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Gas containment for in situ sample analysis on the Moon: Utility of sealing materials in the presence of dust

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Abstract: Lunar dust presents a serious challenge to all operations on the Moon, whether human or robotic. It can be especially problematic in applications where it is necessary to make high integrity, gas-tight seals, such as within payloads designed for in situ analysis of lunar ices and volatiles. The challenge has been addressed within the context of the ProSPA instrument being developed for the Luna-27 mission. Soft sealing materials are preferred in order to minimise the required sealing force to enable use of lightweight actuators. JSC-1A simulant was used to test and compare the sealing performance of the elastomer Kalrez[®] 7075 and of Indium. It was found that both materials were able to seal at dust levels of up to 0.90 mg/cm² with an applied force of up to 400 N. Indium offers the best sealing performance (better than 10⁻⁷ mbar.l.s⁻¹) but Kalrez[®] is capable of operation at higher temperature, which may be beneficial in applications in which samples are heated to release gases for analysis.

Keywords

#ProSPA #Moon #Oven sealing #Lunar dust #Volatile preservation

Highlights

- Indium and Kalrez 7075[®] are able to produce a seal in the presence of lunar dust.
- Indium requires a lower force to seal than Kalrez 7075[®].

- Indium may seal under considerable dust loads.

1 Introduction

One of the most challenging aspects of any mission to the lunar surface, whether crewed or uncrewed, is the presence and properties of the lunar regolith. The extensive impact fracturing of the lunar surface, along with the presence of impact glass-bonded soil particles called agglutinates, creates sharp, angular particles that are highly abrasive (Colwell et al. 2007) and which can interfere with the correct operation of equipment. It is also easily disturbed and electrostatically charged (Grün et al. 2011). Reports from the Apollo missions contained descriptions of dust adhering to spacesuits (Colwell et al. 2007, Grün et al. 2011), optical equipment and batteries (Colwell et al. 2007, Kuznetsov et al. 2017) and even penetrating the hermetic seals of the suits and the living quarters of the astronauts (Kuznetsov et al. 2017).

These properties make lunar dust highly pervasive and problematic in applications where it is necessary to make gas-tight seals in situ on the lunar surface, as there is a high probability that any sealing surface will be contaminated by dust. ESA PROSPECT (Package for Resource Observation and in-Situ Prospecting for Exploration, Commercial exploitation, and Transportation) is an example of one of these applications. This is envisaged to fly on the Russian Luna 27 mission to the lunar South pole with the goal of identifying the presence and distribution of water in the subsurface. The ProSPA (Prospect Sample Processing and Analysis) package (Barber et al. 2018) is a miniaturized chemical analysis laboratory consisting of a gas processing system and two mass spectrometers (one ion trap and one magnetic sector), designed to provide information on the abundance, nature, and distribution of lunar volatiles, as well as act as a demonstrator for In Situ Resource Utilization (ISRU). This requires icy regolith samples to be sealed within miniature ovens and analysed via

stepped or continuous heating. While dust mitigation strategies do currently exist, as used on NASA's MSL rover (Kennedy et al. 2006, Mumm et al. 2008) and the upcoming ExoMars rover (Richter et al. 2015), these involve relatively complex, large-scale sample handling hardware and are therefore prohibited by the mass limits of PROSPECT. Therefore, a low-mass system with heritage design has been selected based on an improved version of the oven carousel from the Rosetta Philae SD2 sampling system (Finzi et al. 2007). This system will provide a sealing force of approximately 400 N, allowing for a performance improvement in comparison to the deformable Pt ovens and zirconium oxide balls used in the COSAC (Goesmann et al. 2007) and Ptolemy (Wright et al. 2007, Morse et al. 2009) instruments on the Philae lander.

2 Lunar simulants

Whilst a large mass of material was brought back from the lunar surface by the American Apollo (382 kg) and Soviet Luna (321 g) missions (Colwell et al. 2007) the precious nature of these samples and the scarcity within most collections makes it challenging to acquire real lunar material for tests. However, there are a wide variety of lunar simulants available which are able to provide a proxy for various aspects of lunar dust. The original engineering simulant, to be used in a wide variety of applications including excavation, construction and dust control, was JSC-1 (McKay et al. 1994), a glassy tuff material produced from the San Francisco volcanic field near Flagstaff, Arizona in the United States (McKay et al. 1994). While the original source of this simulant was exhausted some time ago, a large quantity of simulant from the same quarry is now available under the designation JSC-1A (Liu & Taylor, 2011). This has been well characterised for compositional, textural, mineralogical, and strength characteristics (e.g. Arslan et al. 2010, Hill et al. 2007, LaMarch et al. 2011, Ray et al. 2010) and direct comparisons have been made between the characteristics of JSC-1A and

real lunar material from Apollo 14 and 17 (e.g. Hill et al. 2007, Wallace et al. 2009, Liu & Taylor, 2011). The abundance and availability of this simulant combined with its utility as an engineering proxy and its well-characterised nature make it a good choice for the testing performed herein.

3 Sealing material selection

Seals based on the deformation of a gasket material are commonly used within the larger scale terrestrial mass spectrometers on which ProSPA is based and can broadly be broken down into metal and elastomer seals. The most common metal seals are copper, gold, and silver with other metals such as aluminium, nickel, lead and indium also used (Hoffman et al. 1998). Elastomer seals consist of a wide variety of, normally proprietary, compounds, the most common of which are fluoroelastomers (e.g. Viton[®]), perfluoroelastomers (e.g. Kalrez[®]) and PTFE compounds, of which Teflon[®] is probably the most well-known. These vary substantially in their outgassing rates, resistance to radiation, temperature resistance and strength even within seals of the same compositional category (Hoffman et al. 1998, Heller et al. 1999).

ProSPA combines a selection of challenging technical requirements for volatile preservation within the ovens, a cross section of which can be found in Figure 1. The system must retain a vacuum with a leak rate of 10^{-6} mbar.l.s⁻¹ and be resistant to effects from lunar dust whilst operating on a tapping station which will give a maximum sealing force of approximately 400 N (Barber et al. 2018).

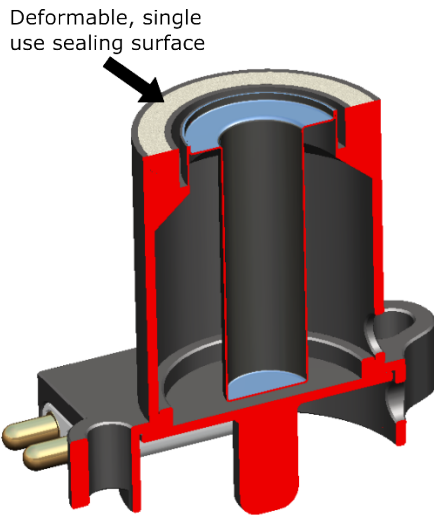


Figure 1: Cross-sectional CAD drawing of the ProSPA oven. The deformable surface at the top of the oven is a single-use surface designed to be deformed via the use of a knife-edge.

This force restriction rules out most of the more common metallic sealing materials as a preliminary study performed on copper, gold and platinum gaskets demonstrated that they require sealing forces in the range of 1-4 kN depending on the exact composition, sealing surface geometry and sealing surface diameter even when clean. This leaves elastomer seals or soft metallic seals such as indium, capable of sealing with very small forces as demonstrated originally by Adam et al. (1957). Indium seals have been used (e.g. The Lunar Sample Preliminary Examination team, 1969) or proposed (e.g. Ming et al. 2018, Zhang et al. 2018) in sample return containers on several lunar missions. Elastomer seals are also an option. As perfluoroelastomer seals appear to have the best combination of temperature resistance, strength and force retention, combined with the lowest potential outgassing rate (Hoffman et al. 1998, Heller et al. 1999) of the elastomers, these were selected for initial tests.

4 Apparatus

A custom apparatus was constructed to test the candidate ProSPA seal materials (Kalrez™ and indium), based on the design of Sheridan (2004), which consists of two main components. The first of these is a support structure with a horizontally mounted (face up) plate with a stainless steel knife-edge (interchangeable 5 mm or 10 mm diameter, 1 mm deep, angled at 45°, finish as machined), onto which the candidate seal material is placed before being loaded by use of a metal rod (Figure 2). The sealing force is increased using a thread mounted plate that can be screwed downwards to progressively increase the load at the sealing interface. The assembly of this system for experiments is shown in Figure 3. The applied force is measured using a Burster™ Model 8524 500 N tension and compression load cell mounted below the knife edge block.

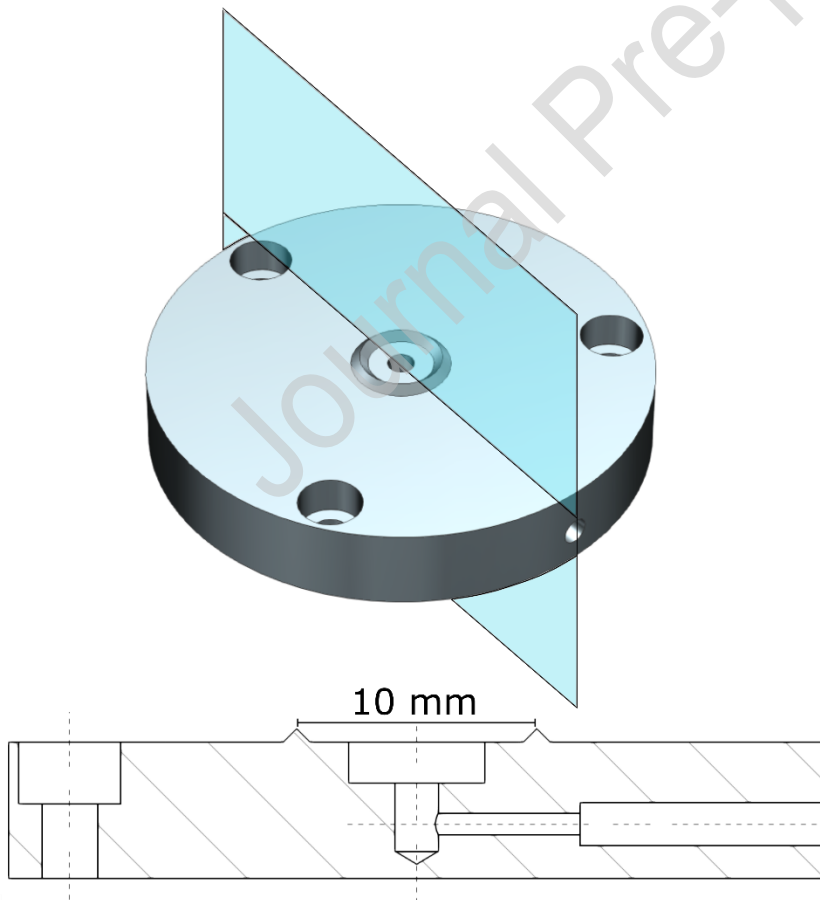


Figure 2: CAD model and cross section of the 10 mm knife-edge block used in the sealing tests. The 5 mm block was identical with the exception of knife-edge diameter.

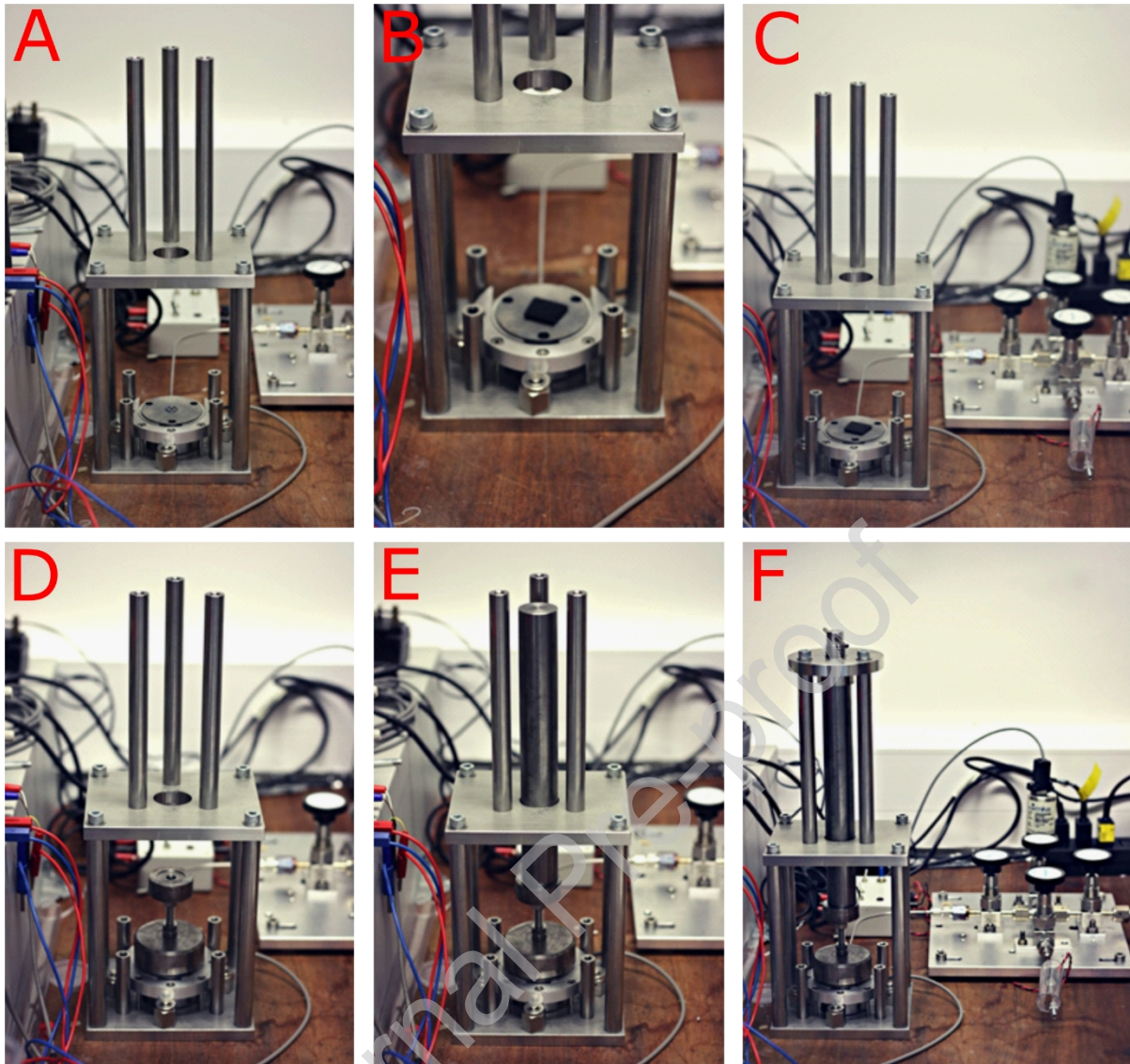


Figure 3: Photos of the experiment setup on the sealing test rig. **A:** Load cell and baseplate are installed into the base of the system. **B:** An area of sealing material sufficient to cover the selected seal geometry is placed in the baseplate. **C:** A complete view of the system with the seal material in place, the measuring apparatus are to the right of the sealing apparatus. **D:** The upper surface of the sealing apparatus is placed over the top of the sealing material. **E:** A rod is placed in order to allow the downward pressure to be exerted onto the sealing surface: **F:** The force is exerted by means of a lead screw attached at the top of the rig.

The knife edge block itself can be swapped in order to test seals of different diameters and contains a welded 2-VCR[®] (1/8") connection which connects into the leak rate measurement apparatus. This is also visible on Figure 3, and a schematic diagram is shown in Figure 4. The leak rate apparatus consists of 4 manual valves attached to a 4-VCR[®] (1/4") cross piece. One

valve leads to a vacuum pump (Pfeiffer HiCube 80), one to the sealing apparatus, and one to an MKS 722A 1 Torr Baratron® capacitance manometer. Valve 4 remains closed and has no purpose in these experiments.

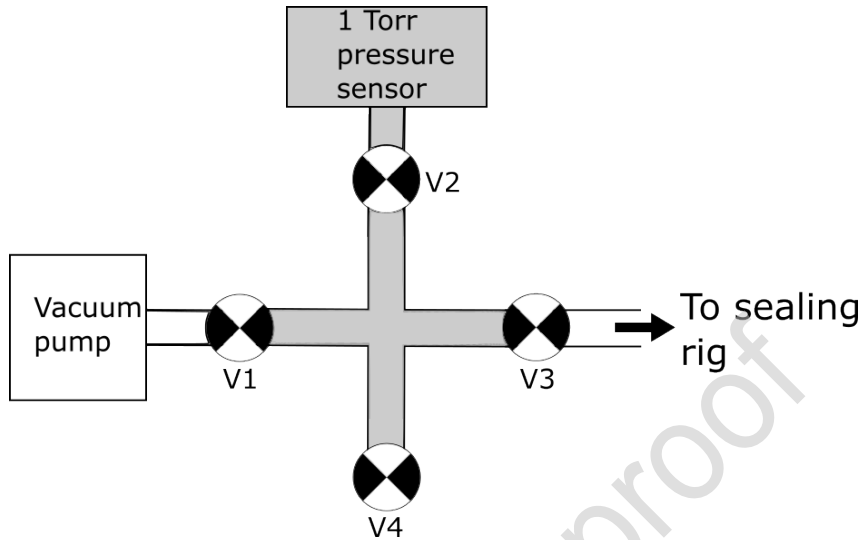


Figure 4: Schematic of rise rate apparatus used in the sealing tests.

The principle of the rise rate apparatus is based on the leak of air into the system through the attempted seal on the sealing test rig using the equation:

$$\text{Leak rate (mbar.l.s}^{-1}\text{)} = \frac{\text{Pressure rise (mbar)} \times \text{volume (litres)}}{\text{time (seconds)}}$$

Where the pressure rise is the measured increase in pressure as measured by the Baratron® during a defined time period after the closure of Valve 1. This time was set to a maximum of 120 seconds during the Kalrez tests, shorter intervals meaning that the Baratron® had become saturated prior to the expiry of the timer. As a result of improved automation and precision during the indium experiments, the timer was reduced to 60 seconds as standard. The volume of the pipework and the valves was determined in advance by using gas equilibration from a known volume and pressure using Boyle's law and was 11.25 ± 0.03 cm³.

5 Preparation and testing of the seals

5.1 Kalrez®

Kalrez® 7075 test blocks were produced by cutting blocks of 40 x 40 x 1 mm and weighing them. The effect of lunar dust contamination was simulated by depositing a measured mass of JSC-1A lunar simulant to the seal and using a spatula to visibly homogenise the dust load as far as practicable. Following this the blocks were reweighed, and the dust load estimated as a concentration based on the difference in mass (Figure 5). Each large block was then cut into four 20 x 20 mm blocks for use in the experiments. It should be noted that the homogenisation procedure was not completely reliable and thus differences can be seen within the dust concentrations in Figure 5. The dust estimates should therefore be taken as an indication rather than an absolute value. The estimated values for dust density are likely to be lower than the real values as the dust is concentrated in the regions where the seal was made, rather than at the margins where it would have not interacted with the knife edge.

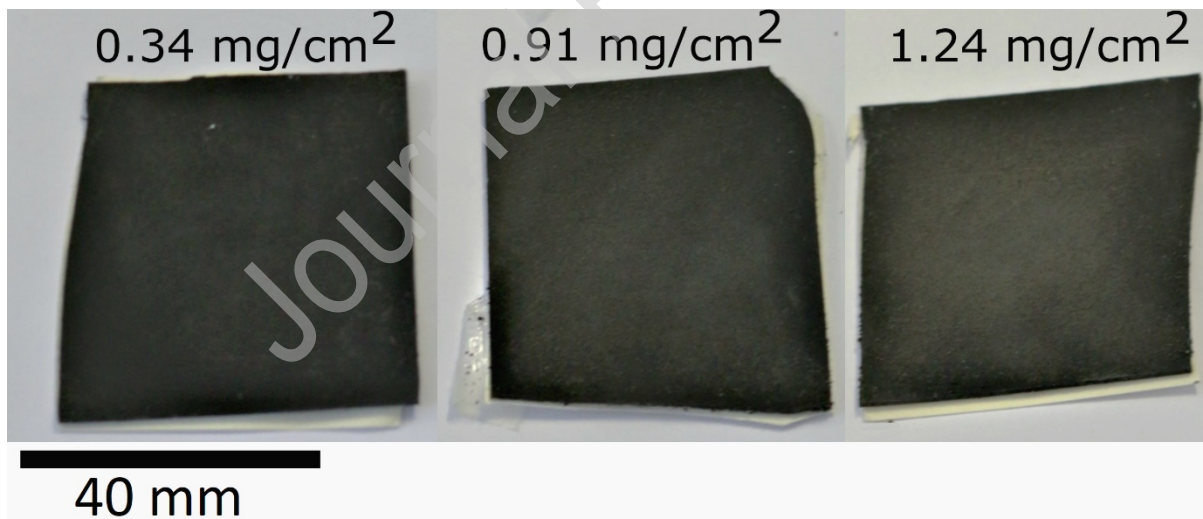


Figure 5: 40 x 40 mm Kalrez® 7075 blocks used to create the dirty seals used in the first phase of tests.

The sealing tests were performed by assembling the sealing apparatus complete with one of the prepared blocks. A force was exerted on the seal and measured using a digital multimeter

connected to the output of the load cell. The valve to the sealing apparatus was then opened to allow the gas already in the system to be evacuated by the vacuum pump. Once the reading from the Baratron[®] had stabilised, Valve 1 was shut in order to isolate the system from the pump and the rise on the Baratron[®], measured over a timed interval of 120 seconds, was recorded. It is important to note that there was some reduction in the indicated force throughout the duration of the experiment, likely as a result of the compression of the seal leading to relaxation of the load. This has been reflected in the use of error bars in the sealing graphs.

5.2 Indium

Given the difficulties of cutting indium without buckling the surface, each 18 x 18 x 0.5 mm, 99.99% purity foil was prepared individually with a set mass of JSC-1A required to create a range of coverages within the suite of samples. These were prepared in the same way as the Kalrez[®] 7075, with homogenisation following weighing of the samples. The samples are shown in Figure 6. The procedure for measuring the indium was also slightly different, with the timing and Baratron[®] handled automatically using National Instruments DAQ hardware controlled through LabView and the measurement time for accumulation reduced to 60 seconds for most of the experiments. Measurements of sealing force were still read manually using a multimeter and experienced the same type of relaxation as the Kalrez[®] 7075 seals, again reflected in the error bars on the sealing force.

0.31 mg/cm² 0.36 mg/cm² 0.47 mg/cm²



0.53 mg/cm² 0.84 mg/cm² 0.90 mg/cm²



5 cm

Figure 6: Individual indium seals used for the indium tests.

6 Results

6.1 Kalrez®

As a baseline test, the duplicate sealing tests of a clean batch of Kalrez® 7075 are shown in Figure 7. This shows that the 10^{-6} mbar.l.s⁻¹ target can be met with approximately an order of magnitude less force than metal seals on the same knife edge. By the projected 400 N sealing force of the ProSPA tapping station, both the 5 mm and 10 mm diameter knife edge seals are comfortably within the 10^{-7} mbar.l.s⁻¹ leak rate range. The 10 mm seal appears to perform slightly better than the 5 mm seal, reproducibly reaching the required leak rate by ~ 150 N.

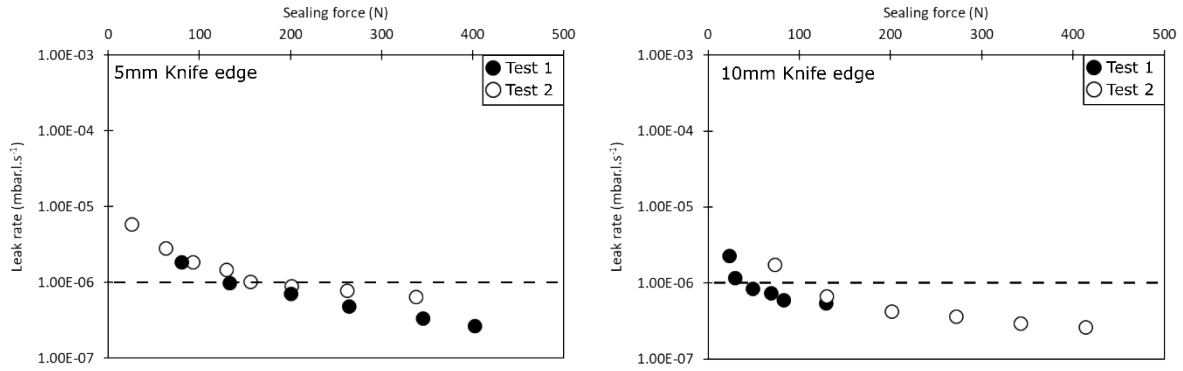


Figure 7: Leak rate as a function of applied sealing force, for 5 mm and 10 mm knife edge sealing into clean Kalrez. The leak rate requirement is highlighted using the dashed line. Error bars are smaller than the symbols.

At the lowest dust level of 0.34 mg/cm^2 (Figure 8) there is a noticeable effect on the seal quality. Both the 5 mm and 10 mm seals require a significantly increased sealing force in order to meet the $10^{-6} \text{ mbar.l.s}^{-1}$ leak rate. A sealing force of $\sim 400 \text{ N}$ is required to produce the necessary seal with the 5 mm knife edge. Only $\sim 200 \text{ N}$ is required for the 10 mm knife edge, although this still represents a significant increase in required force in comparison to the clean seal.

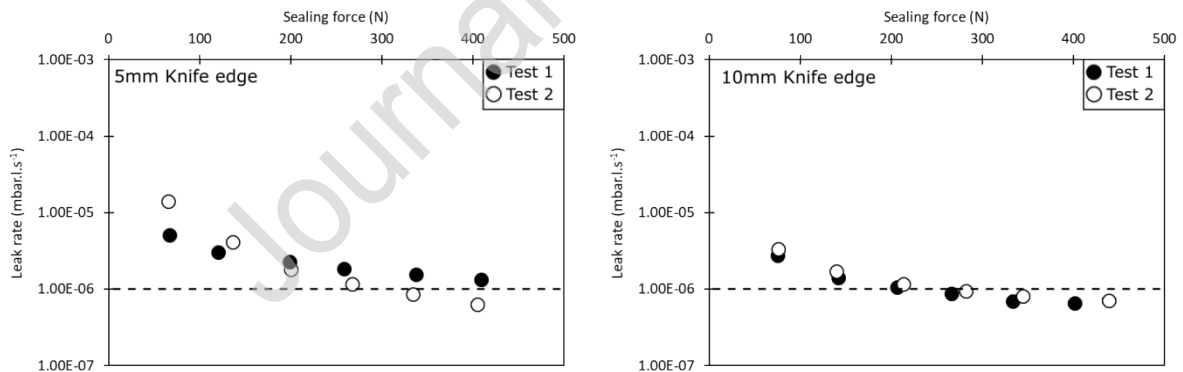


Figure 8: Leak rate as a function of applied sealing force, for 5 mm and 10 mm knife edge sealing into Kalrez loaded with approximately 0.34 mg/cm^2 JSC-1A. Repeats were performed for each experiment. The leak rate requirement is highlighted using the dashed line. Error bars are within the symbols.

The increase in dust density to 0.91 mg/cm^3 (Figure 9) has a similar effect. At this level the 5 mm knife edge can no longer reliably match the required leak rate at the maximum available

sealing force. The 10 mm seal is still able to perform to the required level but requires close to the maximum sealing force to do so.

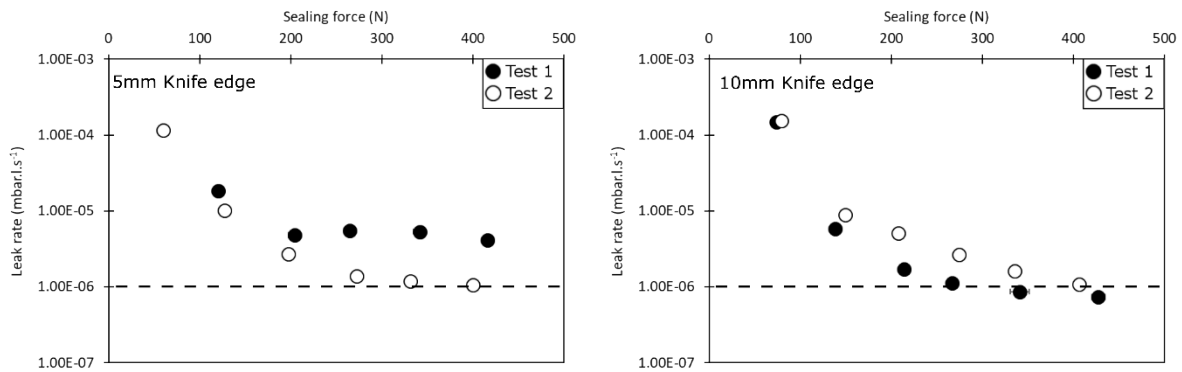


Figure 9: Leak rate as a function of applied sealing force, for 5 mm and 10 mm knife edge sealing into Kalrez loaded with approximately 0.91 mg/cm² JSC-1A. Repeats were performed for each experiment. The leak rate requirement is highlighted using the dashed line. Error bars are usually smaller than the symbols

Finally, at with a dust loading of 1.24 mg/cm² (Figure 10), the seal performance is not achieved with either knife edge. Significant variability is apparent with the 5 mm knife edge with the best result approaching the requirement but the other two sealing at only 10⁻⁴ mbar.l.s⁻¹. The 10 mm knife edge sealed to within the mid 10⁻⁵ range.

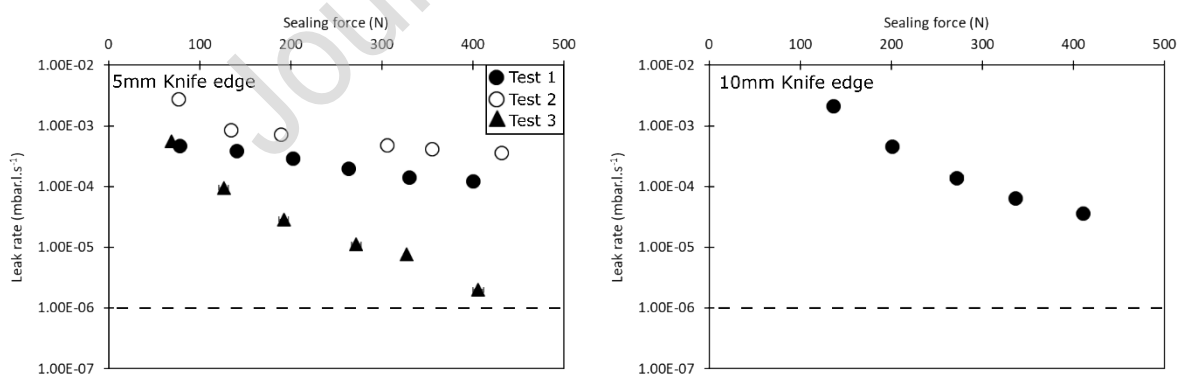


Figure 10: Leak rate as a function of applied sealing force, for 5 mm and 10 mm knife edge sealing into Kalrez loaded with approximately 1.24 mg/cm² JSC-1A. Two repeats were carried out on the 5 mm knife edge as a result of a significant variation between the first and second experiment. A lack of material prevented a repeat on the 10 mm knife edge. The leak rate requirement is highlighted using the dashed line. Error bars are usually smaller than the symbols.

6.2 Indium

Indium performed substantially differently to the Kalrez[®] 7075 when subjected to seal testing. It has not been possible to produce a sealing profile of the clean indium as the leak rate dropped below detection limits with a sealing force of only ~70 N. As a result of the difficulty in preparing dust-contaminated blocks in the same way as the Kalrez[®] 7075, the indium seals were prepared as a spectrum of dust loads rather than identical repeats. Given the noticeably superior performance of the 10 mm knife edge in the Kalrez[®] 7075 tests, the indium was also only tested on this sealing surface.

The results of the different dust loads on Indium are displayed in Figure 11. The effects of the presence of the simulant are clearly visible at the lower sealing forces, a 70 N sealing force only now achieving leak rates of between 10^{-2} and 10^{-4} mbar.l.s⁻¹. At greater pressures however, the seal rapidly improves and, in most cases, achieves the required 10^{-6} mbar.l.s⁻¹ leak rate well within the projected force capacity of the ProSPA tapping station. The 10^{-7} marker on the graph denotes a base level, where there was no measurable leak rate over the duration of the test. Again, most of the Indium seals achieved this level, albeit at different sealing forces. There are two important observations to note from Figure 11. The first is the lack of a trend in the results from different dust loading levels. It would be reasonable to expect the seal quality to degrade progressively with increasing dust levels, but this does not appear to be the case as there is no obvious pattern between seal quality and dust load. This feeds into the second observation, which is that the only sample to not achieve a full seal is the second lowest dust load of 0.36 mg/cm^2 , only managing a leak rate of $\sim 10^{-5}$ mbar.l.s⁻¹ with the maximum sealing force applied. Given the consistently good seal quality of the other 5 samples, containing both equivalent and higher concentrations of dust, it is unlikely that this represents an effect of the dust, but should still be considered.

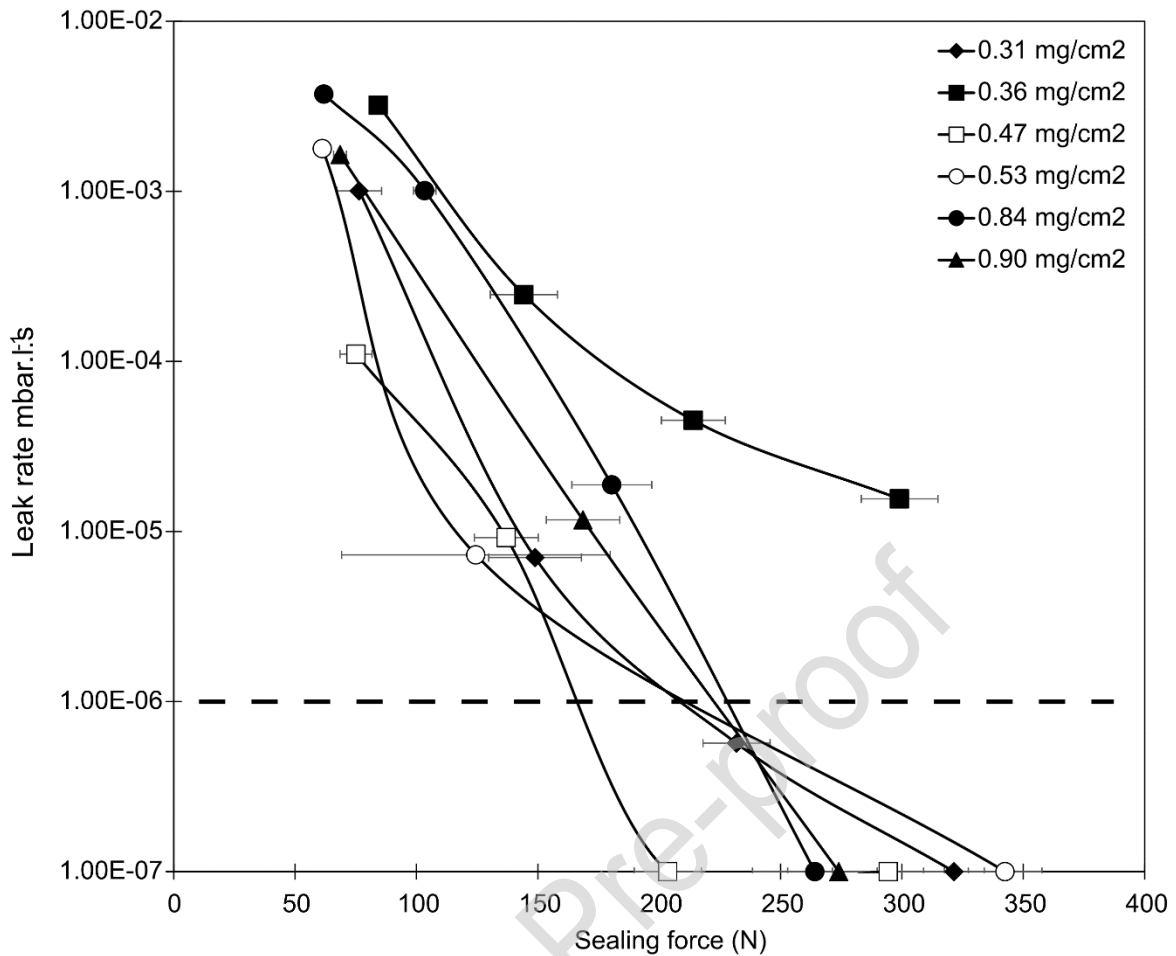


Figure 11: Measured leak rate for seal between stainless steel knife edge (10 mm diameter) and indium sheets dosed with JSC-1A dust loadings, as a function of applied sealing force. Error bars represent stress relaxation as a result of the deformation of the sealing surface. The 10^{-7} mbar.l.s⁻¹ baseline of the graph represents the point at which the leak rates are too low to be measured by the apparatus used.

7 Discussion

The principle behind sealing onto a dust-contaminated surface requires one of two processes to happen: (1) The dust grain needs to be embedded into the seal material in such a way that the seal is able to deform around the grain while maintaining sufficient contact over a sufficient proportion of the sealing surface to prevent gas flow. (2) The knife edge physically cuts through the sealing material tightly enough that the contaminating material is scraped to the margins and the knife edge penetrates to sufficient depth to uncover a relatively pristine

surface that it can seal against. Both mechanisms will be impeded by the presence of dust, but in a different way and to a different extent. In principle, a mechanism based on cutting through the material will be restricted by the thickness of the material and the sealing force available while one based on deformation will depend on the dust load and elasticity of the material.

It is observable from the tests that with the limitations imposed by the deliverable sealing force, the Kalrez® 7075 compound used will barely meet the sealing requirement at dust densities of $\sim 0.9 \text{ mg/cm}^2$. There is also an observable progressive degradation in the quality of the seal as the dust loads are increased for both the 5 mm and 10 mm knife edge seals (Figure 12). This strongly implies that the seal quality is significantly compromised by increasing dust loads, consistent with dust being spread along the sealing surface and implanted rather than being displaced. This means that the mechanism of Kalrez® is likely to be that described by sealing mechanism (1) above. This is in direct contrast to the less well-defined differences between the differing dust loads when using the indium seals, which is suggestive of a sealing mechanism whereby there is an initial barrier to the seal which, once it is overcome, has a much less significant effect on the overall result once the sealing force is increased towards the maximum.

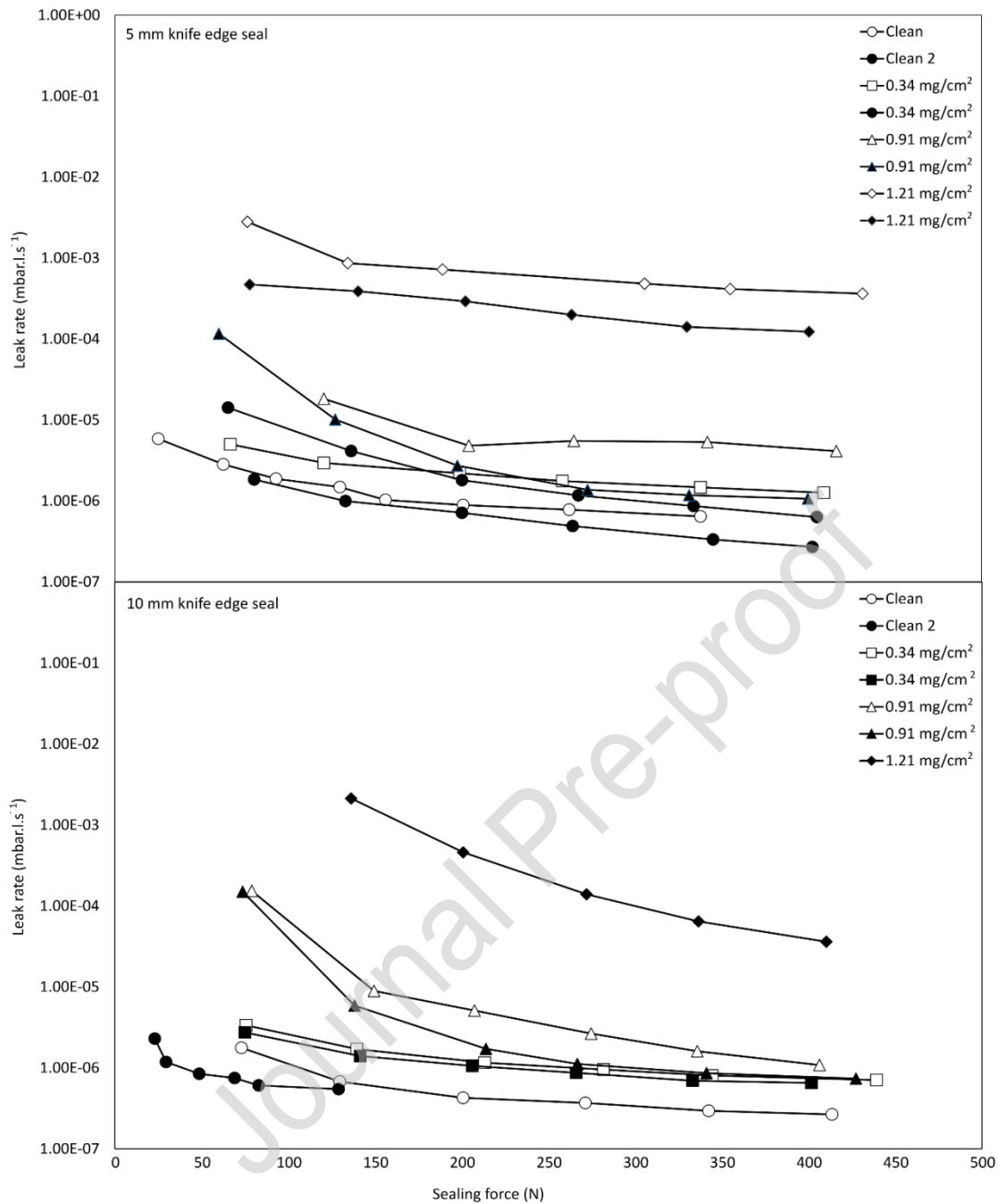


Figure 12: Summary graphs of Kalrez® 7075 sealing performance with different dust loads. Both show a definable difference between the sealing performance at varying dust levels with the 1.21 mg/cm² dust load clearly separated from the lower levels.

The difference of sealing mechanism can be observed directly when the seals are examined after the experiments. Figure 13 shows photographs of Kalrez® 7075 shortly after a sealing

test and of indium after an exploratory test to determine whether it could seal in the presence of a large coverage of JSC-1A. The Kalrez® 7075, as an elastomer, broadly regains its initial shape with a small impression caused by a combination of the indent of the knife edge and implantation of some of the dust grains. In contrast the indium, as is usual for a metal seal, permanently deforms and the broadly uncontaminated metal is visible at the bottom of the indent, with the dust piled at the sides, likely displaced by the knife edge. This suggests that the indium is limited more by the thickness of the seal and the sealing force available, whereas for Kalrez® 7075 the limitation is more likely to be the mechanism by which the material makes the seal.

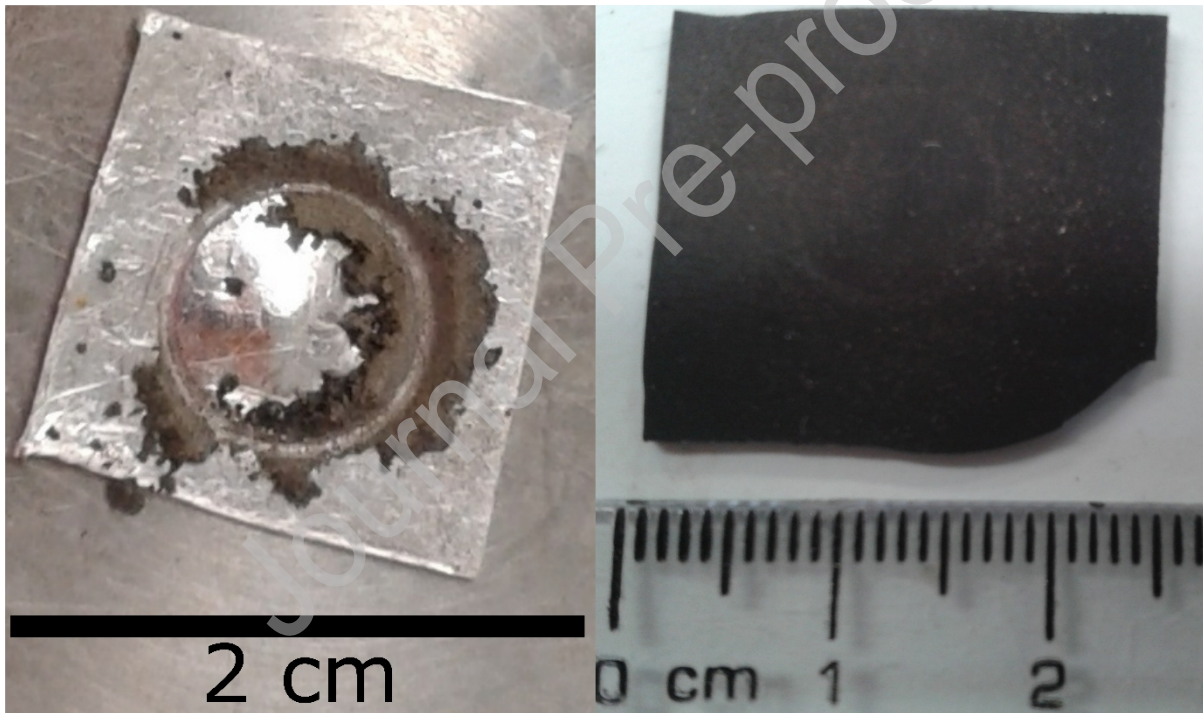


Figure 13: (left) indium after attempts to seal onto a small mound of JSC-1A. (right) Kalrez® 7075 seal after a sealing experiment.

The nature of the indium seal can also provide an explanation for the poor sealing performance in the 0.36 mg/cm^2 test shown in Figure 11. As the foil permanently deforms, minor damage to the surface may result in uneven sealing and a partial gas leak caused by flaws in the foil itself, either the quality or the flatness, rather than the presence of material

interfering with the sealing surface. An alternative is that, as indium is a particularly soft metal, there is the potential for large or slightly obliquely delivered forces to partially tear the metal, creating a small conduit for gas to leak through.

8 Conclusions

The tests have demonstrated that both Kalrez® 7075 and indium are capable of sealing using a ProSPA-like knife edge mechanism and relevant sealing forces, with the quality of seal apparently dependent on the mechanism by which the candidate material makes a seal.

Kalrez® 7075 is able to seal to the required 10^{-6} mbar.l.s⁻¹ leak rate with dust density up to ~ 0.90 mg/cm². The knife edge diameter also appears to play a role in the quality of the seal obtained, as the 10 mm knife edge consistently produced better results than the 5 mm knife edge. In contrast, indium can maintain leak rates of better than 10^{-6} mbar.l.s⁻¹ with the same dust loading and does not seem to degrade much as the dust loading is increased. This appears to be due to the displacement of dust to the margins of the knife edge as force is applied rather than the implantation affects observed in Kalrez® 7075, meaning that the actual seal is always formed on a relatively uncontaminated surface after the dust layer is penetrated. On the basis of sealing with lunar dust contamination, it therefore seems that either option would be capable of performing to the required level.

At present, while it appears that both materials are capable of performing to the required level in the presence of dust, this is only one of the considerations that needs to be made. In an ideal case, the temperature of the ProSPA ovens will range between -100 °C and $+1000$ °C. In contrast, the Kalrez® 7075 used has a maximum rated operating temperature of 327 °C and a minimum operating temperature of ~ -20 °C, at which point it becomes brittle and is no longer able to form or maintain any kind of seal whatsoever, although thawing it does allow it to be used. Indium has a demonstrated track record of sealing in cold lunar environments but

melts at ~ 150 °C. This means that the thermal design of the ovens and sealing interface will need to be examined closely in order to allow these materials to operate effectively.

8.1 Future work

The tests contained within this work provide an overview of the possibilities of Kalrez® 7075 and Indium with respect to sealing ovens on the lunar surface. However, it is clear that the work needs to be progressed in order to produce fully representative results. There are residual questions regarding the thermal and mechanical differences when applying these materials to use within the ProSPA ovens, predominantly surrounding the ability of the materials to operate at the extremes of temperature, and the effect of reduced material mass on the material properties. These questions can both be answered through testing the sealing materials using either ProSPA prototype ovens or thermally and geometrically representative substitutes thereof, which will become available as part of Phase C. In order to accurately reflect the effects of temperature as experienced in a lunar environment, these tests will need to take place in a vacuum chamber to prevent the formation of water ice on the sealing surface.

An additional challenge is to be able to reliably produce homogenous dust loads in large enough quantities to improve the statistical validity of this work. This could be achieved in one of two ways. The first is to input a known quantity of dust into a confined area, use fans to loft and mix the dust particles, and then rely on the subsequent settling of the dust to produce a homogenous layer. The second is to use sieves to distribute a known mass of dust across a known area. In both cases, glass witness plates could be used to measure the dust loading and homogeneity by measuring obscuration of light through the witness plate.

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Editorial handling – Angelo Pio Rossi

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