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An efficient liquid helium / gas-gap switch allowing rapidly servicing low-temperature dynamic nuclear polarization systems

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Abstract. The authors present a novel design for servicing inserts for Hyperpolarising or similar systems operating at temperatures below 4 K. Sample space sleeves that accommodate variable temperature inserts are usually deeply integrated in the cryostat structure and are impossible to remove or to warm up in case of service needs. We present experimental results that show the performance of a liquid helium / gas-gap switch that will be used together with a sample space insert which is in direct contact to a cold plate. This sample space sleeve can be serviced by thermally disconnecting the switch from the cold plate. Furthermore, the additively manufactured heat switch features a novel cold plate interposer for heat transfer that allows the physical separation of the sample sleeve tube from the cold plate enabling a room temperature warm up or complete sleeve removal, if required.

1. Introduction

Hyperpolarized MRI is a new molecular imaging method that allows monitoring of bodily enzymatic and metabolic changes through biochemical methods. The GE Healthcare SPINlab™ C^{13} hyperpolarizing MRI system developed by GE Global Research is an essential, high-end tool for cancer diagnostic research on animals and humans, targeting different types of cancers. SPINlab™ has in particular been developed to serve as a complete stand-alone clinical system using hyperpolarization MRI technology for polarizing pyruvic acid and urea samples for detecting early stages of cancer[1]. Operating this 4-channel hyperpolarizing system involves lowering a small vial into a dedicated polarizing space within the bore of a 5 Tesla magnet. The vial is initially filled at room temperature with 1-^{13}C pyruvic acid and guided into the sample cup where the liquid is frozen and polarized at 0.77 K. 130 °C hot water is then rapidly injected into the vial to dissolve its solid state, maintain its polarization and inject it into a patient. In an effort to further improve ease of operation for the end user and greater serviceability of the cryogenic fluid path design, several key components have been experimentally validated and explained in detail in the following. More specifically, the research and development engineering tasks discussed herein can be broken up into the following steps: design for a straightened rather than curved sample path -> introduce new, removable sleeve -> develop and test a thermal switch for sleeve servicing requirements -> find, select and validate the most appropriate interposing material between switch and cold plate that serves the cryogenic requirements.

[1] For further system details see also Ardenkjær-Larsen [1]

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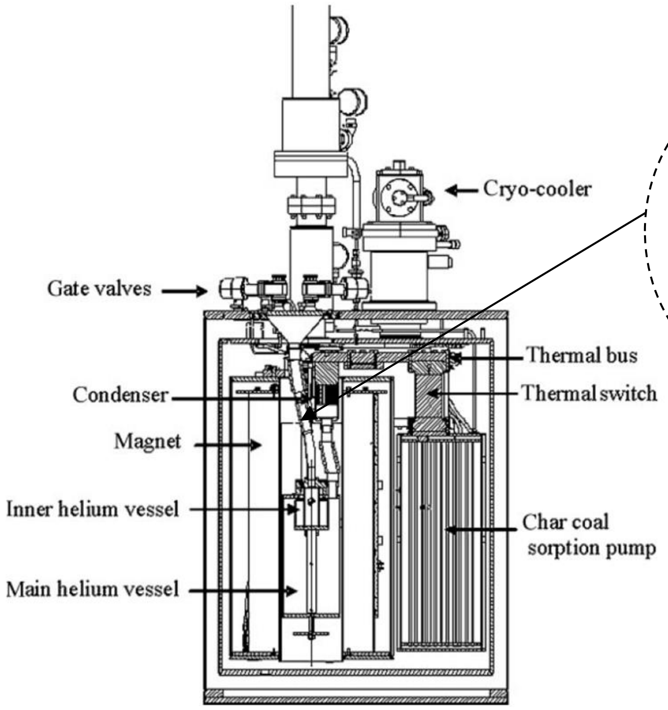
The design change to the curved sample path when lowering the vial into a 1 K sample pot filled with superfluid helium [1] towards a now removable, straight sample sleeve and empty 1 K pot is schematically shown in the figures 1 and 2 below.



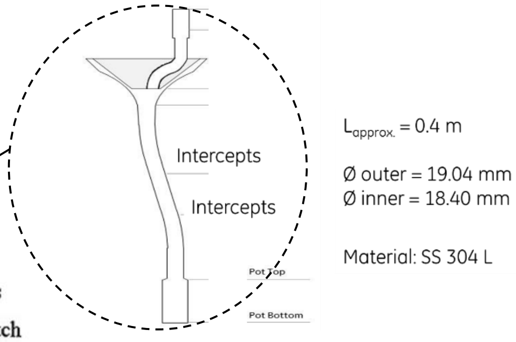
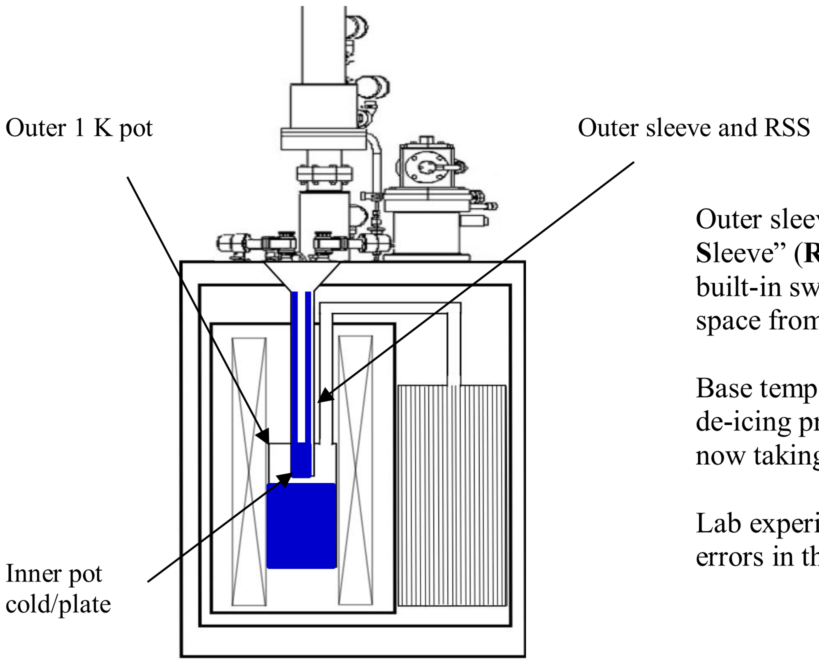


Figure 2 Curved sample path for lowering vials

Figure 2 shows the vial travel path from top of the sleeve with its funnel to the 1 K sample pot filled with liquid helium. The outer pot encloses the inner 1 K pot and is fitted into the warm bore homogeneity region of a 5 Tesla magnet.

Figure 1 Curved sample path (4-vial channel system) for current SPINlab^{TM}.



Outer sleeve with new “**R**emovable **S**ample **S**leeve” (**RSS**/VTI) placed into straight tube with built-in switch to thermally decouple the sample space from the 1 K pot during service;

Base temperature will be maintained < 1 K and de-icing procedures will be greatly simplified, now taking substantially less time.

Lab experiments have shown no vial motion errors in the straight sample path design.

Figure 3 Modified, straight sample path with RSS (removable sample sleeve and internal vial guide stick)

2. Development steps and design parameters to consider

In order to achieve greater resilience of the sample path against any events, such as air ingress or vial break, vial motion incidents or other service related requirements, e.g. sample pot fills or refills and for ease of vial handling, the new, simplified approach as depicted in Figure 2 now requires lowering a **V**ariable **T**emperature **I**nsert (VTI) into the outer sleeve. In this design approach we remove all superfluid helium in the sample pot and utilize the bottom plate of the sample pot as a "cold plate" for heat stationing the vials (within the VTI). The new VTI that is lowered down and in good contact with the cold plate is called RSS (**R**emovable **S**ample **S**leeve). For service, it is further convenient if the RSS can be pulled out completely which also protects the cold plate and the cryostat itself when preparing vials for polarization. To solve these cryogenic challenges for example, a liquid helium gas/gap switch and its thermal interfaces had to be developed. The stringent requirements are summarized below and will be explained in the following sections:

Operational system requirements:

- The ΔT between the two mating, bottom surfaces should be below or equal to 0.1 K (that is 0.77 to 0.87 K) at no thermal load
- Reaching thermal equilibrium across this interface should not take more than 1 to 2 hours.
- The thermal contact conductance between this interface should not change during thermal cycling as this would otherwise affect the thermal balance of the cryogenic component and the operational parameters of the system
- No or very little contact pressure should be applied to the interface and its mating parts
- Thermal contact between these parts should be maintained during cooldown to 0.7 K and remain intact even after multiple warmup/cooldown cycles
- The thermal contact surfaces should be able to recover from "hot" temperature shocks without degradation, whereas the shocks can occur over a period of time of up to 30 minutes, however without reaching room temperature
- Occasionally, it should be possible to completely break those surface contacts without exceeding room temperature (300 K) at the contacts after a cryostat insert warmup or when disengaging the RSS from the cold plate

Cryogenic design requirements

- The temperature profile of the RSS needs to be optimized to minimize stationary heat burden to the 1 K pot (this requires a high strength tube material and small wall thickness)
- Heat intercepts from the outer sleeve need to be thermally linked to the RSS tube
- The interfacing material or component (thermal switch) between RSS and 1 K pot need to be robust and at the same time as thin as possible
- The switch should allow for fast transient operation (e.g. recool, cooldown after a service, e.g. at temperatures > 200 K)
- Thermal contact of the bottom of the RSS tube to the 1 K sample pot cold plate requires implementing a simple and easy to use thermal interposer or so-called TIM (**T**hermal **I**nterface **M**aterial) that fulfils all the above-mentioned requirements.

Switch design requirements

- Choose a flat panel switch structure due to given dimensional constraints in cold sleeve (fitted to thin-walled tube),
- Design for robustness during pressurization and evacuation cycles
- Pre-pressure bearing capable at initial cooldown, without cracking, top/bottom plate deflection or switch fins touching each other
- Given leak-tightness < 1.10^{-10} mbar l / s, no structural microcracking when cycling from room temperature to 4 K
- High fin heat transfer efficiency (switch needs to cool down quickly to 1 K from RT)

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- Switch needs an Off ratio of at least 100 or higher with a 1 W heat load

In this paper we now focus on the cryogenic and engineering aspects for developing the main RSS design details including the thermal switch with the above operating requirements. Galinstan was previously identified as the ideal TIM for this application. Since there are dissimilar materials (copper/vs tested Inconel switch), TIM qualification was done separately and is published in [2], [3]. The switch structure intended to be fitted to the bottom of the RSS is shown schematically in the figures below.

2.1. RSS design overview

In the following Figure 4 the schematics of the setup is given. Figure 5 shows the final configuration with flat panel switch fitted onto the RSS within the sample sleeve and with space claiming vials.

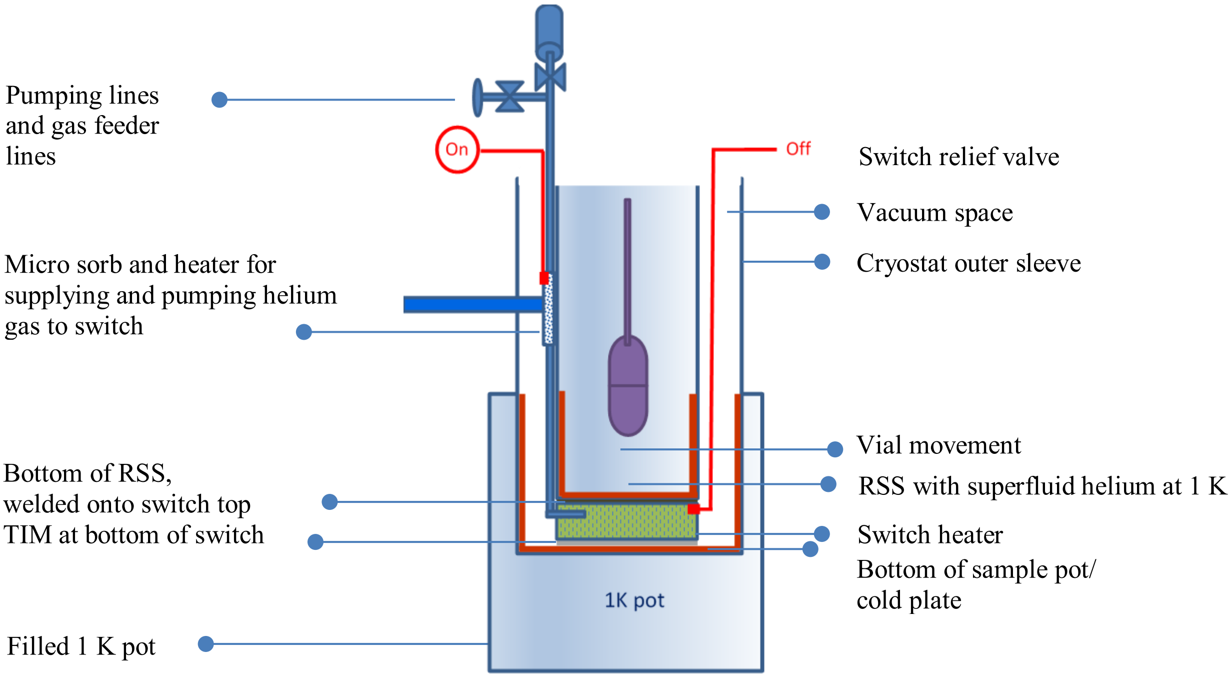
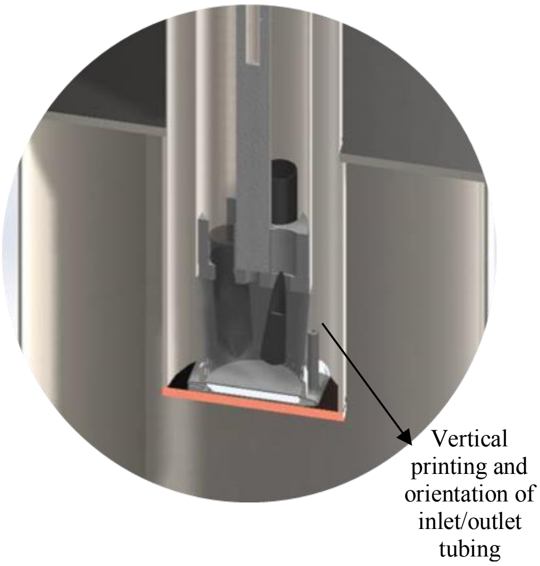


Figure 4 Schematic view of RSS in outer sleeve with attached switch in 1 K pot[2]

[2] Although the temperature is given as 1 K for reasons of simplicity, the actual working temperature can be as low as 0.77 K.

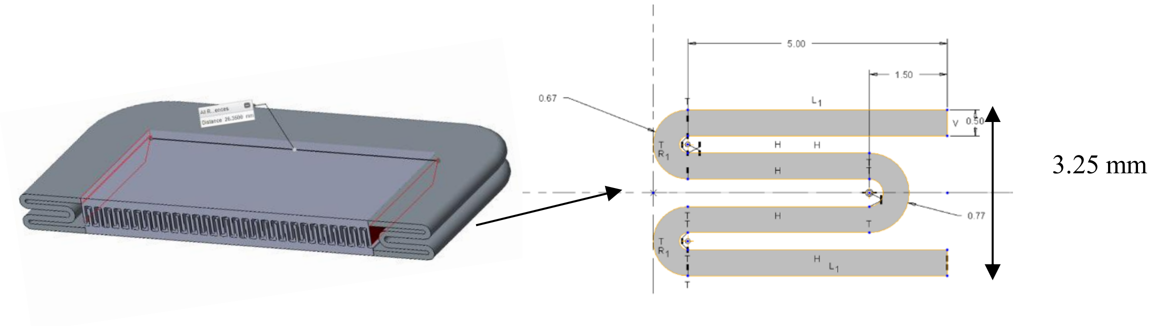


When comparing conventional tubular, copper finned gas-gap switches with respect to compactness, the flat-panel structure, initially developed by Vanapalli and his team for a higher temperature range, immediately looked like an attractive solution [4], [5], [6] that can be fitted to a tube bottom plate. By using an additive manufacturing technique it is now possible to produce a gas gap switch at low cost compared to brazed copper finned switches. 3 material choices were initially looked at, stainless steel, titanium alloy TiAl6V4 and Inconel 718. We chose Inconel over TiAl6V4 since the thermal conductivity of both materials at operating temperature were similar [4] and Inconel tubes can be produced in the required diameter with a wall-thickness of 0.2 mm.

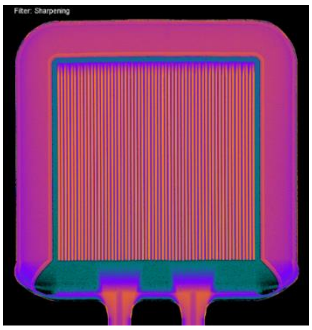
Figure 5 RSS model with vials (black) and envisaged, future thermal switch design (round shape, welded on RSS tube and vertical fill tubes [7]

2.2. *Test switch design*

The following figures show the modeled and manufactured test switch structure as developed for fitting it onto the RSS. The heat path length available in OFF state is given in Table 1 below.



a) b)



c)

Table 1 Thermal parameters for test switch

Thermal test switch	Parameter	Unit
Overall dimensions	3.25 x 37.5 x 37.5 (H x W x L)	mm
Bottom/top plate thickness	0.5	mm
Thermal OFF state length	23	mm
Inner square length	26.35	mm
Fin gap	200	µm
Fin thickness	250	µm
Material	Inconel 718 + copper	-
Liquid helium volume	1.43	ml
Vertical force on switch	300	N

Figure 6 a) Thermal switch design with bottom fill tubes, b) with long heat path length, c) X-ray image[3] showing perfect fin spacing and precise alignment

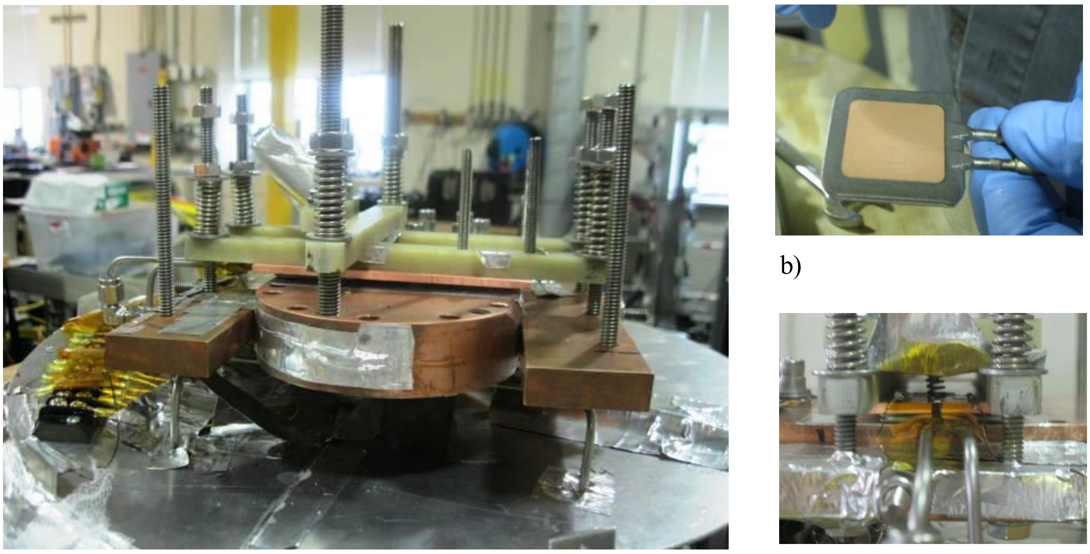
[3] Imaging parameters: Hamamatsu MF 150 tube/DXR250RT detector, 200 µm pixel pitch

Figure 6 shows the internal fin structure and the heat path length in OFF (empty switch) state. The X-rayed switch reveals the internal, unobstructed fin structure with its even spacing and the bottom fill capillary tubes confirming three dimensional, additive processes work well indeed. All parts of the flat panel switch including inlet and outlet tubing as shown in Figures 6 and 7b were additively manufactured in one continuous process. The same can be achieved with the new design that requires vertical printing of the inlet/outlet tubing as shown in Figure 5.

2.3. Switch performance characterization

For the performance characterization the switch was clamped to a copper cold bus that is in direct contact with the second stage of a cryocooler (GE specification). This test setup was previously used for validating TIMs for different cryogenic interfaces. Based on those test results, 3M 425 type aluminum (0.12 mm) and high-purity Indium foil (0.038 mm thick) were chosen as possible interface materials for dissimilar materials (Copper/Inconel).

To achieve optimal contact between switch, TIM and cold bus, both sides of the switch contact surface were copper plated. The switch test setup assembly is shown in Figure 7 below before lowering it into the vacuum chamber. The switch was clamped down onto the cold plate with adjustable metal springs maintaining a compression force of 300 N. A Cernox temperature sensor was held down to the contact surface using small springs.



b)

a) c)

Figure 7 a) Switch test setup, b) copper plated Inconel test switch, c) test switch spring clamped onto cold bus with Cernox temperature sensor and fill tubes

2.4. Turning the switch into ON state

After switch cooldown the cold bus bottomed out at 3 K whereas the top of the switch stayed at 17.5 K indicating a high thermal resistance path length caused by the internal switch metallic structure as shown in Figure 6. Switch loading through the switch fill tubes started by admitting high-purity helium from the gas bottle using a previously evacuated PTFE fill tube with an attached needle valve. The fill pressure was initially set to 5 psi and helium gas was slowly transferred to the switch from the bottle by opening the needle valve for a few seconds.

Figure 8 shows the resulting, immediate switch temperature response and its fast cooldown confirming the efficiency of the Inconel fin heat transfer and the functionality of the gas/gap switch.

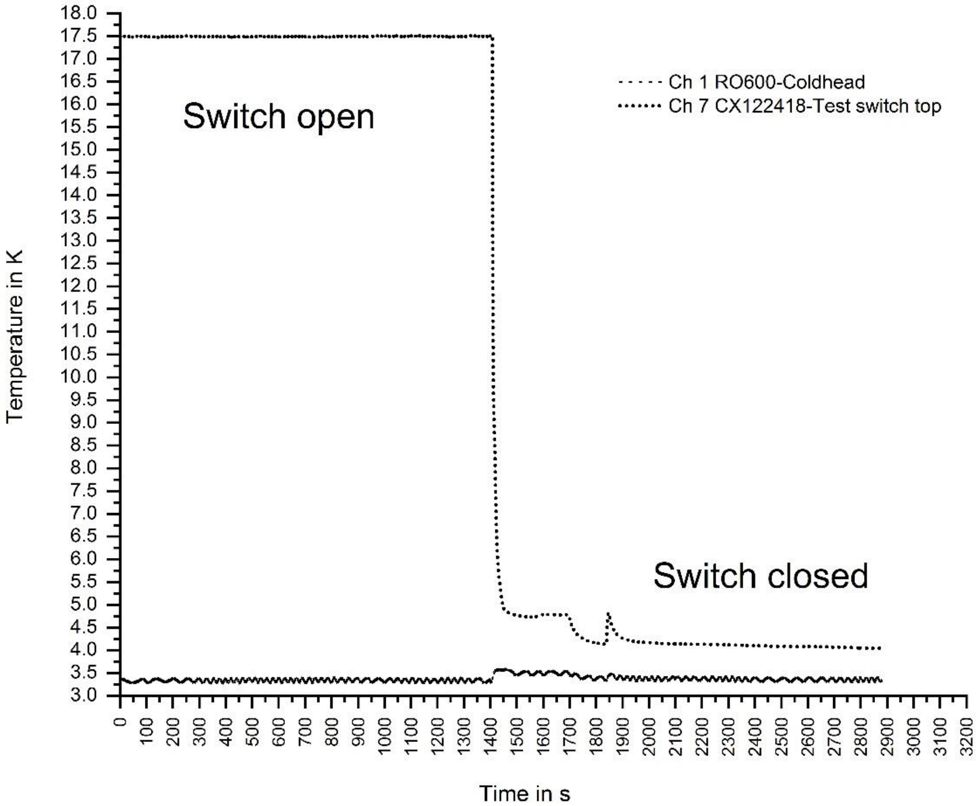


Figure 8 Switch helium fill (partial) and condensation (40 seconds from 17.5 to 5 K)

Since the switch fill volume for the experiment is very small (1.43 ml) the switch was filled manually by opening and closing the needle valves for a few seconds respectively, rather than admitting a continuous flow using a mass flow controller. Figure 9 shows the switch temperature response when opening and closing needle valve and subsequent condensation of helium vapor.
This was repeated until the switch temperature no longer dropped to avoid any switch overfill. To prove that there was indeed a high liquid helium level within the switch and top and bottom fins were at least partially immersed the vapor pressure above the liquid was reduced. Switch and cold plate immediately assumed the same temperature, also further indicating that there is no noticeable temperature difference between the switch and the cold plate.

2.5. Turning the switch into OFF state

Figure 10 shows the switch response when removing helium vapor with a scroll pump. The needle valve was opened for pump out and closed to verify the ON/OFF response time of the switch in the presence of partial helium vapor. Once all vapor had been pumped off, the switch assumed the permanent OFF state. There is no heater required on the switch to turn the switch to OFF state which would normally be mandatory for any automatic operation. A carbon filled microsorb will be used instead to drive the switch.

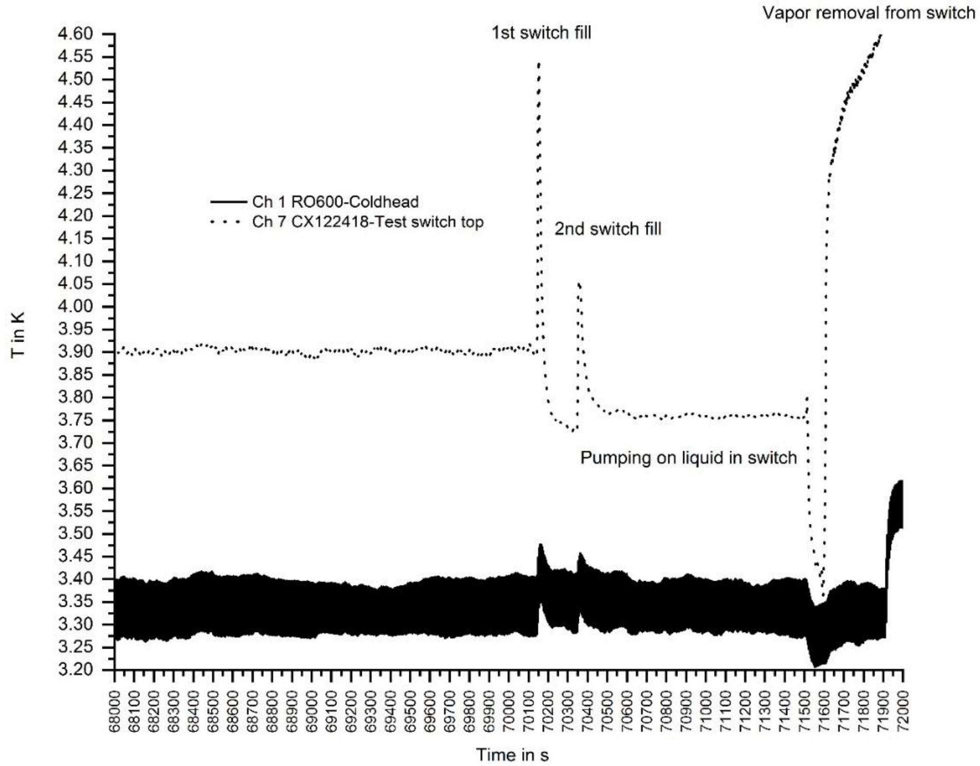


Figure 9 Switch helium fill (partial) and helium condensation

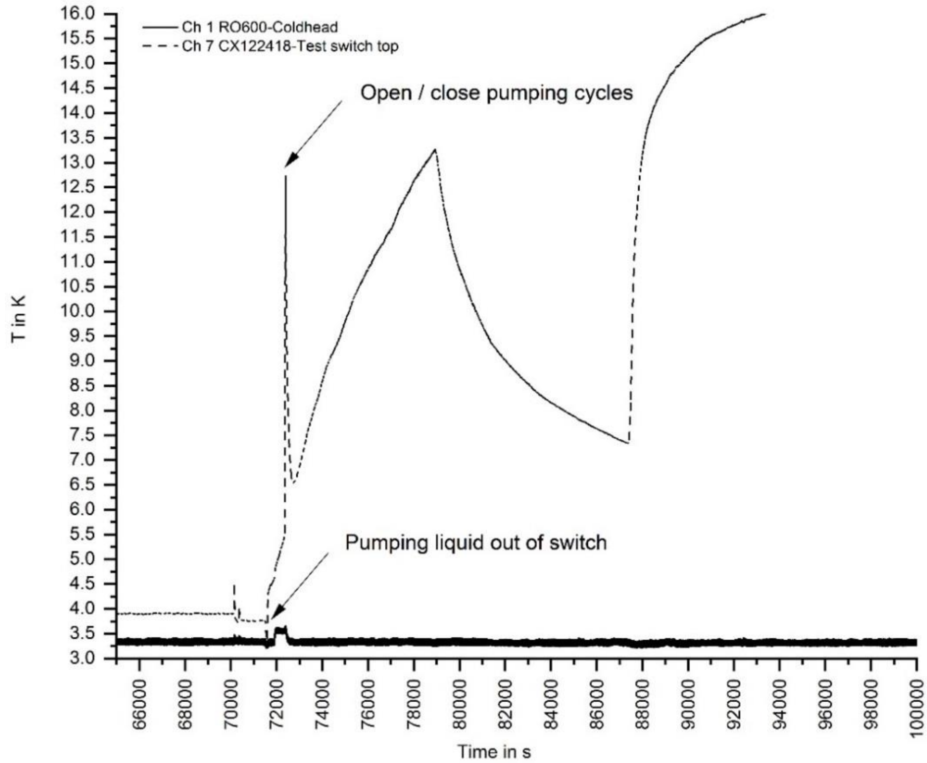


Figure 10 Pump cycles showing the switch ON/OFF state response

2.6. Heat load on switch in OFF state

The OFF state of the switch was defined by applying a constant heat load of 1 W to the switch top plate. The resulting slight increase in cold plate temperature (cryocooler) is shown at the bottom.

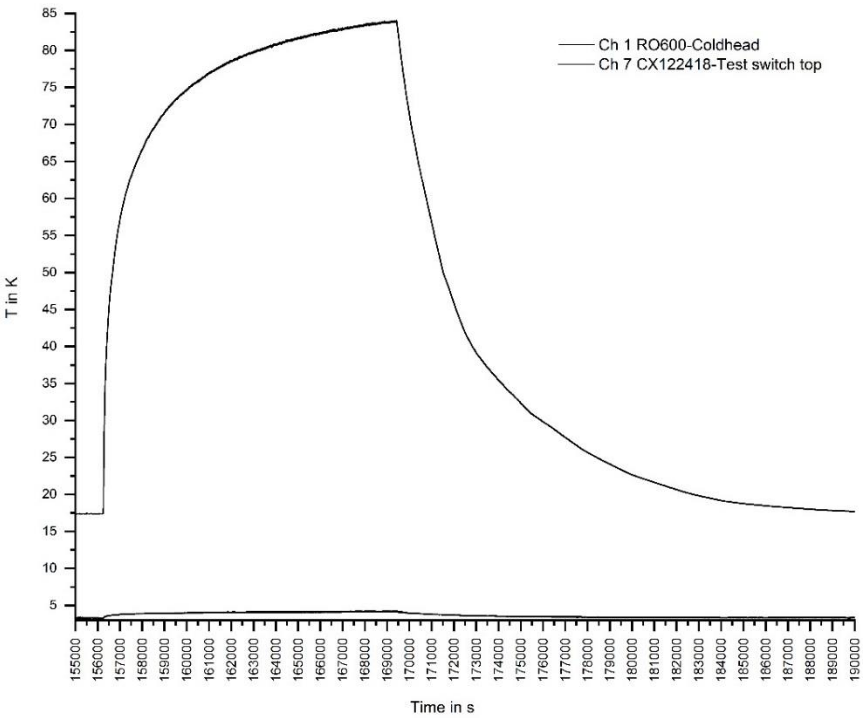


Figure 11 Temperature profile of empty test switch with 1 W applied heat load (T-rise on cold plate)

By applying a constant heat load to the empty switch at the RSS bottom / thermal switch top, the temperature of the cold plate rises by 1 K whereas the switch top temperature elevates to above 90 K with the bottom of the switch remaining at 5 K. The heat load of 1 W for example is sufficient for cleaning or removing nitrogen ice or other contaminants that may have accumulated over time within the RSS. Since the rise is linear with respect to the applied heat load, approx. 3 W of heating need to be applied to drive the sleeve to room temperature all along the tube for removing water ice or other contaminants The cold plate temperature would then rise to 6 K. For transient cleaning heat loads > 1 W the SPINlab^{TM} 1 K pot will be emptied and helium gas transferred back to the main sorb. In this unique cryostat configuration, no magnet ramp down or cryo warm up to room temperature is required for carrying out necessary cleaning work.

2.7. Defining the switch ON/OFF ratio

After completing the measurements, the valves were closed, the gas bottle disconnected and the switch evacuated several times using a scroll pump while the heater was switched off. The set up was allowed to warm up and assumed the same initial start temperature confirming the robustness of the test setup and that interface resistances did not change during repeated thermal cycling. Likewise, during all tests no flow instabilities that could have falsified the measurements were recorded.
If the switch temperature with no heat applied would have been the same as the cold plate temperature there would have been no visible temperature gradient when condensing liquid in the switch and the switch performance would have been poor.

For an applied heat load of 1 W and a switch end temperature of 90 K, the thermal conductance of 1 W / 90 K equals to 0.0111 W / K or to a thermal resistance value of 90 K / W, correspondingly. This is a high value and very well in line with our design target. The ON resistance state is different and cannot be evaluated since no gas phase is present in the switch in actual operating conditions and therefore no heat load can be applied to the liquid in the switch without initiating a phase change [6].

3. Summary

The purpose of this research effort was to develop a new type of RSS/VTI insert that readily fits to a mating cold plate and is easily removable and serviceable. An additively manufactured flat-panel gas-gap heat switch made of Inconel 718 has been qualified and proved to be working reliably in vacuum and in a cryogenic environment well below 4 K. The switch ON / OFF states will be Microsorb driven using activated carbon.

All engineering design goals have been met or exceeded expectations by minimizing sleeve and component heat loads to the cold plate and by validating the thermal switch performance characteristics. The high thermal switch OFF state gives the end user the opportunity to service the sample path without magnet ramp down or system warm up.

The switch design purpose is two-fold: to work as a fast gas-gap switch during service and as an efficient link to the 1 K plate after service has been completed. The latter is achieved by recondensing helium vapor in the switch until the superfluid state is obtained.

A suitable, high conductance, Rohs compliant TIM (Galinstan) has been developed for the chosen interface that enables safe and simple separation of the RSS from switch and cold plate at room temperature, if needed.

4. Acknowledgments

The authors wish to thank S Vanapalli and the 3D systems team for their insight on switch design and additive manufacturing, as well as the GE Global Research Center material's testing and characterization labs. Many thanks also to the technicians working on the tedious cryogenic test setup and to Oxford Instruments (OION). Last but not least, we would like to thank Jonathan Murray of GE Healthcare, Research Circle Technology, for his generous support of this effort.

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