Control of the sealing ability of vacuum materials and sealing rings for vacuum systems

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Abstract. The paper deals with the control of gas permeability of fluoroplast samples from different manufacturers, as well as Viton rings for vacuum systems. To conduct the study, a stand based on a mass spectrometric leak detector is used. According to the results of the study, it can be said that fluoroplast samples can be recommended for use as seals, and Viton seals require regular replacement to prevent leaks of gases and oils.

1. Introduction
The purpose of this work is to control the gas permeability of fluoroplastic samples [1] from different manufacturers, as well as rings for vacuum systems made of Viton.

Fluoroplastic is widely used in technology not only as an insulating and antifriction material, but also for the production of seals and gaskets of vacuum systems [2]. Viton seals are standard vacuum detachable connections. They are used in pumps, leak detectors, microscopes, mass spectrometers, vacuum chambers and other equipment.

2. Stand for gas permeability research
To conduct the study, an experimental stand was developed on the basis of the HELIOT 901W1 mass spectrometric leak detector (figure 1) [3, 4]. The stand is equipped with a vacuum pressure chamber designed for installation of the object of measurement, its preliminary degassing and the creation of a pressure gradient of helium relative to the outer and inner sides of the object of measurement. The effective diameter of the sample through which the diffusion flow in the test chamber passes is 25 mm, the thickness of the sample is 4 mm.

The controlled object is installed in a vacuum pressure chamber, which is attached to the leak detector and pumped out by a vacuum system [5]. Then helium is fed into the chamber, while performing continuous pumping from the opposite side of the object. The quantitative value of the helium flow is measured by the leak detector signal after stabilization of the helium diffusion flow through the object under study.

Check of the gas permeability of the system was carried out with the use of metal screw caps instead of the standard sample (figure 2). For the calibration setup was used a certified etalon – a measure of flow (helium flow) Gelit 2. The leakage through the tooling seals has an intensity three orders of magnitude less than the flow through the sample material. The flow reaches $10^{-10} \text{ Pa}\cdot\text{m}^3/\text{s}$ only after 60 min of testing. Thus, it is acceptable to assume the leakage of the installation seals negligibly small compared to the flow through the samples.
3. Study of the gas permeability of fluoroplastic samples

Characteristics of the samples are shown in table 1. The results of the measurements of the helium flow through the samples of fluoroplastic are shown in figure 3. To build trend lines is used the polynomial approximation for the three instances of each of the samples.

Table 1. Characteristics of the studied samples.

| No. | Brand                | Manufacturer                      | Characteristics       |
|-----|----------------------|-----------------------------------|-----------------------|
| 1   | Fluoroplastic PTFE   | «Flourosils», Italy               | $D = 55$ mm, $h = 4$ mm |
| 2   | Fluoroplastic F-4 planed | «Formoplast», St. Petersburg, Russia | $D = 50$ mm, $h = 4$ mm |
| 3   | Fluoroplastic F-4 planed | «First fluoroplastic plant», Kirovo-Chepetsk, Russia | $D = 55$ mm, $h = 4$ mm |
| 4   | Fluoroplastic PTFE   | «Flourosils», Italy               | $D = 55$ mm, $h = 4$ mm |

Helium flow through the samples reaches saturation after 60 min of testing. After that, the amount of helium flow through the material of the samples is about $10^{-7}$...$10^{-6}$ Pa·m³/s through the samples with a thickness of 4 mm and an effective area for diffusion 1963 mm² (circle with a diameter of 25 mm).
When comparing the gas permeability of the material samples when the gas flow saturation is achieved [6], the following results are obtained, according to the polynomial approximation of the measurement results of three instances of each sample:

- largest was the flow of helium passing through the sample No. $1 = 6 \cdot 10^{-7}$ Pa·m$^3$/s;
- helium flow through samples No. 2 and 4 are identical and equal to $3.5 \cdot 10^{-7}$ Pa·m$^3$/s;
- smallest helium flow is registered through sample No. 3 – $3 \cdot 10^{-7}$ Pa·m$^3$/s. Flow through sample No. 3 is twice less than through the sample No. 1.

4. Investigation of gas the permeability of sealing rings

O-rings are used in technology to seal quick-release joints. The best performance on the tightness of elastomers have rings made of fluoro rubber, patented under the brand Viton. Resistance to gas penetration in Viton rings is high and similar to the resistance to gases diffusion of butyl rubber.

The specific gas permeability of Viton is lower than for other industrial elastomers [7], but it does not completely exclude the penetration of gases under the pressure drop. It should be borne in mind that the diffusion of gas through the seal takes time – from tens of minutes to several hours. Therefore, when blowing helium leak detection leakage is easy to miss.

Figure 4 shows the values of the measured gas flow through Vitan ring (LLC “Vactron Laboratory”, Saint-Petersburg) after the establishment of the diffusion flux at room temperature, the differential pressure of gas at 1 atm and the compression of the ring by 30 %.

According to the results, a product filled with helium (1 atm, 30 °C) and having a Viton ring of ISO-100 standard [8] will lose helium with a flow of $3 \cdot 10^{-7}$ Pa·m$^3$/s. No defects and leaks are taken into account. Helium will leave the product through the Viton seal.

Similarly, atmospheric gases flow through the rings into vacuum chambers. To estimate the flow of gas into the vacuum volume through the Viton ring you should multiply the data shown in figure 4 on the concentration of the gas in the ambient air.

For example, the flow of oxygen to the vacuum chamber through the flange KF-25 will be $2 \cdot 10^{-9}$ Pa·m$^3$/s at room temperature after stabilization of diffusion processes. In this case, the leak detector method of blowing helium will not reveal the leak. It will manifest itself, if you apply helium for a long time, and will be about $6 \cdot 10^{-8}$ Pa·m$^3$/s.

In figure 4 it can be noted that freon-22 has a greater penetrating power through the Viton seal, exceeding even the diffusion capacity of helium [9].

The flows of hydrogen, argon, krypton and xenon through the Viton ring are shown in figure 5 for the compaction temperature of 93 °C. With the increase in the operating temperature of the product, the diffusion flux increases exponentially [4].
Figure 4. Gas flow through the Viton ring after the diffusion flow is established at room temperature, the gas pressure drop of 1 atm and the compression of the ring by 30%.

Figure 5. Gas flow through the Viton ring after the diffusion flow is established at 93 °C, the gas pressure drop of 1 atm, and the compression of the ring by 30%.

5. Conclusion
The studied samples of fluoroplastic can be recommended for use as seals. Production of fluoropolymer sealed volume or body of the product with a large surface area will lead to a breach of the first type of tightness – leaks “on the solid material”.

Viton seals require regular replacement to prevent gas and oil leaks [10]. When applying a long-term pressure drop, such a seal allows the penetration of gases through a material with a flow of up to $10^{-6}$ Pa·m³/s. This should be taken into account when using non-metallic seals as part of highly sensitive analyzers and products that require long-term vacuum preservation.
References

[1]  Sutiy Yu V 1971 *Journal of physical and chemical mechanics of materials* 4

[2]  Kuznetsov V G, Kurbanov T A and Kostrin D K 2017 *Journal of Physics: Conference Series* 872 012006

[3]  Barchenko V T, Lisenkov A A and Vinogradov M L 2014 *Journal of Physics: Conference Series* 872 012002

[4]  Vinogradov M L, Barchenko V T, Lisenkov A A, Kostrin D K and Babinov N A 2015 *Vakuum in Forschung und Praxis* 3 26–9

[5]  Vinogradov M L, Kostrin D K, Karganov M V and Tiskovich V Yu 2016 *Proceedings of the 2016 IEEE North West Russia Section Young Re-searchers in Electrical and Electronic Engineering Conference (EIConRusNW)* 100–4

[6]  Perkins W G 1973 *J. Vac. Sci. Technol.* 10(4) 543–56

[7]  Courtney W J and Schipma P B 1968 *A new handbook for the aerospace engineer – NASA permeability data for aerospace applications*

[8]  Vinogradov M L and Barchenko V T 2015 *Young Researchers in Electrical and Electronic Engineering Conference (EIConRusNW)* 40–4

[9]  AI-AEC-13145 *Design Guide for Reactor Cover Gas Elastomeric Seals* (March 7, 1975)

[10] Vinogradov M L and Kostrin D K 2019 *AIP Conference Proceedings* 2089 020021