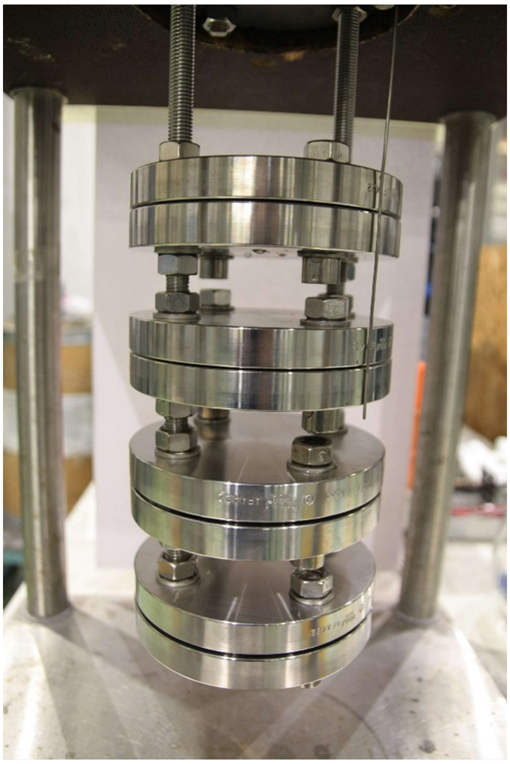
RESEARCH REPORT VTT-R-00724-24



Graphite sealant testing in high temperature water

Authors: Konsta Sipilä, Pasi Väisänen

Confidentiality: VTT Public

Version: 19.12.2024

beyond the obvious

Report's title Graphite sealant testing in high temperature water	
Customer, contact person, address SAFER2028	**Order reference**
Project name Practical solutions for sealant performance issues in nuclear power plants	**Project number/Short name** 137893/PRANCS
Author(s) Konsta Sipilä, Pasi Väisänen	**Pages** 13/
Keywords Graphite, sealant, high temperature water, compression set	**Report identification code** VTT-R-00724-24

Summary

Graphite is used in different types of sealant applications in nuclear power plants, including control rod penetrations, pressurizers, steam generators, pipes, exchangers and valves. In these applications they are subjected to high temperatures and pressure, which cause ageing of the sealants. In this work, a testing setup for graphite sealants based on compression set was designed, constructed and tested. The testing included subjecting four different types of graphite samples to high temperature water. After the exposure the compression set values were calculated for the samples and a suitable acceptance criterion value was discussed. To obtain a suitable acceptance criterion, additional pressure testing should be performed on the samples to correlate the calculated compression set values to the functionality of the sealants.

Confidentiality	VTT Public

Espoo 31.1.2025

Written by	**Reviewed by**
Konsta Sipilä Senior Scientist	Aki Toivonen Principal Scientist

VTT's contact address
P.O.Box 1000. FI-02044 VTT, Finland

Distribution (customer and VTT)

One copy VTT and one copy SAFER_2028.

Approval

VTT TECHNICAL RESEARCH CENTRE OF FINLAND LTD

Date:	
Signature:	Vepsäläinen Mikko 912690851 Digitally signed by Vepsäläinen Mikko 912690851 Date: 2025.03.06 15:27:37 +02'00'
Name:	Mikko Vepsäläinen
Title:	Research Team Leader

Preface

This report has been issued as part of the project “Practical solutions for sealant performance issues in nuclear power plants” (PRANCS) within the SAFER 2028 -research programme. The Finnish state nuclear waste management fund VYR and VTT Technological Research Centre of Finland Ltd are acknowledged for funding this work.

Espoo 31.1.2025

Authors

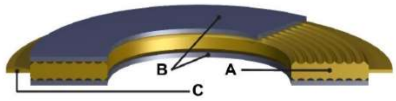
Contents

1. Introduction

Different types of seals, gaskets and sealants are used to ensure the tightness of various types of industrial systems and components, e.g., pumps, valves, structural elements and so on. Applications involving high temperatures and pressures demand high performance from the sealant material and usually exclude the use of polymer-based materials. Graphite-based sealants are a feasible option for these types of environments. Graphite can be used alone or as a part of semi-metallic gaskets, where the gasket is reinforced with a metallic structure. Examples of semi-metallic gasket types are shown in Figure 1.

A=Metal Insert; B=Non-metallic sheet material

Metal Reinforced Non-metallic Gasket



A=Metal Core; B=Soft Facing Material; C=Metal Outer Ring

Kammprofile Gasket

A=Metal Corrugations; B=Soft Facing Material

Corrugated Gasket

A=Outer Ring; B=Filler Material; C=Metal "V" Windings; D=Inner Ring

Spiral Wound Gasket

A=Metal Jacket (Outer Layer); B= Filler Material

Metal Jacketed Gasket

A=Slit Type; B=Folded Type; C= Machined/Square Type

Envelope Gaskets

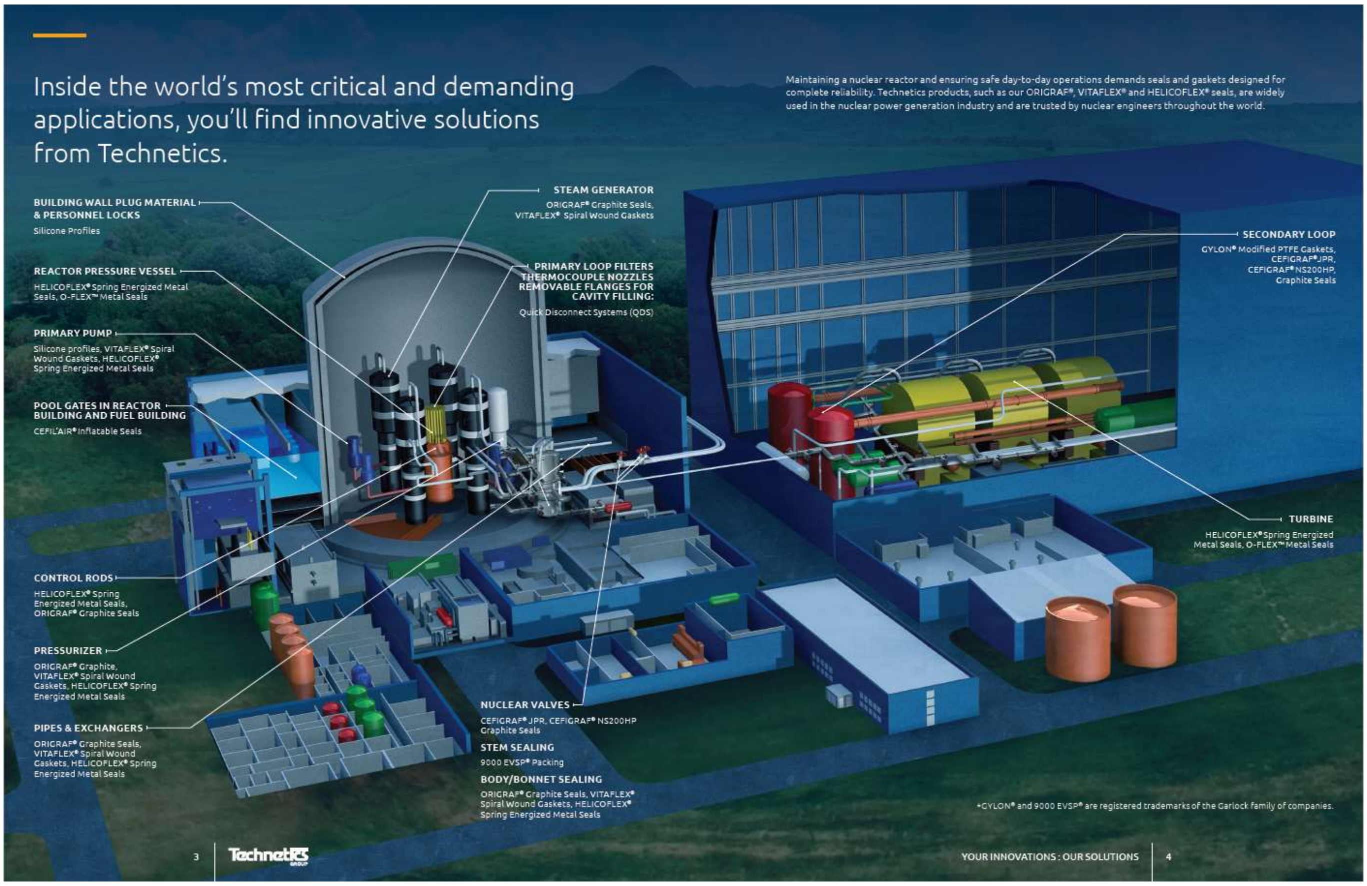
Figure 1. Semi-metallic gasket types (collected and modified from [1]).

The ageing of graphite sealants has been earlier assessed in [2]. High temperature and radiation exposures affect the mechanical properties, chemical reactivity, and dimensional stability of graphite. One sealant type, spiral wound gaskets, is responsible for 46% of all PWR primary leakages. Spiral wound gaskets are typical sealant types that can contain graphite. They seem to be sensitive to the tightening procedure and it can be challenging to ensure the proper tightening level, resulting in compression variability. Too much tightening can result in sealant buckling, while too little tightening can result in gasket relaxation and creep, both of which are accelerated by vibrations or thermal cycling. In addition to the tightening procedure, the application of lubricant, surface roughness and misalignment can accelerate the failure of the gasket. [3] Furthermore, temperature and pressure fluctuations can cause degradation [4].

To study the performance of graphite sealants in high temperature environments, a suitable test setup is required. This work focuses on defining representative testing environment parameters, designing a suitable test setup for graphite sealants, and testing the designed setup. The test setup can be used in the future to test graphite-based sealants.

2. Graphite based sealants and their in-service environments at NPPs

The most obvious service environment for graphite sealants and gaskets would be the hottest parts of light water reactor circuits. Figure 2 shows the systems and components where graphite-based sealants are applied in PWRs. These include control rods, pressurizers, steam generators, pipes, exchangers and valves.



Inside the world's most critical and demanding applications, you'll find innovative solutions from Technetics.

Maintaining a nuclear reactor and ensuring safe day-to-day operations demands seals and gaskets designed for complete reliability. Technetics products, such as our ORIGRAF®, VITAFLEX® and HELICOFLEX® seals, are widely used in the nuclear power generation industry and are trusted by nuclear engineers throughout the world.

Figure 2. Different locations where graphite-based sealants are applied in PWR. [5]

The coolant temperatures in the reactor core are typically around 325 °C but can be significantly varied by the PWR design. For pressurizers, NRC has stated a temperature of 343 °C where primary water stress corrosion cracking has been assessed [6]. In steam generators, the hot leg temperature can be as high as 330 °C and the cold leg temperature 290 °C [7]. Graphite-based sealants have been studied earlier at 350 °C water and 200 bar pressure, although this environment was considered more conservative than that present in NPPs [8]. Thus, it seems that a feasible testing temperature would be above 300 °C and below 350 °C.

The primary side pressure during operation is typically around 16 MPa. There are no major variations expected during normal power operation in the pressure, as the reactor core temperature stays constant. During transients, however, variations in pressure can occur.

3. Experimental testing of graphite sealants

3.1 Design of the experimental setup

3.1.1 Compression set

Based on the environmental parameters discussed in the previous section, compression set testing [9] could be a feasible option for testing graphite-based sealants. The principle of the compression set test is described in Figure 3. The sample with an initial height of h_0 is compressed between two planes to a constant height of h_1. The sample is left in compression for a certain time. After the determined compression time, the compression is removed and the sample recovers. After the recovery time sample height after the compression, h_2, is measured. The compression set (CS) can be calculated by using the equation:

$$CS = \frac{h_0 - h_2}{h_0 - h_1} * 100\% \tag{1}$$

The compression set has a simple working principle and it has been widely used previously in sealant testing [9, 10]. There is also at least one standard available on how to perform a compression set test [11]. The compression can be performed at ambient temperature or at elevated temperature, which makes it ideal also for in situ high temperature testing. The test approach also enables dynamic testing if the compression is momentarily released and reintroduced.

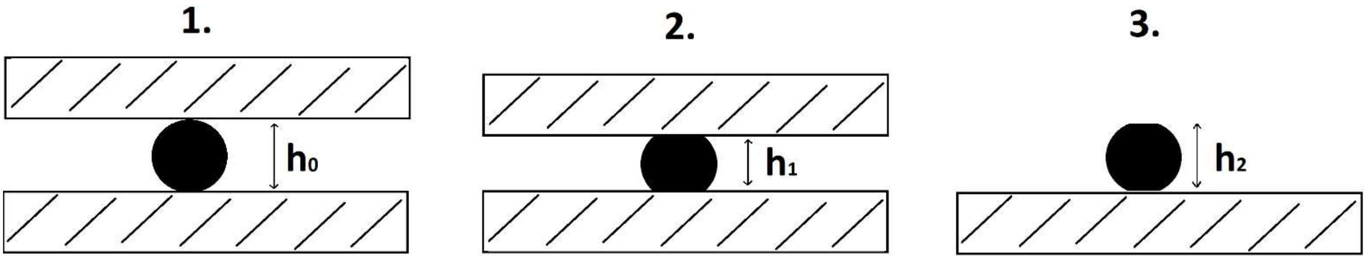


Figure 3. The principle of compression set test. The sample with initial height of h_0 is put between two parallel planes (1). The planes are compressed against each other and the sample is deformed to a certain height h_1 (2). After a certain time period, the compression is removed, the sample recovered and height after the recovering period, h_2, measured (3).

3.1.2 Experimental setup

The tested samples were either O-rings or button samples (see Figure 4 A and B, respectively). Half of the samples had steel reinforcement within them, and half of the samples were plain graphite. A summary of the samples is shown in Table 1.

The experimental setup was constructed of a set of stainless steel flanges. The samples were compressed between two flanges (see Figure 4 C). Calipers (0.85 or 2.3 mm in thickness) were used to determine the correct deformed height h_1 (see Figure 4 D). A total of four of these flange assemblies were piled together and connected to an autoclave lid (see Figure 5). The piled assembly was installed in an autoclave filled with ion-pure water and heated up to 300 °C. The pressure during the exposure was 90 bar and the exposure time one week. After cooling down the autoclave, the assembly was left to dry in ambient air for one week before disassembling the flanges. After releasing the compression, samples were let to recover. The h_2 value was measured 5, 10, 15, 20 and 30 min after the release of the compression (the 30 min time is in accordance with the ASTM D 395-03).

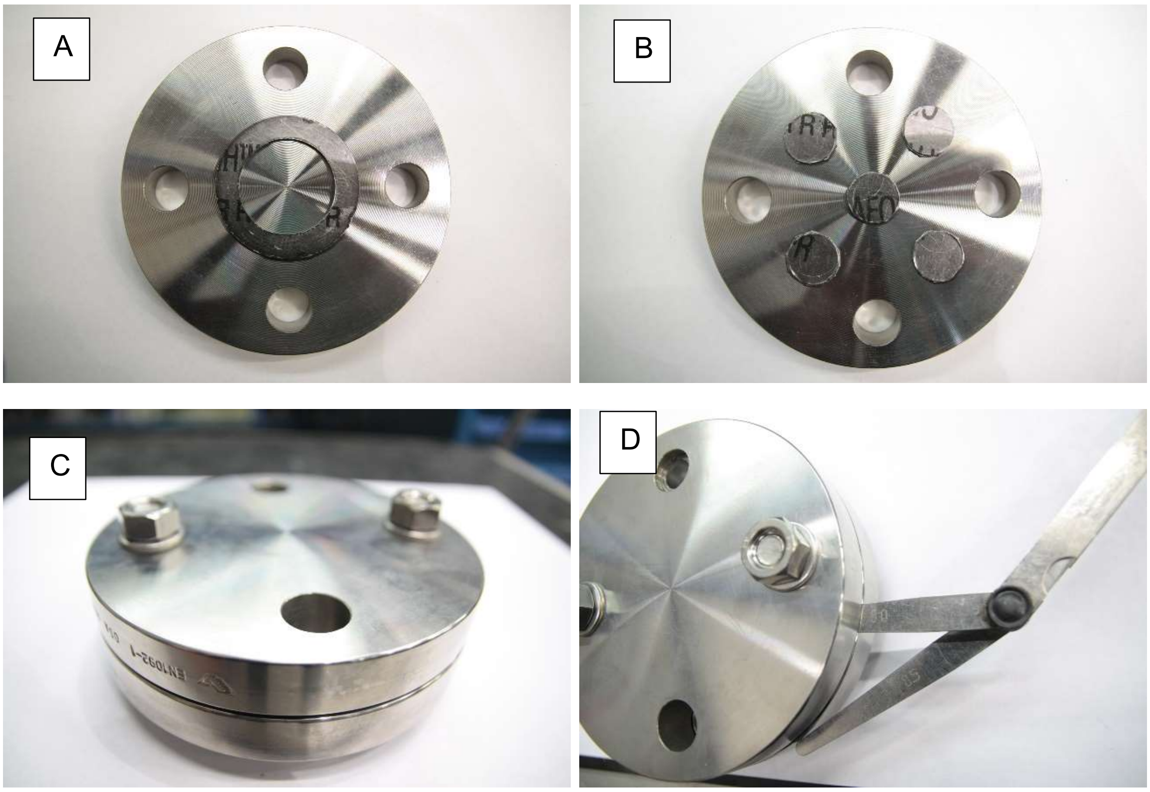


Figure 4. (A) O-ring sample on a flange, (B) button samples on a flange, (C) two flanges compressed towards each other with a sample in between and (D) calipers showing the correct compression state.

Table 1. Summary of the samples tested. The samples with the metallic enforcement were of the first type in Figure 1.

Sample type	Sample amount	Metallic reinforcement	h_0	h_1
Button	5	Yes	1.10	0.85
Button	5	No	3.06	2.30
O-ring	1	Yes	1.10	0.85
O-ring	1	No	3.06	2.30

Figure 5. The fully assembled test setup.

3.2 Results and discussion

When the flanges were decompressed and samples taken out for thickness measurement, it was noticed that most of the graphite button samples without steel reinforcements were torn apart as they were removed from the flange. Only one graphite button sample remained intact after the experiment and could be measured. The use of a lubricant in the case of testing could be considered to avoid tearing of the samples when removing them from the flanges.

The sample thickness measurement results are shown for the various types of samples in Figure 6 to Figure 9. Four out of five reinforced button samples demonstrated a small increasing thickness value until the 30 min time stamp. The reinforced O-ring, however, does not seem to show any difference in the thickness value during the 30 min monitoring period. In the case of the graphite button sample (the only one that remained intact), the thickness seemed to decrease at the end of the 30 min monitoring period, but still being higher than the compressed value. The graphite O-ring on the other hand, displayed obscure behaviour. Although the thickness values seemed to increase after the decompression, the values remained smaller compared to what was the compressed value. This might be due to improper tightening of the flanges, causing the compressed value to be lower than the targeted 2.3 mm.

The calculated compression set values are presented in Figure 10. The compression set value calculated for the graphite O-ring (103%) is not considered to be reliable as there has been some uncertainty regarding the h_1 value of the sample.

The lowest compression set value 54%, was measured for the reinforced button samples. It is smaller than with the graphite button (71%) and the reinforced O-ring sample (72%). The difference between the O-ring and button sample can be reasoned by the shape of the sample. Under the compression O-rings can expand in two directions, while the button samples can expand only in one direction. Thus, the compression is more “severe” to the O-rings than button samples, which could explain the higher compression set value measured with the reinforced O-ring sample.

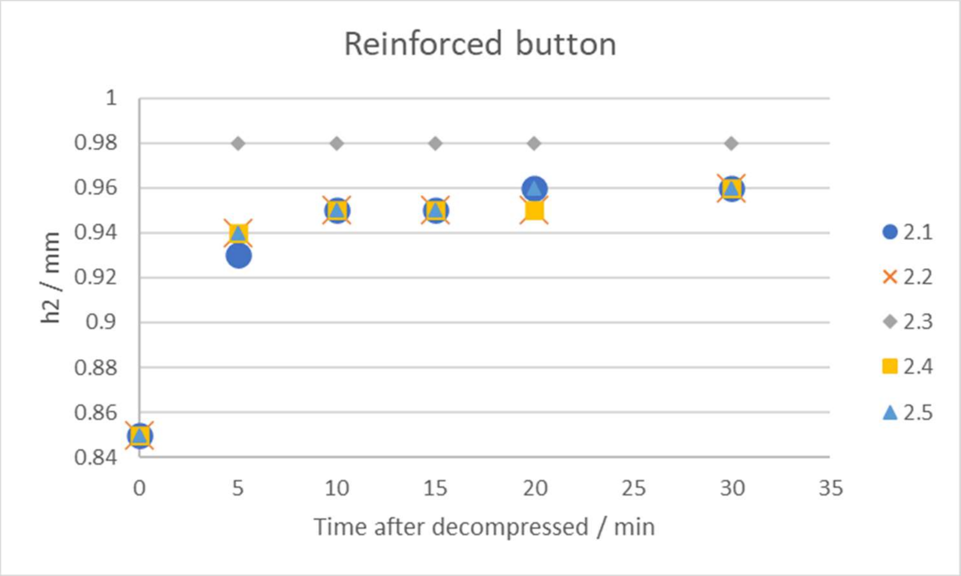


Figure 6. Reinforced button sample thicknesses measured after the decompression.

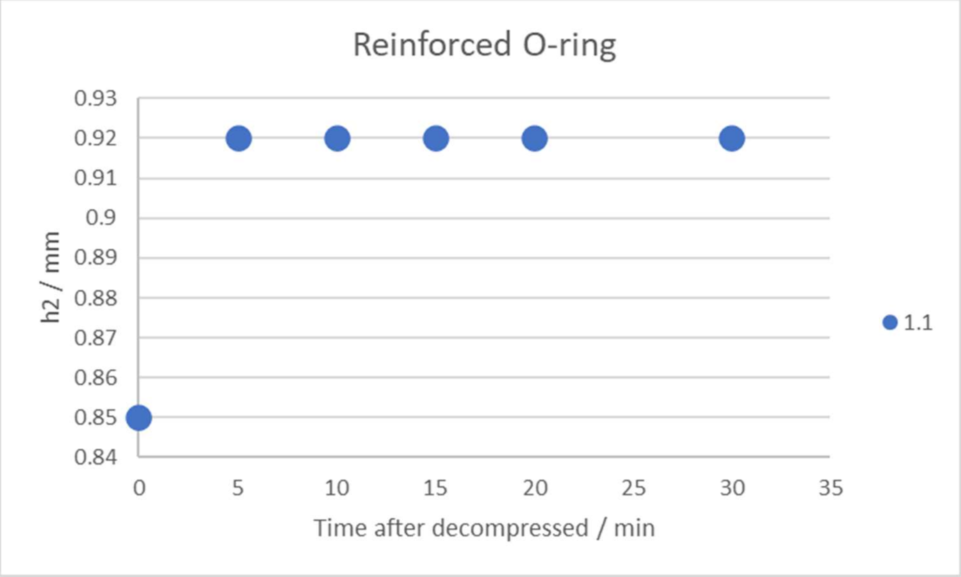


Figure 7. Reinforced O-ring sample thickness measured after the decompression.

Setting up an acceptance criterion based on a compression set would be feasible. 0% compression set would mean that the sample fully recovers from the compression, while 100% indicates that no recovery has occurred. The question remains how to set the acceptance criterion between these values. One approach would be to correlate the compression set value to a certain functional property, e.g., leakage pressure. However, this would require a separate testing arrangement, and it has not been performed in this study. This kind of approach has been adopted elsewhere when setting an acceptance criterion for a nuclear-grade EPDM O-ring [12]. In this specific study, as high as 103% compression set value was proposed for the acceptance criterion. This would indicate that all of the tested samples had an acceptable compression set value.

One possibility for establishing acceptance criteria would be referring to literature data. One sealant manufacturer defines excellent compression set behaviour when the set value is below 20%, moderate set value is between 25-35%, and poor set are values above 40% [13]. According to this scale, all the tested sealants performed poorly.

Unfortunately, no compression set values were found for graphite sealants in a quick literature search, which could have been used in the acceptance criterion assessment. Thus, it seems that additional pressure testing would be required for the tested sealants, or more extensive literature search should be performed.

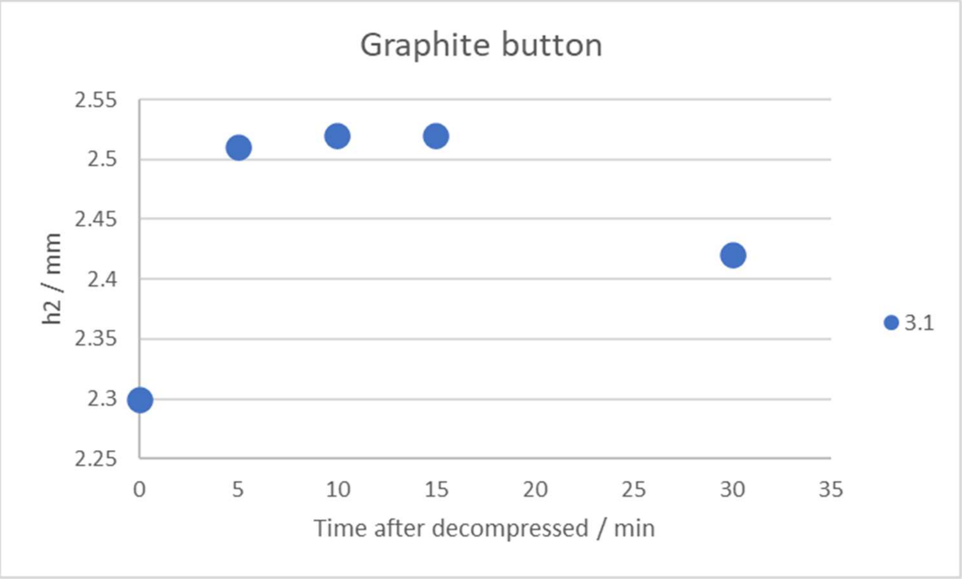


Figure 8. Graphite button sample thickness measured after the decompression.

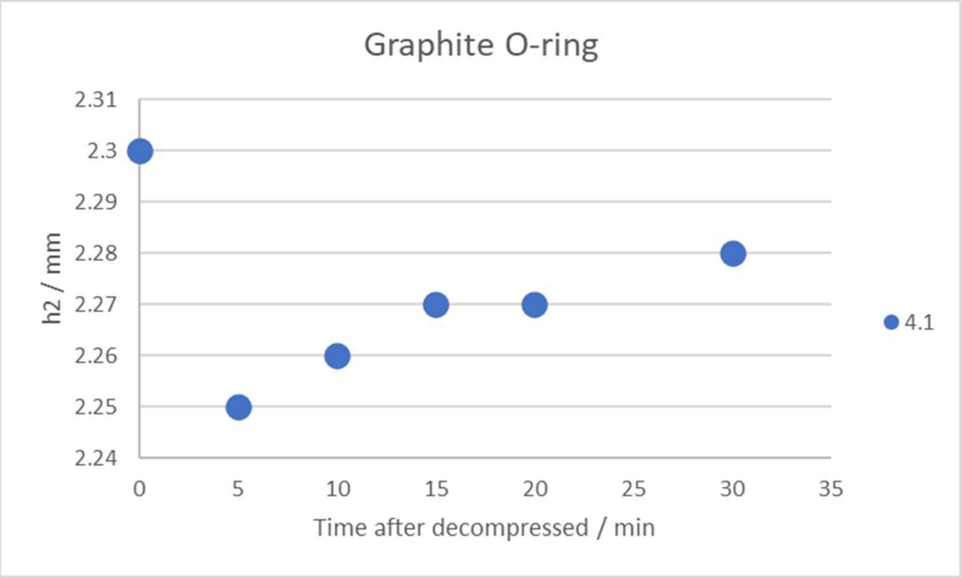


Figure 9. Graphite O-ring sample thickness measured after the decompression.

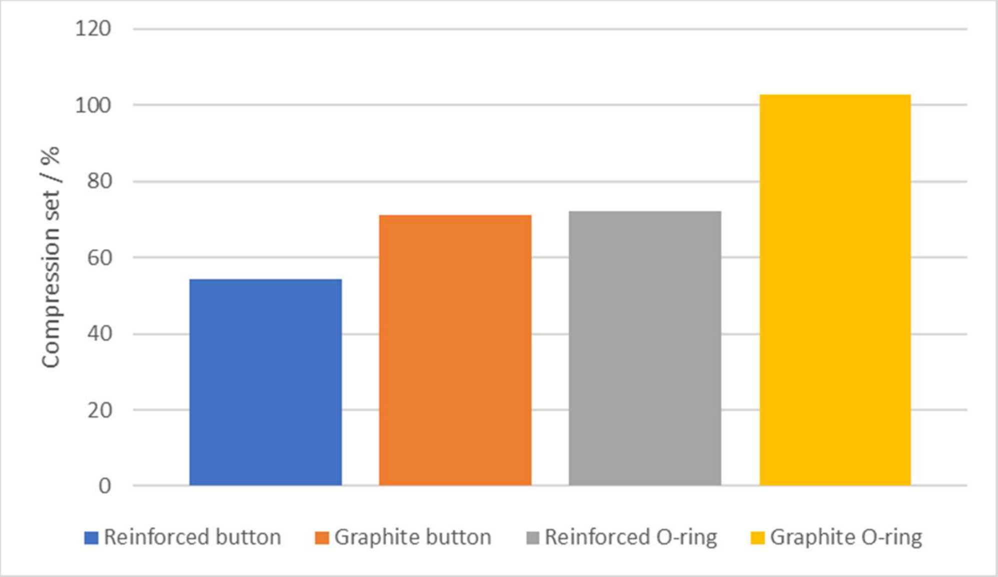


Figure 10. Calculated compression set values for the different types of samples.

4. Conclusions

Graphite is used in many sealant applications in NPPs. As any other sealant material, they are subjected to ageing during their service lives. For graphite-based sealants, the most evident ageing stressors are high temperature and pressure. During this work, an experimental test setup based on compression set was designed for high temperature testing of graphite sealants. Two different types of graphite were used in sample preparation, plain graphite and reinforced graphite, and two different sample designs were tested, buttons and O-rings. The designed test setup functioned well at the high temperature. Application of a lubricant could be considered to ensure smooth removal of the samples from the flanges. The compression set values were successfully calculated for three of the sample types tested. Setting up an acceptance criterion would require additional data. A separate pressure testing of the samples would enable setting up a functionality-based acceptance criterion.

References

1. FSA. 2017. Gasket handbook, 1st edition. FSA, Fluid Sealing Association. ESA, European Sealing Association e.V.

2. Jäppinen, E., Kellock, M., Mosallaei, M., Sipilä, K. 2023. Sealants in nuclear applications: SINAPP-project final report. VTT Research Report VTT-R-00584-23. 56 pp.

3. Jenco, J.M. & Hunt, E.S. 2000. Generic issues effecting spiral-wound gasket performance. International Journal of Pressure Vessels and Piping, 77. pp. 825-830

4. Shen, M., Peng, X., Xie, L., Meng, X., Li, X. 2016. Deformation Characteristics and Sealing Performance of Metallic O-rings for a Reactor Pressure Vessel, Nuclear Engineering and Technology, Volume 48, Issue 2, 2016, pp. 533-544, ISSN 1738-5733, https://doi.org/10.1016/j.net.2015.11.009.

5. Technetics Group. Nuclear brochure. Available: Nuclear Brochure - Technetics Group

6. United States Nuclear Regulatory Commission. 2020. Reactor Pressure Boundary Integrity Issues for Pressurized Water Reactors .Available: Pressurizer Issues | NRC.gov

7. Koistila, T. 2021. Flow accelerated corrosion in nuclear power plant secondary circuit. Master's thesis work, Lappeenranta University of Technology. 88 pp.

8. Vignaud, J. C., Digat, P., Nowak, H. 1992. Water And Steam Tightness Tests of Expanded Graphite Static Gaskets for Nuclear Power Plant Steam Generators. In: Nau, B.S. (eds) Fluid Sealing. Fluid Mechanics and its Applications, vol 8. Springer, Dordrecht. https://doi.org/10.1007/978-94-011-2412-6_10

9. Flitney, R. 2014. Seals and Sealing Handbook (Sixth Edition), Butterworth-Heinemann, ISBN 9780080994161, https://doi.org/10.1016/B978-0-08-099416-1.00009-7.

10. Burnay, S. 2018. DEGRADATION OF POLYMERIC COMPONENTS IN NUCLEAR POWER APPLICATIONS. Energiforsk Report 2018:480. 92 pp.

11. ASTM International. D 395-03 (Reapproved 2008) Standard Test Methods for Rubber Property – Compression Set.

12. Molander, M., Jansson, A., Sandström, J. 2018. Development of condition monitoring methods for polymeric components including low dose rate radiation exposure. Presented at the FONTEVRAUD 9, 17 – 20 September 2018, Avignon, France. Contribution of Materials Investigations and Operating Experience to Light Water NPPs' Safety, Performance and Reliability.

13. Barnwell. Compression set data sheet. Available: What is compression set? Everything you need to know. : Barnwell