

DEVICE FOR HIGH-TEMPERATURE AND HIGH-PRESSURE CYCLIC TESTS OF SEALING AND PRELIMINARY EXPERIMENTAL RESULTS

¹ Daniela MARUŠÁKOVÁ, ¹ Rostislav FUKAČ, ¹ Tomáš MELICHAR, ¹ Jan VÍT

¹ CVŘ – Centrum Výzkumu Řež, Hlavní 130, Husinec-Řež, 250 64, Czech Republic, EU,
daniela.marusakova@cvrez.cz

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Abstract

A characteristic feature of advanced energy devices, such as Generation IV nuclear reactors, is operation combining high temperatures and pressures of working media. One of the key issues is ensuring the tightness of joints of metal components at very high temperatures (above 600 °C). At the same time, it is necessary to ensure tightness during non-nominal operating conditions associated with temporal changes in operating parameters. Commonly, seal (gasket) in Czech Republic is tested according to the ČSN EN 13 555 standard, where the combination of high operating temperature and high pressure changing over time with a chosen rate of change is not considered. For this reason, a unique device for testing gaskets was developed and commissioned at the Research Centre Řež (CVR). The facility allows characterization and tightness tests of different sealing products at wide temperature (up to 700 °C) and pressure (up to 40 MPa) that might also be time dependent. The facility also allows testing of the entire gasket + flange + bolts assembly up to an outer diameter of 150 mm. In this paper, design, capabilities, and the first operational experience are mentioned. In addition, preliminary results from testing gasket with a metallic core are mentioned. The device and the associated research can contribute to increasing the efficiency of sealing metal flange joints and other components and developing methodologies for evaluating sealing elements under extreme conditions, thus increasing the safety of operation of energy or other process equipment operating at high pressures and temperatures.

Keywords: Sealing, tightness, high temperature-high pressure, leak rate, methodology

1. INTRODUCTION

To ensure the safe operation of CVR's high-temperature technologies under pressure (such as UCWL, SCWL, He loops, and other large infrastructures), which are crucial for material research, especially for Generation IV reactors, it is necessary to determine and test suitable seals (gaskets) that can withstand high operating temperatures and pressures (700 °C, 25 MPa). The current approach for evaluating seals is defined by the European Standard ČSN EN 13 555 (ES1). But for high-temperature applications it does not account for non-homogeneous and time-varying temperature fields, nor does it address sealing the entire flange as a complete set. The method for determining these characteristics in a laboratory setting is prescribed by this ES1, for each size and type of seal, these characteristics must be newly determined to perform the design calculations of flange joints [1].

Flange joints and their seals are essential components of any pressure equipment, found across all fields of engineering and industry. A well-designed flange joint and its sealing significantly impacts the safety and economic efficiency of the entire pressure system. Seal failures can lead to unplanned shutdowns and substantial damages. Additionally, the leakage of certain substances into the environment can pose an ecological burden. To mitigate these risks, the European Union has decided to invest more in the design and implementation phases of flange joints to reduce the incidence of seal failures. The design of flange joints for

pressure equipment is now governed by another European Standard ČSN EN 1591 (ES2), which does not rely on empirical relationships to determine the clamping force of the flange based on the type of seal, as other national standards do. Instead, it establishes a procedure for determining flange parameters through iterative calculations based on laboratory-determined characteristics of the clamped seal [2]. To determine the numerical values of the seal characteristics in the tightness test, it is necessary to choose the tightness class of the given flange joint. Tightness classes are denoted as L_N , where N takes values in orders such as 1; 0.1; 0.01 and determines the specific leak rate of the joint in units ($\text{mg s}^{-1} \text{m}^{-1}$). The leak rate of the given tightness class L_N must then take the value N or lower, up to the leak rate value of the next tightness class. [1].

Graphite-based gaskets are commonly used in high-temperature applications, but their sealing performance must be carefully evaluated under thermal and mechanical loads. There exist several types of tests for graphite gaskets. Leakage tests at elevated temperatures are typically performed with helium as a test gas, using mass spectrometry or pressure decay methods to measure leakage rates. These tests, often conducted according to ES1, involve heating in steps up to 600 °C under constant gasket stress to assess tightness as a function of temperature [3]. In addition, graphite gaskets are susceptible to creep—permanent thickness reduction—which can reduce sealing performance. Creep relaxation tests, also standardized in ES1, involve compressing the gasket, heating it to high temperatures, and measuring the remaining gasket stress over time [4]. Oxidation resistance is another critical factor, as graphite can degrade in oxidizing environments at temperatures above 400 °C. This is typically evaluated through thermogravimetric analysis (TGA), where mass loss and oxidation rates are monitored [5]. Finally, thermal cycling tests simulate service conditions by repeatedly heating and cooling gaskets under load to assess stress retention and leakage stability [6]. This paper is focused on leakage testing by newly developed device.

2. EXPERIMENTAL PART

In this section, the design and operational parameters of the experimental device are described.

2.1 Overview of the Horizontal High Temperature Furnace 2

A device for testing seals at high temperatures with the capability of thermal cycling was developed at CVR. The Horizontal High Temperature Furnace 2 (HHTF2) stand with servo-pneumatic pressurizing system, **Figure 1**, can accommodate an entire flange joint or its scaled-down model to study the effects of medium pressure, thermal expansion coefficients of the materials used, leak rate, temperature, and its change. The stand allows for determining the suitability of the design of the tested flange joint and the sealing materials used as a whole sealed joint by establishing its tightness class. The HHTF2 stand cannot determine the tightness diagram for a given seal, but it can determine the tightness class of the entire flange joint under any conditions. Therefore, the HHTF2 stand is not a competitor to specialized equipment designed to measure seal characteristics in accordance with standard ES1, but rather a complement to them in areas where the standardized approach fails.

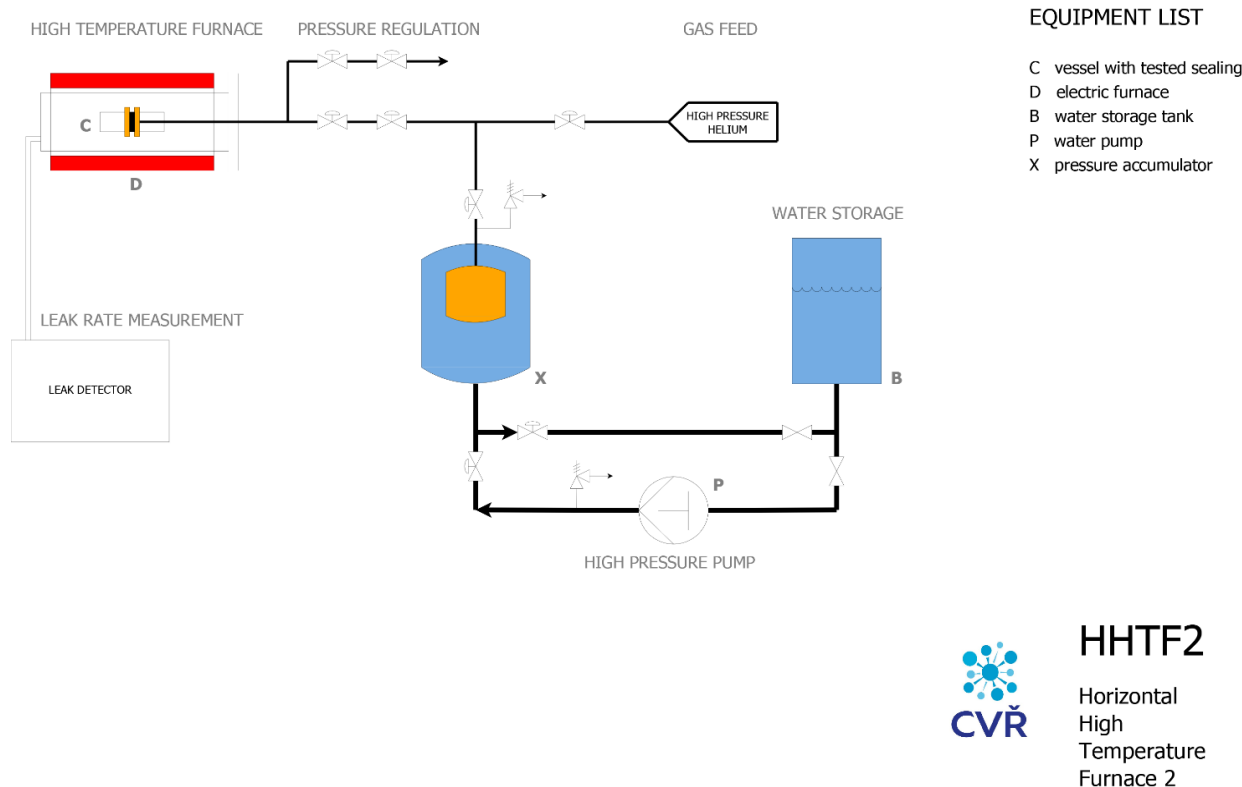


Figure 1 Schema of the Horizontal High Temperature Furnace 2

The main source of helium pressure for leak testing of flange joint is a high-pressure water pump (P). Compressed helium is stored in a pressure accumulator (X) for the experiment and the control system maintains the helium pressure in the flange joint in the vessel (C) throughout the experiment. A leak detector is directly connected to a vacuum vessel with an internal diameter of 150 mm to measure helium leak rate through the flange joint. Minimum detectable leak rate of leak detector is $< 5 \cdot 10^{-12}$ mbar·l·s⁻¹ of ⁴He.

Main parameters of the HHTF2 stand:

- Maximum operating pressure of test helium: 40 MPa.
- Maximum operating temperature of the tested flange joint: 700 °C.
- Maximum diameter of the tested flange joint: 150 mm.
- Maximum length of the tested flange joint: 600 mm (homogeneous temperature field), 1500 mm overall.

2.2 Technical specification of flange joint

A flange joint with a spiral-wound expanded graphite gasket (seal) from Dimer was placed in the vacuum vessel. This is a seal with an internal stabilizing ring with an outer diameter of 30 mm, an inner diameter of 20 mm and a thickness of 5 mm. The flange joint was arranged according to the following **Figure 2**.

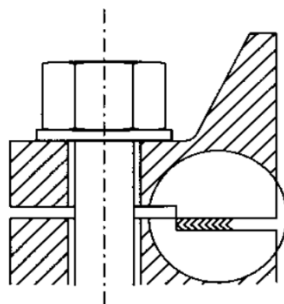


Figure 2 The flange joint arrangement

The material of the flange parts is stainless steel 316L. The four bolted joints of the flange are made with M16 x 1.5 bolts which are made of material 1.4980. The flange joints were pre-tensioned with a tightening torque of 33 Nm before the experiment. Since the flange is placed in a vacuum, these bolted joints were not lubricated before assembly.

3. RESULTS AND DISCUSSION

The first test run of HHTF2 is a part of the commissioning of the experimental device and the main purpose is to prove its operability. The first results of the experimental program are presented in this section.

3.1 Measurement of leak characteristics at room temperature

The leak characteristics of the assembled flange joint in the initial state were measured both under high vacuum and overpressure and are given in **Table 1**. The leak rate of the flange with internal vacuum was measured by connecting it directly to a He analyser. The outside of the flange joint was blown with helium in air (out of pile). The leak rate of the flange joint with internal overpressure was measured in a vacuum vessel HHTF2 (in pile), which is connected to a He analyser as same as in **Chyba! Nenalezen zdroj odkazů..**

Table 1 Initial flange joint characteristics at room temperature

Pressure in flange joint	Temperature (°C)	Pressure of medium (bar)	Leak rate (mbar·l·s ⁻¹)
High vacuum (2.5×10^{-3} mbar)	22	1 (He + air)	$1.4 \cdot 10^{-9}$
Overpressure	21	200 (He)	$1.43 \cdot 10^{-3}$

The measured leak rate 1.43×10^{-3} mbar·l·s⁻¹ corresponds to the requirements for tightness class L_{0.01} according to ES1, i.e. it is a suitable seal for industrial use at room temperature.

3.2 Measurement of characteristics at higher temperature

The tightness characteristics of the assembled flange joint under elevated temperature and pressure were determined at a furnace temperature gradient of 10 °C·min⁻¹, **Figure 1** (D), and a pressure of 200 bar throughout the experiment. Firstly, a temperature of 600 °C was reached with a holding time of approximately 1.5 hours, and then a temperature of 680 °C was reached with a holding time of approximately 1.75 hours. Subsequently, the furnace was switched off and cooled down naturally for approximately 10 hours. Inside the vacuum vessel, the flange joint was heated with a significantly slower temperature change, with a maximum temperature of 550 °C. The temperature change in the flange joint reached its maximum of 250 °C·hour⁻¹. The measured values are in the following diagram, **Figure 3**.

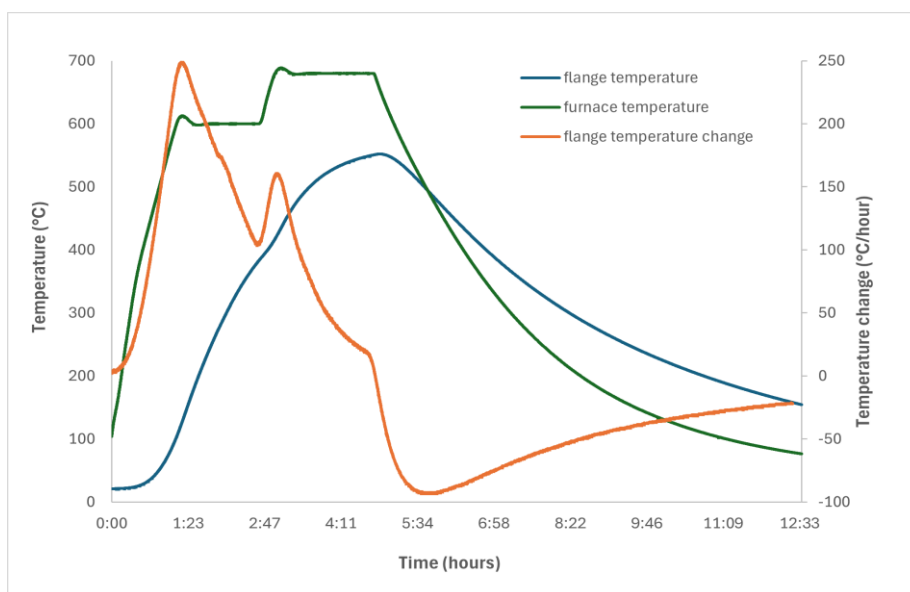


Figure 3 Diagram of furnace temperature, flange temperature and *flange* temperature change in dependence on time

The flange joint leak rate, flange temperature, and flange temperature change during the experiment are shown in **Figure 4**. Changes in the leak rate over time appear to correlate with the flange temperature change over time.

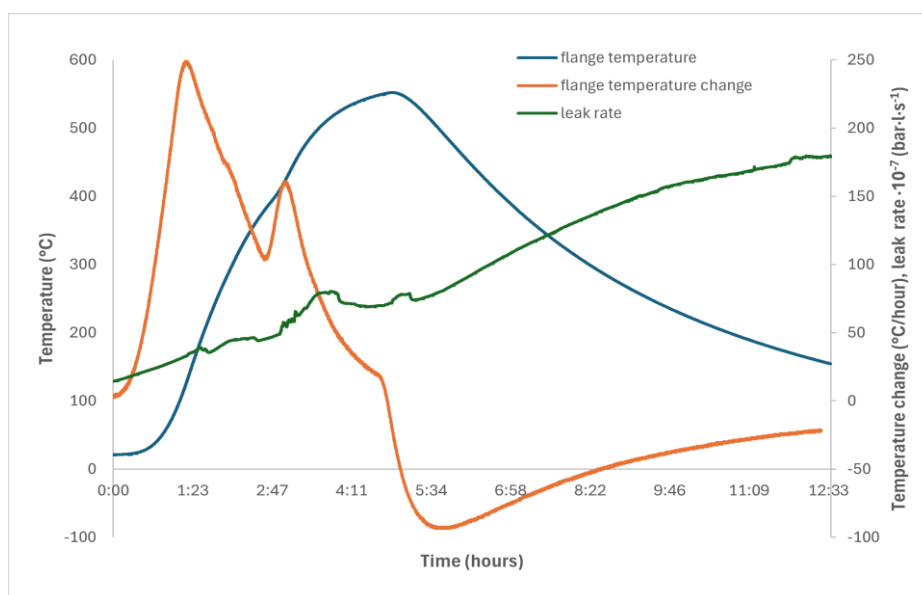


Figure 4 Diagram of flange temperature, flange temperature change and leak rate in dependence on time

The dependence of the flange leak rate and flange temperature change on time is shown in **Figure 5**. It is worth noting that the leak rate does not return to its initial value after its thermal exposure. After 14 hours from the start of the experiment, the flange joint completely cooled down and the leak rate asymptotically approached the value of $2.1 \cdot 10^{-2} \text{ mbar} \cdot \text{l} \cdot \text{s}^{-1}$, i.e. a value approximately 3 times higher than the initial value of the flange joint leak rate.

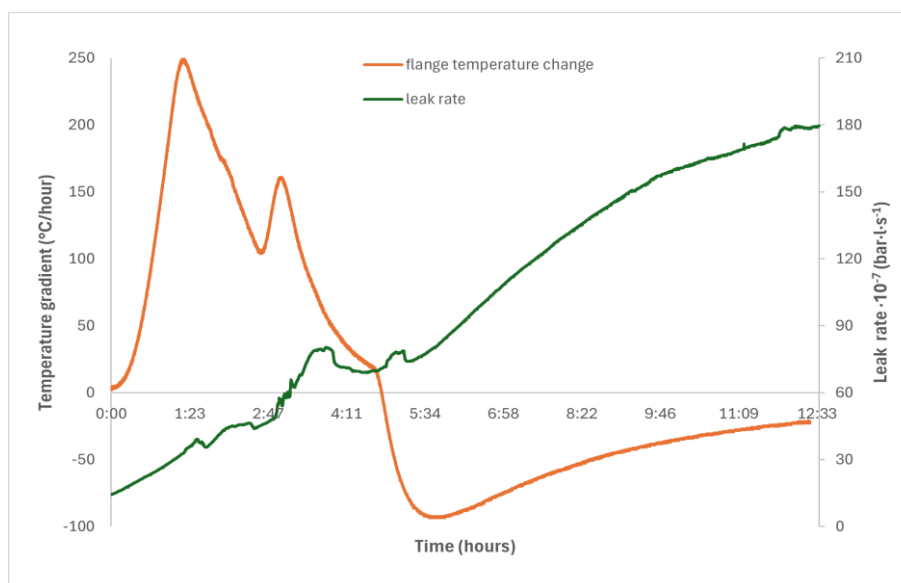


Figure 5 Diagram of flange temperature change and leak rate in dependence on time

4. CONCLUSION

To ensure the safe operation of large technological infrastructures, and to support the development and testing of sealings for advanced energy technologies, a completely unique device, HHTF2, was developed at the Research Centre Řež, which is designed to test the tightness of flange joint at high temperatures and pressures. The functionality of this stand was verified using this test experiment with a commonly available spiral-wound gasket made of expanded graphite. The flange joint leak rate is related, among other things, to the flange temperature change on time. This dependence is evident from the correlations of changes in these quantities over time. And finally, the flange joint exhibits hysteresis when heated. The first test demonstrated sufficient functionality of the tested flange joint with gasket in accordance ES1.

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REFERENCES

- [1] ČSN EN 13555. Příruby a přírubové spoje – Parametry těsnění a postupy zkoušení vztahující se k pravidlům pro navrhování přírubových spojů s kruhovými přírubami a těsněním. Praha, CZ: Úřad pro technickou normalizaci, metrologii a státní zkušebnictví, 2021.
- [2] ČSN EN 1591 – 1. Příruby a přírubové spoje – Pravidla pro navrhování těsněných kruhových přírubových spojů – Část 1: Výpočet. Praha, CZ: Úřad pro technickou normalizaci, metrologii a státní zkušebnictví, 2025.
- [3] WÜTHRICH, W., BEHR, M., GRIESBACH, V. High temperature sealability and creep relaxation of graphite gasket materials. Sealing Technology. 2006, Vol. 2006, No. 6, pp. 6–10. [https://doi.org/10.1016/S1350-4789\(06\)70546-9](https://doi.org/10.1016/S1350-4789(06)70546-9)
- [4] BICKFORD, T., NELSON, G. Characterization of high temperature creep of graphite gaskets. In: Pressure Vessels and Piping Conference. Chicago: ASME, 2008, pp. 295-304.
- [5] CELZARD, A., MARÉCHAL, D., FIERRO, V., FAVRE, T., DELEENS, E., FIÉVET, P. Oxidation resistance of graphite-based gasket materials. Carbon. 2002, Vol. 40, No. 15, pp. 2801–2814. [https://doi.org/10.1016/S0008-6223\(02\)00179-2](https://doi.org/10.1016/S0008-6223(02)00179-2)
- [6] KAWASHIMA, S., ITOH, M., OGAWA, H. Mechanical properties of graphite gaskets at elevated temperatures under thermal cycling. Journal of Pressure Vessel Technology. 2011, Vol. 133, No. 3, pp. 031203-1–031203-7. <https://doi.org/10.1115/1.4003352> LFS je: 504LFS.