Journal of Zhejiang University SCIENCE A
ISSN 1673-565X (Print); ISSN 1862-1775 (Online)
www.zju.edu.cn/jzus; www.springerlink.com
E-mail: jzus@zju.edu.cn

Simple operated multipurpose temperature control cryostat*

ABBAS T.A., OMAR M.S.†
(*Department of Physics, College of Science University of Salahaddin, Arbil, Iraqi Kurdistan, Iraq*)
†E-mail: dr_m_s_omar@yahoo.com
Received May 19, 2006; revision accepted Mar. 5, 2007

Abstract: A suitable simple optical cryostat for optical, magneto-optical, electrical and thermo-electrical measurements was designed. It is suitable for use in a magnetic pool gap as narrow as less than 1 cm. Throughout a long period of time, the heat diffusion process of the cryostat can be easily operated at slow increase in sample temperature in a range 1.25 K/min at 200 K that will be reduced gradually to 0.66 K at room temperature. Liquid nitrogen was used to cool down the temperature. During the operation, the change in the measured energy gap of a semiconductor sample and other physical parameters resulting from the change of temperature can be corrected through the temperature coefficient of that parameter at the corresponding temperature. The cryostat was successfully used for all experiments mentioned above to measure the properties of a single crystal of GaP (Gallium Phosphate) semiconductor.

Key words: Optical cryostat, Electrical cryostat, Magneto-optical cryostat
doi:10.1631/jzus.2007.A0793 **Document code:** A **CLC number:** TB126

INTRODUCTION

It is important to study the properties of any sample at low temperature as such study permits us to investigate processes that cannot be observed at room temperature (Pathinettam Padiyan *et al*., 2000; Hernández *et al*., 2002). Conducting optical experiments using liquid nitrogen suffices for many applications. It is useful to conduct optical cryostat experiments to study the optical properties of a sample at low temperature using liquid nitrogen (Andrews and Boxer, 2000).

Optical cryostats are commercially available, for example, the optical cryostat Model OC 16-03 from (B. I. Verkin Institute for Low Temperature Physics and Engineering of NAS, Ukraine) and CTC-6 from (ABBESS INSTRUMENTS). Generally, they are expensive and difficult to get. For magneto-physical properties, these commercial cryostats need wide gap magnetic poles with intense magnetic field which consequently involves high cost. In order to use narrow air gap magnet poles to study the magneto-optical properties, a special small size thin room cryostat is needed. Such a cryostat was specifically designed in a form so that its sample holder will be suitable for use between magnetic poles gap as small as less than 1 cm. The sample temperature should also be cooled down and controlled through the process of heat diffusion alone, which means that the cryostat does not need electronic temperature control. Such a cryostat is simple to design, can be made for any purpose, is easy to operate, can be made by any simple workshop, and has overall weight of less than 300 g.

DESCRIPTION OF THE CRYOSTAT

The optical cryostat shown in Fig.1 was designed in the shape of symbol Γ. The 19 cm long arm A (2 cm×0.7 cm) cross section is immersed in liquid nitrogen. This part of the cryostat should have large weight in order to transfer a large amount of heat to the liquid nitrogen. The other 21 cm long arm B carries the sample. The first 10 cm of this arm has the same cross section as A. Then the thickness was re-

* Project (No. 3/2/5638) support by the University of Salahaddin in Arbil, Iraqi Kurdistan, Iraq

duced in three steps: the next 1 cm of this arm is 4 mm thick and is used to fix the vacuum sample house; the next following 7 cm is 2 mm thick and is used for the connection wires (thermocouple, hall probes and heater wires); the last 3 cm is about 1 mm thick and is used to carry the sample, sample heater, thermocouples and all other related connections as shown in Fig.1a. To allow the light beam to pass through the sample, the latter was fixed on a circular hole with radius of about 1 mm. In order to pass the thermocouples wires and all other electrical connections to the sample, a suitable canal was made along the cryostat's first and second part of arm B. To allow the heat to distribute more uniformly all over the sample, a 1 cm×1 cm square hole was made between the sample position and the rest of the arm as shown in Fig.1b.

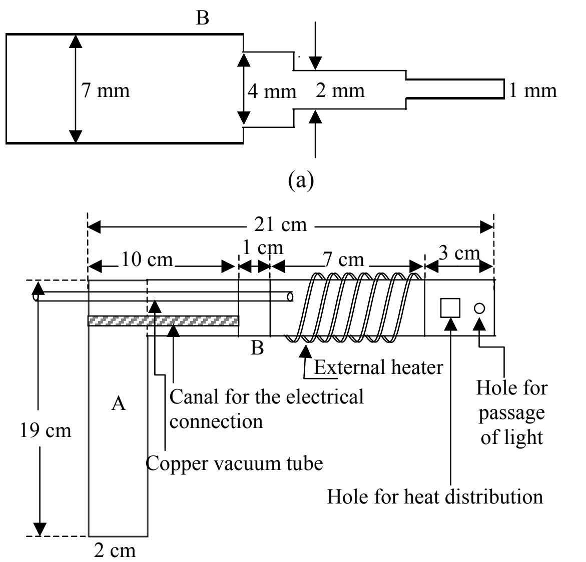

Fig.1 The block diagram for the actual part of the cryostat. (a) Tope view; (b) Side view

For the purpose of vacuuming the sample house, a 1 mm inner diameter copper tube was connected between the sample house and the vacuum system. The sample house was made from 1 mm thick glass slides. The inner dimensions of the glass room (sample room) were 10 cm×2.2 cm×0.5 cm. All the parts of the cryostat except the glass house were isolated from the ambient temperature by pieces of cork.

It is well known that, thermal isolation is important in temperatures mostly below room temperature. In order to trap water vapor inside the sample house, a coil was made as a part of the connection tube to the vacuum unit and with the sample being at room temperature the coil was immersed in the liquid nitrogen in flask 2 as shown in Fig.2. This figure shows the vacuum, the cold trap and the cooling system. In order to reduce the sample temperature, side A of the cryostat was immersed in liquid nitrogen inside flask 1. With this technique, the sample temperature will be cooled down via heat diffusion process. The lowest sample temperature obtained was 200 K. To make the sample temperature be more uniform an external heater was installed around the glass room in part B just before the sample position. This heater works also to control the change of temperature during the experimental operation ΔT, through out all the experimental temperatures. However this cryostat is much simpler than the models OC 16-03 and CTC-6 and that reported by Andrews and Boxer (2000) and also many other cryostats. It needs no temperature control and works in a small magnetic pole air gap.

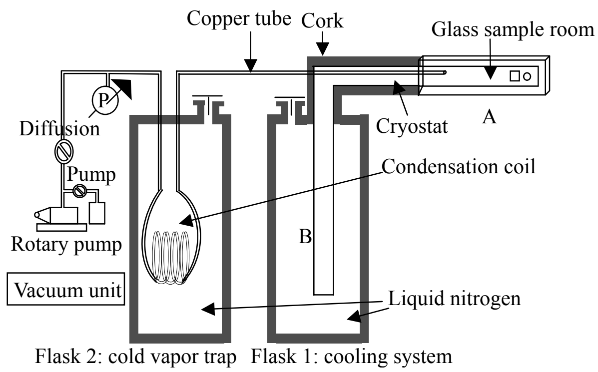

Fig.2 The vacuum and cooling system

OPERATION OF THE CRYOSTAT

The cryostat was operated as follows: the vacuum unit was first operated for about 15 min to eliminate the water vapor from the chamber. Liquid nitrogen was filled in flask 2 shown in Fig.1 as mentioned above. The condensed vapor on the outer surface of the glass house was removed by exposing it to warm air produced by a ventilation unit. Then liquid nitrogen was poured gradually into the cryostat at flask 1 till it was filled. Measurements were started as soon as the lowest sample temperature approached to equilibrium, 200 K in our case.

Due to the relatively long duration time needed for the magneto-optical experiments, accurate optical measurement values are not possible. For this case and for each run, the measurements were corrected by taking into account the temperature effect on the optical parameters. However, the temperature effect arises when the sample temperature changes during the time needed for the optical scanning as well as its repetition under the effects of different magnetic fields. By applying the effects of the mentioned temperature change ΔT on the measurements, the results can be corrected. For example, before the correction, the room temperature of the determined energy gap of a semiconductor GaP (Gallium Phosphate) was found to be equal to 2.2102 eV, while after that it equaled to 2.211 eV (Abbas, 2005). The corrections were operated as follows:

The time needed for one single operation of a light frequency scanning at a temperature T=250 K was 20 s. The temperature change due to this period of time with no magnetic field was in the range of 0.17 K, which changes the value of E_g to be only in the order of 10^{-5} eV and that can be neglected. But the time needed for the preparation and operation of the second experiment due to the first applied magnetic field was about 40 s. This 40 s makes the total time from the beginning of the first run to be about 1 min and the temperature to change in about 0.5 K, which affects the measured energy gap in the second experiment to change to 1.55×10^{-4} eV. For indirect energy gap transition, GaP in this case, was obtained, according to the equation bellow (Camassel and Auverge, 1975):

$$\left(\frac{dE_g}{dT}\right)_B = -3.1\times10^{-4}\ \text{eV/K}. \quad (1)$$

The increase in the number of different magnetic field experiments was accompanied by the increase of experimental duration time and consequently the increase of ΔT. Depending on this and for each corresponding ΔT related to the order of the magnetic field experiment, in this case from 1st to 6th magnetic fields, the correction in the optical energy gap ΔE_g was calculated and added to the measured E_g as given in Table 1. This table shows the correction parameters for different temperature changes ΔT due to experiments related to different applied values of magnetic field at T=250 K. Fig.3 shows the cryostat temperature change ΔT for a duration time of 2.6 min as a function of T which is needed for the operation of a set of zero and the other 6 different magnetic field effect experiments. The almost linear dependence of ΔT on T was obtained by using the cryostat external heater.

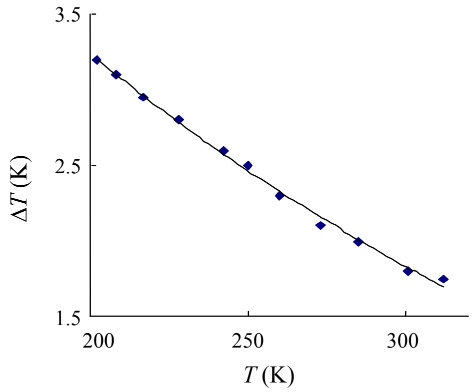

Fig.3 Experimental duration temperature change ΔT as a function of T

To decrease the effects of the temperature change ΔT on the optical properties during the experiment, E_g in this example, the E_g temperature coefficient of GaP which was 3.1×10^{-4} eV was used. For a one run experiment, this value can be ignored, but when several experiments for the same temperature were needed, such as magnetic field effects on the energy gap, the corrections due to the temperature were important as shown in Table 1. Fig.4 shows the corrected effects of the magnetic field on the energy gap of GaP at different temperatures by using the cryostat system shown in this work.

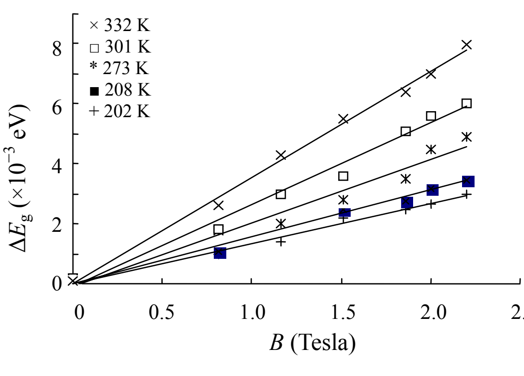

Fig.4 Magneto-optical energy gap shift (ΔE_g) versus magnetic field (B) for GaP at various temperature

Table 1 Correction of the measured energy gap due to the effects of temperature at different magnetic fields when T=250 K

Magnetic field (Tesla)	ΔT (K)	Measured E_g (eV)	ΔE_g ($\times 10^{-4}$ eV)	Corrected E_g (eV)
0	<0.1	2.2320	0	2.2320
0.81	0.4	2.2321	1.24	2.2322
1.16	1.0	2.2322	3.10	2.2324
1.51	1.6	2.2324	4.96	2.2328
1.86	2.0	2.2328	6.20	2.2334
2.00	2.3	2.2333	7.13	2.2340
2.20	2.5	2.2340	7.75	2.2348

CONCLUSION

The cryostat designed in this work can operate at temperature range from 200 K to 330 K and is suitable for measuring the temperature dependence of electrical, thermal, optical, as well as the effects of magnetic field thereon. It is suitable for operating in a magnetic pole gap as small as 1 cm in separation. The cryostat is economical, simple and easy to make it in any research laboratory workshop.

References

Abbas, T.A., 2005. Physical Properties of N-type GaP Single Crystal Semiconductor Compound. Ph.D thesis, College of Science, University of Salahaddin, Kurdistan, Iraq.

Andrews, S.S., Boxer, S.G., 2000. A liquid nitrogen immersion cryostat for optical measurements. *Rev. Sci. Inst.*, **71**(9):3567. [doi:10.1063/1.1287343]

Camassel, J., Auverge, D., 1975. Temperature dependence of the fundamental edge of germanium and zinc-blend type semiconductors. *Phys. Rev. B*, **12**(8):3258. [doi:10.1103/PhysRevB.12.3258]

Hernández, E., López Pescador, A., Durante Rincón, C.A., León, M., 2002. Electrical properties of $CuIn_3Se_5$ bulk crystals at low temperature. *Cryst. Res. Technol.*, **37**(10): 1088-1093. [doi:10.1002/1521-4079(200210)37:10<1088::AID-CRAT1088>3.0.CO;2-D]

Pathinettam Padiyan, D., John Ethilton, S., Paulraj, K., 2000. Protonic conductivity and photoconductivity studies on $H_3PW_{12}O_{40}21H_2O$ single crystals. *Cryst. Res. Technol.*, **35**(1):87-94. [doi:10.1002/(SICI)1521-4079(200001)35:1<87::AID-CRAT87>3.3.CO;2-K]