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DEVELOPMENT OF AN ULTRA-LOW VIBRATION CRYOSTAT BASED ON A CLOSED-CYCLE CRYOCOOLER

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Abstract

Low temperature and low vibration cryostats are useful in a variety of applications such as X-ray diffraction, quantum computing, X-ray monochromators and cryo-TEMs. In this project, we explore an ultra-low vibration cryostat with the cooling provided by a closed cycle cryocooler. Closed-cycle cryocoolers inevitably introduce vibrations into the system, and in this project, flexible copper braiding was used to decouple vibrations and provide cooling at the same time. In order to develop the cryostat, capacity map of a two stage Sumitomo cryocooler was measured as well as vibration transmission through different copper braids using an IR interferometer. This paper covers the capacity map and vibration measurements in the first prototype.

INTRODUCTION

Low temperature sample environment is required for the next generation X-ray sources with a sub-micron beam size. The success of the full potential employment of small coherent X-ray beams will highly depend on the position control of the sample relative to the beam and the ability of sample stabilization. The cryostats currently in use are based on gas-gap barrier principle [1] and are heavy, cumbersome and can only be used at facilities where diffractometers with a through-hole on a phi stage or with an offset chi circle are installed. Many X-ray diffraction facilities utilizing high brightness synchrotron beamlines employ modern diffractometers for specifically designed sample stages which cannot fit cryostats currently available on the market [2, 3].

The project was devoted to the cryostat engineering design development and vibration measurements of a mock-up first generation cryostat. It is necessary to mechanically decouple the cryostat from different sources such as vacuum pumping system, cryocooler, the cryostat mounting table and sometimes from acoustic vibrations. The first generation of the cryostat was manufactured to make vibration measurement with Michelson interferometer. The cryostat was cooled by a closed cycle Gifford-McMahon (GM) cryocooler [4]. Vibration mitigation studies were conducted. The experimental results were used for the development of a full-featured cryostat in Phase I of the project.

SETUP

The capacity map of the cryocooler used for the vibration studies was measured in order to calibrate our system and to precisely predict the heat going to the stages by knowing their temperatures. Two cryogenic resistors were used as heaters.

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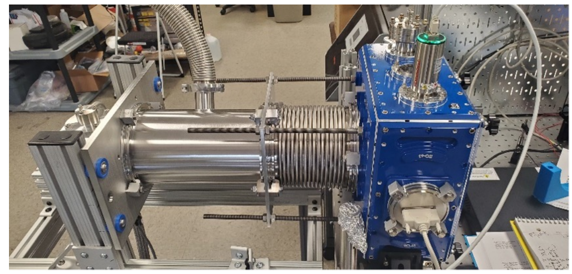

Figure 1: Vacuum envelope with the cryocooler and pumping port on the left and the (blue) instrumentation volume with feed-throughs on the right.

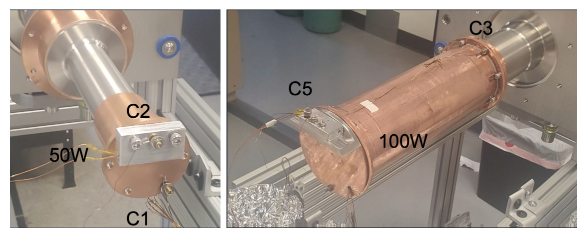

Figure 2: Temperature sensors: left – on the second stage cold head (C1) and heater block (C2); right – on the first stage (C3), at the end of the Cu radiation screen (C4, not seen) and the first stage radiation screen heater block (C5).

One can find out the dissipated heat by knowing their resistance and the current flowing through them. A Sumitomo RDE-418D4 Gifford-McMahon (GM) cryocooler [4] was coupled through a ISO-160 nipple and bellows to a small rectangular vacuum chamber ("vacuum box") with instrumentation feed-throughs as shown in Fig. 1.

The cryocooler consists of 2 stages, the first stage which brings the temperature down to about 30 K, and the second stage which can reach < 4 K. A copper radiation screen was installed around the second stage of the cryocooler and connected to the first stage of the cryocooler to intercept thermal radiation from the room temperature vacuum chamber.

The cooler was instrumented with the following for thermal measurements:

- two heaters built using resistors embedded inside 6061-aluminum blocks – 100/50 W units attached to the radiation screen and the second stage cold head;
- temperature sensors (Cernox) at five locations – on the first and second stage cold heads and the three heater blocks.

These are shown in Fig. 2.

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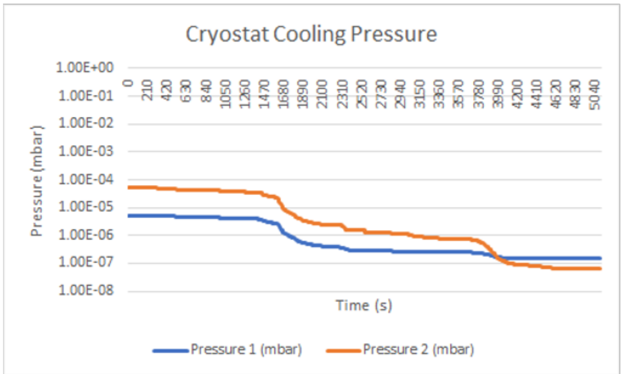

Figure 3: Pressure at the pump end (sensor 1) and the vacuum box (sensor 2).

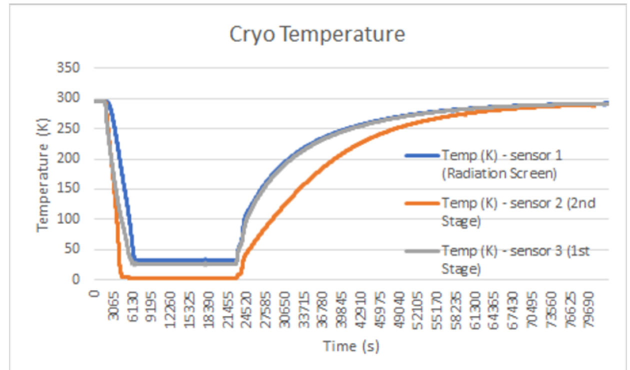

Figure 4: Temperature recorded during system cooldown and warm up with vacuum pump-off at 18000sec mark.

VACUUM STUDIES

Since we were concerned about vibrations coming from the vacuum pump, we wanted to know how high the vacuum pressure could be without causing any cooling performance degradation. To answer this question, we planned to cool down the cryostat pumped by a vacuum station containing a dry scroll pump and a turbo pump. Then, the turbo pump would be turned off, followed by the scroll pump, while monitoring the temperature readings from the sensors. Vacuum pressure curves during cooldown are shown in Fig. 3. Pressure was monitored by a sensor near the turbo pump (sensor 1) and another one on the vacuum box (sensor 2). The first step decrease is due to the cryopumping when the cryocooler is turned but before the turbo pump has reached its nominal speed. Sensor 2, which measured the vacuum in the system, reached lower pressure (few 10^{-8} Torr) than sensor 1 at the vacuum pump, as the cryoocoler became more efficient.

Figure 4 shows temperatures during a cooldown/warm-up test. All the vacuum studies were performed in the cooled-down state, i.e. from 3000 s to 24,000 s. There is a small bump at 18,390 s when the scroll pump was turned off, but the temperatures returned back to the previous values shortly after that event. That was caused by the local pressure burst for a short period of time, but it did quickly recover as presented in Fig. 5 which shows pressure curves after the turbo pump was turned off, followed by the scroll pump.

The pressure level was gradually increasing after the turbo pump was turned off as it took some time for the turbo to completely stop. The pressure reached 10^{-4} and 10^{-6} Torr for gauges 1 and 2, respectively. The scroll pump turn-off caused a pressure burst in the cryostat (see pressure 2), but quickly came back and even went down while gauge 1 either lost connection or malfunctioned for some time: 10^{-8} Torr level. This burst is reflected in a slight increase in the temperature readings. The pressure in the system started to slowly increase and stabilized at 10^{-3} and high 10^{-6} Torr for gauges 1 and 2 respectively, meaning that the cooled cryocooler can maintain a good enough vacuum level by itself and will not introduce vibrations to the system.

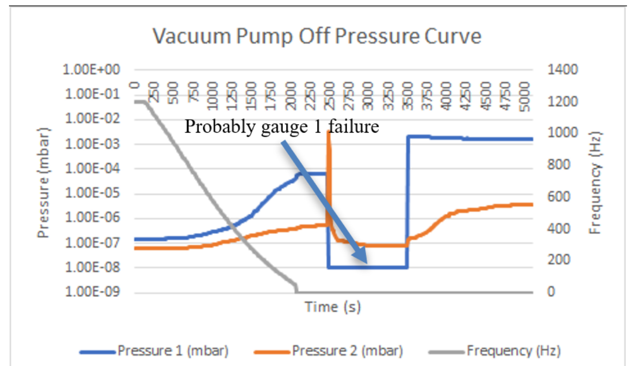

Figure 5: Pressure at the pump end (sensor 1) and the vacuum box (sensor 2).

CAPACITY MAP MEASUREMENTS

The sequence for these measurements was

- pump the system down to about 10^{-3} Torr;
- turn on the cooler and let the system reach <4 K with all the heaters off;
- with no heat input to the first stage, set the second stage heater to: 0, 1.8, 4.4, 8.8, 13.1, 17.4, 21.4, and 31.9 W;
- at each power setting, record the temperature sensor readings once the system is in steady state;
- repeat with the first stage heater set to: 7.5, 10, 20, 40, 60, and 80 Watts.

The results are shown in Fig. 6 and agree with the manufacturer's curves. The capacity of the second stage at 4.2 K is 2 W for a wide range of temperatures of the first stage.

Next, we estimated the measurement uncertainties and investigated the influence of power dissipation in the wires, thermal conductance through the wires, and thermal radiation. The wires that carry electrical current to the heater blocks and temperature sensors also contribute to the heat load, through resistive power dissipation and thermal conduction. At the highest heater power used (80 W to the first stage and 30W to the second stage), we estimate the resistive dissipation contribution in the wires to the first/second stage heat loads to be around 0.19/0.24 W. The corresponding contribution from all five temperature sensors is of order 0.2 mW. Thermal conduction along the heater wires from

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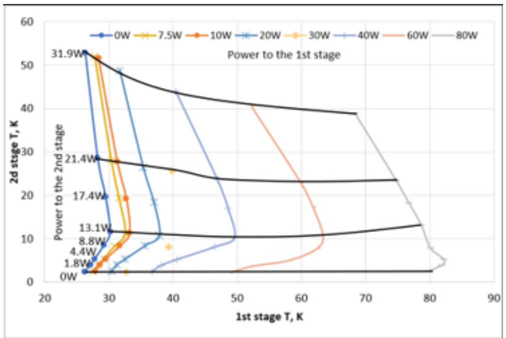

Figure 6: Cryocooler first and second stage temperatures at different input power levels to the stages.

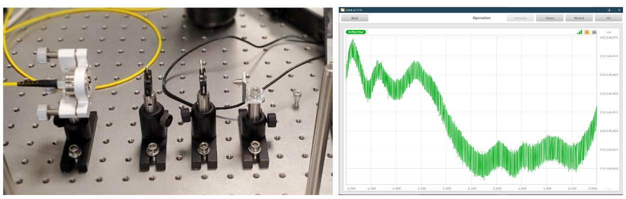
Figure 7: Vibration measurement setup in air (left) and a 5-s sample (right).

room temperature to 30/4 K on the first/second stage is about 60/20 mW and the total amount for the temperature sensors is less than 2.7 mW. Thus, these corrections are negligible. We estimate the radiative heat load on the second stage of the cooler from the radiation screen, which was at around 120 K with 80 W going into the first stage, to be around 0.1 W, also negligible. The radiative load on the screen due to the outer vacuum envelope is of order 5 W, a small correction.

VIBRATION MEASUREMENTS

A simple interferometer was assembled on an optical table to study vibrations and tested in air at room temperature as shown in Fig. 7, from left to right: interferometer head, two irises for alignment, retroreflector (mirror) at the end. The observed short-term (10's of ms) peak-to-peak amplitude was about 5 nm, with a long-term (seconds) drift of order 30 nm (see Fig. 7 on the rigth). The frequency spectrum had the largest component at 60 Hz as expected and a smaller one at about 280 Hz.

When the interferometer was installed in the vacuum cube (Fig. 8 on the left), the drift was eliminated. Air causes drift due to temperature and pressure variations. Vibrations of the vacuum cube placed on the optical table were recorded. Vibrations could propagate to the cube through the vacuum chamber connected to the vacuum pump and also through the cryocooler which was supported by an extrusion frame. The amplitude inside the cube was 20 nm with pump and cryocooler both off (Fig. 8 on the right), increased to 40 nm with the vacuum pump turned on, and up to 200 nm with the cryocooler on. After that, the retroreflector was moved to the radiation screen to record vibrations of the cryocooler relative to the isolated cube. In order to rule out the influence of the bellows, it was disconnected and measurements were

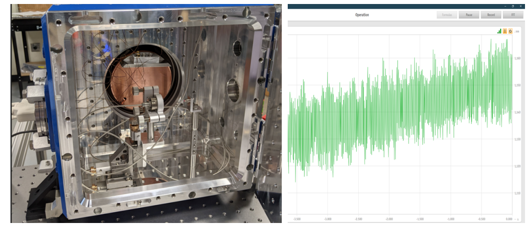
Figure 8: Vibration measurement setup in vacuum (left) and a 3.5 s sample (right).

conducted in air. Cryocooler vibrations were recorded and was 7 um peak-to-peak, which means that the ISO-160 edge-welded bellows provided an order of magnitude vibration decoupling.

CONCLUSION

We have successfully commissioned a test system built around a closed-cycle cryocooler and instrumented for capacity map and vibration measurements. This is an important step toward developing conduction cooled cryostats for applications with low vibration requirements. It was found that the cryocooler can maintain insulating vacuum by itself and no additional vacuum pump is needed which will help to reduce vibrations to the system. The cryocooler capacity map was measured in a wide load range and it was confirmed that the second stage capacity at 4.2 K is 2 W. The vacuum pump introduced 40 nm vibration amplitude through the 3 ft-long KF-40 hydro-formed bellows. The amplitude transmitted through the ISO-160 edge welded bellows due to the operating cryocooler was 200 nm. The future steps will include further design development of the system to integrate a 4 K sample table in the instrumentation box cooled by the cryocooler followed by vibration mitigation studies using IR interferometer.

ACKNOWLEDGMENTS

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REFERENCES

[1] J. Hackley *et al.*, "High-stability cryogenic scanning tunneling microscope based on a closed-cycle cryostat," *Rev. Sci. Instrum.* vol. 85, p. 103704, 2014. doi:10.1063/1.4897139

[2] https://www.nist.gov/laboratories/tools-instruments/synchrotron-x-ray-diffraction-xrd

[3] M. R. Probert *et al.*, "The XIPHOS diffraction facility for extreme sample conditions," *J. Appl. Cryst.* vol. 43, pp. 1415–1418, 2010. doi:10.1107/S0021889810041282

[4] https://www.shicryogenics.com/product/rde-418d4-4k-cryocooler-series