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# RAPID COOLING TECHNOLOGY FOR EXTREME SAMPLE ENVIRONMENT NEUTRON VACUUM FURNACES

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## ABSTRACT

*One of the main limitations of experimental throughput in high temperature neutron experiments is the lengthy cooldown time of the test furnace. Neutron furnaces typically rely on radiation-based cooling under vacuum to reach safe opening temperatures (<100 °C) to change test samples. The lengthy cooldown, often taking 2-3 times longer than the test itself, is due to the furnace design optimized for minimizing heat loss. A rapid cooling technology for neutron furnaces is presented, where closed-loop circulation of low-pressure helium is utilized. The new cooling technology reduces the most impactful 500-100°C cooldown phase from over 2 hours down to as low as < 5 minutes for low thermal mass cases with no sample. The gas mass flowrate had the dominant impact on cooling time, while the system pressure had negligible effects.*

Keywords: Neutron Experiment, Vacuum Cooldown, Radiation, Helium cooling.

## NOMENCLATURE

| | |
|---|---|
| $\rho$ | Density [kg m$^{-3}$] |
| $\alpha$ | Thermal diffusivity [m$^2$ s$^{-1}$] |
| $\nu$ | Kinematic viscosity [m$^2$ s$^{-1}$] |
| k | Thermal conductivity [W m$^{-1}$ K$^{-1}$] |
| h | Heat transfer coefficient [W m$^{-2}$ K$^{-1}$] |
| u | Velocity [m s$^{-1}$] |
| L | Characteristic length [m] |
| T | Temperature [°C] |
| $\dot{m}$ | Mass flow rate [scfm] |
| Re | Reynolds number, $u\,L/\nu$ |
| Pr | Prandtl number, $\nu/\alpha$ |
| Nu | Nusselt number, $h\,L/k$ |

## 1. INTRODUCTION

The unique properties of the neutron, such as electrical neutrality, magnetic dipole moment, and tunable energy levels & wavelengths, make them ideal as an *in-situ* probe for a wide range of materials under both ambient and extreme conditions of pressure, temperature, and magnetic or electric fields. Neutron scattering and related diffraction techniques have been used since the 1970's to obtain information about materials that were impossible with conventional methods such as microscopy [1,2]. However, due to the large logistical and financial cost associated with maintaining a controllable neutron "beamline" source, neutron experiments are generally limited to large research facilities such as the Spallation Neutron Source (SNS) at Oak Ridge National Laboratory (ORNL) and the European Spallation Source (ESS). The broad applicability of neutron experiments for various fields, such as material science, polymers, and medicine, makes those user facilities remain overbooked with lengthy waits for neutron beamline time. In addition to the limited number of facilities, there are further challenges related to experiments in extreme pressure and temperature sample environments. The standard *blue-series* neutron vacuum furnace, AS Scientific Ltd (U.K.) [3], is commonly used for experiments up to 1500 – 1800 °C. The furnace design (Figure 1a), comprised of a resistive heating element surrounded by multiple concentric radiation shields [4], is limited to radiative cooling under vacuum to cool to an opening temperature of ~100 °C. The strong temperature dependence of the radiation heat transfer and the significant thermal mass of the furnace result in a lengthy cooldown time at temperatures below 500 °C. For example, in the HOT-006 neutron furnace at the SNS facility at ORNL (Figure 1b) [5], 90% of the total cooldown time from 1500 – 100 °C is during the temperature range of 500 – 100 °C. One method to reduce the cooldown time that has been adopted by neutron facilities is to back fill the furnace with nitrogen or helium gas and subsequently vacuum down the chamber before repeating the process. This process can reduce the total cooldown time by 2-4 hours, however; the repeated batch fill procedure leads to oscillations in the furnace temperature and is limited to a starting temperature of ~300 °C to avoid oxidation or stress induced deformation to the furnace components [6,7]. There remains a lack of a cooling solution to address the lengthy cooling time for neutron furnaces, especially at a temperature range of 500 – 100 °C where radiation cooling becomes insufficient.


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One approach to increasing the heat transfer rate is helium-based forced convection cooling. In addition to the high thermal conductivity, helium has the advantage of being chemically inert and has a low neutron cross-section, i.e., it doesn't affect the neutron beam operation [8]. With this unique property, helium can enable a new modality for *in-situ* cooling in neutron experiments, which has largely been limited to controlling the radiation rate via the heater input power [9–11].

Goodway et al. [7] achieved a 60% cooling time reduction from 500 – 100 °C by implementing an open-loop continuous helium flow. However, the approach was limited to flow injection outside radiation shields at small flowrates to reduce helium consumption. An improved closed-loop cooling system that actively circulates low-pressure helium inside the furnace is investigated here. Helium is injected inside the radiation shields and directly onto the sample, enabling additional convective-advective heat transfer paths. The increased flowrate and improved flow distribution show a significant reduction in the cooldown time from 500 – 100 °C.

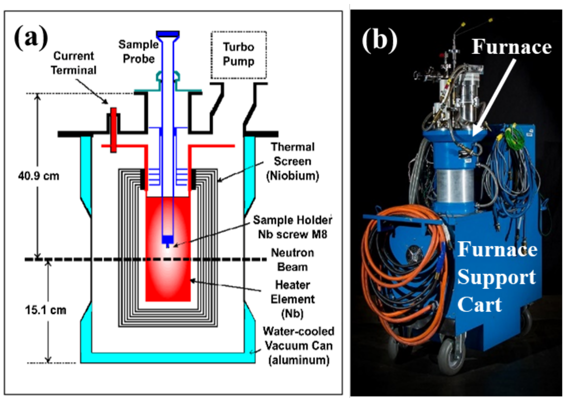


**FIGURE 1:** HOT-006 NEUTRON FURNACE DESIGN (a) [4] AND FURNACE BODY ON TOP OF SUPPORT CART (b) [5].

## 2. MATERIALS AND METHODS

The proposed cooling system is presented in Figure 2. The major components are housed in an external wheeled enclosure: low-pressure helium circulation blower, vacuum pump, refrigerant condensing unit with heat exchanger, and PLC control hardware. An ISO 100 tee with the branch terminated by a feedthrough endcap interfaces the main furnace volume with the external system. Inside the furnace, two helium inlets deliver gas into proprietary nozzles that penetrate the radiation shields to deposit gas between each shield and directly onto the sample location. A single return hose is used to form the closed-loop system. All helium lines have a dedicated isolation valve inside the intermediate adapter box to enable vacuum system isolation and allow independent bypass operation necessary for an oxygen purge. The low-pressure cooling system has an operating range between 4 and 14 psi absolute. The upper limit avoids over-pressurization of the vacuum furnace, while the lower pressure constraint is from mechanical component limitations. The pressure of the cooling system and the furnace during the cooldown is usually near-equilibrium, as the overall pressure drop for the cooling system is minimal (< 0.1 PSI).

Cooldown tests were performed on the HOT-006 neutron furnace at ORNL and in a test furnace at Advanced Cooling Technologies, Inc. (ACT). The test furnace at ACT was designed as a thermal analog to HOT-006 and has been confirmed to have similar thermal characteristics under both radiation and helium-based cooldowns. The mass flow rate was measured using an orifice plate as per the ISO 5167 method [12] with a maximum uncertainty of ±10%. The reported temperature was measured using a K-type thermocouple placed at the typical sample location in the middle of the furnace. A sample was absent during these tests, as the current effort concentrated on the bulk furnace cooldown. The cooling system overview and setup, as tested on the HOT-006 furnace at ORNL, is shown in Figure 2.

Each experimental run started with the furnace being vacuumed down, heated, and held at 500 °C for ~30 minutes to reach thermal equilibrium. The oxygen levels were continuously monitored, and an oxygen purge was performed as necessary to maintain the $O_2$ levels below 200 ppm. At the cooldown start, the main solenoid valves were opened to begin the helium flow into the furnace. The helium continued flowing through the furnace at the prescribed flowrate and pressure during the cooling period. Once the furnace core temperature reached 100 °C, the same valves were closed to isolate the furnace, completing the cooldown cycle. The entire process was fully automated via an Allen Bradley PLC and required minimal user input. The gas flowrate and the overall system pressure were controlled via the combination of pumping speed, gas supply pressure, and vacuum pump engagement, all of which have been automated via the PLC system.

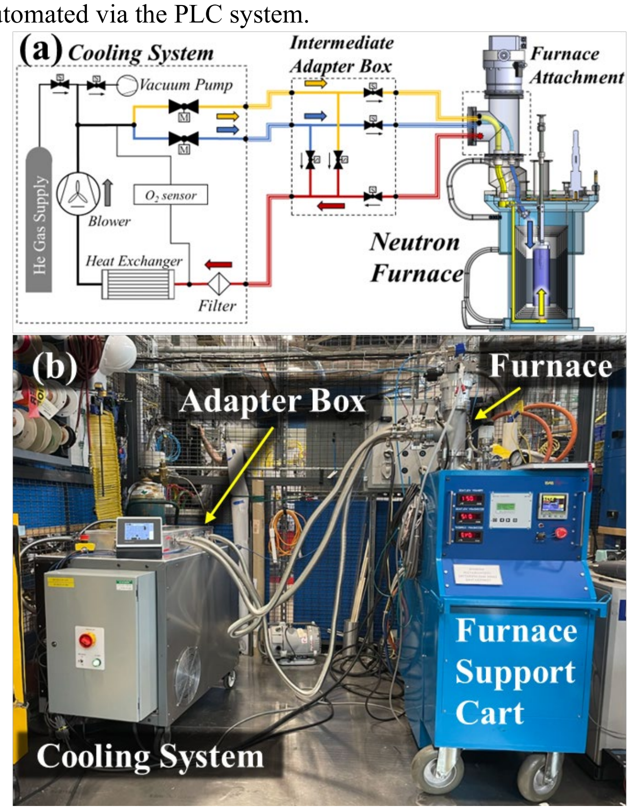


**FIGURE 2:** (a, b) COOLING SYSTEM (LEFT) WITH INLET AND RETURN HOSES CONNECTED TO FURNACE (RIGHT).


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## 3. RESULTS AND DISCUSSION

Figure 3 shows the comparison of cooldown times of the HOT-006 furnace with the radiation and helium-based cooling.

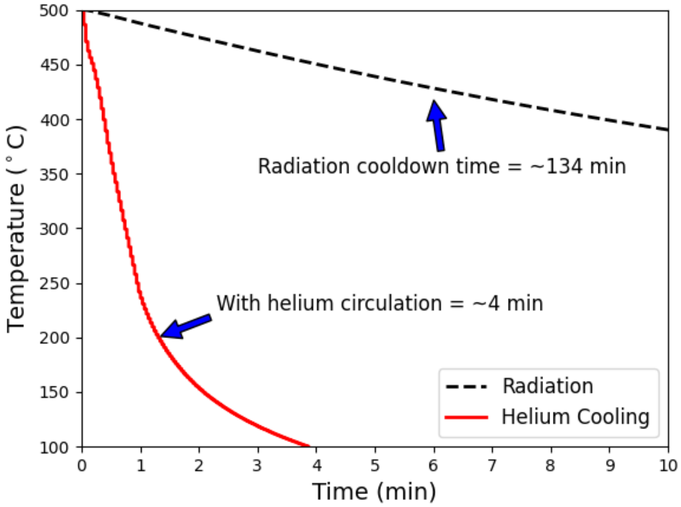


**FIGURE 3:** COOLDOWN COMPARISON FOR HOT-006 FURNACE. THE COOLING SYSTEM REDUCES THE COOLDOWN TIME FROM 134 MINUTES TO 4 MINUTES.

The cooldown time from 500-100 °C at a helium flow rate of 6 scfm was reduced from 134 minutes to approximately 4 minutes, a ~33.5x improvement. The results demonstrate the ability of the added cooling system to drastically decrease the turnaround time of neutron experiments by reducing the post-experiment cooldown time. Since the facility neutron beamline remains in continuous operation regardless of the experiment status, the lengthy instrument blackout during the cooldown represents a period of valuable beam-time losses that otherwise could be utilized for performing more experiments.

The rapid cooldown system has been extensively tested with the HOT-006 and ACT test furnaces. No apparent degradation to the furnace components was observed from the 500 °C repeated cooldowns throughout the experimental period. The following sections investigate the impact of helium flowrate and system pressure on the observed cooldown performance.

### 3.1 Impact of Flowrate

Figure 4 shows the effect of different helium mass flowrate on the cooldown time in the thermal analog test furnace at ACT. A larger helium flowrate was found to correspond to a faster overall cooldown time, with the effect especially pronounced below 400 °C. The cooldown time with 1 scfm of helium was approximately 21 minutes. Doubling the flowrate to 2 scfm reduced the time to 13.5 minutes, an improvement of ~ 35%. A further increase to 4 scfm reduced the cooldown time to ~ 9 minutes, another improvement of 33%. Finally, the cooldown time for the test furnace at the highest flow rate of 6 scfm is ~5.3 minutes, comparable to the cooldown time in the HOT-006 furnace at ORNL (~4 minutes) reported in Figure 3.

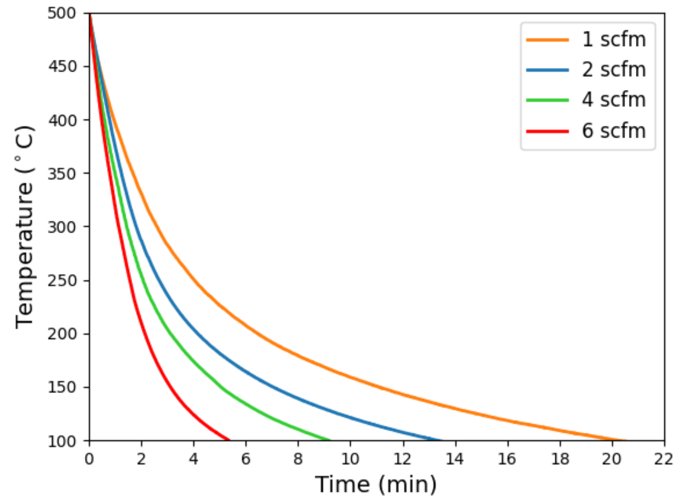


**FIGURE 4:** EFFECT OF HELIUM FLOW RATE ON COOLDOWN

The results show that the gas flow rate can control the cooldown time. This contrasts with previous findings by Goodway et al. [7], where flowrate did not significantly impact the cooldown time. The discrepancy is likely from the substantially lower flowrate range employed by Goodway, less than 0.2 scfm at most, and the flow injection that didn't directly impact onto the sample. The current closed-loop approach avoids the loss of expensive helium gas, hence there are no practical differences in the operating costs at higher flow rates. The cooldown improvement with flowrate shown in Figure 4 indicates a significant benefit to using higher flow rates. Considering the substantial cost of neutron beamline time (upwards of $30k/hour as reported by the ORNL facility), the maximum achievable system flow rate should be used to achieve the fastest cooldown possible.

Another appealing aspect of the investigated cooling system is the ability to control the cooldown rate in neutron furnaces precisely. One of the advantages of performing neutron experiments in such furnaces is the ability to examine the sample properties under precisely controlled heating rates. Various studies on high-strength steels [13,14], Co-Re base alloys [15], and Fe-Cr alloy [7], etc., are carried out under specific heating profiles. However, current neutron furnaces, which can control the *in-situ* heating rate, lack the analogous control of the *in-situ* cooldown rate. Such novel capabilities are desirable for future experiments. The presented results indicate that the cooling system has the ability to control the cooldown rate by adjusting the flow rate of helium through the furnace.

### 3.2 Impact of System Pressure

This section compares the effect of different system pressures on the cooldown time. The mass flow rate at different system pressures is maintained constant by varying the gas velocity to compensate for the pressure-dependent gas density. Representative results, shown in Figure 5, indicate that the system pressure has a negligible impact on the cooldown time under the same mass flow rate. At a flow rate of 3.5 scfm, both 8 psia and 12 psia cases have an approximate cooldown time of 10


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minutes. The same trend is seen at the lowest pressures tested, as both 5 psia and 6 psia cases have the same cooldown time of ~20 minutes at a flow rate of 1.5 scfm. We also observed similar results in other operating conditions where cooldown time remained unchanged at a constant mass flow rate despite a pressure difference.

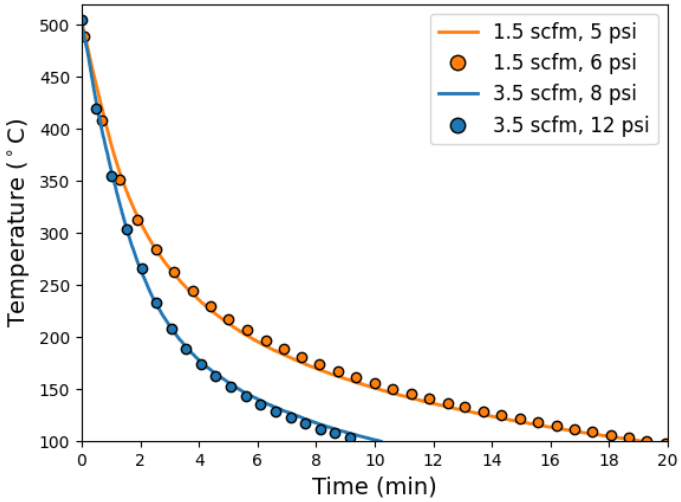


**FIGURE 5:** EFFECT OF SYSTEM PRESSURE ON COOLDOWN

The theory of forced convection in internal flow provides insight into observed results. The heat transfer coefficient and Nusselt number can be described via an empirical correlation of the form:

$$Nu = \frac{hL}{k} = CRe^{m}Pr^{n} \quad (1)$$

where *C*, *m*, and *n*, are empirical constants [16], *Re* is flow Reynolds number, *Pr* is Prandtl number, *L* is characteristic length, and *k* is gas thermal conductivity. The Reynolds number remains constant at a given mass flow rate, as the density change of helium with pressure is accompanied by an inversely proportional change in the flow velocity. Meanwhile, the Prandtl number and thermal conductivity are gas properties independent of flowrate. Figure 6 plots the Prandtl number and thermal conductivity of helium at different pressures based on the correlations by Petersen [17]. The thermal properties of helium are a weak function of pressure over the evaluated pressure range, explaining the negligible effect on the overall cooling performance shown in Figure 5. Nonetheless, this generalization only holds true at sub-atmospheric pressures. In contrast, for high-pressure systems, such as gas quenching furnaces operating at 50-200 psia, the gas properties have a stronger pressure dependence, as also shown in Figure 6. In these systems, it is well known that the pressure can significantly impact the cooling performance [18].

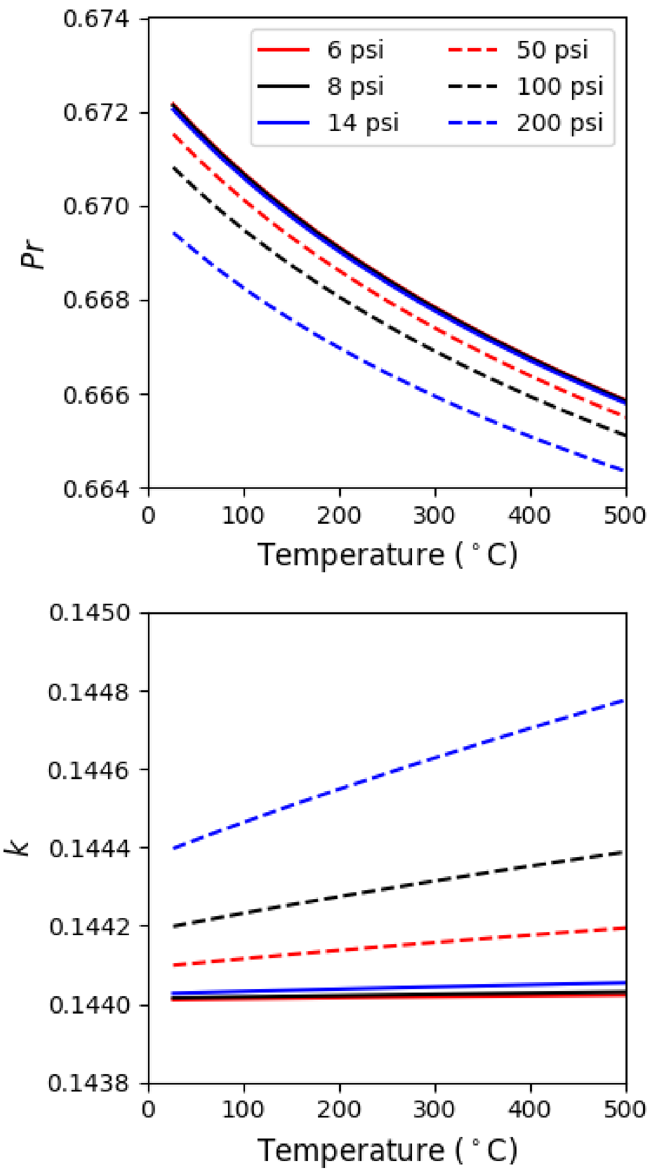


**FIGURE 6:** HELIUM PRANDTL NUMBER (TOP) AND THERMAL CONDUCTIVITY (BOTTOM).

## 4. CONCLUSION

A novel closed-loop helium circulation cooling technology for neutron vacuum furnaces was presented. The cooling system shows strong potential to improve the experimental throughput in high-temperature neutron facilities by reducing the post-experiment cooldown time. The overall furnace cooldown time for the HOT-006 furnace from 500 – 100 °C was significantly reduced from 134 minutes under vacuum to 4 minutes with 6 scfm of helium flow. The cooldown time was primarily affected by the gas flow rate, while the effect of system pressure was negligible at the range considered. Doubling the flow rate in the 1-6 scfm range led to a reduction in cooling time of 30-35%. Notably, it was found that a similar cooling performance can be achieved at the same mass flowrate over a range of pressures.


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## ACKNOWLEDGEMENTS

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