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Research paper

Design and development of a helium injection system to improve external leakage detection during liquid nitrogen immersion tests

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\textbf{A R T I C L E I N F O}

\textbf{Article history:}
Received 16 May 2016
Received in revised form 15 June 2016
Accepted 1 July 2016
Available online 2 July 2016

\textbf{Keywords:}
Helium injection
Nitrogen boiling suppression
Cryogenic thermal control system
Cryogenic external leakage testing

\textbf{A B S T R A C T}

The testing of assemblies for use in cryogenic systems commonly includes evaluation at or near operating (therefore cryogenic) temperature. Typical assemblies include valves and pumps for use in liquid oxygen-liquid hydrogen rocket engines. One frequently specified method of cryogenic external leakage testing requires the assembly, pressurized with gaseous helium (GHe), be immersed in a bath of liquid nitrogen (LN\textsubscript{2}) and allowed to thermally stabilize. Component interfaces are then visually inspected for leakage (bubbles). Unfortunately the liquid nitrogen will be boiling under normal, bench-top, test conditions. This boiling tends to mask even significant leakage.

One little known and perhaps under-utilized property of helium is the seemingly counter-intuitive thermodynamic property that when ambient temperature helium is bubbled through boiling LN\textsubscript{2} at a temperature of \(-195.8\) °C, the temperature of the liquid nitrogen will reduce.

This paper reports on the design and testing of a novel proof-of-concept helium injection control system confirming that it is possible to reduce the temperature of an LN\textsubscript{2} bath below boiling point through the controlled injection of ambient temperature gaseous helium and then to efficiently maintain a reduced helium flow rate to maintain a stabilized liquid temperature, enabling clear visual observation of components immersed within the LN\textsubscript{2}. Helium saturation testing is performed and injection system sizing is discussed.

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1. Introduction

1.1. Boiling suppression using gaseous helium

In a report issued by the United States National Bureau of Standards (subsequently National Institute of Standards and Technology) “Suppression of bubbling in boiling refrigerants” published in Nature [1] the authors found that the presence of bubbles in a body of boiling liquid nitrogen (LN\textsubscript{2}) may be eliminated by blowing helium, hydrogen or neon gas over the surface of the liquid (at rates high enough to break the surface). They reported that the bubbling in the liquid gradually diminished and eventually ceased. The temperature of the liquid had dropped by several degrees and boiling had stopped. They found that the temperature reached by the liquid nitrogen was that at which the saturated vapor pressure of nitrogen equaled the partial vapor pressure of the gas above the liquid. The conclusion was that nitrogen was evaporating into the helium gas. They found the injection of air, nitrogen or argon gases had little or no effect on the bubbling. They followed these experiments by injecting helium below the surface of the liquid nitrogen and found it took 1/6–1/8 of the mass of helium to produce the same temperature reduction as was required when blown across the surface.

Takayoshi et al. in their technical note “The boiling suppression of liquid nitrogen” [2] referenced the 1957 Nature article to show that the cooling effect was known, but they state that at the time of writing their technical note (2009) a comprehensive quantitative study had not been performed. This is perhaps surprising as the Nature article was published in 1957 – a 32 year gap.

The Takayoshi study involved injecting gases into liquid nitrogen contained in a double walled glass Dewar flask mounted on top of a laboratory scale. They measured mass loss of liquid nitrogen over time at flow rates of 1.0, 1.5, 2.0 and 5.0 l/min gaseous helium (GHe). Their experimental results showed that the mass loss of nitrogen over time and the rate of temperature reduction over time increased with greater helium flow rate. The measured temperature reduction correlated well with the predicted temperature reduction based on the mass of nitrogen evaporated, latent heat of vaporization for nitrogen and the thermal masses of the
liquid and glass of the Dewar. They concluded that these tests confirmed that the LN$_2$ was being cooled due to forced evaporation. Xu et al. showed that liquid nitrogen could be cooled to the triple point (63.1 K) by using helium injection [3]. They commented that this convenient method of cooling did not appear to be widely known or practiced by others. The visual clarity improvement brought about by cooling liquid nitrogen below boiling temperature through the injection of gaseous helium has not been previously investigated.

1.2. Leakage measurement challenges

When the immersion method of cryogenic testing is employed liquid nitrogen will tend to be boiling in the containment bath. This boiling is due to heat input from the surroundings, including heat from convection and radiation, but primarily conduction from pressure test lines, possible electrical cable connections, conduction through the containment bath walls and floor and conduction from the surrounding air. Additionally, there may be heat conduction from ancillary components: from energized electrical or hydraulic components (solenoids, servo valves etc.). Heat is also introduced whenever ambient temperature helium is brought into the immersed test component.

This boiling of the LN$_2$ in the containment bath can visually mask even significant leakage at the component interfaces, making it very difficult to determine seal integrity. Additionally bubbles tend to form at nucleation centres such as on the joints and corners of fittings, flanges and couplings – which exacerbates the problem of determining whether bubble formation is caused by test component leakage or by boiling LN$_2$.

Helium has several properties that lead to it being the gas-of-choice for pressurizing components during cryogenic testing. Helium is inert. Helium will diffuse through a leakage path at approximately three times the rate of nitrogen (or air) and so will be able to detect smaller leakages [4]. Helium boils at a temperature of $-268.9 \, ^\circ C$ at ambient pressure [5]. This is well below the boiling point of nitrogen ($-195.8 \, ^\circ C$) and so helium will not liquefy during assembly immersion in LN$_2$. Because of the preference for helium as a cryogenic test pressurizing gas it will generally be readily available for use in the cooling system proposed here.

1.3. Evaporation rate and saturation testing

The most efficient thermodynamic utilization of supplied helium in the injection process occurs when the helium gas is fully saturated with nitrogen prior to reaching the surface of the liquid – all potential for evaporative cooling has been utilized. Increasing the depth of the liquid nitrogen above the depth required to obtain full helium saturation will not improve cooling efficiency, in fact the expansion of the helium as it rises will cause a (minor) heating effect as helium at $-195.8 \, ^\circ C$ has a negative Joule-Thomson (JT) coefficient [6].

Tests performed by Takayoshi et al. [2] included a comparison of equal flow rate bubble streams (1.5 l/min) from different diameter tubes (5 mm, 10 mm and 15 mm) at one liquid nitrogen depth for all helium injection tests. The measured rate of weight loss of liquid nitrogen was similar for all tube sizes. The conclusion was that, because the bubble streams produced by the different tubes had different configurations (bubble size and geometries, bubble rates, bubble surface area and flow characteristics etc.) and yet produced a similar rate of weight loss of liquid nitrogen, all flow streams were fully saturated prior to bubbling from the surface of the liquid nitrogen. The depth of liquid (pool depth) above the injection point was not stated. However the depth can be estimated from the internal diameter of the Dewar (15 cm) and the mass of liquid in the Dewar during testing (1.2 kg), both specified. Assuming the Dewar has a hemispherical base the maximum liquid level above the base would be 11 cm. The method described does not determine actual depth required to obtain full saturation of the helium.

The aim of saturation testing as part of this project is to evaluate the depth of liquid nitrogen required to produce full saturation of a bubble stream using one consistent injector diameter with a constant helium flow rate while varying the pool depth. The helium would be considered to be saturated when an increase in pool depth no longer produces an increase in the rate of liquid nitrogen weight loss. This test injector diameter would then also be utilized in the injection control system and the full saturation depth information obtained would aid in optimising the cooling test system configuration.

2. Saturation depth test

2.1. Helium injection tube

An injection tube was constructed from ¼” O.D. copper tubing. A brass cap was silver soldered to the mandrel bent tube. The brass cap was drilled with a single centralized hole of 1 mm diameter, see Fig. 1. This diameter was chosen to match the 1 mm diameter of the final test system manifold holes and hence a similar bubble stream would be produced for a given helium flow rate. A 1 mm hole is sufficiently large to prevent significant back-pressure build up between the holes and the flow control orifices under all expected flow conditions.

2.2. Dewar calibration

The inner surface of the Dewar to be used for saturation testing was not uniform and so a calibration was performed to produce a depth vs weight Look Up Table (LUT). A Precisa XB 3200C balance with RJ45 RS232 serial port output was connected to a laptop with Precisa Balint V 5.0 data collection software installed. The Dewar was placed on the balance. To calibrate the Dewar, de-ionised water was added to the Dewar at approximately 1 cm height intervals. At each addition of water a measurement of depth down from a reference plane was taken using a depth Vernier gauge and the weight was recorded, see Fig. 2.

Fig. 1. Injection tube.
With the tube installed with the injection hole a known distance (17.3 cm) below the reference plane and compensating for the volume of the helium injector, this LUT was used to calculate pool depth above the injection point. Linear interpolation was incorporated to generate depths between calibration measurement heights. The LUT was converted when used for LN$_2$ testing by including a water/nitrogen density conversion factor.

### 2.2. System calibration verification

Water was added to the Dewar to a measured level of 8.3 cm above the location of the face of the brass cap. Using the weight measurement and Dewar profile LUT the calculated value of water height was 8.35 cm. Given the nature of the testing, where the surface of the liquid will be disturbed during testing, 0.5 mm is considered an acceptable depth error.

### 2.3. Saturation depth test

Configuring the Dewar to act as a depth gauge greatly simplified testing and data collection: when liquid nitrogen was poured into the Dewar a real time indication of liquid depth above the orifice could be seen in Excel: effectively a digital depth gauge. Liquid nitrogen was added to the Dewar, using the depth over the orifice indication in Excel as a real-time guide. The temperature was allowed to stabilize and data collection was initiated. Helium gas flow of 2 l/min was initiated 30 s after the start of data recording. This 30 s delay prior to helium injection was to enable analysis of the stabilized rate of weight loss for each experiment prior to helium injection. Helium flow was stopped 120 s after the start of data recording and data collection was halted after a total of 300 s. The system was configured to record weight and time and to calculate and plot the following graphs in real time:

- Weight vs time (weight automatically set to zero at initiation of data recording).
- Liquid depth above the orifice vs time.
- Rate of weight loss vs time.

Sufficient liquid nitrogen (approximately 76 g [94 ml]) was added to the Dewar to increase the pool depth by approximately 1 cm. The weight vs time measurement process was then repeated. An initial series of 9 tests were run at pool depths ranging from 1.6 cm to 10.0 cm.

A typical set of graphs is shown for the tests performed with a pool depth of 4.91 cm above the injection point. Variation of weight with respect to time is shown in Fig. 3, the variation of pool depth with respect to time is shown in Fig. 4 and the variation of rate of weight loss with respect to time is shown in Fig. 5. The nominal depth above the injection point was calculated from the average of the depth at 50 s and the depth at 110 s. These times were chosen to give a reliable average while excluding any test start-stop anomalies.

The rate of weight loss vs time graph (Fig. 5) provides significant information about the injection process:

- The short-duration weight increase seen at the initiation of helium flow is produced as the helium ejects the liquid nitrogen from the injection tube.
- The rate of weight loss prior to injection is larger than the rate after cessation of helium flow. This difference is due to the liquid boiling (and at a constant temperature) prior to injection and then, immediately after injection has stopped, being below...
boiling temperature (but increasing in temperature). The boiling rate (prior to injection) is such that heat removed from the system, which is the rate of mass loss multiplied by the latent heat of vaporization \( \text{LN}_2 \), is equivalent to the heat entering the system, thus maintaining a constant liquid temperature.

The rate of weight loss during the injection period (30–120 s) can be seen to reduce. A factor in this rate change is the reduction in saturated vapor pressure as the liquid cools.

There is considerable turbulence within the Dewar during the injection period, primarily caused by the injected bubble stream. This turbulence is reflected in fluctuations in the data. It can be seen that turbulence is greatest during injection, less prior to injection (steady-state boiling) and least after injection has ceased (no boiling).

2.4. Rate of weight loss vs time

The following tests were performed to investigate whether there was a depth above which the helium stream was fully saturated prior to bubbling from the surface when injected from a tube with the same hole diameter as the cooling system manifold holes. This full saturation would be indicated by a levelling of the rate of weight loss vs pool depth trace as the pool depth increased. None of the data shown in Fig. 6 indicates a leveling trend: the rate of weight loss of nitrogen with a helium injection rate of 2 l/min is still increasing at a system-limited pool depth of 10 cm, therefore it is clear that with the single 1 mm diameter orifice saturation was not occurring with a pool depth of 10 cm or less. If a full-saturation depth had been established the manifold of the control system would have been installed with the flow holes facing upward and a pool depth matching the saturation depth. Because of this result the flow manifold was subsequently installed with the holes facing down, close to the bottom of the containment tank to maximize GHe/LN\(_2\) residence time. Further testing will include increasing pool depth and varying injection hole diameter and configuration to optimise evaporation rate and verify actual saturation depth. It is expected that for a given flow rate smaller diameter holes will produce smaller bubbles in the stream with a greater surface area to volume ratio and therefore a greater nitrogen evaporation rate will be expected. Sintered injectors (including additively manufactured porous structures) will also be investigated.

3. Helium injection system

3.1. Materials and methods

The principle injector system components are shown in Fig. 7. The complete test assembly is shown in Fig. 8.

3.1.1. Manifold and valve system

The helium supply system includes an aluminium manifold with four separate chambers. Solenoid shutoff valves control the flow of helium gas to each of the four chambers. The gas pressure at the inlet of each solenoid valve is the same. Each manifold chamber has 1 mm diameter GHe injection holes. The chambers have one, two, four and eight holes, see Fig. 9. The doubling of the number of holes, in combination with binary step control (see Section 3.1.2) enables the manifold to produce 15 discrete flow rates. The system was sized to provide controlled but rapid...
cooling of the Styrofoam test box including liquid nitrogen, test manifold assembly and test components.

Flow control orifices are installed between the solenoid valves and the four manifold chambers. The standard orifices are chosen to give as near as possible a factor of two between the flow rates of successive orifices. For example the orifice installed on the manifold section with 4 holes will have twice the flow rate of the orifice installed on the manifold section with two holes. This will provide as close to uniform per-hole flow rate for all holes on the manifold, and hence similar bubble stream configuration. Fig. 10 shows the orifice manufacturer’s flow data in standard litres per minute (SLPM) for the supplied orifices with helium at 25 psig (172 kPa). The ideal characteristic flow rate is shown.

Pressure gages are installed upstream and downstream of orifice number 3 to enable the calculation of system helium flow rate, see Fig. 11.

3.1.2. Binary step control system

In general, if n is the number of manifold sections with the number of holes doubling from one section to the next, then the number of discrete output steps, and hence discrete flow levels, S, is given by 

\[ S = \left(\frac{2^n}{C_0}\right) - 1. \]

For a manifold with four sections the number of output steps will be \((2^4) - 1 = 15\) steps, see Table 1.

In comparison to this binary control system with four chambers, if each chamber contained the same number of holes then fifteen chambers and fifteen solenoid valves would be required to obtain the same individual step resolution. Flow rates for the 15 open steps, utilizing the individual orifice flow data, with 25 PSIG system pressure are shown in Fig. 12.

3.1.3. Closed loop control

The cooling system is closed loop, employing temperature feedback from a type E thermocouple. This type was chosen because, of all standard thermocouples suitable for cryogenic applications, (type E, J, K, N, T) type E has the greatest \(\Delta\) voltage/\(\Delta\) temperature ratio between \(-190^\circ\text{C}\) and \(-200^\circ\text{C}\) (26.4 \(\mu\text{V/}^\circ\text{C}\)) [7]. The microcontroller system, see Fig. 13, initially opens all solenoid valves to reduce the temperature as rapidly as possible from boiling temperature \((-195.8^\circ\text{C}\)). As the temperature reduces the system cycles through the binary control matrix, gradually reducing the helium flow until the system stabilizes when the heat into the system is balanced by the heat removed through the evaporation of liquid nitrogen. The microcontroller is configured to provide a linear bar-LED display of the system flow rate. This visual indication is helpful because the solenoid driver Mosfets are driven in binary sequence so the flow rate corresponding to the Mosfet LED indicators is not intuitive.
3.2. Tests and results

The manifold was immersed with the holes facing down to maximise gas/liquid residence time. Clearance between the manifold and the floor of the Styrofoam container was maintained by the heads of M5 bolts screwed into the four corners of the manifold. A \( \frac{1}{4} \) in. stainless steel compression fitting (approximately 4 cm long), resting on a plastic disk (to aid visibility) was immersed in the liquid together with the manifold assembly. This fitting was not pressurised at any point during the experiment, but was placed to illustrate the improvement in clarity of the liquid nitrogen as the temperature reduced. Once thermally stabilized the liquid level was brought up to 30 mm above the top surface of the manifold, 55 mm above the floor of the container. Photographs were taken after thermal stabilization of the system, Fig. 14, and of the manifold and fitting, Fig. 15. The manifold and fitting were not pressurized. All bubbles were due to the liquid boiling. A large bubble can be seen forming at the (non-pressurized) fitting opening. If this fitting had been attached to a port of an assembly under test it would be very difficult to verify if bubbling formation here was due to leakage or boiling.

Table 1
Solenoid array positions.

<table>
<thead>
<tr>
<th>Step number</th>
<th>Solenoid valve position</th>
</tr>
</thead>
<tbody>
<tr>
<td>15 (-195.8 °C [Max flow])</td>
<td>Open Open Open Open</td>
</tr>
<tr>
<td>14</td>
<td>Closed Open Open Open</td>
</tr>
<tr>
<td>13</td>
<td>Flow reducing as the temperature reduces</td>
</tr>
<tr>
<td>12</td>
<td>Closed Closed Open Open</td>
</tr>
<tr>
<td>11</td>
<td>Open Open Closed Open</td>
</tr>
<tr>
<td>10</td>
<td>Closed Open Closed Open</td>
</tr>
<tr>
<td>9</td>
<td>Open Closed Closed Open</td>
</tr>
<tr>
<td>8</td>
<td>Closed Closed Closed Open</td>
</tr>
<tr>
<td>7</td>
<td>Open Open Open Closed</td>
</tr>
<tr>
<td>6</td>
<td>Closed Closed Closed Closed</td>
</tr>
<tr>
<td>5</td>
<td>Open Closed Open Closed</td>
</tr>
<tr>
<td>4</td>
<td>Closed Closed Open Closed</td>
</tr>
<tr>
<td>3</td>
<td>Open Open Closed Closed</td>
</tr>
<tr>
<td>2</td>
<td>Closed Open Closed Closed</td>
</tr>
<tr>
<td>1</td>
<td>Open Closed Closed Closed</td>
</tr>
<tr>
<td>Closed (No Flow)</td>
<td>Closed Closed Closed Closed</td>
</tr>
</tbody>
</table>

Fig. 11. Manifold system.

Fig. 12. Flow rate vs open step number.

Fig. 13. Micro-controller and solenoid driver board.

Fig. 14. Stabilized test system. LN₂ is boiling (helium shut-off valve is closed).
The system was pressurized to 25 PSIG GHe and the cooling was initiated. A video recording was made of the cooling process. The timing of the step sequence as the system cycled through the binary sequence was taken from the LED display as recorded on the video. The system reached thermal equilibrium approximately 55 s after initiation of helium flow. The injection system cycled between open level three and open level four to maintain thermal equilibrium. These open levels correspond to helium flow rates of 2.0 and 3.4 standard litres per minute (SLPM) respectively. The stabilized temperature was $-197^\circ$C and $-195.8^\circ$C. Each step is 0.1 $^\circ$C.

Fig. 16 shows the step sequence over time. Fig. 17 shows the helium flow rate vs time. Fig. 18 shows the approximate LN$_2$ temperature vs time. The temperature steps on the chart were scaled by dividing the difference between the two measured temperatures ($-197^\circ$C and $-195.8^\circ$C) by the number of steps (12) between the two temperatures. Each step is 0.1 $^\circ$C.

After 123 s the helium flow was shut off. The liquid nitrogen was now clear. Photographs were taken of the system and of the submerged manifold and fitting, see Figs. 20 and 21. No bubbles were visible on or around the manifold or the fitting. External leakage would now be easily discernable for any pressurized component immersed in the liquid.

The temperature increased after the helium valve was shut off. It can be seen from Fig. 16 that the system responded to the increasing temperature by stepping back through the valve sequence. As
the temperature increased the liquid nitrogen began to boil, the temperature stabilizing at $-195.8\, ^\circ C$.

4. System sizing

4.1. Cooling system capability

The cooling system heat-removal capability can be calculated by subtracting the non-helium injection heat removal rate from the heat removal rate during helium injection. The heat removal rate can be calculated from the mass rate of evaporation of liquid nitrogen multiplied by the latent heat of vaporization of liquid nitrogen. As an example, during the single orifice injector testing at a pool depth of 5.5 cm the measured non-injection bath evaporation rate was 0.07 g/s and the evaporation rate with 2 l/min helium flow rate was 0.22 g/s.

The rate of heat removed from the system due to forced evaporation is given by Eq. (1).

$$\frac{dQ}{dt} = \left( \frac{dM_{N_2}}{dt} - \frac{dM_{N_2m}}{dt} \right) \times \Delta H_{vap}$$

where:

- $\frac{dM}{dt}$ = heat removal rate due to injection (J/s).
- $\frac{dM_{N_2}}{dt}$ = evaporation rate during helium injection (kg/s).
- $\frac{dM_{N_2m}}{dt}$ = stabilized no-injection evaporation rate (kg/s).
- $\Delta H_{vap}$ = latent heat of vaporization (N$_2$) = 199 kJ/kg.

Therefore, at a 5.5 cm pool depth with a helium flow rate of 2 l/min the rate of heat removal due to helium injection is given by:

$$\frac{dQ}{dt} = \left( \frac{0.22}{1000} \right) - \left( \frac{0.07}{1000} \right) \times 199 \times 10^3 = 29.9\, J/s$$

4.2. Time to temperature

The helium flow rate through the manifold assembly with all solenoids open was approximately 11 l/min (see Fig. 12). Using the rate of heat removal obtained for the single injector at a flow rate of 2 l/min calculated in Section 4.1. (29.9 J/s) and assuming an equivalent heat removal rate for a given flow rate, the heat removal rate for the manifold would be 164 J/s. Using this rate of heat removal (assuming there are no other sources of heat and thermal mass and assuming instantaneous thermal conduction through the block and liquid) the time-to-temperature for the manifold system with a pool depth of 5.5 cm, cooling a 2 kg aluminium block immersed in 3 l of liquid nitrogen by $-176\, ^\circ C$ would be less than one minute. The calculation corresponds well to the test manifold experimental time-to-temperature shown in Fig. 18.

Once the cooling system has been characterized for rate of heat removal at a series of flow rates and pool depths, matching the injection system to an application simply requires two pieces of information:

- The stabilized rate of heat input into the liquid nitrogen test system bath, most easily calculated from the mass (or volume) loss of liquid nitrogen over time.
- The test system thermal mass.

4.3. Reduction in saturation vapor pressure

As the temperature of the liquid reduces during helium injection the saturated vapor pressure and hence the evaporation rate also reduce. The reduction in saturation vapor pressure can be calculated using the Antoine equation, Eq. (2).

$$P = 10^A \times \frac{T}{B + C + T}$$

where:

- $P$ = vapor pressure, mmHg
- $T$ = temperature, $^\circ C$
- $A$, $B$ and $C$ = Antoine coefficients, for nitrogen:
  - $A = 6.72531$, $B = 285.573$, $C = 270.087$ [8]
- At $-195.8\, ^\circ C$ (boiling temperature)
  - The saturated vapor pressure = 729.3 mmHg
- At $-179\, ^\circ C$ (stabilized temperature)
  - The saturated vapor pressure = 645.6 mmHg

This reduction in saturation vapor pressure of 11% would lead to a slightly higher stabilized flow-rate but would have minimal effect on system sizing, which is based primarily on full-flow conditions.

Fig. 20. System after cooling.

Fig. 21. Manifold and fitting in the cooled LN$_2$. 
5. Conclusions

It has been shown that the residence time obtained when injecting helium at a flow rate of 2 l/min through an upward-facing orifice of 1 mm diameter with a liquid nitrogen pool depth of 10 cm is not sufficient to achieve full saturation: evaporation of nitrogen into the helium is still occurring after this residence time and so a greater pool depth would be required to produce fully saturated helium. This information was used to aid the configuration of the injector manifold of a novel helium injection control system described here.

Through the use of a helium injection control system it has been shown that liquid nitrogen may be cooled efficiently to a controlled temperature that suppresses boiling and enables clear visual observation of components immersed within the liquid. This will aid in the inspection of many critical cryogenic components for which external leakage may have serious consequences, such as liquid oxygen and liquid hydrogen valve and pump assemblies for use in rocket engines. A simple experiment to measure the nitrogen weight or volume loss over time provides the basic information required for sizing the cooling system for a particular application. Estimates of thermal mass and discussions of approximate time-to-temperature should be the only other information required for finalizing the system configuration. The 15:1 ratio between the maximum flow rate and minimum flow rate of the system should make this system suitable for many combinations of stabilized heat input and thermal mass. The system is easily modified for various flow rates by configuring orifice diameter and inlet pressure. Future work will include evaporation and hence heat-removal characterization of the manifold injection system at a variety of pool depths and flow rates. Further investigation of pool depth and bubble stream configuration to obtain full helium saturation will be investigated.

Acknowledgements

AT gratefully acknowledge the UK’s Engineering and Physical Sciences Research Council (EPSRC) funding of the EPSRC Centre for Innovative Manufacturing in Advanced Metrology (Grant Ref: EP/I033424/1).

Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.cryogenics.2016.07.002.

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