure, the leakage was measured using a helium detec-

Figure 5: Results of the thermal analysis, a) temperature distribution, b) heat flux

Figure 6: Results of the thermo-structural analysis: a) distribution of the equivalent von Mises stress, b) total deformation.

tor. The measurement was terminated when the leakage of the helium was stabilized. The effect of stabilization of the leakage of helium at particular pressure was presented in Figure 7. After the measurements were completed, the chamber was opened, the gasket was removed and measured again at three points around the circumference. The entire test procedure followed in several basic steps:

1. Measuring the geometry of the gasket: inside and outside diameter as well as thickness at three points around the circumference.
2. Installation of the gasket in the joint, tightening the nuts with the proper torque value,
3. Submerging the flanged joint in liquid nitrogen.
4. Placing the joint in a sealed chamber.
5. "Pumping" of the vacuum.
6. Loading the joint with helium pressure.

7. Measurements of helium leakage.
8. Decompression of the chamber.
9. Disassembly of the joint.
10. Measurement of the gasket.

Figure 8 shows the photo of the test rig according to the authors concept. In Figure 9 a) and 9 b), the photo of the joint before and after cooling to 77 K were presented respectively.

Figure 7: An example of stabilization of helium leakage from a flanged joint gasketed with expanded graphite.

Figure 8: Experimental test rig to assess the leakage from the flanged joint.

6 Measurement results

The result of the test were the characteristics showing the dependence of the helium leakage from the joint as a function of its pressure - Figure 10.

Based on figure 10 it can be seen that at ambient temperature the lowest leakage rate in the whole helium pressure range was obtained in case of kammprofile gasket with PTFE layers. At a maximum pressure of 8 bar, the leakage rate in this case was $1 \cdot 10^{-6}$ mg/m·s. The leakage values obtained for the kammprofile gasket with a graphite layer and a soft-material expanded graphite gasket were similar. Their leakage at helium pressure of 2 bar was nearly the same $5 \cdot 10^{-5}$ mg/m·s and at 8 bar the leakage of the kammprofile gasket whit graphite was $4 \cdot 10^{-4}$ mg/m·s and for graphite was $3 \cdot 10^{-4}$ mg/m·s.

The situation was drastically changed in case where the joint was cooled down to the temperature of 77 K. The leakages of this case were presented in Figure 11. Compared to the leakage obtained at ambient temperature, significant changes occurred in case of the kammprofile gasket with PTFE layer – Figure 12. The leakage at pressure of 2 bar was $5.75 \cdot 10^{-4}$ mg/m·s and increased by two orders of magnitude compared to the leakage measured at ambient temperature. At pressure of 8 bar leakage increased to $4.38 \cdot 10^{-3}$ mg/m·s. In case of the two other gaskets the changes were small – Figures 13 and 14. However, they show that freezing the flanged joint to 77 K causes an increase of the leakage.

For a better comparison of the gaskets behavior, their leakage characteristic at ambient and 77 K temperature were collected and presented in the Figure 15.

7 Summary and conclusions

The main conclusion from the work are following:

- The proposal of the new test rig to carry out the gaskets both at ambient and 77 K can safe work under internal pressure of 40 bar and the same it may be subjected to temperature range from 293 K to 77 K.
- The all gasket subjected to ambient temperature exhibited a good sealing properties. In all cases the leakage rate did not exceeded the value of $1 \cdot 10^{-2}$ mg/m·s.
- The kammprofile gasket layered with PTFE exhibited the best solution at the room temperature and pressure range of 2 to 8 bar.
- The leakage test carried out at the temperature of 77 K caused that the leakage of all tested gaskets increased.
- The kammprofile gasket layered with PTFE exhibited the worse sealing property at 77 K and it shouldn't be recommended in cryogenic applications.

Figure 9: Tested flanged joint; a) before cooling, b) after cooling to 77 K.

Figure 10: Characteristics of helium leakage from the flanged joint gasketed with particular gaskets at room temperature.

Figure 11: Characteristics of helium leakage from the flanged joint gasketed with particular gaskets at 77 K.

During the test, several fundamental problems were noticed:

- The stabilization time of the helium leakage rate both ambient and cryogenic temperature depends on the level of joint tightness. The higher tightness of the joint the longer time of the leak stabilization.
- The gasket should not be mounted on cooled flanges. This is due to the fact that the flanges at

the cryogenic temperature quickly become frozen because they collect moisture from the surrounding air, covers the sealing surfaces with the ice crystals. This effect prevents the gasket from being properly formed and consequently causes a higher leakage. Therefore, to provide the correct assembly of the bolted flange joint with gasket the mating surface should be properly clean and dry.

Figure 12: Characteristics of the helium leakage from a flanged joint gasketed with the kammprofile layered of PTFE at ambient and 77 K.

Figure 13: Characteristics of helium leakage from the flanged joint gasketed with the expanded graphite gasket at ambient and 77 K.

Figure 14: Characteristics of helium leakage from a flanged joint gasketed with the kammprofile gasket layered of graphite at ambient and 77 K.

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Figure 15: The compression of the leakage rate of particular gaskets at ambient and 77 K temperature.

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