MEDSI 2004 proceedings

FLOW CRYOSTATS FOR USE IN SYNCHROTRON FACILITY

Ricardo Steinmann
European Synchrotron Radiation Facility - BP 220 - 38043 Grenoble- France
Fernando Bartolomé
ICMA, CSIC Universidad de Zaragoza, 50009 Zaragoza - Spain

Abstract

The improvements in synchrotron beam stability made in recent years have allowed for a substantial increase in measurement quality. The low temperature environment given by cryocoolers becomes the bottleneck in data precision because of their limited temperature range and elevated vibration level. An alternative solution for sample cooling –as low as 1.5 K- is the use of specially designed continuous flow cryostats. We present two versatile, fully operational cryostats, developed at the ESRF.

1 Mini-cryostat for XRMS experiments

In the last years, there has been a wide development of new X-ray techniques due to a substantial increase in measurement quality resulting from the improvements achieved on the beam stability. Amongst others, X-ray magnetic resonant scattering (XRMS) is a relatively new X-ray technique to analyze the magnetic properties of materials. XRMS combines the crystallographic information available by means of an X-ray diffraction technique with sensitivity to the local magnetization in the sample. The experimental geometry of a XRMS experiment on a ferromagnet imposes the requirement that the applied field be perpendicular to the (horizontal) polarization direction [1]. We have constructed a versatile and low-cost cryostat specifically suited for XRMS measurements allowing a wide scattering angle available for diffraction experiments in the liquid Nitrogen temperature range.

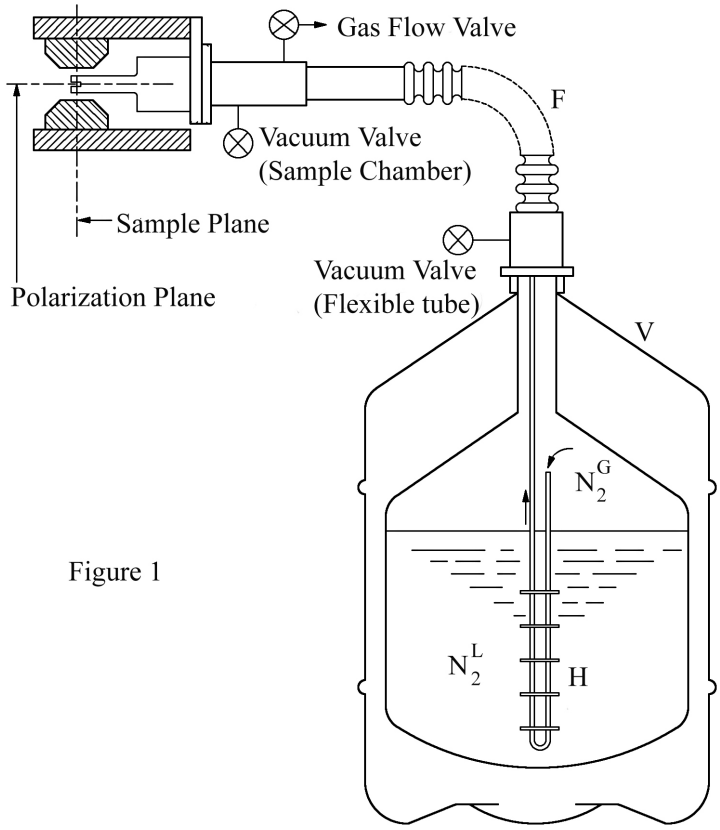


Figure 1

Figure 1 shows the experimental arrangement and Figure 2 a longitudinal cut of the sample chamber. The storage vessel (V) is slightly pressurized (about 0.2 bar over the atmospheric pressure) to generate the gas circulation. The warm gas coming from the upper part of the vessel is cooled down, all through the heat exchanger (H), close to the liquid temperature and reach the sample chamber (1 in figure 2) by the way of the pipe (F). The delivery pipe F is a double wall type made from two coaxial flexible tubes; the inner tube was enveloped with 26 layers of superisolation.

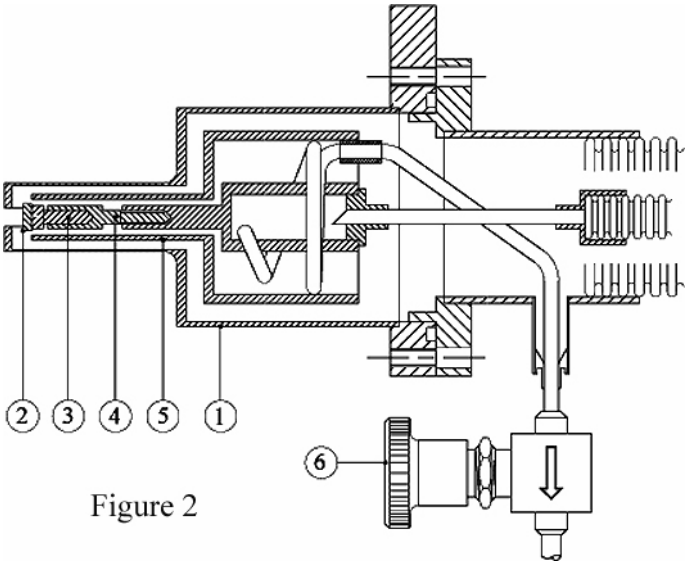


Figure 2

Sample (2) temperature is conditioned by means a cold finger (4) equipped with a heater (RuO2 resistive chip) and a thermometer (Cernox). The outgoing gas cools the copper screen (5) surrounding the sensitive cold parts. The valve (6) allows the flow regulation and thus, the cooling power setting.
The sample chamber, made in gold plated stainless steel (type 316), has 12 mm OD to fit into the gap ferromagnet. On the cylindrical rounded end a 3 mm slit was made in order to glue a Kapton window of 50 microns thickness. The sample holder (3), made in copper, can hold a sample of 6 mm diameter.

The goal of this mini-cryostat is the versatility. Beyond the facility for positioning (any angle, any direction), it can be used as a continuous (gas or liquid) flow type in several configurations letting different temperature ranges and stabilities at the sample. For example, in order to obtain the best temperature regulation ($\Delta T/T \leq 0.5\%$) a coarse approach is made by the gas flow regulating valve (cooling power setting) and the precise temperature is set by the use of a temperature controller. In those cases in which a high precision temperature setting is not required but a good stability (ΔT best than 0.1 K) is required, the addition of a metering valve in parallel with the flow valve allows for a fine cooling power control.
We performed a temperature-dependent XRMS experiment on Nd2Fe14B [2,3] using this device. The lowest well-stabilized temperature reached was 86 K using a copper cold finger. The cooling down time, from room temperature, was about 20 min.
The wide scattering angle available for diffraction and the handiness of this mini-cryostat stand for a large spectrum of potential applications by simple change of the shape of a few pieces.
Preliminary tests were made using liquid Helium as cold source and gas Helium as flowing fluid. In spite of its specific design for the liquid nitrogen temperature range, a temperature of around 12 K was easily reached.
At present, a liquid Helium temperature range mini-cryostat is in construction

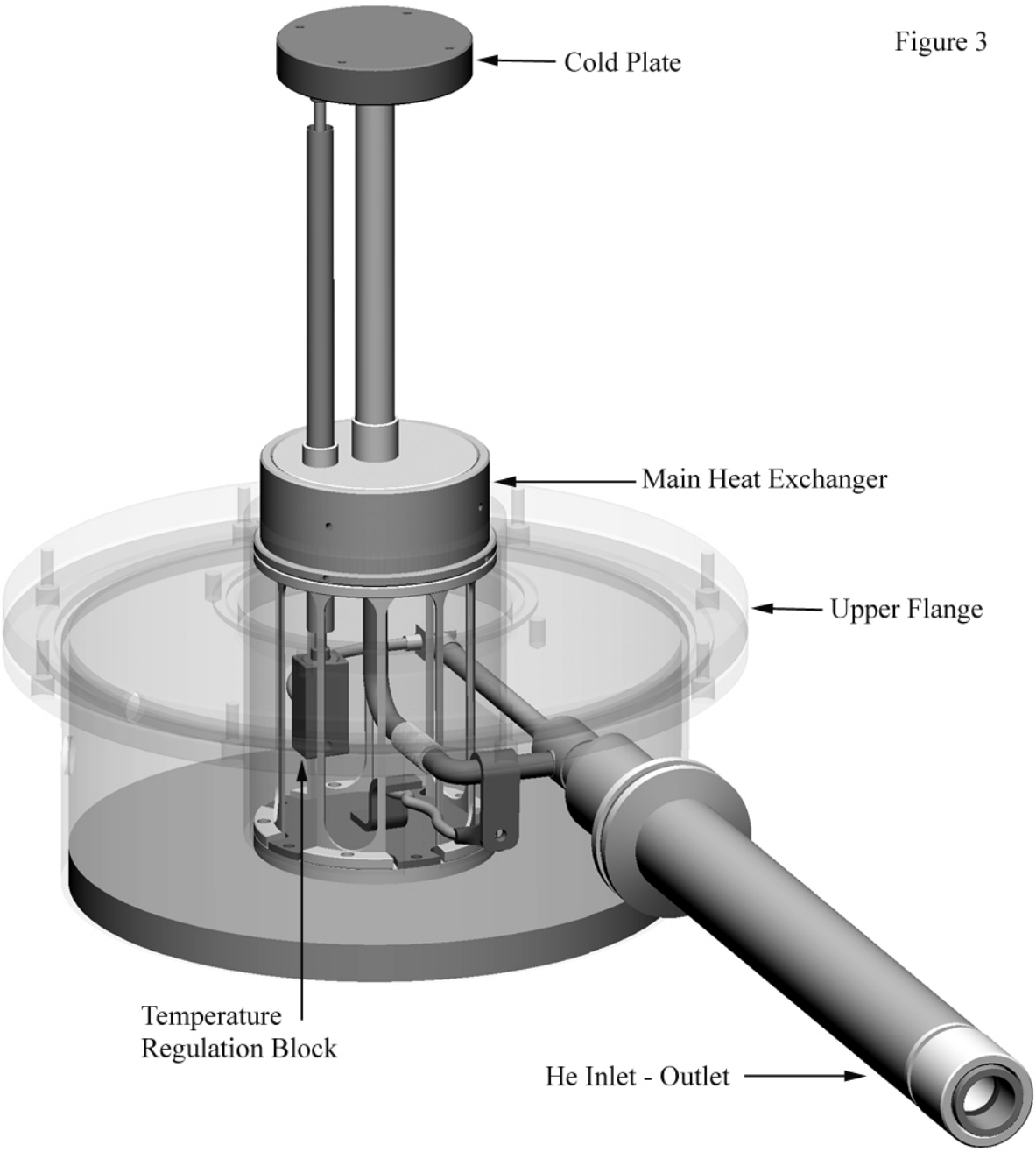
2 Multipurpose continuous flow cryostat.

Notwithstanding their simple and friendly use, piston cryogenerators become the bottleneck in the measurement and data collection process because of their substantial vibration level compared to the beam stability. Amplitude vibrations of a few micrometers at a wide frequency spectrum are harmful for the finest applications as topography, EXAFS, etc.

The cryostat we present here was specially designed to replace a piston type cryocooler.
Taking exactly the same geometrical dimensions, it allows the use of most of the existing dedicated parts as measurement chamber, screen and vacuum chamber while improving temperature range, stability, cooling rate and above all vibration level.

A drawing of the cryogenic components of the cryostat is shown in figure 3.

Figure 3



Liquid or gas He coming from the storage dewar -through a gas cooled transfer tube LLT600 from Oxford Instruments- is conditioned in the temperature regulation block and flows (inside a 2 mm ID tube) to the cold plate where the measurement chamber is mounted (not shown here). Gas He pumped through the gas cooled transfer tube goes through the main heat exchanger where the excess gas enthalpy is used to cold down two aluminum screens (not shown here): the first one covering the lower part of the cryostat, support and the temperature regulation block and the other enveloping the sample chamber. Finally, the remaining outgoing cold gas screens the incoming He from the room temperature radiation.
He inlet and outlet pipe is a modified Johnston-Cox coupling to allow it to work horizontally.
The cold plate and the side of the main heat exchanger have been gold plated to improve the thermal contact. The vacuum chamber, mounted on the upper flange (shown in figure 3 in transparence), has the windows adapted to each particular experiment according to the specific sample chamber used.

The operation of this cryostat [4] is like a typical flow cryostats, the only difference is that in this device it is possible to use the temperature regulation block in the same way as the evaporator in a classical Helium bath cryostat allowing temperatures ~1.5 K during a short lapse (2 or 3 min depending on the thermal load). The lowest working temperature depends on the thermal load and the Helium consumption depends on the working temperature, as is show in figures 4 and 5 respectively.

Figure 4

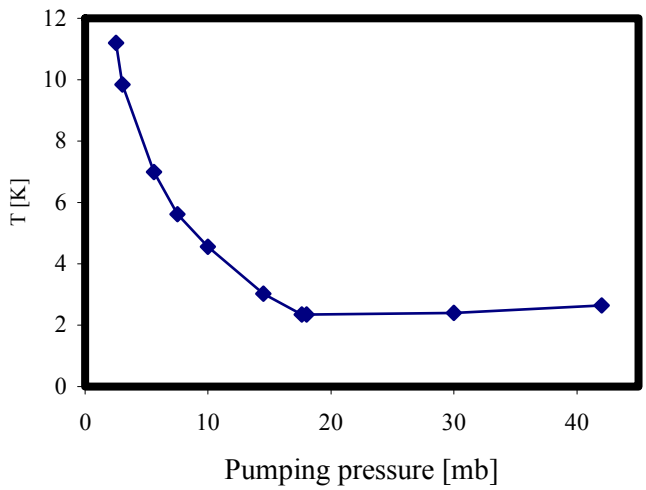
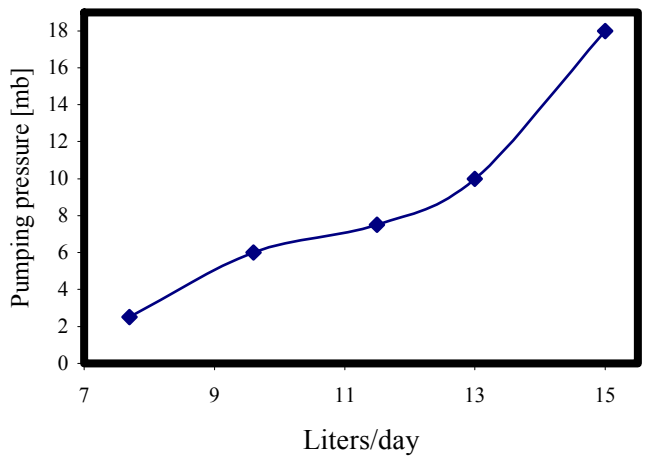


Figure 5



The temperature range is 2.1 K – 360 K and its stability is $\Delta T/T \sim 0.1\%$
Cooling down from room temperature to 2.1 K takes 18 min and a 10 K step about 2 min.
With a static thermal radiation load of around 10 W the He consumption is 0.625 l/h at 2.1 K, 0.32 l/h at 12 K and 0.06 l/h at 150 K.

3 References

[1] F. de Bergevin et al, Phys. Rev. B, **46**, 10772 (1992)
[2] F. Bartolomé et al, J. Appl. Phys. **87** (9), 4762, (2000)
[3] This work was partially financed by Fundación Ramón Areces
[4] R. Steinmann, internal communication BM29 (2002), ESRF