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LOW TEMPERATURE HALL AND SHUBNIKOV-
DE HAAS EFFECTS

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CINCINNATI, OH 45221

DEC 1992

FINAL REPORT FOR 09/01/90-04/30/92

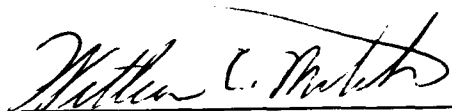APPROVED FOR PUBLIC RELEASE; DISTRIBUTION IS UNLIMITED.

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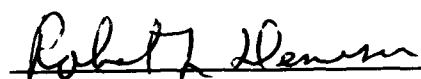WRIGHT PATTERSON AFB OH 45433-6533

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     The goals of this program were to develop the facilities necesasary to measure the quantum Hall effect and Shubnikov – de Haas effects at magnetic fields up to 9 Tesla and at temperatures down to 0.5 Kelvin or below. These goals were accomplished and AlGaAs/GaAs high electron mobility samples were measured, showing the effectiveness of the high fields and low temperatures. A 3He refrigerator was designed and constructed for this project and coupled with a superconducting magnet. A PC based computer control program was witten in QuickBasic to control the experiment.

QUANTUM HALL EFFECT MEASUREMENT, SHUBNIKOV - DE   16
HAAS EFFECTS, MAGNETIC FIELDS, HIGH ELECTRON
MOBILITY SAMPLES, SUPERCONDUCTING MAGNET,

UNCLASSIFIED   UNCLASSIFIED   UNCLASSIFIED   UL

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1. INTRODUCTION

Improved epitaxial growth techniques for semiconductors (molecular beam epitaxy [MBE] and metal organic chemical vapor deposition [MOCVD]) now enable us to fabricate heterostructures with atomic layer resolution. The ability to fabricate such structures has resulted in many advances in electronic and optical devices. For example, two dimensional electron gases are routinely produced at AlGaAs/GaAs interfaces in high electron mobility transistors (HEMT's). These are now becoming commercially available. Many of these advances have been made within the lattice matched AlAs-GaAs system and its alloys, but MBE and MOCVD make possible the fabrication of psuedomorphic HEMT's, AlGaAs/InGaAs/GaAs, in which the electron gas is located at the AlGaAs/InGaAs interface, and the InGaAs layer is under biaxial tension due to its larger bandgap. Strain can have significant effects on the properties of these materials and on the band structures in particular. Among these effects, and pertinent to our interest, is a possible decrease in the hole effective mass in the strained layer. Such an effect could result in devices usable for very high speed complementary logic circuits. In conjunction with the strain effects, piezoelectric fields can be important in III-V strained heterostructures if the growth axis is different from [100]. In particular, polarization electric fields can be produced at the interfaces in strained superlattices grown in the [111] direction.[1]

These effects have produced an interest in superlattices and heterostructures grown in orientations other than the standard [100]. Significant anisotropies are expected in the hole effective masses in two-dimensional hole gases for different growth axes due to the non-parabolicity of the valence bands. Accurate measurement of these anisotropies are necessary to calculate band structures and to model optical and electronic device performance. Shubnikov - de Haas (SdH) effect measurements can give the two-dimensional effective mass but extremely low temperatures are required.

In this program, we have developed a ^{3}He refrigerator capable of achieving temperatures down to 0.4K in conjunction with a SdH experiment, also developed under this program.

This report is divided into seven sections. In the section after this introduction, the theory of the SdH effect is explained with particular emphasis on its use in the measurement of effective masses. In the following section the SdH experiment developed for this program is described in detail, followed by a description of the ^{3}He refrigerator. The experiment's computer control is discussed in the next section, results on some simple HEMT structures are presented and finally conclusions and recommendations are presented.

2. SHUBNIKOV - de HAAS EFFECT IN SEMICONDUCTOR HETEROSTRUCTURES

The Shubnikov-de Haas effect is ideally suited for the study of the "anomalous" electronic properties of GaSb/InAs based heterostructures. Measurements of these quantum mechanical oscillations of the magnetoresistance provide a wealth of information about the material under study, as is discussed below. This technique is particularly useful for the study of two-dimensional systems, such as the electron gas at the GaAs/AlGaAs interface in single heterostructures, and in superlattice structures, where transport parallel to the growth axis is significantly reduced relative to that in the plane of the individual layers.

The standard treatment of the Shubnikov - de Haas effect in two-dimensional systems is given in the review by Ando, Fowler, and Stern[2] and the paper by Fang, Fowler, and Hartstein.[3] Ando, Fowler, and Stern give an expression for the longitudinal magnetoconductivity σ_{xx} in the low magnetic field limit, $\omega_c\tau < < 1$, where $\omega_c = eB/m^*$ and τ is a scattering time. Since the magnetoresistance ρ_{xx} is usually the experimentally determined quantity, rather than the more easily theoretically calculated σ_{xx}, the conductivity tensor is inverted to obtain ρ_{xx}. The expression for the oscillatory portion of the magnetoresistance for a *single* band of carriers in a two-dimensional sample is:

$$\rho_{xx}(osc) = CONST. \times \frac{(\omega_c\tau)^2}{1+(\omega_c\tau)^2} \exp\left(\frac{-\pi}{\omega_c\tau}\right) \frac{X}{\sinh X} \cos\left(\frac{2\pi\epsilon_F}{\hbar\omega_c}\right), \tag{1}$$

where $X = 2\pi^2kT/\hbar\omega_c$. This equation, while strictly valid only in the low field limit $\omega_c\tau < < 1$, has been shown to be useful as long as the oscillations remain sinusoidal.[4]

The concentration of carriers in the band, n, is determined from the frequency of the oscillations through $f = nh/2e$. It can be seen from Eq. 1 that varying the temperature only changes the magnitude of the oscillations, not the period. The temperature dependence is contained predominantly in the term $X/\sinh(X)$, assuming that τ is a slowly varying function of temperature. Fitting the temperature dependence of one or more oscillation maxima gives the effective mass m^* of the carriers in the band producing the oscillation. Once the effective mass is known, the magnetic field dependence of the oscillations determines τ, or equivalently, the Dingle temperature T_D, defined by $\tau = \hbar/2\pi k T_D$. Note that the scattering time determined in this manner is not the same scattering time determined from low-field mobility measurements. The latter is a transport time and is not significantly affected by low-angle
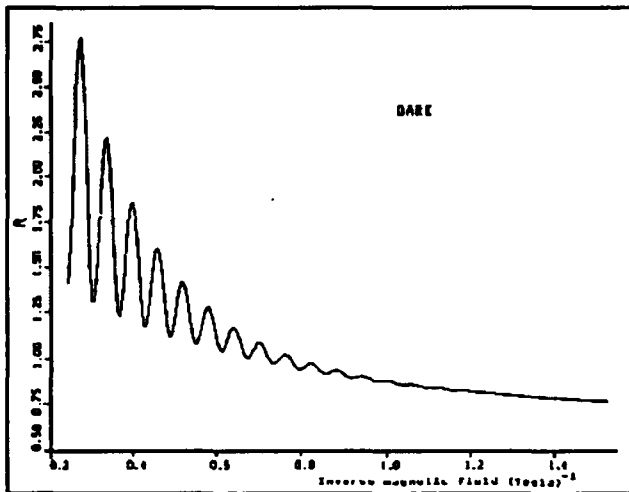

Figure 1. Shubnikov-de Haas oscillations for one electron sub-band.

scattering, while the Shubnikov - de Haas time includes the effects of scattering from all angles.

The above simplified analysis assumes only one band of carriers, which is not ordinarily the case in the semimetallic GaSb/InAs structures of interest here. To understand the complexity introduced by more than one band, we display some typical Shubnikov - de Haas results in Figure 1 and Figure 2. These data were measured in our laboratories at 2 K.
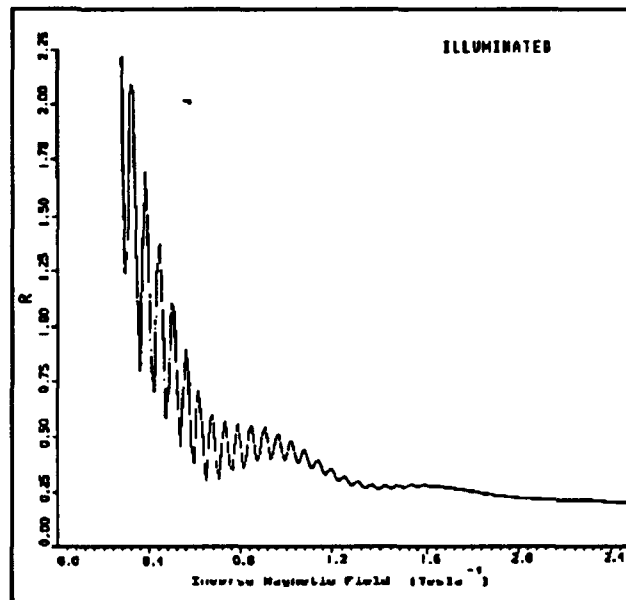
Figure 1 shows the Shubnikov - de Haas

Figure 2 Shubnikov - de Haas oscillations for the same specimen as Figure 1, with two electron sub-bands.

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oscillations for one electron sub-band of the AlGaAs/GaAs system. A single oscillation frequency is observed.

Figure 2 demonstrates the effects of slightly populating the second electron sub-band in the same AlGaAs/GaAs structure by inducing a persistent photocurrent with illumination. The presence of multiple carriers is usually exhibited by a beat pattern in the Shubnikov - de Haas oscillations,[5] from which the individual oscillations can be extracted from Fourier analysis. A second oscillation period is clearly seen in Figure 2, but the concentration of electrons in the second band is not high, so the frequency is not sufficiently close to the frequency of the first sub-band to produce a complete beat pattern. In general, data such as shown in Figure 2 can be analyzed using a simple Fourier decomposition to determine the concentrations of the carriers.

The effective masses of the carriers are needed to both identify the types of carriers and for comparisons to theoretical models. A complete separation of the oscillations and measurement of their individual temperature dependences enables one to determine these masses.

The previous examples have shown Shubnikov - de Haas effects in the relatively simple n-type AlGaAs/GaAs single interface structure typified by the high mobility single-band GaAs conduction band. The Shubnikov - de Haas effect in GaSb/InAs quantum wells is much more complex,[6] since both electrons and holes are present. Washburn et al[7] extended the experiments of Mendez et al[6] to lower temperatures and higher mobilities and found several anomalous effects in the Shubnikov - de Haas oscillations. These were attributed to interactions between the electrons and holes rather than to oscillations directly related to hole concentrations. Because of the large effective mass of the holes compared with the electrons ($0.33m_o$ to $0.037m_o$) in this system, direct observation of holes will be difficult for the magnetic fields available to us. Further complications arise due to the zero field spin-splitting of the electron sub-band, which is induced by asymmetries in the well structure.

This effect produces a beating in the low field oscillations since two electron bands are populated.

In summary, Shubnikov - de Haas experiments are particularly well suited to the study of radical Type-II heterostructures as shown by the experiments at other laboratories. Unlike the conventional low field Hall effect, Shubnikov - de Haas is specific to two-dimensional carriers. Even when there are three-dimensional carriers present, the two-dimensional carriers produce distinct oscillations which can be uniquely identified by rotating the sample in the magnetic field. In this way we can ensure that we are only looking at the two-dimensional carriers, and not observing effects due to carriers in the bulk. Multiple carrier types also can be identified as each produces its own oscillations if the mobilities of each are high enough. Shubnikov - de Haas becomes even more effective when coupled with other experiments, such as cyclotron resonance, as we propose here. Cyclotron resonance is more useful for determining the effective masses of the carriers present, but it is not as useful in determining precise concentrations, where Shubnikov - de Haas excels. These two experiments complement one another, and provide a powerful combination for the study proposed here.

3. SHUBNIKOV - de HAAS EXPERIMENT

The purpose of this project was to develop the necessary equipment to study the Shubnikov-de Haas effect at very low temperatures. The magnetoresistance oscillations were studied in fields to 9.0 T and to temperatures as low as 0.5K. The high temperature limit was determined by the quality of the samples, since the Shubnikov - de Haas oscillations smear out as temperature increases due to broadening of the Landau levels; we expect to observe oscillations to about 10 K. A ^{3}He refrigerator with a 9T superconducting magnet was developed for this experiment. Standard four-terminal AC resistance measurements were used, with frequencies kept below 50 Hz. Measurement currents were kept below a microamp and care was taken to ensure that self-heating was prevented. We found that

currents as low as one nanoamp can be used. The sample temperature was measured with a calibrated germanium thermometer at zero field, and controlled electronically with a relatively field-insensitive carbon-glass thermometer during field sweeps.[8] The magnetic field was stepped through the required range under computer control, and the computer was used to record current, voltage and field values for each temperature point for later analysis. For Fourier transform analysis the field points were evenly spaced in inverse field. Ohmic contacts to the samples were formed by alloying indium dots.

4. ^{3}He REFRIGERATOR

The design of the ^{3}He refrigerator constructed here is based on the standard models presented in Richardson and Smith[9] and White[10] with only minor modifications. Cooling with ^{3}He is advantageous over that by the more common ^{4}He because ^{3}He does not have a superfluid transition until the temperature is reduced to millikelvin, so a bath of ^{3}He can be boiled under pressures of 10^{-3} Torr or less which correspond to temperatures of less than 0.3 K, while the lowest practical temperature obtainable by boiling ^{4}He is about 1.2 K. In most applications, including the design used here, the ^{3}He is liquified from the gas by contact with ^{4}He at a temperature of less than 3.19 K (the boiling point of ^{3}He at atmospheric pressure). After condensation, the ^{3}He is collected in a "pot" in thermal contact with the sample stage and pumped on (with a standard diffusion pump and a sealed mechanical pump) to reduce the temperature. The ^{3}He that evaporates is recycled. With a totally sealed system, the ^{3}He can be returned to the condensing chamber or stored in tanks for reuse at a latter date.

A schematic of the cryostat design developed under this program is shown in Figure 3. The cryostat is enclosed in a vacuum can, I, that is immersed in the 4.2 K bath. The ^{4}He pot, G, inside the vacuum can, is filled with liquid and pumped down to 3 K or lower. The ^{3}He gas from the storage tank, L, is then bled into the condenser, F, which is in thermal contact with the ^{4}He pot and condensed. A capillary tube connects the condenser with the ^{3}He pot, E. Once the pot is filled with liquid ^{3}He, the closed cycle diffusion pump and mechanical pump, J and K, reduce the pressure in the pot to 10^{-3} Torr or lower, lowering the sample

A: Sample
B: Sample Mount
C: Ge Thermometers
D: Cu Sinter
E: He-3 Pot
F: He-3 Condenser
G: He-4 Pot
H: Needle Valve
I: Vacuum Can
J: Diffusion Pump
K: Sealed Mechanical Pump
L: He-3 Storage Tank
M: Superconducting Magnet
N: He-3 Return Line

Figure 3 ^{3}He Cryostat
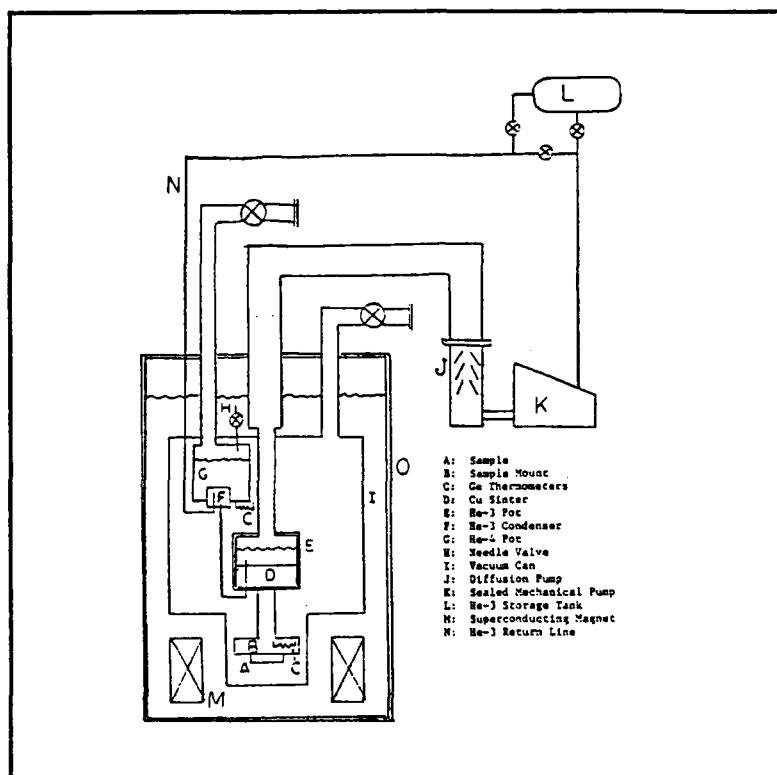
temperature to 0.5 K. By proper valving, the ^{3}He gas is returned to the condenser from the back of the sealed mechanical pump for continuous operation. Ge resistance thermometers, C, on the sample stage and the ^{4}He pot monitor the process.

The ^{3}He pot is filled with sintered copper powder, D, to increase the surface area in contact with the liquid. This is necessary because the Kapitza resistance can be quite high at very low temperatures. This is discussed in Richardson and Smith[9] on page 159. To ensure that the pump can get the ^{3}He pot down to 10^3 Torr, a resistance between the condenser and the pot is required. This is made by inserting a stainless steel wire into the stainless steel capillary tube connecting the condenser and the pot. Since the resistance has to be quite high, the wire should be the same diameter as the inside diameter of the capillary and about 6" total length is required.

The gas handling panel for the ^{3}He is shown in Figure 4. Two storage tanks, S1 and S2, are used so that gas can be removed from one while it is being returned to the other. Initially, the gas is bled into the ^{3}He pot of the cryostat directly through the pump line from tank S1 via valve 7 and the diffusion pump outlet A. Once the charge is condensed, valves 7 and 19 are closed and valves 1, 8, 10-12 are opened, the pumps are turned on and the gas is returned to the panel via line B through valve #1. With valves 2, 4, 13 and 14 closed, the gas is cycled through the LN$_2$ cold trap to remove any pump oil or other mists that might have collected in the gas lines and then returned to the condenser in the cryostat through

valve 12 and line C. The pressure gauges on either side of the cold trap monitor the pressure to indicate if the trap is filling up. The pressure relief valve, 4a, ensures that the gas is returned to the storage tanks should there be a catastrophic evaporation, from a loss of vacuum or some other system failure. At the end of the run, the gas is returned to the opposite storage tank by closing valves 10-12 and opening valve 4 with the pumps running. The other access ports are for pumping out the system, leak checking or back filling with inert gas.
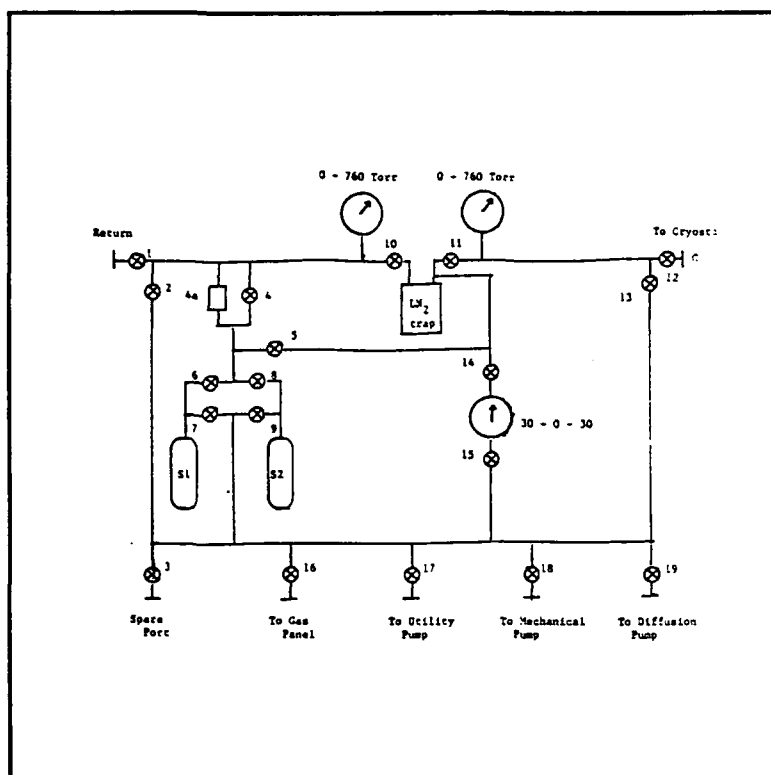
Figure 4 ^{3}He Gas Handling Panel

5. COMPUTER CONTROL OF THE EXPERIMENT

We developed a complete computer control system for this experiment. We employ a 486-based PC with a IEEE-488 bus controller. All of the instruments used to take the data, control temperatures, ramp and monitor the magnetic field, and plot the results are controlled by the computer.

The computer program first allows the user to set the important experimental parameters - AC measurement frequency, sample current, temperature, number of data points, starting and ending magnetic field, inverse or direct field, etc. The controller then sends the requisite parameters to the instruments and performs the necessary calculation for the current ramp for

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the magnet. After stabilizing the temperature the data is taken in the following sequence. First the magnetic field is ramped to the proper level. The temperature is then checked and the controller sent the needed information to re-stabilize the temperature. Then the Hall and longitudinal voltages are measured with two lock-in amplifiers and recorded on disk. The entire sequence is then repeated until the required number of points is taken. The field is then ramped down to zero and the data is plotted.
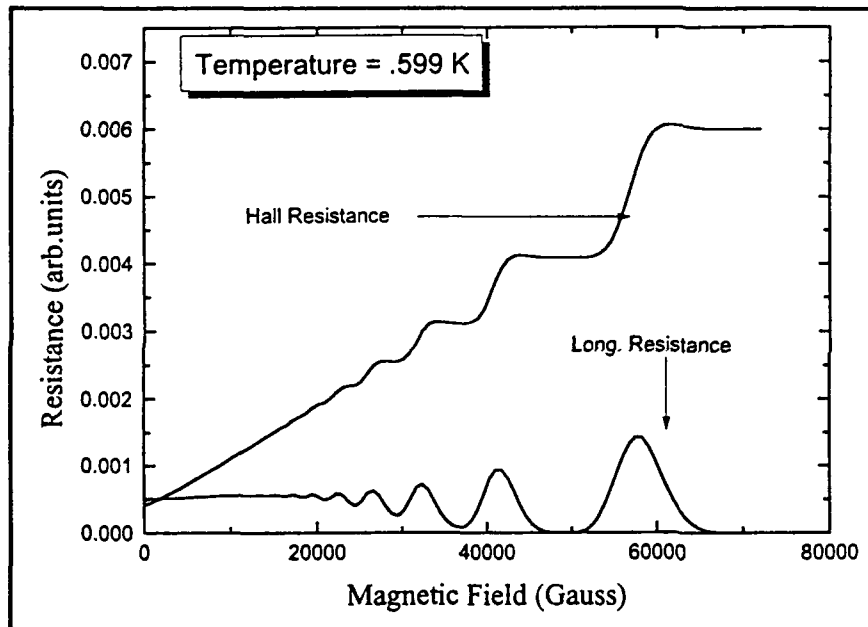
6. SHUBNIKOV - DE HAAS RESULTS

Measurements were made on several samples of GaAs/AlGaAs heterostructures provided by Wright-Patterson Air Force Base. Measurements down to 0.4 K and fields as large as 9 T were made. Typical results are shown in Figure 5. Shubnikov-de Haas oscillations are clearly seen in the data, and are sharper and clearer than the oscillations previously seen in these samples at higher temperatures and lower fields. . This is indicative of full operation of the system which now meets and exceeds all specifications. Measurements are currently being extended to other systems being provided by Wright-Patterson Air Force Base.

Figure 5 Typical results of a Shubnikov-de Haas experiment.

7. CONCLUSIONS AND RECOMMENDATIONS

Temperatures below 1.0K can significantly improve the resolution of Shubnikov - de Haas effect experiments. The ^{3}He system developed here has been shown to be a useful tool in the material evaluation of such heterostructures, particularly in the evaluation of effective masses and scattering times. The lower temperatures obtainable with ^{3}He compared with the 1.1K limit of ^{4}He systems permits the measurement of SdH effect in samples of marginal quality. This is most valuable during the development of new materials when growth processes have not been optimized. Even on higher quality samples, the extended temperature range over which the SdH effect can be measured with a ^{3}He system improves the accuracy of effective mass and scattering time measurements, allowing for the investigation of more subtle effects, such as variation in effective mass with strain.

New studies of SdH effect in strained p-type structures such as the InGaSb-InAs system or the Si-Ge system should be undertaken to verify if the strain does in fact lift the light hole mass above the heavy hole mass. Some measurements of mobilities in p-type structures have suggested that this is the case but a measurement of the light hole effective mass would conclusively decide this issue. Studies of mercury cadmium telluride superlattices should also be undertaken. The quality of these soft II-IV structures is not as good as similar structures in the III-V system and the lower temperatures obtainable here might permit the measurement of SdH oscillations.

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