Improvement of Sample Containment/Handling for Volatile Analysis

Oven sealing – Executive Summary

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1 EXECUTIVE SUMMARY

A common method of extracting compositional information from solid samples is through heating within a sealed container. This allows for the extraction and analysis of any volatile components that may be either directly released from the structure of the sample or evolved through reaction with other gases, for example oxygen. This is a straightforward process in terrestrial laboratory settings but becomes considerably more challenging when applied to in-situ analysis of other planetary bodies. The need for automation, and the mass and power restrictions required to operate a spaceflight analytical instrument, place severe constraints on how such devices can operate. In addition, the presence of contaminants in the form of pervasive dust can significantly complicate the process of forming and maintaining system integrity to the degree required to effectively collect and analyse gases. A number of recent missions have addressed this in different ways. The Curiosity and ExoMars rovers use complex, large scale sampling hardware to mitigate the impacts of dust, but these are prohibitively large and expensive for use in regular, small-scale investigations. Rosetta's SD2 system used a much smaller, extremely low power system, but this, unsurprisingly, produced comparatively poor sealing performance and a lack of reusability. More recent work with the ESA PROSPECT package has built on the heritage of SD2 in order to improve the sealing performance of a low mass mechanism, but there is still the lack of understanding regarding the performance and the best way to deploy such a mechanism on the surface of other planetary bodies. The scope of this work was therefore to investigate the concept of a reusable sealing and heating mechanism that could operate with relatively low compressive force in the presence of dust contamination while allowing for a sample to be heated to 1000°C.

As the current development for low mass sealing mechanisms is focused on PROSPECT, it was considered that this would be the most suitable baseline from which to develop and test the sealing concepts for this project. As part of the development of the ProSPA gas analysis package for PROSPECT, an oven has been designed for the purposes of allowing a sample to be heated to 1000°C whilst keeping the temperature of the sealing interface, and therefore the gasket material, to a maximum temperature of approximately 300°C. This allowed the focus of the project to be predominantly confined to testing different sealing materials and concepts. Preliminary work identified perfluoroelastomer (specifically Kalrez), indium (pure and alloyed), and PTFE as potential candidate materials. Later discussions also resulted in filled compositions of PTFE being investigated. Several knife-edge profiles were also identified, based on existing profiles used in sealing vacuum systems; these are shown in Figure 1.

Figure 1: Machined knife-edge attachments. L-R: 90º, VCR-Type, CF-type

In order to perform the sealing tests, a system, termed the Environmental Sealing Breadboard, was designed and built. The purpose of this system was to allow the entire process of sealing an oven-like volume, pressurising it with gas, and measuring the consequent leak rate to take place under vacuum conditions in order to simulate real performance as closely as practicable. To this end, a box vacuum chamber was procured and fitted with a linear actuator which translates vertical motion into the chamber. By driving a rod assembly (referred to as the Force Application Rod) which incorporates the knife edges shown in Figure 1, the system was able to simulate the movement of a tapping station. A set of interchangeable dummy ovens, also fitted with knife edges to seal into the lower surface of the gasket material, were used to simulate real ovens. These were set within a thermally controlled stage which was operated between -100°C and + 350°C in order to approximate the temperature range of a ProSPA oven. Monitoring of the force application was accomplished through the use of a vacuum compatible load cell placed within the Force Application Rod. Thermocouples were used throughout the system to monitor temperatures and ensure that no sensitive components were operated above their rated temperatures. An image of the interior setup of the box chamber is shown in Figure 2.

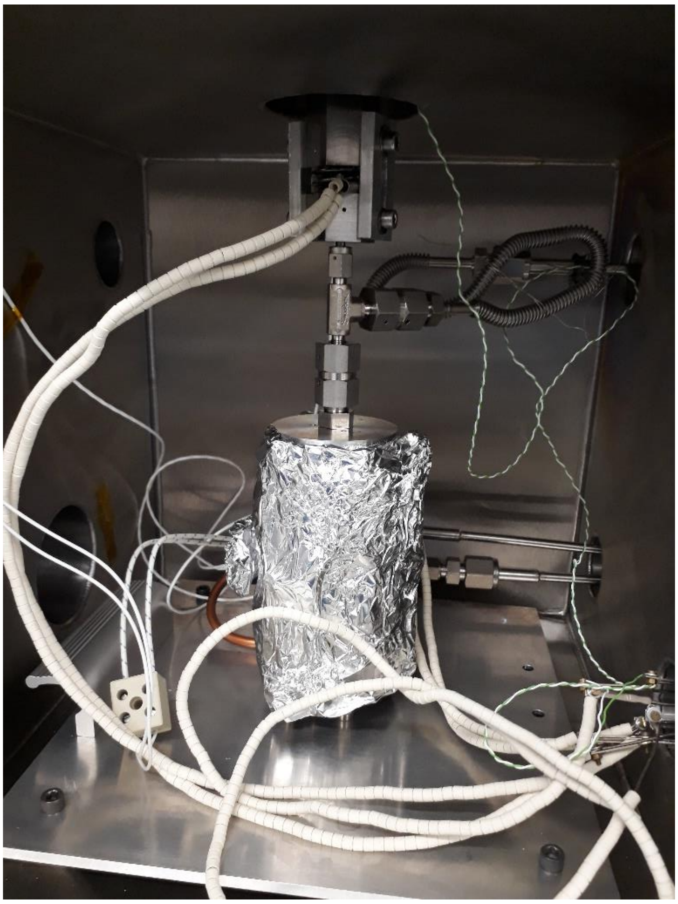

Figure 2. Interior setup of the ESB. The load cell cannot be seen as it is within the flange at the top of the image.

The method of measuring the leak rate was iterated over the course of the instrument construction. The original concept was to use a high sensitivity baratron to directly measure the rise in pressure within the system after the main chamber was isolated from the vacuum pumps. However, outgassing of components within the chamber, particularly during heating, meant that these results were not representative of the real leak rate through the seal. As a result, it was decided that a quadrupole mass spectrometer, calibrated to known flow rates of helium, would be a more effective method of measurement. Aside from the physical switching of the laboratory helium supply and the changing of sealing gaskets, the system was set up to operate automatically via a LabVIEW programme.

In order to fit the tests into the contracted time available, it was decided to first perform tests that could eliminate possible pathways quickly. Kalrez was considered due to its promising performance in early stages of testing for ProSPA when a single knife-edge was used. However, it was found that the use of two knife edges caused the material to cut through, making it unsuitable for further testing. The higher operating temperature range of PTFE with respect to the other remaining materials meant that it was prioritised for testing. The different PTFE compositions, plain, glass-filled, and carbon-filled, were compared using the baseline 90° knife edge with no dust coverage. This resulted in leak rate performance of ~1-2 orders of magnitude lower than the plain PTFE leak rates. A strong correlation between increasing temperature and worsening leak rate was observed in the plain PTFE which was not seen to the same extent in the glass and carbon filled compositions. However, this was never sufficient to take the observed leak rate of plain PTFE above that of the filled compositions.

A second suite of tests focused on the relative performance between the different knife-edge profiles, again with the 90° profile and 0% obscuration dust loads treated as the baseline case. In this case, the CF-style knife edge was found to perform worse than either the baseline case or the VCR-style edge by approximately an order of magnitude. It also showed an accelerating trend towards worsening leak rates at higher temperatures, rather than the linear trend shown by the 90° edge. The performance of the VCR-style edge and the baseline 90° edge was comparable and so the VCR edge was deemed to offer no real benefit.

After establishing that plain PTFE sealed by a symmetric 90° knife edge produced the best sealing performance in the absence of dust, samples were tested for performance when contaminated. Dust coverage from 0 – 9%, measured by obscuration, was achieved by using a Palas RBG 1000 dust aerosolisation unit. The resultant samples were grouped and tested in three categories: 2%, 5%, and 9%. There was an observable worsening in the results from clean to contaminated, approximating half to one order of magnitude change in the leak rates (low 10^{-7} mbar.l.s^{-1} range to low 10^{-6} mbar.l.s^{-1} range) as soon as contamination was introduced. These data continue to show the trend towards increased leak rates at higher temperatures, as seen in the clean material. From the results it does not appear that raising the level of dust contamination from ~2% to ~9% measurably impacts the results further. It may be the case that even higher dust loads would start to impact the seal performance but loads higher than those tested do not seem likely in the ProSPA-like scenario on which this activity is based.

The lack of a measurable difference between the different dust loads in the system meant that there was no need to perform cold tests on the intermediate dust loadings, only the dust-free and highest load. Aside from some outliers, the seal quality continued the trend linking leak rate performance to temperature, with leak rates reducing as the seal became colder. This trend holds down to temperatures of approximately -50°C, with most samples also displaying good performance down to -80°C. However, the results at -100°C are mixed with the majority showing worse performance at this temperature than at -80°C. In approximately 50% of cases, the seal quality degraded by approximately 2 orders of magnitude at -100°C relative to -50°C. This was interpreted as being the result of phase transitions in PTFE, which start to become significant at ~-80°C, combined with some lateral wobble of the Force Application Rod induced by vibrations during movement. The former effect will have caused the gasket to become more resistant while the latter effect could have resulted in a sub-optimal angle of engagement between the knife-edge and the seal gasket. It is likely that any effects of this kind would be masked at higher temperatures as the PTFE would have been able to deform more easily to accommodate variation in the angle of engagement. This also explains why the tests which show a poor performance at -100°C generally display standard performance above -50°C. A summary of the cold tests is shown in Figure 3.

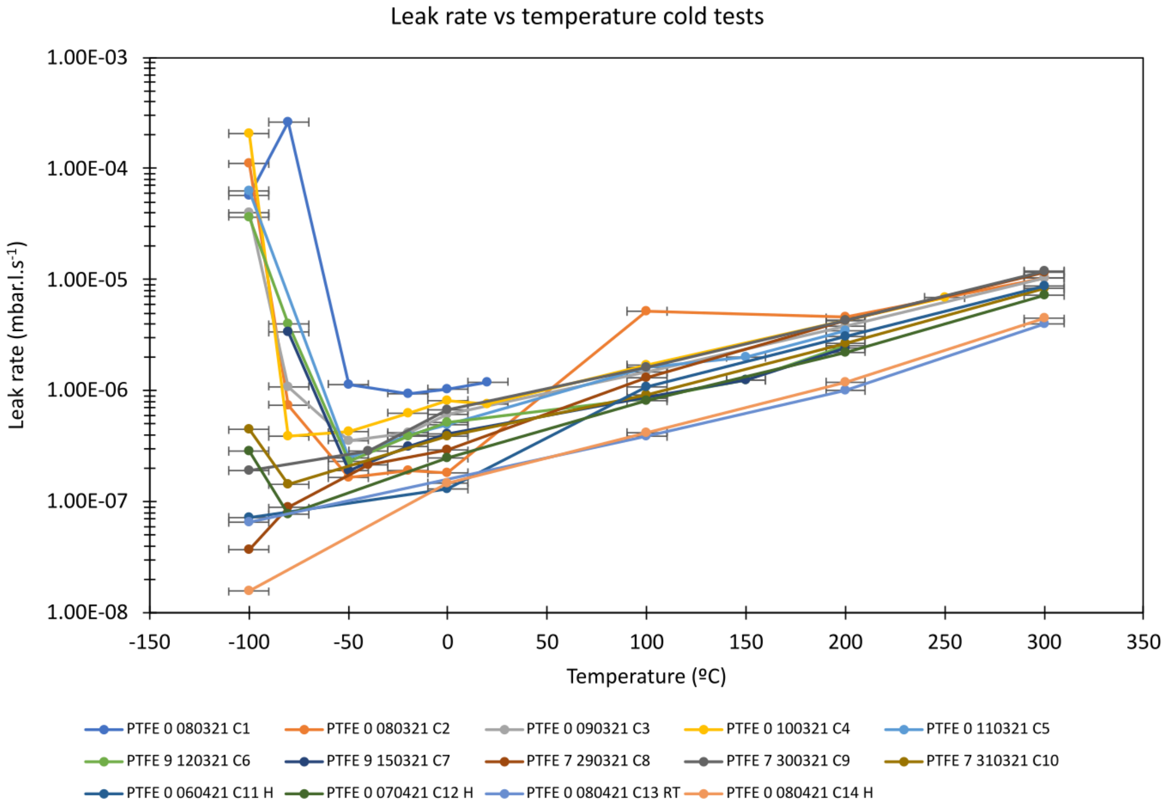

Figure 3. Results of cold tests on PTFE gaskets. Dust level is denoted by the number immediately following "PTFE" in the sample identifier. The suffix "H" or "RT" denotes a hot or room temperature (ambient) descending knife-edge respectively. The marked error bars are not uncertainties in measurement, but instead serve to show the maximum over/undershoot of the heating and cooling ramps.

The final set of sealing tests concentrated on the ability of the gaskets to withstand multiple sealing events. It was found that the initial thickness of the gaskets (1 mm) was insufficient as prolonged compression over the course of a temperature ramp caused them to become exceptionally thin and even break in places. Thicker PTFE gaskets were tested for their ability to reseal and were found to be capable of performing acceptably through at least 5 full cycles of a room temperature to 300°C heating ramp. This was found to be at a small cost to overall sealing performance, particularly at the higher temperatures. The impact of thickness was not investigated in full, but it can be inferred that the optimum thickness will be 1-2 mm.

In addition to the seal testing, a number of the gaskets were subject to surface metrology and X-ray computed tomography scans in order to ascertain the effects of the sealing process in terms of the knife edge indent and the behaviour of dust during the process. Surface metrology data showed that the knife-edges penetrated approximately 500 µm at both sides, regardless of the thickness of the gasket. It also served to identify some variability across the knife edge indent consistent with an offset in the force application rod. CT data was used to reconstruct 3D images of the gasket in order to determine the locations of dust particles. An example scan section is shown in Figure 4, where the dust grains have been highlighted in false colour. From these images it is clear that the intrusion of the knife edge pushes the dust grains into the PTFE, which subsequently deforms around them and allows the system to remain gas tight.

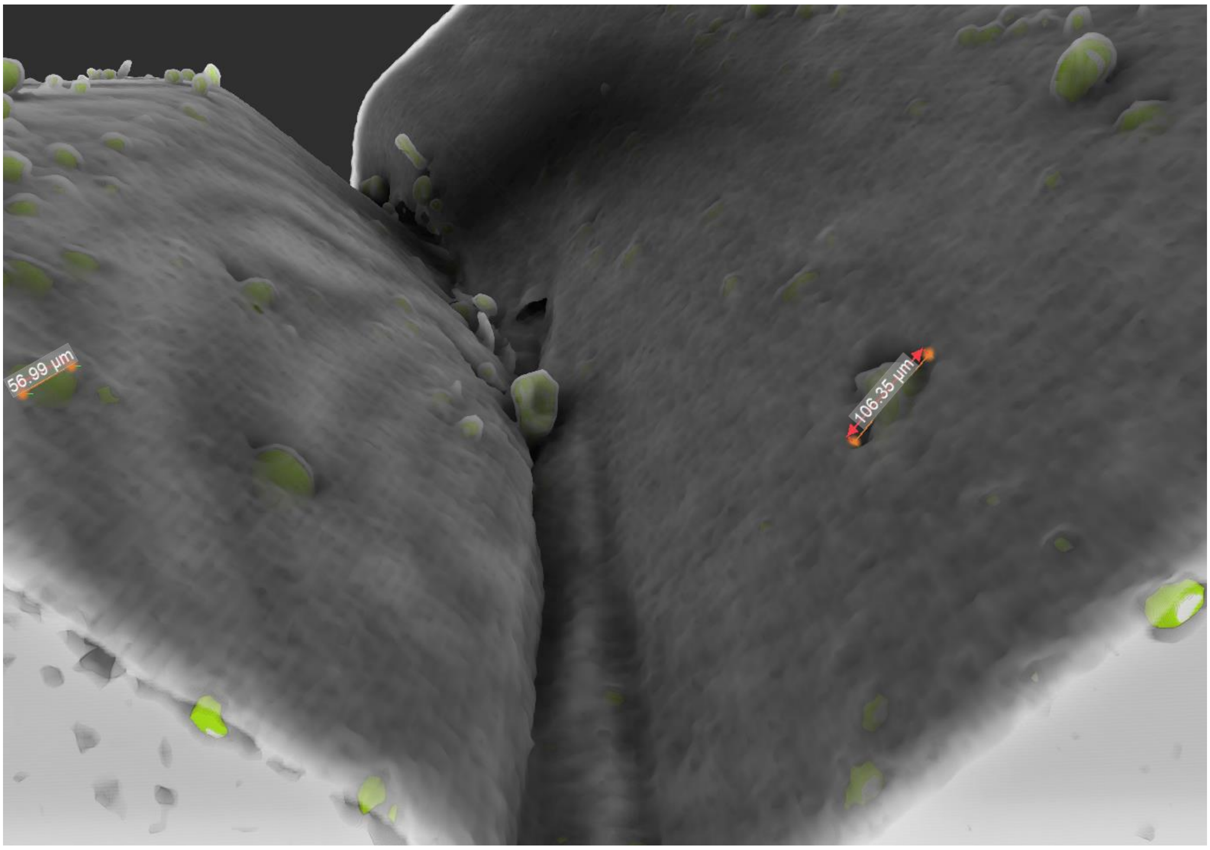

Figure 4. Behaviour of dust particles in the walls of the knife-edge indent in sample PTFE 9 120321 C6.

From the results of this activity, it is possible to draw a number of conclusions and recommendations for future application of low mass, low force sealing mechanisms. It would appear that PTFE of approximately 1.2 – 2 mm thickness would be suitable for operation in a ProSPA-like scenario. At the thicker end of this range, it should be possible to produce reusable ovens, but this would be subject to further testing to optimise the thicknesses. Despite the heat resistance of PTFE to temperatures of up to 327°C, it would be advisable to modify the thermal design of the oven to further minimise the temperature in order to reduce the degradation of leak rate that occurs as the temperature rises. Finally, all potential causes of misalignment of the knife edge should be considered if the intention is to seal an oven at or below -80°C. This not only includes obvious sources such as vibration of the spacecraft, but also considerations regarding components such as the flexibility of the sample carousel.