A Cryogenic Optical Fiber Irradiation System

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ABSTRACT

This paper examines the feasibility of using a cryogenic optical fiber link for an irradiation study of the quantum Hall effect (QHE). Commercially available $GaAs_{1-x}P_x$ light-emitting diodes (LED's) were studied at temperatures between 4.2 and 300 K to examine their suitability for use in a cryogenic environment. Results for light output versus current and temperature, including freeze-out temperatures, are presented along with I-V characteristics at 4.2, 77, and 300 K. A surprising form of negative resistance is observed in infrared GaAs and yellow GaAs_{0.85}P_{.15} LED's at low temperatures. Further, an attempt to measure optical fiber attenuation as a function of temperature showed that for fiber lengths under 5 m the changes were smaller than the resolution of the experiment. The optical link as a whole provided light powers between $6X10^{-7}$ and $1X10^{-5}$ milliwatts, adequate to extend a previous QHE study.

I. INTRODUCTION

The recently discovered quantum Hall effect $(QHE)^1$ in which a 2-dimensional electron gas exhibits quantized transverse resistance at very low temperatures and in high magnetic fields has generated great interest. Recent experiments, performed in Professor Kirk's laboratory has examined the effect of light on a quantum Hall sample.² Layered n-AlGaAs/GaAs samples were irradiated with light from red light-emitting diodes (LED's) and the resultant changes in the QHE were measured. The incident photons inject free electrons into a previously undoped layer of GaAs. The induced carriers allow conduction in a region parallel to the 2-dimensional layer in which the quantum Hall effect is observed. The resultant Hall voltage measurements show a degradation of the pure quantum effect. To extend this study it is useful to determine how changes in the quantum Hall effect depend on the wavelength of light used. By varying the wavelength of light we can probe the electronic structure of the Hall sample in a manner analogous to probing atomic transitions or chemical bonds. The purpose of the study was to investigate a possible method of irradiating the Hall samples with light of various wavelengths.

The original experiment used LED's placed inside the sample chamber, cooled to about 0.75 K. The LED's were run at 0.5-5 mA in successive pulses of 3-1200 msec. The simplest way to extend this experiment using other wavelengths of light would be to place LED's of various colors inside the cryostat. The performance of LED's at low temperatures, however, is not well understood. It has been established previously that red GaAsP LED's function in liquid helium (4.2 K)³, while green GaP LED's do not.⁴ An obvious alternative to placing the LED's in the sample chamber is to locate a bank of LED's at some warmer temperature and channel the light to the Hall sample with optical fibers. Since the Hall sample must be maintained at temperatures on the order or 10 millikelvin, it is also desirable to have the optical fibers as close as possible to the Hall sample to minimize the heat leak into the sample chamber. To build such a system requires an understanding of the performance of light-emitting diodes and optical fibers at low temperatures. The purpose of this project then was to establish the requirements of such a system and then determine experimentally if an optical fiber link with cooled LED's could be used.

II. EXPERIMENT

A. APPARATUS TO DETERMINE LIGHT-EMITTING DIODE PERFORMANCE

1. Overview

In this experiment the light output and I-V (current vs. voltage) characteristics of red, yellow, green and infrared $GaAs_{1-x}P_x$ light-emitting diodes (LED's) were studied as a function of temperature and current. Table 1 summarizes the characteristics of the diodes used.

Tab.	le 1.	Characteristics	of	$GaAs_{1-x}P_x$	LED's	$(300 \mathrm{K})$) ^{5,6}
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<u>x [mole frac.]</u>	Color	<u>Peak wavelength</u>	Light output (@20 mA)
0.00	IR	880 nm	1.5 mw
0.40	Red	$650 \mathrm{nm}$	$0.32 \mathrm{mw}$
0.85	Yellow	580 nm	0.091 mw
1.00	Green	$560 \ \mathrm{nm}$	$0.026 \mathrm{mw}$

A schematic of the experimental apparatus is shown in Fig. 1. To measure the relative light output, diodes were coupled to plastic 140- μ diameter optical fibers which carry the

light to a photodiode detector. The lamps were put in a brass vacuum chamber at the base of a cryostat which was then immersed in liquid nitrogen or liquid helium for cooling. Additional details of fiber preparation, light detection, temperature and current control and data acquisition are discussed below.

2. Optical fiber link

Small holes (approximately 0.25 mm) were drilled in the top of the light-emitting diodes to accommodate optical fibers. Using a small gauge syringe, the holes were filled with clear Emerson Cumings 1266 epoxy and the fibers were glued in place. On the other end the fibers were bundled and cast into a 5-mm long, 1-mm diameter cylinder with five minute epoxy. The fiber ends were then polished. The final epoxy cast then fit into a Teflon cover attached to the photodiode detector.

Relative light output was measured with a United Detector Technologies PIN 10DF photodiode detector placed at the top of the cryostat at room temperature. The detector was filtered to provide a constant response at a given light power level, independent of the wavelength of light. This was especially important since the wavelength of light output from an LED changed with temperature. Use of this detector allowed accurate measurements of relative light power output independent of the original color of the LED and independent of any shifts. Light striking the detector produced a current of 0.1 amps per watt of light power. Typical currents were on the order of 10^{-9} amps. This current was shielded at the feedthrough and along the cable. The detector was connected in series



Fig. 1. Experimental apparatus

with a 8 megaohm reference resistor. The voltage across the resistor, usually millivolts, could be measured. This voltage was a measure of relative light output, rather than absolute. Only a fraction of the light produced by the LED was coupled to the fiber and this fraction could not be carefully controlled with the technique mentioned above. Using data book values⁵ for room temperature luminous intensity and comparing these to light outputs discussed below, it was estimated that coupling efficiency varied between 5×10^{-6} and 5×10^{-5} . This method prevented an accurate comparison of the light output from one diode to another, but it allowed comparisons of light output of the same diode at various temperatures and currents.

3. Temperature control

The LED's with fibers attached were placed in a copper sample holder and thermally anchored to the copper with Apeizon "N" grease. The sample holder rested on the bottom of the brass vacuum chamber while nylon legs and spacers on the sample holder prevented direct thermal contact between the copper and the brass chamber walls. The sample holder and LED's were then cooled by filling the vacuum chamber with helium gas at about 1 atm to allow heat transfer to the walls of the vacuum chamber, which was externally cooled with liquid helium. The temperature was determined by measuring the resistance of a calibrated germanium thermometer. Because of the characteristics of the resistance vs. temperature curve for the thermometer, temperatures could only be measured up to 100 K. When the temperature of the sample holder reached liquid helium temperature (4.2 K), the helium gas in the brass vacuum chamber was pumped out using a diffusion pump, which effectively isolated the sample holder temperature from that of the surrounding



Fig. 2. Current control and data acquisition system



Fig. 3. Power booster (current control circuit)

brass chamber. Resistive wire was wrapped around the copper sample holder and was then used to heat the sample holder to higher temperatures. In practice, the small heat leak through the nylon legs and spacers required running a heater current continuously to maintain temperatures above 4 K. To cool the heater was turned off and helium exchange gas was let back in to the vacuum chamber.

4. Current control and data acquisition

During the original room temperature (293 K) and liquid nitrogen tests (77 K), the current was controlled by hand using a variable output power supply. This method required running the LED's continuously for about 20 seconds for each data point. The heating due to this continuous current caused temperature uncertainties on the order of 1-2 K. Since many measurable properties of semiconductors depend on the absolute temperature, such an uncertainty at around 4 K could create large errors. For this reason a computer control and data acquisition system was assembled for experiments at liquid helium temperatures. A schematic of this system is shown in Fig. 2.

A HP-9835A computer with an IEEE-488 bus handled current control, temperature measurement, I-V characteristic measurements, and light outputs. Variable currents from 0.01 mA to 80 mA were produced by generating voltages at small currents using the D/A converter and then converting them to calibrated currents with a simple transistor power booster, shown in Fig. 3. The "compliance voltage," the voltage necessary to produce the current set by the D/A was measured using a Keithley 179 multimeter. Output voltages from the photodiode detector and temperatures were measured with a HP-3478A multimeter. At high temperatures the LED's were turned on for 1 second for each data point and turned off for 2 seconds between readings. At very low temperatures, where heating from the light and resistive dissipation could cause temperature uncertainties, the LED currents were pulsed for 50 milliseconds with 10 seconds between readings.

B. RESULTS OF LED TESTS

1. Light output vs. temperature

Plots of light output vs. temperature for several LED's are shown in Figs. 4.1-4.3. Since temperatures could only be measured up to 100 K, the curves between this temperature and room temperature are rough interpolations. As shown in Fig. 4.3, yellow and green LED's ceased operation at about 75 and 85 K respectively. Since carrier density in gallium compounds is due to doping rather than thermal activation⁷, we do not expect carrier freezeout to be a problem. Both the yellow and green LED's have indirect minima in the energy vs. momentum structure at higher momenta. Recombination between localized holes in the valence band and higher momentum electrons in indirect minima must be phonon-assisted to conserve momentum.⁶ Although at low temperatures the carrier density may still be high and essentially independent of temperature, we expect the phonon density to drop sufficiently so that indirect transitions become improbable. Infrared and red LED's, however, continue to function down to 4.2 K. Both of these types of diodes produce light by direct recombination without phonon assistance and have carrier densities essentially independent of temperature. Hence, they do not suffer freezeout even at 4.2 K. In all cases



Fig. 4.1 Light output for infrared LED vs. temperature

LIGHT OUTPUT FOR INFRARED LED VS. TEMP.

LIGHT OUTPUT (VOLTAGE ACROSS 8MQ [mV]) 20.0 15.0 17.5 12.5 10.0 0.0 2.5 5.0 7.5 0.0 T 8 30.0 60.0 90.0 120.0 TEMPERATURE [K] 150.0 180.0 210.0 I=40 mA = 10 mA240.0 1 1 1 ł 270.0 1 300.0 6 b

Fig. 4.2 Light output for red LED vs. temperature

LIGHT OUTPUT FOR RED LED VS. TEMP



Fig. 4.3 Light output for green and yellow LED's vs. temperature

LIGHT OUTPUT FOR GREEN AND YELLOW LED'S VS. TEMP.

the diodes became more efficient at producing light as they were cooled. Table 2 shows the increase in light production for each LED at 100 and 4.2 K for compared to its room temperature value.

Table 2. Light output for LED's at 100 and 4.2 K.

(Room temperature output normalized to one at I=40 mA)

Lig	ht	Ou	$\mathbf{t}\mathbf{p}$	out
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LED	<u>100 K</u>	<u>4.2 K</u>
IR GaAs	1.67	2.05
Red $GaAs_{0.6}P_{0.4}$	1.95	7.01
Yellow $GaAs_{0.15}P_{0.85}$	6.29	-
Green GaP	6.13	_

2. Light output vs. current

Graphs of relative light output versus current for red, yellow, green and IR LED's are shown in Figs. 5.1-5.3 at 300, 100 and 4.2 K (where applicable). As expected, room temperature light outputs exhibit essentially linear current dependences for all diodes studied, though slight saturation effects can be seen at higher currents. The linear dependence was preserved for red and IR LED's even to liquid helium temperatures. Note that the yellow LED showed a serious drop in efficiency at 78 K when the current exceeds 10 mA.

LIGHT OUTPUT (VOLTAGE ACROSS 8MQ [mV]) 80.0 10.0 20.0 60.0 70.0 30.0 40.0 50.0 0.0 0.0 9 9 19 9 10.0 0 D Room temp. 78 K 4.2 K 20.0 . De 30.0 LED CURRENT [mA] 40.0 50.0 60.0 70.0 80.0

LIGHT OUTPUT FOR INFRARED LED VS. CURRENT

Fig. 5.1 Light output for infrared LED vs. current





Fig. 5.2 Light output for red LED we amand

OUTPUT VOLTAGE ACROSS 8 MQ RESISTOR [mV] 10.0 15.0 20.0 12.5 17.5 0.0 2.5 5.0 7.5 0.0 Т T 5.0 10.0 0 \triangleright Green LED, 300 K Yellow LED, 300 K Yellow LED, 77 K 15.0 ٩ LED CURRENT [mA] 20.0 9 25.0 30.0 35.0 40.0 ١

Fig. 5.3 Light output for green and yellow LED's vs. current

LIGHT OUTPUT FOR GREEN AND YELLOW LED'S

I-V curves for LED's at 300, 77 and 4.2 K (again, where applicable) are shown in Figs. 6.1-6.3. We note the very surprising result for the I-V curves of infrared LED's at 4.2 K and for yellow LED's at 77 K. For infrared diodes at room temperature and even in liquid nitrogen, the I-V curves were not surprising, exhibiting positive exponential characteristics. At 4.2 K, however, as the current was increased the compliance voltage jumped discontinuously above 3 volts. Then, as the current was further increased, the compliance voltage dropped, indicating a negative dynamic resistance. The location of the voltage discontinuity was not fixed, even for the same diode, varying over a range of 2-5 mA. Negative resistance was also observed for yellow LED's at 77 K, shown in Fig. 6.2, however the transition is made without a discontinuity. Note that this is just above the freeze-out temperature for the yellow LED's. The negative resistance characteristic is reproduced for infrared LED's using very short current pulses on the order of 50 milliseconds with 10 seconds between pulses. The yellow LED's showed serious instability for such short current pulses, but exhibited reproducible negative resistance for 1 second pulses. For both diodes, it did not depend whether the current was scanned increasingly or decreasingly.

The negative resistance does not appear to be a tunneling phenomenon. A sample I-V curve for a tunnel diode is shown in Fig. 7. Because of a peak in tunneling current at low voltages and an increase in thermal current at higher voltages, I-V curves for tunnel diodes show multiple voltage values at a fixed current. The I-V characteristics for yellow and infrared LED's in Figs. 6.1 and 6.2, however, show multiple current values at fixed voltage. Note that the axes have been interchanged. Scanning the current of a tunnel



I-V CURVES FOR INFRARED LED

Fig. 6.1 I-V characteristics for infrared LED's at 4.2 K, 78 K and 300 K





Fig. 6.2 I-V characteristics for yellow and green LED's at 77 K and 300 K

COMPLIANCE VOLTAGE ACROSS LED [V] 2.4 2.2 2.0 0.0 0.2 0.4 0.6 0.8 1.6 1.8 1.4 1.2 1.0 0.0 T Т Т Т T Т Т Т ۰ - ۰ - ۰ - ۰ - ۰ - ۰ - ۰ - ۰ - ۰ - ۰ 5.0 9 þ ф ф 10.0 ١ o 300 K □ 77 K △ 4.2 K 15.0 0 цЦ LED CURRENT [mA] 20.0 0 25.0 þ 54 ١ ١ ۱ 30.0 -0-₽ ł ١ ۱ 1 35.0 0, ۱ ١ I 40.0 l

I-V CURVES FOR RED LED

Fig. 6.3 I-V characteristics for red LED at 4.2, 77, and 300 K.

diode, instead of the voltage, one would expect to see a discontinuity in the voltage after the current exceeded the peak. After the peak, however, the voltage should increase with current, not decrease. At present the reason for a negative resistance is not understood. Due to instrumentation limitations, high speed voltage scans (instead of current scans) could not be performed, but such experiments are planned for the future.

The red GaAsP LED's exhibited more conventional I-V curves at 300, 77 and 4.2 K, as shown in Fig. 6.3. When the diode was cooled to liquid nitrogen temperature, the turn-on voltage (and hence gap energy) increased. At liquid helium temperatures, however, the increase in turn-on voltage for low currents was small. At higher currents, the forward voltage was actually less than at 77 K. The change in slope may indicate the beginnings of a transition to a negative resistance state as seen in the infrared LED's. Attempts were made to fit the high temperature I-V curves to the simple model of a diode where the current through a diode is given by the implicit relation (Ref. 8):

(1)
$$I = I_d exp[\frac{e(V - IR_s)}{kT}] + I_r exp[\frac{e(V - IR_s)}{2kT}],$$

where e is the electron charge, V is the voltage across the diode, R_s is the series resistance due to bulk resistivity of the semiconductor and to metal-semiconductor contacts. I_d and I_r are the saturation diffusion and recombination currents. Recombination current is responsible for photon emission and the ratio of I_d to I_r is a measure of the efficiency of the LED. Note for even small negative biases the reverse current tends quickly to $I_d + I_s$. Infrared GaAs LED's do not obey this rule with reverse bias. Instead reverse breakdown



SAMPLE IV CURVE FOR A TUNNEL DIODE

occurs at potentials less than 1 volt, with exponentially increasing currents on the order of 50 microamps. I attempted to fit room temperature and liquid nitrogen data for positive potentials to this theory using the chi-square minimization routine MINUIT from the CERN library to extract the parameters I_d , I_r , and R_s , solving the implicit relationship for I using a simple binary search. The experimental curves, however, fit the theory only marginally well, with increasing deviations as the temperature drops. In all cases, the current data better fit a lower order exponential curve. Because of the $exp[\frac{1}{kT}]$ dependence, this situation may indicate that the LED's, even for short current pulses, operate well above ambient temperature. It has, however, been previously demonstrated that GaAs LED's do not exactly follow Eq. 1, especially at higher current densities.⁹

Since accurate fits could not be made, parameters like gap energy, related to the saturation current and the temperature, could not be extracted. Qualitative statements, however, can be made. In particular, the fits showed that saturation currents decrease with temperature and gap energies increase as expected. Further, IR LED's appear to show almost exclusive recombination current effects, even at room temperature.

C. OPTICAL FIBER MEASUREMENTS

1. Set-up

The purpose of this experiment was to determine the attenuation of optical fibers as a function of temperature between 4.2 K and 300 K. A bundle of six commercially available fibers, length 4-5 m, were coupled to infrared TIL906-1 LED's by the technique described in Section II.2. In this case, however, the LED's were kept at room temperature. The bulk

of the fiber was then wrapped around a 2.5-cm diameter copper sample holder which was placed in the brass vacuum can as before. In this way, the temperature of all but 1 m of the fiber could be accurately controlled. The fiber ends were cast into epoxy and coupled to the photodetector at the top of the dewar as before. The LED's were then turned on and the light traveled through a single fiber into the detector.

The relative attenuation can be determined by the following relation (Ref. 10):

(2)
$$\alpha_T = \frac{10}{l} (\log(I_{Rm}/I_T))$$

where α_T is an additional attenuation coefficient in dB/m due to temperature effects. I_T is the light output measured at temperature T, I_{Rm} is the room temperature output, and l is the length of fiber.

2. Results

For the fiber lengths used, the change in attenuation between room temperature and liquid nitrogen temperature (77 K) was found to be less than the resolution of the experiment. A previous study¹⁰ confirms this measurement, citing attenuation changes on the order of 0.15 dB/m in liquid helium. For 3 meters of cooled fiber, this would give a 1% change in measured intensity, roughly the accuracy of the experiment.

III. DISCUSSION

Using the scaling factor for red LED's given in Table 2 and a first order approximation, I have estimated the light incident on the previous Hall sample to be 1.1×10^{-8} milliwatts. To replicate and extend the quantum Hall effect experiment, we need light power of at least this level. From results in Section II.B., room temperature light output from 1-m optical fibers ranged between 6×10^{-7} and 1×10^{-5} milliwatts. These figures increase by as much as a factor of six at 100 K due to improved LED performance. Clearly, optical fiber attenuation changes were negligible for fiber lengths studied in this work. So an optical fiber link with LED's cooled to 100 K will provide ample light to allow irradiation studies at four different wavelengths.

To determine a safe operating point for the LED's, I measured the temperature distribution in a liquid helium dewar. As expected from the heat equation for steady state temperatures, the relation is:

(3)
$$T_z = T_{LHe} + \frac{z}{D}(T_r)$$

where T_z is the temperature at a height z above the liquid level, T_{LHe} is the temperature of the liquid helium bath (4.2 K), D is the distance from the top of the dewar to the liquid level and T_r is room temperature. From this relation it is easy to calculate a point in a dewar, given a maxmimum liquid level, which is always above 100 K.

IV. SUMMARY

In summary, the characteristics of commercially available $GaAs_{1-x}P_x$ light-emitting diodes have been established for temperatures between 4.2 and 100 K. Green and yellow LED's cease operation at around liquid nitrogen temperatures, while red and infrared LED's function even at liquid helium temperatures (4.2 K). In all cases, except at the freezeout temperature, light output increased as the diode was cooled. Turn-on voltages, and hence gap energies, also increased as the diodes were cooled. Surprisingly, yellow and infrared LED's were found to exhibit a form of negative resistance at low temperatures which is apparently not due to tunneling. The mechanism for the negative resistance is not understood, but more experiments are planned. Among these, we intend to investigate the temperature dependence of the negative resistance and examine any wavelength shifts and resistive heating changes at the discontinuity. From a practical viewpoint this study shows that a cryogenic optical fiber link with cooled LED's can be used to extend the previous quantum Hall effect irradiation studies.

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