Design and performance of closed cycle sample cooling stage for angle resolved photoemission spectroscopy capable of reaching temperatures below 2 K

Benjamin Schrunk  
_Iowa State University and Ames Laboratory, bschrunk@ameslab.gov_

Lunan Huang  
_Iowa State University and Ames Laboratory_

Yun Wu  
_Iowa State University and Ames Laboratory, yunw@iastate.edu_

Daixang Mou  
_Iowa State University and Ames Laboratory_

Kyung chan Lee  
_Iowa State University and Ames Laboratory, kclee@iastate.edu_

Follow this and additional works at: [https://lib.dr.iastate.edu/ameslab_manuscripts](https://lib.dr.iastate.edu/ameslab_manuscripts)

Part of the [Materials Science and Engineering Commons](https://lib.dr.iastate.edu/materials-science-engineering-commons), and the [Physics Commons](https://lib.dr.iastate.edu/physics-commons)

**Recommended Citation**

Schrunk, Benjamin; Huang, Lunan; Wu, Yun; Mou, Daixang; Lee, Kyung chan; Jo, Na Hyun; and Kaminski, Adam, "Design and performance of closed cycle sample cooling stage for angle resolved photoemission spectroscopy capable of reaching temperatures below 2 K" (2019). _Ames Laboratory Accepted Manuscripts_. 494.  
[https://lib.dr.iastate.edu/ameslab_manuscripts/494](https://lib.dr.iastate.edu/ameslab_manuscripts/494)

This Article is brought to you for free and open access by the Ames Laboratory at Iowa State University Digital Repository. It has been accepted for inclusion in Ames Laboratory Accepted Manuscripts by an authorized administrator of Iowa State University Digital Repository. For more information, please contact digirep@iastate.edu.
Design and performance of closed cycle sample cooling stage for angle resolved photoemission spectroscopy capable of reaching temperatures below 2 K

Abstract
We have designed, constructed, and tested a unique cold finger suitable for angle resolved photoemission spectroscopy. This design is based on in situ helium reliquification and utilizes pulse tube cryocooler. The pulse tube can be removed for baking without breaking Ultra High Vacuum (UHV). This design also allows the use of non-UHV heater that can be replaced without the need to vent the system. The cold finger has minimal vibration, operates over a temperature range of 1.7 K–400 K, and has no measurable residual magnetization. In continuous mode, it can maintain a sample temperature of 2.6 K, while in single shot mode (by pumping on liquid helium), it can reach temperatures down to 1.8 K for a period of several hours.

Keywords
Angle-resolved photoemission spectroscopy, Ultra-high vacuum, Mechanical instruments, Photoelectron spectroscopy, Vacuum apparatus, Superfluids, Magnetic fields, Pulse tube refrigerator

Disciplines
Materials Science and Engineering | Physics

Authors
Benjamin Schrunk, Lunan Huang, Yun Wu, Daixang Mou, Kyung chan Lee, Na Hyun Jo, and Adam Kaminski

This article is available at Iowa State University Digital Repository: https://lib.dr.iastate.edu/ameslab_manuscripts/494
Design and performance of closed cycle sample cooling stage for angle resolved photoemission spectroscopy capable of reaching temperatures below 2 K

Benjamin Schrunk, Lunan Huang, Yun Wu, Daixang Mou, Kyung chan Lee, Na Hyun Jo, and Adam Kaminski

ARTICLES YOU MAY BE INTERESTED IN

A variable-temperature scanning tunneling microscope operated in a continuous flow cryostat
Review of Scientific Instruments 90, 093702 (2019); https://doi.org/10.1063/1.5118676

An open-source high-frequency lock-in amplifier
Review of Scientific Instruments 90, 094701 (2019); https://doi.org/10.1063/1.5083797

Optothermally pulsating microbubble-mediated micro-energy harvesting in underwater medium
Review of Scientific Instruments 90, 095004 (2019); https://doi.org/10.1063/1.5097298
Design and performance of closed cycle sample cooling stage for angle resolved photoemission spectroscopy capable of reaching temperatures below 2 K

Cite as: Rev. Sci. Instrum. 90, 093105 (2019); doi: 10.1063/1.5113722
Submitted: 6 June 2019 • Accepted: 25 August 2019 • Published Online: 13 September 2019

Benjamin Schrunk,1,2 Lunan Huang,2 Yun Wu,1,2 Daixang Mou,1,2 Kyung chan Lee,1,2 Na Hyun Jo,1,2 and Adam Kaminski1,2,a)

AFFILIATIONS
1 Division of Materials Science and Engineering, Ames Laboratory, Ames, Iowa 50011, USA
2 Department of Physics and Astronomy, Iowa State University, Ames, Iowa 50011, USA
a)kaminski@ameslab.gov

ABSTRACT
We have designed, constructed, and tested a unique cold finger suitable for angle resolved photoemission spectroscopy. This design is based on in situ helium reliquification and utilizes pulse tube cryocooler. The pulse tube can be removed for baking without breaking Ultra High Vacuum (UHV). This design also allows the use of non-UHV heater that can be replaced without the need to vent the system. The cold finger has minimal vibration, operates over a temperature range of 1.7 K–400 K, and has no measurable residual magnetization. In continuous mode, it can maintain a sample temperature of 2.6 K, while in single shot mode (by pumping on liquid helium), it can reach temperatures down to 1.8 K for a period of several hours.

Published under license by AIP Publishing. https://doi.org/10.1063/1.5113722

I. INTRODUCTION

Cryogenic cooling has been used as a tool to aid scientific research for decades,1,2 as well as being the subject of scientific research.3 Advances in cryogenic cooling have made attaining and maintaining low temperatures widely available and relatively inexpensive compared to just a few decades ago. There are several commercially available designs of cryogenic coolers used in research, each with drawbacks and benefits. For example, the Gifford-McMahon (GM) coolers can be used in any orientation with minimal cooling power losses. The GM type tends to have larger amplitude vibrations (due to use of movable displacer/regenerator) than some other designs and are thus unsuitable to applications sensitive to vibrations.6,7 By contrast, the Pulse Tube (PT) design has significant power losses in orientations other than vertical,8 but has much smaller amplitude vibrations and is therefore suited to some applications for which the GM style is not.9 Therefore, for any given application, careful consideration must be taken to ensure the correct style is used and that any weaknesses of the design are mitigated as much as possible.

Angle Resolved Photoemission Spectroscopy (ARPES) experiments present a unique set of requirements with respect to sample cooling. Those include low residual magnetic field (especially important in ultrahigh resolution laser based spectrometers), low vibrations, and relatively high cooling power due to the need for a wide angle sample access. Typically, liquid helium based ARPES cold fingers have rather high helium consumption, which cost preventive except for large scale user facilities. Closed cycle refrigerators were utilized in ARPES measurements for decades, but they typically could reach sample temperatures down to ~12 K. Newer generation of closed cycle refrigerators use low melting point alloy in heat exchangers and those cannot be heated up above 80 °C. This poses a serious problem, as typically ARPES system requires baking at 120 °C for an extended period of time.
In order to address these challenges, we designed, constructed, and tested a cold finger that meets all of these requirements. The design is based on the principle of in situ helium reliquification. The use of helium as an exchange gas is a well-established method for thermal contact, especially useful when vibrations must be minimized for the measurement technique to succeed.\textsuperscript{2}\textsuperscript{10}–\textsuperscript{13} By accumulating a sufficient amount of liquid helium at the bottom of the cold finger and pumping using a combination of molecular and roughing pumps, this design can achieve sample temperatures down to 1.8 K for several hours. Using helium exchange gas/liquid for heat transfer allows us to mechanically separate the PT from the Ultra High Vacuum (UHV) volume and its disassembly for baking of the ARPES spectrometer without breaking the UHV. Additional benefit is ability to install the sample heater outside of the UHV. Such simplified design eliminates the need for UHV compatible heaters and substantially limits the outgassing during warm up. The cold finger described above is currently operational in our lab based tunable laser ARPES system.\textsuperscript{5}

II. DESIGN

The key design goals were based on the minimizing residual magnetic field, minimizing vibrations, and allowing removal of the PT without breaking UHV. The schematic drawing of the design is shown in Fig. 1. We used Cryomech PT415 pulse tube with CPA1110 compressor for helium reliquification. The PT415 has base temperature of second stage of 2.8 K and cooling capacity of 1.5 W at 4.2 K. The upper portion of the cold finger body is made out of stainless steel (SS316) and welded to inner part of 10 in. Conflat flange (CFF). The top side of the flange is machined to accommodate mounting the PT415 head such that the cooling stages are inside the cold finger/gas/liquid exchange volume. The first stage of PT contacts the bottom of the larger diameter tube via the spring loaded ring that is connected with copper braids to the cold finger at the place, where main thermal shielding is mounted on the UHV side. Springs and braids ensure good thermal contact between the first stage of PT and thermal shield. This is important when pumping on liquid helium to lower the temperature below 2 K. In such a case, the He pressure at the first stage is too low to effectively cool the thermal shield. The second stage of PT is located inside of the smaller diameter SS tube without any mechanical contact. This tube has a perimeter welded SS316 flange (Kimball Physics) at the bottom. A long, thinner titanium tube is attached at that point using CFF flange machined out of titanium and thicker, copper gasket machined out of hard tempered OFHC copper. We used titanium because it is nonmagnetic and has very low thermal conductivity. This poses bit of a challenge as, unlike copper, it cannot be welded to stainless. We had to resort to using CFF flanges and copper gaskets to make UHV grade connections between titanium and stainless. At the bottom of titanium tube, there is a set of titanium flanges that secure the tip of the cold finger made out of OFHC copper to which sample is attached. The upper portion of the tip is machined to act as a gasket, creating UHV seal between the copper tip and titanium CFF flange welded to titanium tube. All titanium hardware (screws, nuts etc.) are used to minimize residual magnetic field. The copper tip is partially hollow to allow better heat exchange with liquid helium. The sample is located only a few inches from the liquid and connected by 1.5 in. diameter copper rod. On the exchange gas side of the copper tip, we mounted small copper block with attached heater (Lakeshore 50 Ω, 25 W). This allows us to rapidly heat the sample stage without risk of contamination of the UHV from the heater. It is also possible to replace the heater without the need to vent the UHV part of the system.

III. OPERATION

The cold finger can be operated in several modes. In “high temperature mode,” all helium gas is pumped out and PT can be switched off. By using the heater the sample temperature can be increased to the limit of materials used. Since we use indium for sample thermal contact and sensor soldering, this limit is 400 K. This temperature can be substantially increased by avoiding the use of indium. In the “intermediate temperature mode,” we fill the exchange volume with small amount of helium gas. This facilitates cooling of the sample to ~10 K but allows fast temperature changes. In the “regular mode,” we fill the exchange volume with helium so that most of it is liquified at the bottom of the copper tip. Any evaporated gas is reliquified by the second state of PT and cooled to 2.6 K. The typical sample temperature is 3 K. This is a continuous mode of operation and sample can be maintained at this temperature as long as the compressor is powered without helium consumption. In the “low temperature mode,” we would add more helium, and so most of the titanium tube is filled with liquid. We would then
use combination of the molecular pump and roughing pump to
decrease pressure above the liquid and cool it down. This allows us
to reach sample temperature of \(\sim 1.7\) K for extended period of time
( several hours). When all liquid helium evaporates, we can repeat
the liquefaction process, which takes about an hour and cool the
sample back to \(\sim 1.7\) K. The use of copper braids connecting the
1st stage of the PT to shielding is essential, as the gas heat transfer
while pumping is not sufficient to maintain the temperature of the
shielding.

IV. PERFORMANCE

Even though PT heads have significantly lower vibration than
GM coolers, we opted for version with remote motor to further mini-
imize the vibration. With PT head on the measured level of vibra-
tion is roughly \(7\) \(\mu\)m as measured by external microscope and CCD
camera. The vibrations due to PT can be eliminated completely for
extended periods of time by liqquifying sufficient amount of helium
in situ and turning the PT off. This allows it to maintain tempera-
tures below \(4\) K for several hours that is typically sufficient for low
temperature ARPES measurements. With PT off, the vibrations in
the system are below \(2\) \(\mu\)m and due to external causes (floor, build-
ing, etc). The temperatures of various portions of the setup in nor-
mal mode of operation are as follows: 1st stage of PT and shielding
\(~52\) K, 2nd stage of PT is at \(2.6\) K, and sample is at \(2.8–3\) K. The
time plot of sample temperature during introduction of helium gas
after previous pumping is shown in Fig. 2. Figure 2(a) shows the full
temperature curve and the labels above the close-up boxes indicate
the total amount of helium gas introduced to the cold finger. After
pumping out all of previously introduced helium gas, the tempera-
ture rises quickly to \(60\) K within first 10 min. Adding the first liter of
He gas results in cooling of the sample to \(\sim 10\) K [Figs. 2(b) and
2(c)]. After adding about \(40\) l, enough liquid forms at the bottom of
copper tip to maintain the temperature of the sample at \(4.1\) K
[Figs. 2(d)–2(f)]. Figure 2(g) shows the temperature stabilization
after a total of \(40\) l was added. Figure 3 shows the temperature of
the sample, after the liquefaction of helium and subsequent pump-
ing with combination of roughing and molecular pumps (Agilent
IDP-7 and Adixen ADP-5011). The sample temperature of \(1.8\) K
can be maintained for period of \(\sim 3\) h. A calibrated chip version of
cernox sensor (Lakeshore CX-1030-BC-HT) is mounted at the bot-
tom of the cold finger for measuring the temperature. We found
out that the encased version of cernox (e.g., CX-1030-SD) produces
sufficient magnetic field at low temperature to affect photoelectrons
paths at low photon energies (\(\sim 7\) eV). Our sample holder has electrical
connections for 4-point contact measurement of resistivity, and
thus, we can mount another calibrated cernox sensor and measure
the temperature at exact sample location and verify that it agrees
with sensor mounted on the cold finger. We also independently ver-
ifified the temperature at the sample position by mounting a thin
indium wire at the sample position and attaching it to 4-point con-
tact connector. The indium sample was mounted to mimic the con-
ditions of ARPES measurement (e.g., thermal contact, amount of
thermal radiation, etc.). The measured temperature dependence of
resistance of indium is shown in Fig. 4. The sharp decrease of resis-
tance is observed below \(3.4\) K is due to transition to the supercon-
ducting state. Such a measurement of \(T_c\) (superconducting critical
temperature) provides assurance of accuracy of sample temperature
measurement.

**FIG. 2.** (a) A cooldown temperature curve starting from evacuated exchange volume and several stages of adding helium gas. (b) Zoom-in on curve during adding first liter of helium gas with temperature stabilizing at \(11\) K. [(c) and (d)] Second liter added resulting in temperature about \(8.5\) K. (e) Another liter for a total of three liters and \(7.3\) K. (f) Fourth liter added resulting in temperature of \(6.6\) K. (g) Finally, with \(40\) liter in the cold finger the temperature reaches \(4.1\) K and liquid helium is accumulated at the bottom of cold finger.
FIG. 3. (a) Temperature curve showing the effects of pumping using combination of roughing and molecular pumps. The low temperature achieved is about almost 1.8 K with for a duration of around 3 h. (b) Heating curve showing increase of the temperature from about 5 K to 100 K by using 25 W heater with 30 l of He gas as exchange medium. (The heating time depends on the amount of He gas in the system.)

FIG. 4. Temperature dependence of resistance of a thin indium wire demonstrating superconducting transition below 3.4 K. Small offset of resistance is due to calibration of nanovoltmeter.

V. CONCLUSION

In conclusion, we have designed, built, and tested a cold finger uniquely suited for ARPES measurements. It offers undetectable residual magnetic field, low vibrations, compatibility with UHV, and ability to remove the PT head for bakeout as well as heater mounted externally to UHV and temperature range between 1.8 K and 400 K. The cold finger is now in routine operation in our vacuum ultraviolet (VUV) tunable laser spectrometer.

ACKNOWLEDGMENTS

Research was supported by the US Department of Energy, Office of Basic Energy Sciences, Division of Materials Science and Engineering. The Ames Laboratory is operated for the US Department of Energy by the Iowa State University under Contract No. DE-AC02-07CH11358.
REFERENCES