ISBN: 978-3-95450-248-6 ISSN: 2673-5490 doi: 10.18429/JACoW-IPAC2025-THPS059

COMPARISON OF VARIOUS OUTGASSING RATE MEASUREMENTS FOR UHV SYSTEMS

P. M. Suherman, M. C. Bellachioma
GSI Helmholtzzentrum für Schwerionenforschung GmbH, Darmstadt, Germany

Abstract

Outgassing rate is one of the most important criteria for vacuum acceptance of various components used in ultra-high vacuum (UHV) systems. There are numerous methods that can be used to measure the outgassing rate of UHV components. One of the most common techniques is the so called 'pressure rise' method. In this method, the component under test is enclosed in a system and isolated from the pumping system. The outgassing rate is calculated from the pressure rise rate that occurs due to the outgassing of the component. Comparing with other techniques, the pressure rise method is more straightforward and allows easier analysis of the data. Nevertheless, the outgassing rate obtained from the pressure rise method tends to be much lower than the actual outgassing rate.

This paper presents an investigation of another approach to analysing the data obtained from the pressure rise method. The objective of this approach is to provide a greater accuracy in the outgassing rate measurement, as well as to understand the reason behind the large error obtained using the pressure rise method. The new approach of calculating the outgassing rate from the 'pressure rise' method is then compared to the normal pressure rise method, as well as throughput method.

OUTGASSING RATE MEASUREMENT

The outgassing rate of vacuum chambers and vacuum components can be obtained using different methods [1, 2]. The use of the methods depends on the components being investigated. The most common technique used is the pressure rise method, where gas is accumulated in an enclosed chamber isolated from the pump. The pressure rise method is straightforward and not time consuming. The suppliers of the vacuum chamber or vacuum components normally present the outgassing rate based on this pressure rise method. Unfortunately, the outgassing rate obtained from this technique tends to be inaccurate.

Another common technique used to determine the outgassing rate is so called throughput method. In this method, the test chamber is separated from the pump by a baffle with a known conductance.

EXPERIMENTAL SETUP

Figure 1 shows the experimental setup used to measure the outgassing rate in this study. The setup can be used in-situ for two techniques, i.e., the pressure rise and throughput methods. The outgassing rate measurement was conducted on stainless-steel chambers with different dimensions. Some commercial samples that are commonly used for high vacuum systems were also tested: PEEK, Macor, and Vespel.

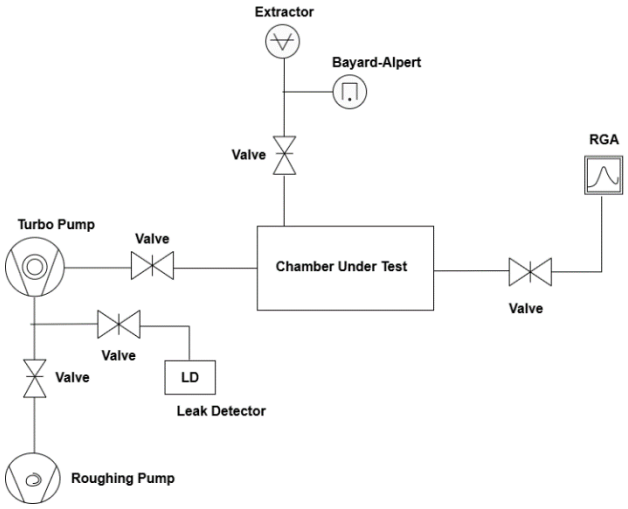

Figure 1: Experimental setup for in-situ pressure rise and throughput methods.

METHODS OF COMPARISON

Figure 2 shows a typical pressure rise behaviour over a certain time, when the chamber under test was isolated from the pumping system. The pressure rise rate shown in Fig. 2 was obtained from the measurement of stainless-steel chambers before and after bake-out treatment.

There are three major phases observed in Fig. 2: the first phase (Phase I) - where the initial pressure rise occurs very quickly for a short period after the closing of the valve, the transition phase shown as a 'curved part' on the graph, and the final phase (Phase II) where the pressure rise rate either gradually decrease or rises again almost linearly.

In an ideal condition, the pressure should increase linearly with time, when the chamber under test is isolated from the pumping system. In practice, however, after the chamber isolation from the pumping system, there is a probability of various events, besides the outgassing of the test sample. The most common occurrence after an extended chamber isolation is gauge pumping and re-adsorption from the chamber wall. In practice, a spinning rotor gauge (SRG) should be used when conducting the pressure rise method, to avoid any gauge pumping or additional outgassing from the gauge filaments. However, the pressure measurement range of a SRG is limited to about 10^{-7} mbar [3].

ISBN: 978-3-95450-248-6 ISSN: 2673-5490 doi: 10.18429/JACoW-IPAC2025-THPS059

As shown in Fig. 2, for a stainless-steel chamber after bake-out, the valve closing is clearly indicated by a vertical pressure rise, then followed by a brief linear pressure rise before gradually decreasing to almost constant rate rise. The constant rate rise after prolonged isolation from the pump might be due to the equilibrium between the outgassing rate and the re-adsorption of the chamber surface. It could also be due to the gauge pumping.

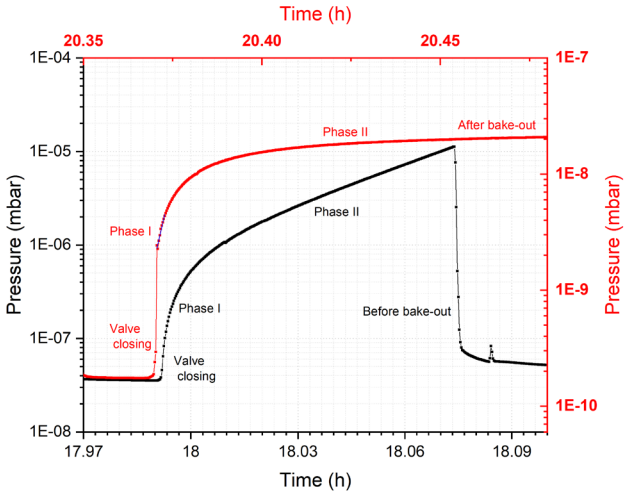

Figure 2: The rate of pressure-rise for a stainless-steel chamber, before and after bake-out treatment, represented by black and red graphs, respectively.

For an unbaked chamber, the elimination of the valve outgassing is not as straightforward, because the valve closing effect is quite obscure. As shown in Fig. 2, the effect of the valve closing for unbaked chamber, is mixed with the initial pressure rise of the chamber itself and then followed by a linear pressure rise again. The second linear rate rise might be due the water outgassing that was more dominant than the re-adsorption and gauge pumping effect.

The measurement time chosen for the linearity of pressure rise in Phase I is very brief. The measurement time is actually the characteristic pumping time, which depends on the volume of the chamber under test and the residual pumping speed [2].

Figure 3 shows the measured outgassing rate for stainless steel chamber, before and after bake-out treatment. The comparison of the pressure rise method and throughput method is also shown in Fig. 3. The outgassing rate taken from both Phase I and Phase II during the pressure rise measurement was also compared. The Phase I calculation was taken directly after the chamber under test was isolated from the pumping system. The outgassing rate obtained from Phase II was taken after the pressure rises passing the 'curved part' of the graph. The calculation taken from this phase is the commonly employed in the pressure rise method, which normally gives a large error. In this study, a correction of outgassing rate based on Phase II was employed by taking account of the residual pumping speed due to gauge pumping and/or re-adsorption of the chamber wall.

Figure 3 shows that the pressure-rise method is comparable with the throughput method for the baked stainless-steel chambers, when the pressure-rise calculation was taken from Phase I. This was not the case, when the calculation was taken from Phase II. When the calculation was taken after some extended isolation from the pump, the outgassing rate calculated obtained from the pressure rise method is much lower than the throughput method. However, after including the residual pumping speed into the calculation from Phase II, the outgassing rate calculated was relatively good agreement with the throughput method.

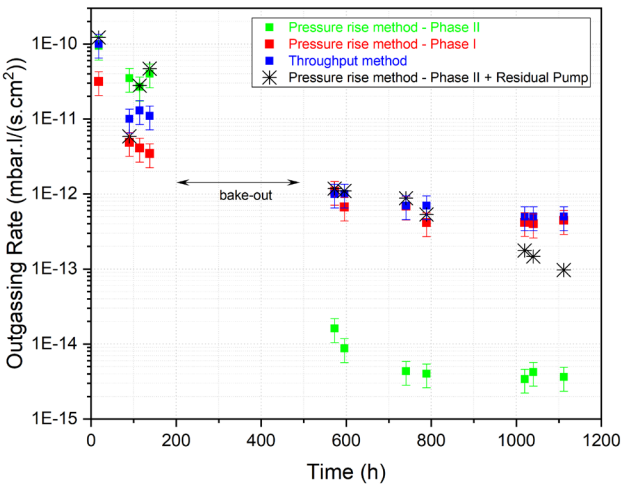

Figure 3: Comparison of outgassing rate measurement of baked and unbaked stainless-steel chamber using different methods.

For unbaked stainless-steel chambers, there is less difference between the calculations taken from Phase I or Phase II. Unlike the baked stainless-steel chamber, the outgassing rate obtained from Phase II for the unbaked chamber is slightly higher than the outgassing rate obtained from the throughput method (Fig. 4). It might be that for the unbaked stainless-steel chamber, the effect from gauge pumping and/or re-adsorption was less dominant than the outgassing rate of the sample itself. This might also explain the linear increase of the pressure in Phase II for the unbaked stainless-steel chamber. The comparison with the throughput method for the unbaked stainless-steel chamber is also not in good agreement, when the calculation was taken from Phase I.

The same trend was also observed for unbaked Macor (Fig. 5) and Vespel samples. Even after bake-out, the pressure-rise method calculated based on Phase I or Phase II for PEEK and Vespel samples, did not agree well with the throughput method. It might be that the pressure rise method based on Phase I cannot be used, when the samples under test have a high outgassing rate or a high-water content, such as unbaked stainless chambers and polymers. The higher water content of PEEK and Vespel is confirmed with the RGA spectra.

ISBN: 978-3-95450-248-6 ISSN: 2673-5490 doi: 10.18429/JACoW-IPAC2025-THPS059

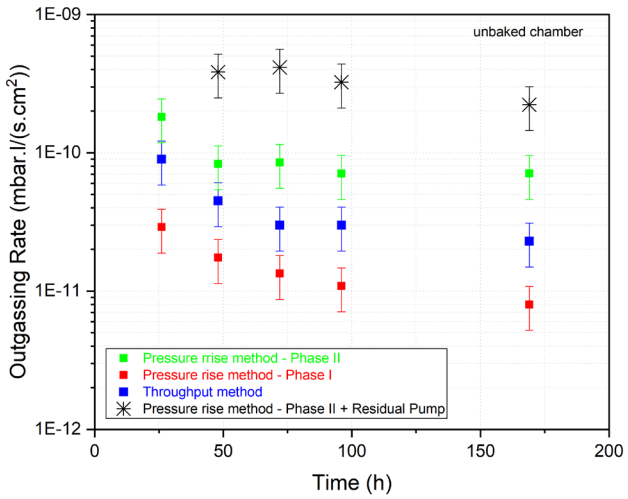

Figure 4: Comparison of outgassing rate measurement of unbaked stainless-steel chamber using different methods.

After bake-out, the pressure rise rate in Phase II for the PEEK and Vespel samples also increased linearly with time. Unlike the Macor sample, the different linearity in the curvature was not observed (Fig. 6). For the PEEK and Vespel samples, there was no noticeable difference in the behaviours of pressure rise rate before and after bake-out. It might be that the bake-out cycles were not sufficient enough, so water was still the dominant outgassed component from the polyamide contained in the PEEK and Vespel samples.

Unlike stainless-steel chambers, the pressure rise behaviour for the Macor, PEEK, and Vespel samples did not undergo a decreased rate in Phase II. The outgassing of the samples may be still dominant compared with the gauge pumping and re-adsorption of the wall chamber. Hence, the residual pumping speed was not included in the outgassing rate calculation.

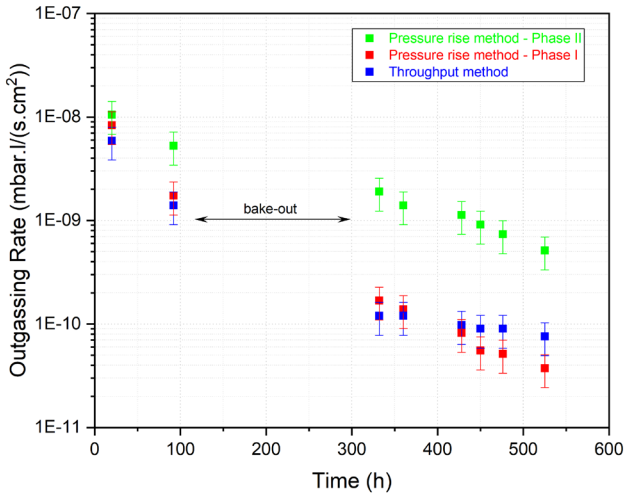

Figure 5: Comparison of outgassing rate measurement of baked and unbaked Macor sample, using different methods.

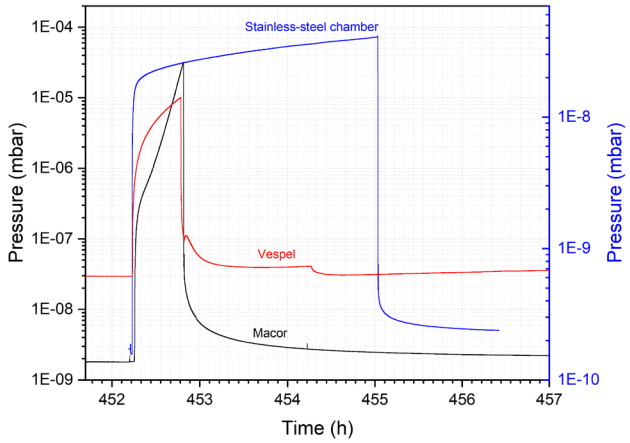

Figure 6: Time dependent pressure of Macor and Vespel after bake-out, during pressure rise method, compared with baked stainless-steel chamber.

CONCLUSION

This study showed that the pressure rise method is comparable to the throughput method for baked stainless-steel chambers and baked Macor sample, if the calculation of the pressure rise method was taken from the very initial rate rise before the re-adsorption phase occurred. There is not enough evidence to conclude, if the pressure rise method calculated from the initial rate rise is only valid for low outgassing rate samples, or samples with a certain dominant composition. The RGA spectra showed that both the baked stainless-steel chamber and Macor samples have hydrogen as a dominant element, but the outgassing rate of the baked Macor was much higher than the outgassing rate of baked stainless-steel chamber.

If the pressure rise method was taken after an extended time of pump isolation, care should be taken for any effect of gauge pumping and re-adsorption from the chamber walls. Applying the residual pumping due to these effects on the outgassing rate measurement is necessary to produce reliable results.

More study needs to be done, to investigate the condition or constraint for the pressure rise method, in terms of the calculation based on Phase I, especially the study for polymer samples that are normally used for UHV systems.

REFERENCES

[1] R. Grinsham and A. Chew, "A Review of Outgassing and Method for its Reduction", in *Applied Science and Convergence Technology, 26(5)*, 2017, pp. 95-109. doi: 10.5757/ASCT.2017.26.5.95

[2] P. Chiggiato, "Outgassing properties of vacuum materials for particle accelerators", *Proceedings of the 2017 CERN-Accelerator School course on Vacuum for Particle Accelerators,* Glumslov, Sweden, 2017, pp. 143-189. doi: 10.48550/arXiv.2006.07124

[3] https://www.sens4.com/blog/vacuum-technology-2/spinning-rotor-gauge-explained-15.