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RPPR Final Report
as of 09-Aug-2021

Agency Code: 21XD

Proposal Number: 75675MSRIP **Agreement Number: W911NF-20-1-0066**
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Final Report for Period Beginning 15-Jun-2020 and Ending 14-Jun-2021

Title: Acquisition of Helium Reliquefiers for Investigation of Novel Two-Dimensional Magnetoelectric Devices
Begin Performance Period: 15-Jun-2020 **End Performance Period:** 14-Jun-2021
Report Term: 0-Other

Submitted By: Venkat Chandrasekhar Email: v-chandrasekhar@northwestern.edu
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Distribution Statement: 1-Approved for public release; distribution is unlimited.

STEM Degrees: **STEM Participants:**

Major Goals: This proposal was for the acquisition of two local helium reliquefiers that can be attached to our existing low temperature cryostats to enhance and extend our capability to perform low temperature experiments on a variety of materials and devices. The acquisition of this instrumentation immediately impacts our ARO funded work on magneto-electric devices, but will also impact other ongoing projects on complex oxides, superconducting hybrids, quantum information science and quantum materials. The expected lifetime of the equipment is 10 years, but it is expected that the equipment will pay for itself in terms of reduced helium costs in less than two years.

Accomplishments: Two helium reliquefiers were purchased and installed at Northwestern University. Due to delays at the manufacturer and some issues with the design, the instruments were delivered in late March 2021 and only became operational in early June 2021, at the end of the project period. Nevertheless, we have started to use both reliquefiers, and the benefits in running low temperature experiments in terms of considerably reduced helium consumption as well as continuous operation are already evident.

Due to the delayed delivery of the instruments, no publications have so far resulted from the grant, but we expect the instruments to impact almost all projects requiring access to liquid helium temperatures and below.

Training Opportunities: Three graduate students: Patrick Krantz, Kevin Ryan and Max Wisne were closely involved in the design and installation of the instruments, and are now experts in their operation. As such instruments are likely to get more common in the future due to shortages in helium, this experience is invaluable for their future careers.

Results Dissemination: Nothing to Report

Honors and Awards: Nothing to Report

Protocol Activity Status:

Technology Transfer: Nothing to Report

PARTICIPANTS:

Participant Type: PD/PI

RPPR Final Report
as of 09-Aug-2021

Participant: Venkat Chandrasekhar
Person Months Worked: 1.00 **Funding Support:**
Project Contribution:
National Academy Member: N

Participant Type: Graduate Student (research assistant)
Participant: Patrick Krantz
Person Months Worked: 1.00 **Funding Support:**
Project Contribution:
National Academy Member: N

Participant Type: Graduate Student (research assistant)
Participant: Kevin Ryan
Person Months Worked: 1.00 **Funding Support:**
Project Contribution:
National Academy Member: N

Participant Type: Graduate Student (research assistant)
Participant: Maxwell Wisne
Person Months Worked: 1.00 **Funding Support:**
Project Contribution:
National Academy Member: N

Partners

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I certify that the information in the report is complete and accurate:
Signature: Venkat Chandrasekhar
Signature Date: 8/9/21 10:00AM

Final report on "Acquisition of Helium Reliquefiers for Investigation of Novel Two-Dimensional Magnetoelectric Devices."

Award Amount: $152,620
Project Period: June 15, 2020 – June 14, 2021

This proposal requested funding to obtain two helium reliquefiers in support of our DoD funded research into the magnetoelectric properties of two-dimensional (2D) van der Waals magnetoelectric devices. In addition, the availability of this instrumentation was expected to impact several other federally funded research projects requiring measurements at liquid helium temperatures and below.

Northwestern University has a helium recovery system, but even with 100% recovery, the direct cost of liquid helium is $6.50 a liter; more typically it is $9-10/liter with ~90% recovery. With each cryostat utilizing approximately 20 liters a day, and successful experiments running continuously for 1 month or more, expenses associated with liquid helium quickly add up. Consequently, the goal of this grant was to obtain local reliquefiers, instruments that take the liquid helium gas boil-off from the cryostat dewar, reliquefy it in a cold head immediately adjacent to dewar, and return it to the dewar. In an ideal system, there would be no loss while the experiment is running, and the only cost for liquid helium would be for the initial cool-down, most of which could be recovered by venting the dewar to the University's recovery system at the end of the experiment. In addition, the continuous reliquefication of the gas boil-off means that one does not have to transfer helium every 2-3 days (depending on the particular cryostat), an operation that usually disrupts the ability to perform measurements for 3-4 hours, in addition to contributing to unrecoverable helium loss.

The only company that manufactures such an instrument is CryoMech. They have various models that differ in the amount of helium that they can reliquefy per day. We chose the CryoMech PT-415. This instrument can liquefy 15 liters of helium per day from room temperature gas, and up to 24 liters per day from cold gas. Given the typical helium consumption of our cryostats of about 20 liters of liquid helium per day, this was the model best suited to our requirements.

The CryoMech reliquefiers are based on a pulse tube mechanism that generates significant vibrations that couple to the cryostats and can adversely affect sensitive measurements, particularly at millikelvin temperatures. Consequently, we also opted for the remote motor option that places the cold head motor 2 ft from the cold head itself, significantly reducing the vibrations coupled to the cryostat.

In addition to the cost of the two reliquefiers themselves, the project also covered the costs of installation at Northwestern, which included items such as tubing to connect the house chilled water supply to the instrument compressors and support structures for the cold head and remote motors. The total costs are outlined below:

1. Two CryoMech PT-415 helium reliquefiers, including remote motor stands, vertical supports and shipping:
   $146,606
2. Costs for installation, including Unistrut supports, tubing for water cooling of the compressors, and electrical connections to power. All installation was performed by graduate students in my group, except connection to electrical power, which needed to be performed by licensed electricians per code.
   $3,014

Total Cost $152,620

Installation and Operation

The standard reliquefiers that CryoMech sells are meant to liquefy helium gas stored at room temperature (typically in recovery balloons) into standard transfer tanks, from which the liquid helium can then be transferred to a cryostat. Instead of this arrangement, we desired to have the reliquefier attached directly to the cryostat. This improves the liquefaction rate and has the added benefit of eliminating the need to periodically transfer liquid helium into the cryostat. The two reliquefiers purchased were targeted towards two specific cryostats in my group: a Kelvinox MX 100 dilution refrigerator, and a dewar with a two-axis superconducting magnet that can be used with several different cryogenic inserts. Images of the reliquefiers installed on these two systems are shown below.

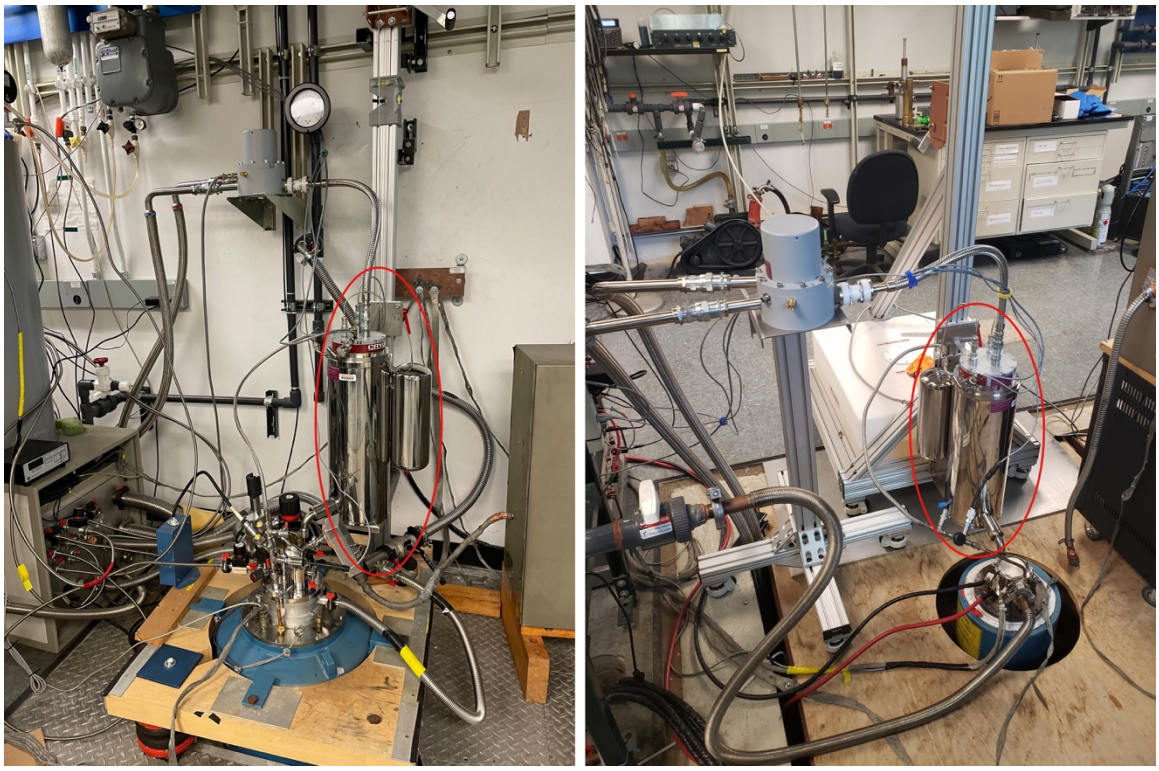

Figure 1. Left panel: Kelvinox MX100 dilution refrigerator with reliquefier cold head (circled in red) supported on the wall. Right panel: Two-axis magnet dewar with reliquefier cold head supported by mobile stand. The compressors in both installations are one floor below.

The purchase order for the machines was received by CryoMech on June 30, 2020, shortly after the grant started, and delivery was expected to be in 6 months, roughly by the end of 2020. However, the custom design and manufacturing took much longer than expected, and the units were shipped only at the end of March 2021. Unfortunately, the custom designs by CryoMech were not tested before shipping, and apparently the compressor units were also not thoroughly tested, so that we encountered many problems in installing the machines. For example, the mobile cart shown in the right panel in the Fig. 1 is part of CryoMech's stock setup for reliquefying into a standard transfer dewar roughly 4 ft in height. This stock design was modified by CryoMech to transfer into a dewar at floor height, as shown in the figure. This required modifying the winch that raises and lowers the cold head so that it could be lowered to the floor. However, this mechanism did not work, and it had to be modified by us to enable it to function. Similarly, the winch mechanism for the system shown in the left panel also did not work as designed and needed to be modified. Finally, the compressor for the system shown in the right panel had a leak as delivered and had to be shipped back to CryoMech for repair. It is anticipated that it should be repaired and returned in early September.

Current use

With the problems mentioned above, we did not have the systems installed and running until the first week of June 2021. Nevertheless, the substantial savings in liquid helium consumption are already evident. For the two-axis magnet dewar (shown in the right panel in Fig. 1), we usually had to transfer ~60 liters of liquid helium every 3 days. With the reliquefier, apart from the initial transfer, there was essentially no loss of the liquid helium level while the reliquefier was running. For the dilution refrigerator (left panel in the figure), which is currently cold, we normally have to transfer liquid helium every 2.5 days. With the reliquefier, the measured loss of the liquid helium is about 3% a day, so that we should not have to transfer for approximately 1 month. The loss rate in the dilution refrigerator is due to the liquid helium required to cool the 1K pot, which is not returned to the reliquefier, but vented to the central recovery system. We are considering the feasibility of returning the helium gas output of the 1K pot pump to the reliquefier that would eliminate even this small loss. The problem is to ensure that oil and potential air leaks in the 1K pot do not contaminate the return gas flow.

Since we have just started using one of the reliquefiers on a regular basis (while awaiting repairs on the second system), no publications have yet resulted from this grant, but we anticipate that this grant will significantly impact the low temperature measurements of the magnetoelectric devices that we will shortly be measuring as part of our DoD funded research, as well as other experiments requiring access to low temperatures. Three graduate students: Patrick Krantz, Kevin Ryan and Max Wisne, have been intimately involved in installing the reliquefiers, and all are now well trained in their operation.