Helium exchange gas based variable temperature insert for cryogen-free magnet system

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Abstract: A cryocooler based variable temperature inserts (VTI) has been designed and developed for measurement of physical properties at low temperature and high magnetic field. The VTI, designed using the helium exchange gas principle, needs to be integrated in the warm bore of an existing 6 T cryogen free magnet system. The lowest temperature achieved at the sample is 5 K at 34.5 kPa (~5 psi) gaseous helium environment in the sample space. The equilibrium temperature of the sample, at the vacuum condition, is 8.7 K. The cool-down time of the sample at vacuum environment is 9 hrs whereas it takes 7 hrs in presence of helium exchange gas. The temperature of the sample was varied up to 325 K. The stability of the temperature achieved is less than 50 mK. The cooling and heating curves has been studied to estimate time required for a complete cycle of experiment. This paper will briefly present the design and performance of VTI system in temperature range of 5-325 K.

1. Introduction

The cryogen-free systems, in which the liquid cryogens utilization is completely eliminated by using direct thermal linking of cold head with the sample is called as ‘dry’ system [1] and system in which evaporating helium is liquefied or cooled by a cryocooler (CCR) and supplied back to a sample is called as re-condensing systems [2]. These systems are replacing the conventional bath cooled systems because helium supply problems and safety issues [3]. Cryocooler based variable temperature insert (VTI) system makes system compact and efficient with lesser power consumption [4-6].

The low temperature has now become basic requirement to study the physical properties of material especially for magnetic materials, due to the freezing of motion of atoms. [7]. The high magnetic field at lower temperature gives us more scope to study low temperature properties as well as magnetic properties of materials. Nowadays, integrated VTI system and high field superconducting magnet systems are commercially available for studies of physical properties at low temperature and high magnetic field [8].

A VTI system has been designed and developed for the temperature range of 5 K to 325 K for measurement of physical properties at low temperature and high magnetic field (6 T). The cryocooler based VTI system has been designed using the helium exchange gas principle. The VTI system needs to be integrated in the warm bore of an existing 6 T cryogen free magnet system (CFMS). The existing cryogen-free superconducting magnet having warm bore, hence the variation of temperature was not possible for the sample inside the working bore. The deep (0.5 m) and narrow warm (50 mm) bore of
existing CFMS [9] poses a complexity in the designing of VTI to achieve the desired temperature at the sample space, which is deep inside warm bore. A double stage GM cryocooler (model – SRDK 408 of SHI Cryogenics, Japan) has been used for the VTI system. For any low temperature physics experiment, it is more crucial to achieve the precise control of sample temperature in the entire range and its stability at any particular temperature. End user will need to raise and lower the sample temperature during any experimental studies. Hence, both cooling and heating curves has been studied to estimate time required for a complete cycle of experiment. Integration of the VTI system with the CFMS would make the system Variable field and variable temperature (VFVT). This paper will briefly present the design and performance of the VTI system.

2. Design of VTI

The schematic of the VTI system is shown in the Figure 1. The system consists of GM cryocooler (a) is fixed on the vacuum jacket (b) so as to be thermally linked with the thermal shield (c) made of electrolytic-tough-pitch (ETP) copper. The bottom part of thermal shield is inserted into the warm bore of the CFMS. The top-loading sample probe (d) is inserted into the sample bore of 25 mm size, whose warmer section (e1) is made of stainless steel (SS-304) and the colder section (e2) is made of copper (Cu). The warmer section of sample bore is thermally anchored to 1st stage of the CCR to reduce conduction heat load from the ambient. The colder copper section of the sample bore is thermally anchored at 120 mm below the ConFlat (CF) flange coupling (f) by copper braids as a thermal link (g) to the 2nd stage of the CCR.

Figure 1. Schematic of the VTI system.
The schematic of the sample probe is shown in the Figure 2. The sample probe having sample holder at bottom of 1.27 m stainless steel (SS-304) sample tube and an instrumentation port is attached at the top of SS probe tube. A set of radiation baffles were silver brazed on the sample probe tube to reduce the thermal radiation load from the top. The sample probe can be positioned at the center of the magnetic field. A Cernox temperature sensor has been fixed on the sample probe to measure the perspective sample temperature even at high magnetic field. A 25 Ω non-inductive resistive heater of 100 W capacity has also been fixed on the sample probe to vary the sample temperature. The sample bore space is connected with the helium gas buffer vessel. The helium buffer vessel will provide necessary exchange gas to the sample space to make thermal linking to the sample holder. The sample bore is also connected with a small vacuum pump, which can be used to evacuate, if necessary.
The VTI needs to be integrated in the warm bore of the existing 6 T cryogen-free magnet system to make it a VFVT system, as shown in Figure 3. To characterize the thermal behavior of the VTI system, five calibrated silicone diode (DT470, Lakeshore Cryotronics) temperature sensors (T1-T5) are placed at different location along with a Cernox sensor (T6) for the sample. The location of the temperature sensors are given in the following:

- T1: Thermal shield of the VTI chamber
- T2: 2nd stage of the GM Cryocooler
- T3: Sample bore on SS tube
- T4: Sample bore on copper tube
- T5: Bottom of sample bore
- T6: Sample

3. Performance of the VTI system
The cooldown characteristics of the system have been experimentally studied for different environment in the sample space such as vacuum, atmospheric pressure, 34.5 kPa and 69 kPa helium.

3.1. Cool Down of the VTI System:
Initially, the sample space is evacuated to $10^{-2}$ mbar vacuum level. After evacuation, the sample space is purged with helium gas. The VTI chamber has also been evacuated to $10^{-4}$ mbar. The CCR has been started for cooling down the VTI system. Figure 4 shows the cool down profile of the VTI system at 34.5 kPa gaseous helium (GHe) environment. The thermal shield of the VTI chamber reaches to the steady state temperature of 34 K in 7 hrs. Whereas the 2nd stage of the CCR (T2) takes 6 hours to reach the steady state temperature of 3.4 K. The heat loads on 1st & 2nd stage of GM cryocooler are ~20W & ~800 mW respectively. The helium convection medium allows to reach the lowest sample temperature down to 5 K at the exchange gas pressure of 34.5 kPa. Due to the thermal anchoring with the 2nd stage.
of the CCR, the temperature of the copper bore (e2 in Figure 1) i.e T4 reaches minimum temperature of 4.75 K.

![Cool-down profile diagram](image)

Figure 4. The cool-down profile of the helium exchange gas based VTI system at 34.5 kPa sample space environment.

The similar kind of system cooldown readings were taken for sample space at vacuum condition. The system reaches equilibrium condition in 9 hours with the lowest sample temperature of 8.7 K, which is 3.7 K higher than the steady state temperature of the sample at 34.5 kPa gaseous helium environment. This higher temperature occurred due to heat transfer has been taken place by radiation whereas in above mentioned case through convection. The 1st & 2nd stage of GM cryocooler settled to 3.3 K and 34 K temperature with heat loads of 800 mW and 20 W respectively. In Figure 5, the cooling of sample with two different sample environment were compared. The sample takes 2 hours more to reach equilibrium condition in vacuum condition than 34.5 kPa gaseous helium atmosphere.
3.2. Heating and cooling curve

The low temperature physics experiments relies on the achievement of the precise control of sample temperature in the entire temperature range and its stability. End user will need to raise and lower the sample temperature during any experimental studies. Hence, both cooling and heating curves have been studied to estimate time required for a complete cycle of experiment.
The temperature of the sample probe has been controlled by Lakeshore temperature controller (Model 331). The heater (25 Ohm of 100W capacity) and the Cernox sensor on the sample holder are connected with the Lakeshore controller for close loop control of the temperature. Figure 6 shows the comparison of the heating and successive cooling curves of the sample respectively for vacuum and 5 psi GHe condition. Figure 6 signifies the heating and cooling of the sample in continuous mode. The temperature of the sample can also be raised or cooled in steps. Continuous heating takes approximately 15 minutes to raise the temperature of the sample from 8.7 K to 325 K in vacuum while in GHe environment at 5 psi, it requires 35 minutes to heat the sample from 5 K to 325 K. The heat load is 5 W with highest temperature of 8.5 K on 2nd stage of GM cryocooler in vacuum heating whereas 14.5 K with heat load of 11 W in GHe environment at 34.5 kPa. Both natural cooling curves have been taken at heater power off mode. The cooling of the sample takes 2 hours 30 minutes to reach 8.7 K in vacuum condition while it takes 3 hours in GHe environment at 34.5 kPa to reach 5 K.

In both cases, the heat load on 1st stage is 20 W with rise of temperature from 34 K to 37 K. The major heat load incorporates on to 2nd stage of cryocooler through exchanging helium gas, but it provides precise controlling environment to maintain the sample temperature while heating and cooling.

3.3. Sample Resistance-Temperature (RT) measurement:-

The thin film of sample (20% Cadmium(Cd) in Zinc oxide (ZnO)) made by sol-gel spin coating method in IUAC is verified with VTI setup, the results of the variation of resistance with temperature in heating and cooling are plotted in Figure 7. The sample is showing expected behaviour of negative temperature coefficient as it is semiconductor material at low temperature and constant resistance at room temperature. The measured value of the resistance of the sample is in well agreement with the values measured earlier in other measurement system.

4. Conclusion:-

The experiments have been successfully conducted and compared to get the minimum sample temperatures 5 K and 8.7 K in 34.5 kPa GHe environment and vacuum condition respectively. The gaseous He atmosphere provides better cooling of sample as compare to vacuum condition, which indicates convective heat transfer allows to quick cool down of the sample with lower temperature than
the vacuum condition. The thermal anchoring of the cold copper bore with 2nd stage of GM cryocooler is one of the reason to get minimum temperature at sample space. This VTI setup is successfully integrated with previously developed cryogen-free magnet system and able to provide variable sample temperature range (5 K to 325 K). This integrated system can also provide variable temperature with variable magnetic field.

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