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SAMPLE PROBE WITH EMBEDDED TWO-PHASE COOLING FOR CRYOSTATS

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ABSTRACT

Cryogen-free ("dry") cryostats are widely used to achieve as low as mili-K scale temperature, a common environment for neutron scattering experiments. In top-loading dry cryostats, the samples are usually held in a sample can or sample plate at the bottom of a long sample probe, and the sample space is filled with low-pressure helium exchange gas. The cooling of the sample relies on the natural convection of the helium gas, which is eventually cooled by a cryocooler. The inherent inefficiency of this cooling mechanism, as well as low cooling power of cryocoolers, causes up to 8 hours of cooling time from room temperature to base temperature, with typically more than 50% (or even 87%) of this time spent for cooling from room temperature to around 80 K, leading to time-consuming sample change process and waste of high-cost beam time. Here, a sample probe with embedded two-phase nitrogen cooling for top loading cryostats is presented. A 1/4" tube is implemented to a conventional 3/4" (diameter) sample probe for nitrogen transport. A full-scale prototype was fabricated and tested. Cooling from room temperature to around 80 K was achieved in less than 10 minutes due to the efficient two-phase cooling mechanism.

NOMENCLATURE

h	Heat transfer coefficient
T_{sur}	Surface temperature
T_{sat}	Fluid saturation temperature
ΔT	Superheat, $T_{sur} - T_{sat}$

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1 INTRODUCTION

Cryogenic temperatures, usually achieved using cryostats, are common environments in neutron scattering experiments [1]. In these experiments, the sample is either a solid crystal or powder inside a dedicated can. A crystal sample or sample can is mounted on a holder at the bottom of the sample probe, which facilitates loading/unloading, usually from the top of a cryostat. Cryostats that use cryocoolers as the cold source is called cryogen-free ("dry") cryostats, or closed cycle refrigerators (CCR). In dry cryostats, the sample is usually housed in a variable temperature insert (VTI), which is thermally connected to a cryocooler using copper strips and filled with low-pressure helium exchange gas. In this configuration, the cryocooler cools the VTI through heat conduction, which in turn cools the helium exchange gas via inefficient natural convection [2]. The helium gas cools the sample holder through the same convection process, and the sample is ultimately cooled to the base temperature, as low as mili-K scale, by its holder via conduction (Figure 1).

Dry cryostats become the mainstream equipment for low temperature environment in neutron scattering experiments due to the low maintenance cost, simple operation, and, most importantly, not consuming more and more of the expensive liquid helium. However, the cool-down time from room temperature to base temperature ($<$4 K) is time consuming due to the inefficient heat transfer path and low cooling power of cryocooler. As shown in Figure 2, it typically takes $>$3 hours, up to 8 hours to complete, with typically $>$50% (even up to 87%) of the cooling time spending on cooling from room temperature to around 80 K [3–5]. After 80 K, the cooling rate surges until $\sim$10 K, primarily due to the dramatically reduced materials' specific heat

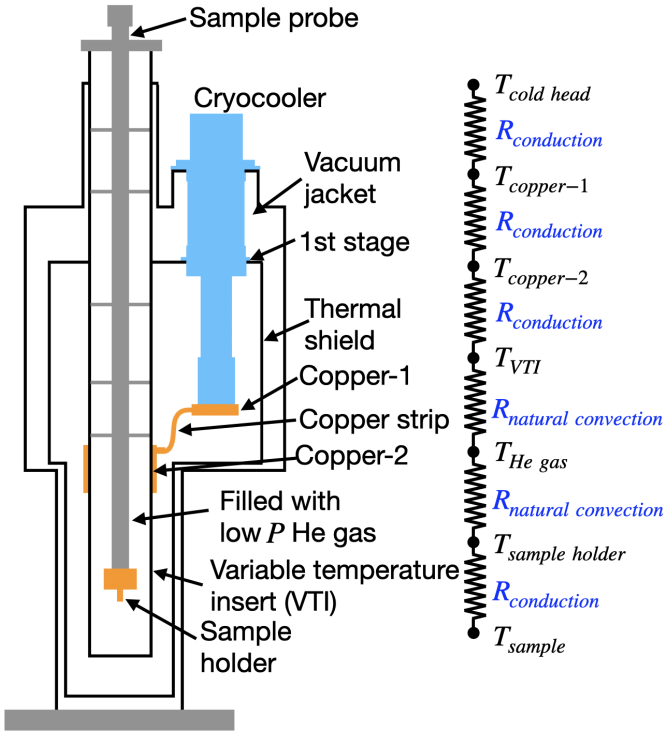

FIGURE 1. A dry cryostat using cryocooler as cold source.

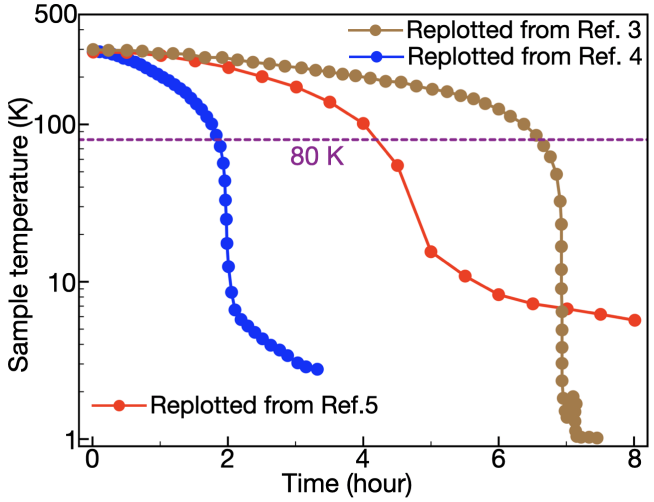

FIGURE 2. Cooling curves of dry cryostats [3–5].

and thus the thermal mass when temperature is below 80 K [6].

Neutron scattering experiments are usually available in large shared facilities and the neutron beam costs in the order of tens of thousands of dollars per hour. The users often face strict time constrains, with valuable beam time lost during sample changes. Consequently, reducing sample change time is of critical significance to the community. In current work, the effort is focused on reducing the cooling time from room temperature to ∼80 K down to less than 10 minutes, compared to hours in a normal operation. The fast cooling is achieved by involving boiling of liquid nitrogen, after the sample is pre-cooled down to 80 K, the cooling will be handed over to cryocooler.

2 SAMPLE PROBE DESIGN AND FABRICATION

2.1 Sample Probe with Two-Phase Nitrogen Cooling

A new sample probe is designed to aid the initial sample cooldown from room temperature to ∼80K in minutes by involving boiling of liquid nitrogen, an efficient heat transfer mechanism utilizing latent heat for heat transfer [7, 8]. Unlike conventional sample probes, which use a central shaft with multiple baffles to host the sample at the bottom, this design incorporates a small tube (1/4” diameter) embedded within a larger 3/4” central shaft. The inner tube transports liquid nitrogen to a chamber near the sample position at the bottom of the probe (Figure 3). The current sample probe design is suitable for all top-loading cryostats. The dimension of a specific cryostat will affect the baffles diameter and sample probe length. The former reduces heat transfer between sample and ambience through radiation to mitigate heat leak and will not affect the cooling performance; while the latter may vary <4” for various cryostats, leading to a few seconds’ difference of cooling time [9], which is negligible compared to the cooling time scale (from hours to minutes).

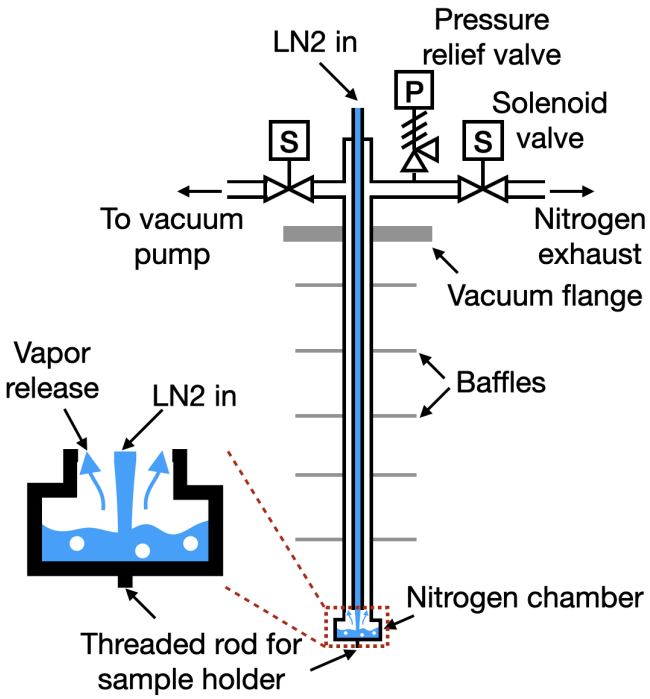

FIGURE 3. Sample probe with two-phase nitrogen cooling.

The chamber, which holds liquid nitrogen, rapidly cools the sample holder through the boiling process of the liquid nitrogen. The cooling process includes four stages of different heat transfer mechanism depending on the surface temperature [9,10]: film

boiling, transition boiling, nucleate boiling, and single-phase convection. The heat transfer coefficients for each mechanism can be estimated using correlations as shown in Eq. 1 [11].

$$
\begin{aligned}
h &= 125 + 0.069 \times \Delta T, \qquad 52 \leq \Delta T \leq 214, \\
h &= 13087.8 - 723.04 \times \Delta T + 13.48 \times \Delta T^2 \\
&\quad - 0.084 \times \Delta T^3, \qquad 19.6 \leq \Delta T < 52, \\
h &= 82.74 - 131.22 \times \Delta T + 37.64 \times \Delta T^2 \\
&\quad - 1.13 \times \Delta T^3, \qquad 4 \leq \Delta T < 19.6, \\
h &= 21.945 \times \Delta T, \qquad 0 \leq \Delta T < 4.
\end{aligned} \tag{1}
$$

The nitrogen vapor generated during boiling is vented to the ambient environment via the annular space between the inner transport tube and the central shaft. A solenoid valve regulates the exhaust release, and a pressure relief valve is included in the system to prevent overpressure. An additional connection at the top allows for attachment to a vacuum pump, which evacuates the line before liquid nitrogen is introduced. This pre-evacuation step removes moisture and prevents nitrogen ice formation inside the sample probe. Once the sample cools to approximately 80 K, the sample probe is re-evacuated to remove any remaining nitrogen vapor and to provide thermal insulation between the sample and the external environment. To further minimize radiative heat transfer, six reflective baffles are installed along the length of the sample probe.

2.2 Sample Probe Fabrication

To minimize heat leakage to the surroundings through conduction in the sample probe, stainless steel is chosen for the central shaft due to its relatively low thermal conductivity [12]. In contrast, copper is used for the nitrogen chamber to ensure efficient heat transfer to the sample holder. One significant challenge in fabricating a designed sample probe is achieving a hermetically sealed connection between the stainless steel and copper components.

To address this, a stainless-steel-to-copper bimetallic joint was manufactured in-house using friction welding [13]. During the process, a stainless tube was rotated at a speed of 1500 rpm while a stationary copper cylindrical piece was pressed against it. The friction generated at the interface produced sufficient heat to soften the materials in the contact region. Upon cooling, this resulted in a strong, metallurgical bond. The fabricated bimetallic joints passed the leak check under vacuum and pressure test at 30 psig. Then the welding interface was visually inspected under a microscope by purposely cutting a window at the joint position, and a clean interface line without apparent defects has been observed (Figure 4).

Then, a full scale prototype sample probe was fabricated. An ultra-Torr fitting for a 3/4" OD tube was used to accommodate

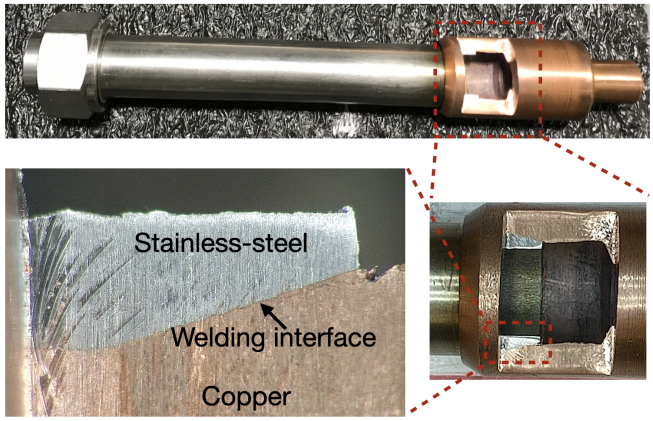

FIGURE 4. Stainless-steel-to-copper bimetallic joint fabricated by friction welding.

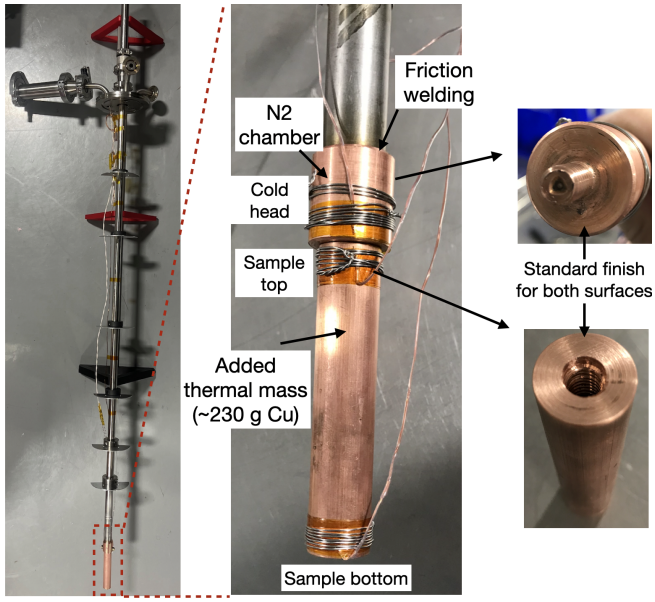

FIGURE 5. Full scale sample probe with embedded two-phase nitrogen cooling.

the 3/4" central shaft with an adjustable length of ±8". The top of the sample probe has an ISO 100 flange for a 100 mm cryostat mounting and includes three KF25 feedthroughs for wires, thermocouples, and other sensors. Six baffles in total are used to minimize heat loss to the ambient. The baffles are fixed on the central shaft using shaft collars with the flexibility of adding more or removing a few if needed, as well as the locations of the baffles. An additional 0.25" ID equalization tube connects the space below the ISO flange and the sample space. At the bottom of the sample probe, a stainless-steel-to-copper bimetallic joint is employed for the copper nitrogen chamber (named cold head) for sample holder cooling. A short 5/16-18 threaded rod was included at the bottom of the cold head to accommodate a sample holder for the neutron scattering experiment. A ∼230 g cooper

rod (name sample) was added during experiments to mimic the thermal mass of the actual sample holder, and the sample can. Both contact surfaces of the cold head and the copper rod have a standard finish (Figure 5). Three thermocouples were included to measure temperatures at the cold head, the sample top and the sample bottom.

3 RESULTS AND DISCUSSION

3.1 Cooling Process Control

The prototype sample probe was tested using a custom-built 4" vacuum chamber to simulate a 100 mm cryostat (Figure 6). In addition to the thermocouples for temperature measurement, a vacuum gauge, three cryogenic solenoid valves (SV), and a pressure relief valve (PRV) were used control the cooling process. The cooling procedure is designed to achieve three goals: (a) minimize cooling time to 80 K; (b) prevent liquid nitrogen (LN_2) from reaching the exhaust due to safety concerns and to reduce liquid usage; (c) minimize the nitrogen boil-off time, which is vital to avoid neutron scattering signal contamination from nitrogen and N_2 solidification due to the rapid temperature drop of VTI below 80 K. Therefore, the sample is cooled by three steps.

1. Initial cooldown: SV #1 fast cycle open/close, SV #2 and #3 closed. LN_2 is supplied gently to cool the sample step by step until it reaches a desired temperature; pulsed LN_2 supply limits the amount of liquid Nitrogen stored in the nitrogen chamber inside the cold head.
2. Structure cooldown: SV #1 slow cycle open/close for N_2 supply and boiling off respectively, SV #2 and #3 closed. The sample is expected to reach 80 K before the VTI does. Thus, more LN_2 is supplied after the liquid in the cold head boils off, hence maintaining the sample temperature at 80 K.
3. Unused nitrogen removal: SV #1 closed, SV #2 cycled with a helium gas supply, SV #3 cycled with a vacuum pump. When the VTI is cooled to a target intermediate temperature, the remaining N_2 in the cold head must be removed before further cooling below 80K. LN_2 is boiled off, and the space is purged with helium gas, followed by the final evacuation.

An electrical box (Figure 7) was built to control three solenoid valves using the temperature readings from thermocouples as feedback. An Arduino Uno is used as the central processing unit, and a relay shield is added to control the valves via three 120 VAC receptacles. The temperature is first measured by an NI thermocouple card (NI 9211) directly, recorded, and then converted to a 0 – 5 VDC signal fed to Arduino Uno by an NI analog output module (NI 9263). An in-house C++ Arduino code uses the temperature signal to guide the program's execution.

The Arduino is programmed to have manual and automatic modes (Figure 7). A user can directly control each solenoid state in the manual mode via a PC serial communication with the Arduino. In automatic mode, the microcontroller would independently control the solenoids based on various setpoints a

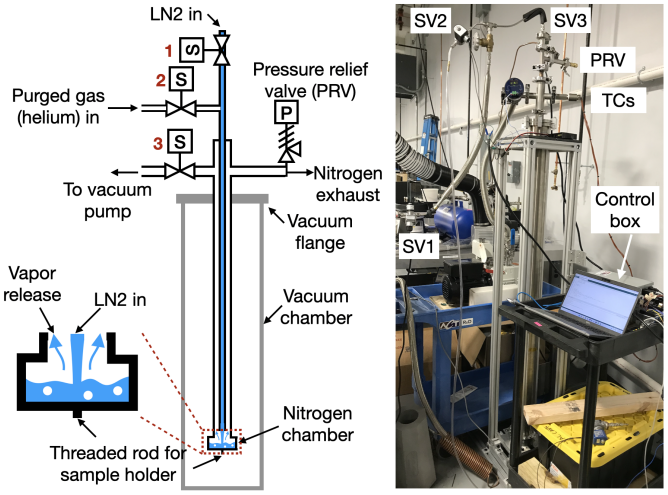

FIGURE 6. Schematic and picture of experimental setup.

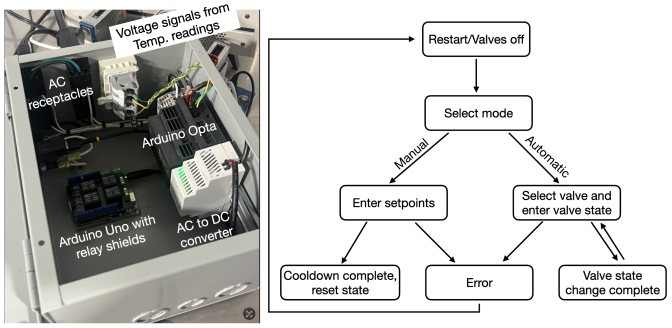

FIGURE 7. Electrical box and the flow chart of the C++ code to achieve the desired cooling process.

user defines before a cooldown: an initial temperature setpoint (SP_I), final temperature setpoint (SP_F), iteration count (I_C), and a vacuum-out temperature setpoint (SP_V). The temperature setpoint will be stepped from SP_I to SP_F with 30-second intermediate steps in the amount of I_C steps. For example, with a combination of SP_I = 105, SP_F = 80, I_C = 5, and SP_V = 97, the controller would use following setpoints 105 K, 100 K, 95 K, 90 K, 85 K, and 80 K to gradually supply LN_2, avoiding liquid in the exhaust, while expediently cooling the sample down to 80 K; then the sample is maintained at 80 K for a few minutes to mimic the process of waiting for VTI cooling. Once the VTI temperature reaches 97 K (SP_V), the helium purge followed by gas evacuation takes place. In an actual operation, SP_V would be the temperature reading from VTI to initiate purging and evacuation.

3.2 Sample Cooling in Manual Operation under Vacuum

The prototype sample probe was tested firstly using a fully manual approach with 4" cryostat simulator under full vacuum. The initial goal was to prove the concept and find reasonable setpoints to avoid liquid Nitrogen at the exhaust. Manual setpoints of 105 K, 95 K, and 85 K were used in sequence until the cold

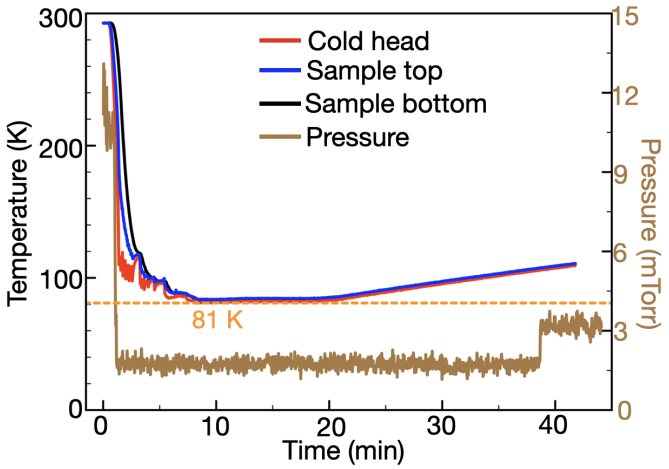

FIGURE 8. Cool-down curve of prototype sample probe under vacuum in a fully manual operation.

head reached 81.2 K after just 10 min (Figure 8). Meanwhile, the sample reached 83.8 K simultaneously, just a 2.6 K temperature difference from the bottom. This indicates adequate thermal contact between the cold head and sample, implying that the standard surface finish is sufficient. At ∼15 minutes, the LN_2 supply was closed, but the cold assembly remained around 80 K for another 4 minutes due to the leftover liquid nitrogen pool inside the cold head. A slow temperature increase afterward indicates the remaining LN_2 has fully evaporated. An active evacuation of the sample probe's internal space started when the cold head was warmed up to 87 K. No temperature drop was observed during the evacuation, further supporting the absence of LN_2. The pressure in the 4" chamber was also monitored during the experiment. The vacuum pump was shut off and isolated when the chamber reached the base pressure of ∼12 mTorr, then dropped to ∼1.5 mTorr during the cooldown. During the post-experiment warmup, the pressure jumped to 3 mTorr around ∼100K, possibly because some condensed gases in the cryostat simulator evaporated. The well-maintained vacuum level further verify the hermetical seal of bimetallic joint down to 80 K.

3.3 Sample Cooling in Automatic Operation under Vacuum

The prototype sample probe was then tested under fully automatic operation mode with the cryostat simulator under vacuum. All three cooldown steps were executed automatically with predetermined process setpoints, as previously described. Although not visible, the 3 setpoint steps (105K, 95K, and 85K) were utilized to portion the LN_2 influx. As shown in Figure 9, the cold head reached ∼82 K within 6 min, with a ∼3 K temperature difference between the cold head and the sample (region I). The sample then rested for 6 minutes without any fresh LN_2 and only heated up to 85K (region II). Subsequently, the room temperature helium gas purged the sample probe space 5 times, leading to a temperature rise to ∼95 K (region III). Finally, the

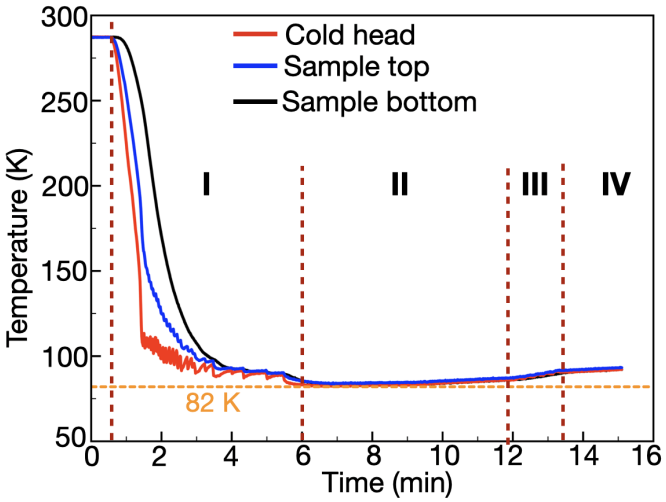

FIGURE 9. Cool-down curve of prototype sample probe under vacuum in a fully automatic operation.

sample probe space was evacuated and, once under vacuum, isolated from the vacuum pump being off (region IV); hence, it was ready for hypothetical further cooldown.

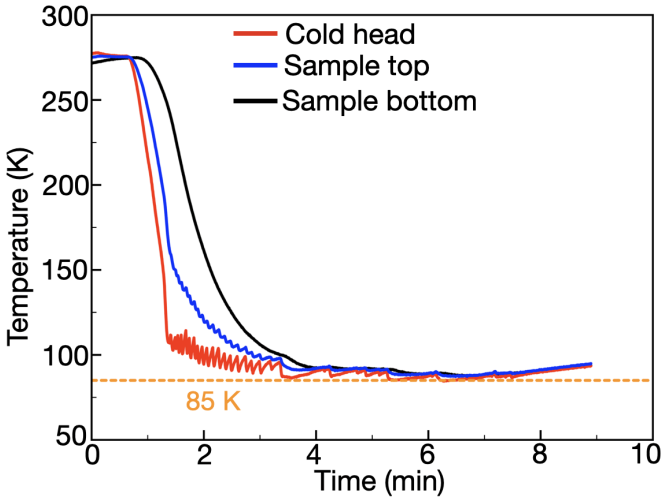

FIGURE 10. Cool-down curve of prototype sample probe under vacuum in a fully automatic operation.

3.4 Sample Cooling in Automatic Operation with Helium Exchange Gas

Last, the prototype sample probe was tested under fully automatic operation mode with the cryostat simulator filled with ∼10 kPa helium exchange gas at room temperature, to mimic a real operation condition in a neutron scattering experiments. All three cooldown steps were executed automatically with predetermined process setpoints: 105K, 95K, and 85K. As shown in Figure 10, the cold head reached ∼85 K within 3 min, with

a $<$2 K temperature difference between the cold head and the sample. The presence of helium exchange gas slightly facilitated cooling and reduced the temperature difference between the cold head and sample. Helium exchange gas increase the heat leakage, indicated by the more disrupted temperature. However, the temperature rase immediately when the LN_2 supply was stopped, implying the liquid build-up was successfully prevented in the cold head. The sample probe space was evacuated after $\sim$7 min, and the sample was warmed up due to heat leakage.

3.5 Future Work

The prototype sample probe would be tested in an actual CCR system. To reduce liquid nitrogen consumption and further reduce time, several strategies to enhance film boiling heat transfer and to increase Leidenfrost point will be explored.

4 CONCLUSIONS

Reduction of the cool-down time of the sample in cryostats is of great significance for fast sample change in neutron scattering experiments. Inconveniently, it currently takes hours for current dry cryostats to cool the sample from room temperature down to $\sim$80 K. A new concept of a sample probe has been presented that embeds liquid nitrogen transport line to achieve fast cooling by involving boiling of liquid nitrogen. The concept was proved by successful fabrication and tests of a full scale prototype. The experiments demonstrate a fast cooling time from room temperature to $\sim$80 K within $<$3 min. The nitrogen supply was finely controlled so that the build-up of liquid inside the sample probe was successfully prevented.

ACKNOWLEDGMENT

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