### A NEW INSTRUMENT TO MEASURE

## FAR INFRARED REFLECTANCE AND TRANSMITTANCE

By

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A Thesis

Submitted to the School of Graduate Studies

in Partial Fulfillment of the Requirements

for the Degree

Master of Science

McMaster University

October 1996

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# MASTER OF SCIENCE (1996) McMaster University (Physics) Hamilton, Ontario TITLE: A New Instrument to Measure Far Infrared

Reflectance and Transmittance

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NUMBER OF PAGES: vii, 73

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#### ABSTRACT

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A new instrument to measure far infrared transmittance is described. The operation of a Martin-Puplett spectrometer which provides the light for the instrument is reviewed. The data acquisition is also discussed as is the method used to extract transmittance spectra from the raw data. A detailed description is given of the cryostat which holds the detector and the sample. The detector is a silicon bolometer with an operating temperature of 0.3 K, and the sample can be cooled to temperatures between 1.2 K and 100 K. Provision has also been made to allow application of a magnetic field as high as 18 T using the high field superconducting magnet at the University of Toronto. In the future, an insert will be built to allow the instrument to measure reflectance.

In order to test the apparatus, transmittance measurements with no applied magnetic field were made on a CuGeO<sub>3</sub> single crystal. The same crystal was measured using a previously well tested Fourier transform interferometer and a 1.2 K bolometer. Comparisons of the two measurements were made, and reasonable agreement was obtained. Two different filters were used to demonstrate their effect on the range of frequencies over which the transmittance can be measured. Measurements down to 5 cm<sup>-1</sup> was easily achieved.

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#### ACKNOWLEDGEMENTS

I would like to thank my supervisor, Professor T. Timusk, who came up with the initial design for the cryostat and who provided me with plenty of advice for the many modifications and additions which were made. I also thank the machinists at the McMaster University machine shop, Clee Berwick and Michael Roberts, whose high quality work was absolutely vital to the success of the project.

I would also like to thank Andy Duncan for all the work he put into the computer program and Anton Puchkov for writing the Fourier transform routines. Thanks also go to Carlos Zarate for his help with the spectrometer and H.A. Dabkowska for providing the CuGeO<sub>3</sub> crystal which was used to test the instrument. I thank in advance D.A. Bonn for his efforts with the reflectance insert and T.E. Mason for making his 18 T magnet available.

I also thank all the other people whose advice was helpful at one time or another, Gord Hewitson, Dimitri Basov, Tatiana Startseva, Rob Hughes, Andy Duft, Jim Garret, and others. Finally, I would like to thank my parents for their unconditional support and love.

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#### 1. INTRODUCTION

This thesis is concerned with the design, construction, and initial testing of a new apparatus to perform reflectance measurements in the far infrared. Reflectance is simply the fraction of light energy which reflects off the surface of a material as a function of frequency. If the reflectance is known at all frequencies, it is possible to calculate the optical constants for the material such as the index of refraction, the dielectric function, and the optical conductivity. Knowing these can provide valuable insights into the nature of the material. The frequency of the light used determines the energy of processes which can be probed. The far infrared corresponds to energies that are relevant to collective states, such as superconductivity, which are among the most interesting states of matter.

All of these collective states, however, occur at low temperatures making it necessary to be able to do reflectance measurements with a helium cooled cryostat. It is also true that many collective states have magnetic properties so that the ability to apply a magnetic field is desirable. Fields as high as 18 T can be achieved, but this capability has never been combined with low temperature far infrared reflectance measurements. These magnetic fields correspond to energies which can be probed by far infrared light. The ultimate goals for the new system are to allow far infrared reflectance measurements at low temperatures and high magnetic fields. The use of a Martin-Puplett spectrometer and a 0.3 K detector should allow measurements from about 120 cm<sup>-1</sup> down to 2 cm<sup>-1</sup>. The experiment takes place in a cryostat submerged in liquid helium making temperatures down to 1.2 K possible. The sample chamber has also been designed to fit inside a high field superconducting magnet which reaches fields as high as 18 T. As a first step, the cryostat has been tested in a configuration which allows measurement of far infrared transmittance which is experimentally somewhat simpler than reflectance.

Figure 1.1 shows a diagram of the overall setup. A computer sets the position of the movable mirror of a Martin-Puplett spectrometer. The light is chopped as it leaves the spectrometer and enters the cryostat where the light is transmitted through the sample and then detected. The detected signal is sent to a lock-in amplifier which, since it is locked to the chopper frequency, is able to eliminate much of the signal's noise. Finally the signal is returned to the computer which moves the mirror one step, and the cycle is repeated. The data collected as the mirror moves over its entire range is called an interferogram. By taking the Fourier transform of the interferogram, a spectrum of transmitted light as a function of light frequency is obtained.



Figure 1.1 A computer moves the mirror of a Martin-Puplett spectrometer. The light from the spectrometer is transmitted through a sample and detected inside the cryostat. The lock-in amplifier removes noise from the signal.

In chapters two, three, and four the operation of this system will be described in detail. In chapter five the results of initial testing will be reported, including far infrared transmittance data on the spin-Peierls compound CuGeO<sub>3</sub>. This data will be compared with measurements of the same crystal made on an existing, well tested Fourier transform spectrometer. Finally, in chapter six, future plans for the instrument are discussed, including the capability to measure reflectance and apply high magnetic fields to the samples.

#### 2. MARTIN-PUPLETT SPECTROMETER

The Martin-Puplett spectrometer provides the light which is transmitted through the sample. Within the spectrometer the light is polarized and manipulated in a way similar to a Michelson interferometer which allows a spectrum to be obtained by taking the Fourier transform of the light intensity as a function of the position of a movable mirror. The spectrometer functions well from  $2 \text{ cm}^{-1}$  to about 125 cm<sup>-1</sup>.

#### 2.1 Description

The Martin-Puplett spectrometer (Martin and Puplett, 1969) is depicted in figure 2.1. Light produced by a mercury arc lamp is directed by a parabolic mirror into the input polarizer. The input polarizer consists of many wires stretched side by side over a circular frame. The wires are oriented approximately 45° from vertical. Only the component of the light with the electric field vector perpendicular to this direction passes through the polarizer since the conducting wires will reflect light with the electric field parallel to them.



Figure 2.1 The Martin-Puplett spectrometer shines light onto a beam splitter which splits the light into vertically and horizontally polarized components. The components are recombined after reflecting off roof mirrors.

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The light leaving the input polarizer, now linearly polarized at 45°, next comes to the beam splitter, also a series of wires stretched over a circular frame but this time with the wires oriented vertically. Since the incoming light is polarized 45° from vertical, half the light will pass through the beam splitter and have horizontal polarization, and the other half will be reflected with vertical polarization. The beam splitter is tilted so that the directions of travel of the transmitted and reflected beams are perpendicular to each other.

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Each beam comes to a roof mirror which reflects the beam back on itself while rotating its polarization by 90°. The beam which passed through the beam splitter with horizontal polarization is reflected back toward the beam splitter with vertical polarization. When it reaches the vertically oriented wires of the beam splitter it is reflected toward the chopper. Similarly the beam which originally reflected off the beam splitter with vertical polarization is reflected back toward the beam splitter with horizontal polarization. It passes through the beam splitter and is recombined with the other now vertically polarized beam traveling toward the chopper.

The relative phase of the two beams depends on the difference between the distances traveled by the beams. One of the roof mirrors is mounted on a translation stage so that this path difference can be adjusted by moving the mirror.

Consider for a moment the situation where the light source produces only one frequency. When the path difference is zero or a multiple of the wavelength of the light, the two beams are in phase. Since their polarizations are perpendicular to each other, the combination of the two beams is linearly polarized 45° from the vertical. If the path difference is increased from zero to one quarter of a wavelength, the phase difference goes from zero to 90°. This causes the polarization to go from linear to circular. If the phase difference is increased again to 180°, the polarization goes from circular back to linear, but this time perpendicular to the original polarization. Increasing the phase difference another 180° reverses the process bringing the polarization back to the original state. See figure 2.2 for a summary.

In order to detect these changes in polarization, the recombined beam passes through a chopper which is a series of wires stretched over a rotating circular frame. In any given position the chopper only transmits the component of the beam with the electric vector perpendicular to the chopper's wires. Each time the chopper rotates through 180° it samples the degree of linear polarization of the beam in all possible directions. If the beam is completely linearly polarized, as it is for zero phase difference, the transmission goes from 100% to zero and back to 100% as the chopper rotates through 180° since the electric vector goes from being perpendicular to the wires to being parallel and back to being perpendicular again. If the beam is circularly polarized, as it is for 90° phase difference, then 50% of the light is transmitted no matter what the angle of the chopper. Hence, in going from zero phase difference to 90° phase difference, the light transmitted through the chopper goes from maximum to zero modulation. At 180° phase difference, the modulation is at a maximum again, but the phase of the modulation is 180° relative to that of the modulation at zero phase difference.

The degree of modulation is a direct indication of the phase difference between the two beams. If the phase difference is known as a function of path difference which is given by the mirror position, then the frequency of the light can be calculated. The degree of modulation is measured using a lock-in amplifier locked to the frequency of the chopper with the phase set to read maximum modulation at zero path. If the light source produced a single frequency, then scanning the mirror would cause the lock-in signal to be a cosine. Taking the Fourier transform of this cosine would give the light frequency. If the light source produces a spectrum of many frequencies, then the lock-in signal as a function of mirror position, called an interferogram, is a superposition of several cosines. Taking the Fourier transform of the interferogram separates the cosines and gives the light intensity as a function of frequency.



Figure 2.2 If the light source produces a single frequency, then the relationship between the phase difference between the separate beams, the polarization of the recombined beam, and the lock-in amplifier signal is relatively simple.

2.2 Alignment

The first step in aligning the Martin-Puplett interferometer (Lambert and Richards, 1978) is to adjust the input parabolic mirror and the tilt control of the stationary roof mirror so that, with the beam splitter removed, the light reflecting off the roof mirror is focused back onto the source. With a mylar beam splitter installed, it should be possible to look at the scanning roof mirror through the beam splitter and see the image of the roof line of the stationary mirror. The scanning mirror is in the position of zero path difference when it is coincident with this image.

The next step is to adjust the tilt, lateral sheer, and rotary sheer of the stationary roof mirror illustrated in figure 2.3. If a flat mirror is used to direct the beam of a He-Ne laser into the spectrometer in place of the source, and a piece of white paper is placed into the recombined beams which leave the beam splitter, it should be possible to see two separate lines caused by the roof lines of the two mirrors. These lines must be made coincident for correct alignment. The tilt and lateral sheer should be iteratively adjusted with the laser aimed down the optical axis, while the rotary sheer should be adjusted with the laser beam as far from the optical axis as possible while still producing two lines on the white paper. The lines should remain coincident even when the white paper is moved closer to or farther from the beam splitter.



Figure 2.3 A roof mirror has two perpendicular flat reflecting surfaces and rotates polarization by 90°. The stationary roof mirror has three degrees of freedom including tilt, rotary sheer, and lateral sheer.

At this point, the spectrometer is ready to operate in polarizing mode. The He-Ne laser and the mylar beam splitter are removed, and the polarizing beam splitter is installed. The mercury arc lamp and the chopper are turned on, and the light is directed into a far infrared detector. The signal from the detector goes to a lock-in amplifier which is locked to the chopper frequency. The mirror is scanned to find the peak corresponding to zero path difference, and this signal is maximized by adjusting the tilt, lateral sheer, and rotary sheer bearing in mind that adjusting the tilt may change the position of zero path difference.

It is also necessary to adjust the phase of the lock-in amplifier and the angle of the input polarizer. The lock-in phase is first adjusted so as to minimize the interferogram modulation, and then increased by 90°. This is the correct phase, but the resulting interferogram has a baseline which in probably not zero. The baseline is the signal far from zero path difference and must be adjusted to zero by changing the angle of the input polarizer even if this decreases the peak modulation. This tradeoff is made since the noise produced by the light source is larger than that produced by the detector. In systems where the reverse is true, maximizing the modulation is more important than having zero baseline. Figure 2.4 shows the signal when the lock-in amplifier is at the correct phase and when it is 90° from this phase.



**Figure 2.4** The lock-in amplifier phase is adjusted for minimum modulation giving a signal like the top graph and then increased by 90° producing a signal like the bottom graph. The baseline should be minimized by rotating the input polarizer.

#### 3. COMPUTER PROGRAM

The computer used in the system is a 90 MHz Pentium with 24 megabytes of RAM and a 300 megabyte hard drive running DOS 6.2 and Windows 3.1. The software is written in a programming language called Labview version 3.1.1. The computer has two main functions. First, it must coordinate data acquisition with the motion of the scanning mirror, and second, it performs preliminary data manipulation such as calculation of Fourier transforms.

#### 3.1 Stepper Motor Control

The stepper motor which moves the scanning mirror is controlled by a Hurst EPC-013 motor control board which moves the motor one step every time it receives a pulse. The pulses are sent by a National Instruments PC-TIO-10 timer card which has ten counters. The computer program provides the user with two modes for stepping the motor. The first is a manual mode in which the timing is controlled by the computer. This mode allows the user to quickly move the mirror to any position and to perform a rough scan in order to find the location of zero path difference. Once it has been located, data acquisition can be done using the second mode which turns over the timing of the scan to the timer card.

The computer configures the timer card to produce four signals which are shown in figure 3.1. The first simply acts as a gate for the second. As long as the first signal remains high, the second signal consists of a series of downward and upward edges with one pair of edges for each data point. The downward edge triggers a delayed single pulse in the third signal, while the upward edge triggers the lock-in amplifier to collect data. The fourth signal is the 300 Hz series of pulses which actually moves the mirror. Each pulse causes the motor control board to move the motor one step. These pulses are gated by the pulses in the third signal which are just long enough to pass the right number of pulses to move the mirror from the location of one measurement to the next.

In the timer controlled mode, the time between the end of the burst of pulses which moves the mirror and the triggering of the lock-in amplifier to take data is kept constant. This is vital because it takes time for the system to reach equilibrium after the mirror is moved. Vibrations need to settle down, and the bolometer needs to adjust to any large changes in signal. If the time allowed for this adjustment is not constant, then the measured interferogram may be affected. These precautions are not taken, however, in the manual control mode where the first two timer signals are not used. The computer simply triggers a pulse of the appropriate length in the third signal whenever the mirror is to be moved.



Bursts of Pulses to Move Mirror between Measurements



In both modes, once triggered the lock-in amplifier begins to sample data at a rate of 512 samples per second, storing the samples in a buffer. The more samples collected, the longer the integration time of the measurement. Once the lock-in amplifier starts collecting data, the computer repeatedly queries it to find out how many samples have been taken. When at least enough have been stored in the buffer, the computer downloads the correct number of samples from the buffer, clears the buffer, and enables the lock-in amplifier to be triggered again. The samples which have been downloaded to the computer are averaged together to produce a data point for the interferogram.

#### 3.2 Cutoff and Resolution

A perfect interferogram would be a continuous curve. In practice, it is only possible to collect a discrete set of data points separated by a finite distance. This means that any frequencies in the interferogram with a wavelength less than twice this distance will be missed. For example, if the mirror is moved 20  $\mu$ m between data points, then the path difference changes by 40  $\mu$ m since the light travels to the mirror and back. Any frequencies with wavelengths less than 80  $\mu$ m will be missed, and the spectrum will only extend to the cutoff frequency of 1/80  $\mu$ m = 125 cm<sup>-1</sup>. Since the spectrometer doesn't function well above this frequency anyway, 20  $\mu$ m is an appropriate step size. Another property of a perfect interferogram is that it extends to  $\pm\infty$ . Since the mirror can only be moved a finite distance, the true interferogram is actually truncated. The truncation can be thought of as an apodization function, A(x), by which the true interferogram is multiplied. For example, if x represents path difference, then A(x) could have a value of one between  $x = \pm L_1$  where data is actually taken, and a value of zero everywhere else. The inverse Fourier transform of the true interferogram  $\gamma(x)$  is the desired spectrum P( $\omega$ ).

$$\frac{2}{\pi}\int_{-\infty}^{\infty}\gamma(x)e^{-i\omega x}dx = P(\omega)$$
(3.1)

Since it is the product of  $\gamma(x)$  and A(x) which is measured, and since the Fourier transform of a product of functions is the convolution of the Fourier transforms of the individual functions, then what we actually get is

$$\frac{2}{\pi}\int_{-\infty}^{\infty}\gamma(x)A(x)e^{-i\omega x}dx = \left[P(\omega)\right]*\left[\frac{\sin(\omega L_1)}{2\pi\omega}\right]$$
(3.2)

The result is a convolution of the desired spectrum with a sinc function. This limits the resolution of the spectrum to about  $1/L_1$ . To increase the resolution, the mirror must be moved farther from zero path difference increasing the value of  $L_1$ .

#### 3.3 Fourier Transform

Yet another property of a perfect interferogram is that it is perfectly symmetric. This means that all the frequency components have the same location of zero path difference and that the Fourier transform of the interferogram is purely real. In general, it turns out that frequency dependent phase shifts  $\phi(\omega)$  are introduced by imperfections in the spectrometer (Porter and Tanner, 1983) so that

$$\frac{2}{\pi}\int_{-\infty}^{\infty}\gamma(x)A(x)e^{-i\omega x}dx = \left[P(\omega)e^{i\phi(\omega)}\right]*\left[\frac{\sin(\omega L_1)}{2\pi\omega}\right]$$
(3.3)

These phase shifts can be caused by differences between the two arms of the spectrometer either due to misalignment or to imperfections in the optics.

Phase error can also be caused by the finite separation between data points. It is likely that no data point will fall exactly at zero path difference so that zero path difference does not correspond to the interferogram maximum. If the assumed location of zero path difference is not correct then a phase error proportional to frequency is introduced. This type of error can be minimized by fitting a quadratic curve to the interferogram maximum and the points on either side. If the remaining phase error is assumed to be a slowly varying function of  $\omega$ , then it can be moved outside the convolution in equation 3.3 giving

$$\frac{2}{\pi}\int_{-\infty}^{\infty}\gamma(x)A(x)e^{-i\omega x}dx = e^{i\phi(\omega)}\left\{\left[P(\omega)\right]*\left[\frac{\sin(\omega L_{1})}{2\pi\omega}\right]\right\}$$
(3.4)

The convolution of the spectrum with the sinc function is easily obtained by calculating the amplitude of equation 3.4.

In practice, there is another complication. In order to get higher resolution, the apodization function used in not usually A(x), but B(x) which has a value of one between  $x = -L_1$  and  $x = L_2$  where  $L_2$  is larger than  $L_1$ . This corresponds to scanning the mirror farther on one side of zero path difference than the other. This is an asymmetric function with a complex Fourier transform. The real part of the Fourier transform comes from the even part of B(x) while the imaginary part comes from the odd. These are shown in the upper half of figure 3.2. It is not obvious how to proceed, but one method is to multiply the interferogram by a ramp which goes linearly from zero at  $x = -L_1$  to one at  $x = L_1$ and has a value of one between  $x = L_1$  and  $x = L_2$ . This changes B(x) to a new apodization function, C(x). C(x) and its even and odd parts are shown in the lower half of figure 3.2.

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Figure 3.2 The asymmetric apodization functions, B(x) and C(x), can be divided into even and odd parts. The even part of C(x) is just a symmetric high resolution truncation.
Notice that the even part of C(x) is just a symmetric truncation like A(x) but with higher resolution since it extends between  $x = \pm L_2$ . This gives

$$\frac{2}{\pi}\int_{-\infty}^{\infty}\gamma(x)C(x)e^{-i\alpha x}dx = e^{i\phi(\alpha)}\left\{\left[P(\alpha)\right]^*\left[\frac{\sin(\omega L_2)}{2\pi\omega} + \operatorname{Im}\left(\frac{2}{\pi}\int_{-\infty}^{\infty}C(x)e^{-i\alpha x}dx\right)\right]\right\}$$
(3.5)

Unlike equation 3.4, the phase error cannot be removed by simply finding the amplitude because of the presence of the imaginary part of the Fourier transform of C(x). The solution is to use the low resolution interferogram with A(x) to calculate the phase error  $\phi(\omega)$ . This is used as an approximation for the high resolution phase. By taking the Fourier transform of the interferogram with C(x), multiplying it by  $e^{-i\phi(\omega)}$ , and taking the real part, the high resolution spectrum is obtained.

$$\operatorname{Re}\left\{e^{-i\phi(\omega)}\frac{2}{\pi}\int_{-\infty}^{\infty}\gamma(x)C(x)e^{-i\omega x}dx\right\} = \left[P(\omega)\right]^{*}\left[\frac{\sin(\omega L_{2})}{2\pi\omega}\right]$$
(3.6)

The Fourier transform routines used in the program require interferograms with the number of points equal to a power of two. To accommodate this, both the low and high resolution interferograms are placed into arrays with  $2^n$  points with n the smallest positive integer such that  $2^n$  is greater than or equal to twice the number of points between x = 0 and  $x = L_2$ . The unfilled portions of the arrays are filled with zeros. A symmetric apodization function which goes smoothly to zero at  $x = \pm L_1$  or  $x = \pm L_2$  is usually applied to remove any sudden jumps between the data and the artificially added zeros. The data is then shifted so that x = 0 is the first point in the array. Appendix A contains a seven step summary of the procedure for obtaining a high resolution, phasecorrected spectrum (Porter and Tanner, 1983).

# 4. THE CRYOSTAT

Figure 4.1 shows a schematic of the cryostat. Light is guided by a polished brass pipe to the bottom of the cryostat where it enters a sample chamber. The sample chamber currently contains a transmittance insert which guides the light through a polarizer and a sample. The light then enters a bolometer chamber where it is detected by a He<sup>3</sup> cooled bolometer. The entire cryostat sits in a dewar with its lower half submerged in liquid helium.

# 4.1 Transmittance Insert

The transmittance insert which has been built to fit inside the sample chamber is shown in figure 4.2. A polished brass pipe directs the light to the bottom of the insert where it is reflected up into a gear box. The gear box contains sample and polarizer wheels which can be independently rotated to place different samples into the path of the light and to change the polarization of the light hitting the samples. The rotation is accomplished by turning 1/4 inch thin wall stainless steel tubes which extend to the top of the cryostat. After being transmitted through the sample the light is directed by a polished brass pipe out of the sample chamber and to the bolometer.



Figure 4.1 Light enters the cryostat and is guided down into the sample chamber where it interacts with the sample. It is then guided up out of the sample chamber, through a filter, and into the bolometer chamber to be detected.



**Figure 4.2** The transmittance insert fits inside the sample chamber. Light guided by polished brass pipes reflects off a mirror at the bottom of the insert, passes through a polarizer and sample, and exits the sample chamber.

It is important to be able to polarize the light which interacts with the sample since many materials of interest are anisotropic. However polarizing the light decreases its intensity by a factor of two and it may be desirable to use unpolarized light, especially if the sample is isotropic. The polarizer wheel is shown in figure 4.3. Polarizing film is stretched over the long semicircular slot which covers half the wheel, and the hole on the other half is left open. When the wheel is turned, different parts of the semicircular slot are rotated into position allowing all possible polarizations. The hole on the other half of the wheel can also be rotated into position in order to let the light pass through unpolarized.

The sample wheel has five positions where samples can be mounted and is shown in figure 4.3. It consists of a stainless steel gear, a thermally insulating Teflon layer, a brass wheel onto which the samples are mounted, and a thin brass plate which holds the samples in position. The brass wheel is wrapped with constantan wire through which current is passed to raise the sample temperature above 1.2 K when desired. For redundancy, two thermometers are mounted on the wheel to monitor the sample temperature. The thermometers have temperature dependent resistances and resistance to temperature conversion tables are contained in Appendix B. All electrical wires as well as the stainless steel rods are thermally anchored to the He<sup>4</sup> bath to keep them from conducting heat from the top of the cryostat to the sample wheel.



Figure 4.3 The gear box contains two wheels which can be rotated via gears. The polarizer wheel allows light to be polarized at any angle. The sample wheel allows different samples to be rotated into position.

4.2 Silicon Bolometer

The device used to detect the light is a silicon bolometer. A bolometer has a resistance which is very sensitive to temperature. When light strikes the bolometer, it is absorbed causing a small increase in the bolometer temperature which is reflected by a change in resistance. This change in bolometer resistance is a direct indication of the intensity of the light. In order for the bolometer to function properly, the detected light must be properly filtered and the bolometer must be at the proper temperature and bias.

4.2.1 Filters

Before entering the bolometer chamber, the light passes through filters designed to allow only certain frequencies of light to reach the bolometer. There are places for up to eight different filters on a filter wheel which can be rotated to bring different filters into place. The wheel is rotated by turning a 1/4 inch thin wall stainless steel tube which extends to the top of the cryostat. The filter wheel is in a box which is not sealed so that the filters are actually in direct contact with the liquid helium in which the cryostat is submerged. This means that the filters remain cold even when exposed to thermal radiation from the heated sample. Were they to warm up, they would reradiate this radiation toward the bolometer. The filters allow measurements to be made down to very low frequencies where the amount of light produced by the spectrometer rapidly decreases with decreasing frequency. The intensity of the light produced by the lamp fluctuates which introduces noise into the measurements. The ratio of intensity to noise is similar over all frequencies which means that the largest fluctuations occur at the most intense frequencies. If the light was not filtered, then these large fluctuations would dominate the noise in the bolometer signal. These noise levels may be insignificant at some frequencies, but they become comparable to the signal at the lowest frequencies. The way to make low frequency measurements is to filter out higher frequencies so that only the relatively small fluctuations of the low frequency light contribute to the noise.

Of the eight positions in the filter wheel, three are currently occupied. One contains a brass plug which completely blocks the light. This allows bias tests to be done on the bolometer and provides a check that no light leaks around the filter wheel. Another position contains a piece of gray plastic from the cap of a Kodak film container. This material cuts off frequencies above 120 cm<sup>-1</sup>. The other filter is a 1/128 inch thick glass microscope slide cover slip with a cutoff of 60 cm<sup>-1</sup>. These filters become transparent at high frequencies, so a piece of black polyethylene which cuts off frequencies above 1000 cm<sup>-1</sup> is also used. It is placed before the sample to prevent heating of the sample by ambient radiation. 4.2.2 Cooling System

The operating temperature of the bolometer is 0.3 K. Such a low operating temperature gives the high sensitivity needed to measure weak low frequency signals, but it also necessitates the fairly complicated cooling system shown in figure 4.4 which involves two isotopes of helium, He<sup>4</sup> and He<sup>3</sup>. Pumping on a chamber containing liquid He<sup>3</sup> causes its temperature to drop to the required temperature of 0.3 K. Figure 4.4 shows the liquid He<sup>3</sup> chamber thermally connected via a copper plate to the bolometer. At room temperature, 50 psi of He<sup>3</sup> is stored in gaseous form in a chamber at the top of the cryostat. It condenses down into the liquid He<sup>3</sup> chamber when the temperature of the chamber becomes about 1.2 K.

This is accomplished using liquid He<sup>4</sup> which, when pumped on, goes from 4.2 K to 1.2 K. The liquid He<sup>3</sup> chamber is connected via a 3/8 inch stainless steel tube to a charcoal absorption pump which is submerged in liquid He<sup>4</sup> outside the evacuated bolometer chamber. Although the part of the tube which is inside the bolometer chamber is thin walled, it still supplies some thermal contact between the liquid He<sup>3</sup> chamber and the He<sup>4</sup> bath. Hence, by pumping on the entire He<sup>4</sup> bath and bringing its temperature to 1.2 K, the liquid He<sup>3</sup> chamber will also be cooled causing He<sup>3</sup> to condense inside. Provision has also been made to allow condensation of He<sup>3</sup> without pumping on the He<sup>4</sup> bath. Figure 4.4 shows that there is a second small chamber contained within the bolometer chamber which can be filled with liquid He<sup>4</sup> from the bath by briefly opening a needle valve. The needle valve is operated by turning a 1/4 inch thin wall stainless steel tube which runs to the top of the cryostat. This He<sup>4</sup> chamber is thermally connected via a copper plate to the tube which comes from the liquid He<sup>3</sup> chamber. Once filled with liquid He<sup>4</sup>, the He<sup>4</sup> chamber can be pumped on through a 1/4 inch thin wall stainless steel tube to decrease its temperature and that of the liquid He<sup>3</sup> chamber to 1.2 K.

The pump used to decrease the liquid He<sup>3</sup> temperature to 0.3 K is a charcoal absorption pump. This pump, the liquid He<sup>3</sup> chamber, and the storage chamber are all connected to form a closed system. The absorption pump is surrounded by an insulating vacuum jacket and contains activated charcoal which absorbs He<sup>3</sup> when cooled below about 50 K. The pump is first heated above this temperature by passing current through wires wrapped around the pump. The temperature can be measured using a thermometer which is mounted on the pump. Once the He<sup>3</sup> is condensed into the liquid He<sup>3</sup> chamber, the current to the pump is shut off. The He<sup>4</sup> bath then slowly cools the charcoal so that it pumps on the He<sup>3</sup> decreasing its temperature to 0.3 K. The He<sup>3</sup> can be recycled by warming up the charcoal, recondensing the He<sup>3</sup>, and cooling down again.

chamber which is connected to the charcoal absorption pump. light onto the bolometer. The bolometer is in thermal contact with the liquid He<sup>3</sup> Figure 4.4 The bolometer chamber contains a polished brass cone which directs



There are three thermometers in the cooling system. Like the two on the sample wheel, they have temperature dependent resistances. Tables to convert resistances to temperatures for all five thermometers in the cryostat are contained in Appendix B. A step by step procedure for bringing the system from room temperature to 1.2 K and to cool the bolometer to 0.3 K is given in Appendix C. The wiring of the system is described in detail in Appendix D.

4.2.3 Bias Circuit

Like the thermometers in the system, the bolometer is a temperature dependent resistor. The difference is that the bolometer is far more sensitive and has a faster reaction time. The bolometer circuit is shown in figure 4.5. One end of the bolometer is attached to ground, and the other is connected to a 40 M $\Omega$  load resistor. A bias voltage is applied to the load resistor so that a small current flows through the resistor and bolometer which are in series. The voltage at the bolometer, V<sub>B</sub>, is thus proportional to the bolometer resistance.

In order to decrease the signal noise, the output impedance is decreased by using a JFET as a voltage follower with a 12 k $\Omega$  output resistor. The output of the JFET, V<sub>s</sub>, is equal to V<sub>B</sub> plus a small offset. The bias for the JFET is supplied by a 9 V battery. This battery also supplies current to a heating resistor which is in

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thermal contact with the JFET. This is necessary since the JFET will not operate at the low temperatures in the bolometer chamber where it is mounted. Its temperature must be raised to at least 60 K. Thermal isolation is accomplished by mounting the JFET and heater on the end of a low conductivity rod. This assembly is surrounded by a cold metal can lined on the inside with a black radiation absorbent coating. This prevents thermal radiation from the hot JFET from reaching the bolometer.

The bolometer itself is suspended by its two wires inside a brass hemispherical cavity. Light from the sample chamber is concentrated on the cavity by a polished brass cone. The bolometer is very small, but it absorbs much of the light as it bounces around inside the cavity. To increase the absorption without increasing the bolometer's size and thus its thermal time constant, the bolometer is attached to a 3 mm  $\times$  3 mm diamond substrate with a thin conducting layer of nichrome on one face. The diamond has a low heat capacity and high thermal conductivity, while the nichrome layer is an efficient absorber. The bolometer substrate combination is shown in figure 4.6.



Figure 4.5 The bolometer bias circuit applies a bias to a 40 M $\Omega$  load resistor which is in series with the bolometer. The voltage at the bolometer is the signal, but a JFET is used as a source follower to decrease the output impedance.



Figure 4.6 The bolometer is suspended by its wires inside a hemispherical cavity. To increase its effective area without increasing its thermal time constant, the bolometer is attached to a larger diamond substrate with a nichrome coating.

## 5. INITIAL TESTING

First, the bolometer itself was tested by measuring its DC load curve. Then the ability of the system to measure transmittance was tested by comparing measurements of CuGeO<sub>3</sub> with results from another transmittance apparatus.

### 5.1 DC Load Curve

The DC load curve can be measured by monitoring the voltage drop across the bolometer,  $V_B$ , as the bolometer bias voltage,  $V_{BIAS}$ , is changed.  $V_{BIAS}$ and  $V_B$  can be used to calculate the current through the bolometer,  $I_B$ , since the bolometer is in series with a 40 M $\Omega$  resistor. With the voltages measured in volts,  $I_B$  in nA is  $I_B = (V_{BIAS}-V_B)/0.04$ . From the DC load curve which is the plot of  $I_B$ against  $V_B$  shown in figure 5.1, important operating parameters for the bolometer can be deduced. The power dissipated in the bolometer, P, is given by the product of the current and the voltage at the point on the load curve where the bolometer is actually operated. The responsivity, S, which is a measure of how sensitive the bolometer is to light, is given by dividing V', the intercept of the line tangent to the curve at the operating point, by 2P. The thermal conductance, G, is given by dividing P by 0.1T where T is 0.32 K, the operating temperature of the bolometer.



Figure 5.1 The DC Load Curve is a good indicator of bolometer performance. It can be affected by both the bolometer temperature and the amount of background radiation, and is useful for choosing an appropriate bias voltage.

5.2 CuGeO3

CuGeO<sub>3</sub> is the first inorganic compound to exhibit a spin-Peierls transition. A spin-Peierls transition is the transformation of a system of uniform Heisenberg antiferromagnetic linear chains to a system of dimerized or alternating antiferromagnetic linear chains. Spin-phonon coupling between the one-dimensional spin and three dimensional phonon systems causes this dimerization to become energetically favorable below some temperature. The transition is second-order and has been observed in measurements of magnetic susceptibility (Hase *et al*, 1993) as well as in neutron and x-ray experiments (Pouget *et al*, 1994) to occur at 14 K in CuGeO<sub>3</sub>.

The crystal structure shown in figure 5.2 (Hirota *et al*, 1994) consists of antiferromagnetic copper-oxygen chains separated by germanium-oxygen chains. The changes in lattice positions in the copper-oxygen chains corresponding to the dimerization are also shown. The coppers move about 0.01 Å in alternating directions on the c axis along the chains doubling the unit cell in the c direction. The shifts in copper positions are accompanied by shifts in the positions of those oxygens which separate the coppers in the antiferromagnetic chains. These shifts are in the a-b plane and result in a doubling of the unit cell in the a direction.



Figure 5.2 CuGeO<sub>3</sub> consists of copper-oxygen chains separated by germaniumoxygen chains. Below the transition temperature, the atoms in the copper-oxygen chains shift causing a doubling of the unit cell in the a and c directions. Arrows represent the directions of the shifts (after Hirota *et al*, 1994).

CuGeO<sub>3</sub> also has interesting properties in an applied magnetic field. The magnetic phase diagram is shown in figure 5.3 (Hase *et al*, 1993). In the absence of a magnetic field, the transition to the dimerized phase occurs at about 14 K. This transition temperature decreases with increasing applied magnetic field until it reaches about 10 K at 12 or 13 T. Above this field, the dimerized phase is replaced by another magnetic phase. Transitions between the high temperature phase and the other phases are second order. The transition between the dimerized and high field phases is first order.

CuGeO<sub>3</sub> has been chosen as an appropriate material with which to do preliminary testing of the transmittance apparatus for three reasons. First, single crystals grown from CuO flux by H.A. Dabkowska at McMaster University have been made available to us. Second, the presence of features in the transmittance spectra in a magnetic field (van Loosdrecht *et al*, 1996) make it desirable for future magnetic field experiments. Third, the transmittance spectrum at zero field contains a large temperature dependent feature, a phonon, at about 50 cm<sup>-1</sup> which falls in the range of the apparatus. The transmittance from 20 cm<sup>-1</sup> to 110 cm<sup>-1</sup> for a sample measuring  $2 \times 3 \times 0.5$  mm was obtained using a far infrared rapid scan Fourier transform spectrometer and a 1.2 K bolometer and is shown in figure 5.4. These results will be taken as the true spectra and will be compared to measurements of the same crystal with the new apparatus.



Figure 5.3 The magnetic phase diagram of CuGeO<sub>3</sub> has three phases. A uniform phase exists at high temperatures while a dimerized phase exists at low temperatures. A third phase is also present at low temperatures and high field (after Hase *et al*, 1993).



Figure 5.4 The transmittance of CuGeO<sub>3</sub> features a strong phonon at 50 cm<sup>-1</sup>. This data was measured using a well tested system consisting of a rapid scan Fourier transform spectrometer and 1.2 K bolometer. The resolution is 5 cm<sup>-1</sup>.

#### 5.3 Results

The same CuGeO<sub>3</sub> sample that was measured with the 1.2 K bolometer was also used to test the new apparatus. Figure 5.5 shows one of the interferograms which were produced. This particular one consists of 119 data points with 20  $\mu$ m between points giving a cutoff frequency of 125 cm<sup>-1</sup> and a resolution of 5 cm<sup>-1</sup>. Each point is the average of data sampled by the lock-in amplifier over 0.6 s at a sampling rate of 512 samples per second. The amplitude of the signal at the peak is about 0.15 mV. The sample is oriented with its c-axis along the optical axis of the instrument, and since the a-b plane is nearly isotropic, no polarizer was used.

Spectra are obtained by taking the Fourier transform of the interferograms. Examples are shown in figure 5.6. Measurements are made of both the reference and the sample. The reference is a 1/16 inch diameter open hole, and the sample is the CuGeO<sub>3</sub> crystal seen through an identical 1/16 inch aperture. Results were obtained for two filters. The top graph shows spectra which were obtained with the filter which cuts off light above about 120 cm<sup>-1</sup>. The bottom graph shows spectra obtained with the filter which cuts off light above about 120 cm<sup>-1</sup>. The bottom graph shows spectra obtained with the filter which cuts off light above with the reference in place while the sample gives less signal since its transmittance is less than 100%.



Figure 5.5 This is an example of an interferogram produced by the new apparatus. This particular one will give a spectrum with a resolution of  $5 \text{ cm}^{-1}$  and a cutoff frequency of 125 cm<sup>-1</sup>.



Figure 5.6 The top spectra have a cutoff of about  $120 \text{ cm}^{-1}$  while the bottom spectra only extend to  $60 \text{ cm}^{-1}$ . The three spectra in each set correspond to the reference, the sample, and the leakage. The resolution is  $5 \text{ cm}^{-1}$ .

Transmittance, which is the fraction of light energy which is able to pass through the sample, is obtained by taking the ratio of the sample spectrum to the reference spectrum. For this to give correct results, however, all light reaching the bolometer must pass through the sample or reference. Errors are introduced if some light reaches the bolometer by a different path. In the two graphs in figure 5.6, the smallest curves are the signal when the sample wheel is rotated to a position which should completely block the light. Clearly, a substantial signal is still detected, particularly at lower frequencies. This is due to light leaking around the sample wheel or through empty holes in the sample wheel. This will eventually be corrected by making small modifications to the transmittance insert, but here this error will be accounted for by subtracting the leakage spectrum from the sample and reference spectra before taking the ratio.

Just as the sensitivity of the bolometer is affected by changing its bias voltage, it can also be affected by the average amount of radiation which reaches it. At reasonable bias voltages, changes in the size of the modulated signal do not significantly change the bolometer sensitivity, but the unmodulated radiation from the room temperature apparatus at the top of the cryostat, as well as that from the sample and reference when heated, can be significant. This is a problem if the bolometer sensitivity when the sample is in position is different from the sensitivity when the reference is in position. This can be checked for by simply measuring the DC bolometer voltage since this determines where the device is operating on the load curve. If the voltage is not the same with the sample as it is with the reference, then the measured transmittance will not be accurate. The problem can be solved by using filters which block high frequency thermal radiation. Increasing the bolometer bias voltage will also help. Table 5.1 shows the DC bolometer voltage for a bias voltage of 1 V. With the 60 cm<sup>-1</sup> filter, the values are the same for sample and reference at all temperatures. With the 120 cm<sup>-1</sup> filter though, the values are not the same. The bolometer is more sensitive when the sample is in position then when the reference is in position. Also, with this filter the bolometer becomes less sensitive with increasing sample wheel temperature so that measurements at 100 K are not even possible. Improvements could be made by increasing the bias voltage and adding filters, but for illustrative purposes, results will be presented without doing so.

Temperature	V <sub>B</sub> with 60 cm <sup>-1</sup> Filter		V <sub>B</sub> with 120 cm <sup>-1</sup> Filter	
	Sample	Reference	Sample	Reference
1.2 K	166 mV	166 mV	141 mV	123 mV
10 K	165 mV	165 mV	141 mV	122 mV
20 K	162 mV	162 mV	147 mV	139 mV
100 K	138 mV	138 mV	5.6 mV	5.2 mV

Table 5.1 Measurements of DC Bolometer Voltage, VB.

Figure 5.7 shows the 5 cm<sup>-1</sup> resolution CuGeO<sub>3</sub> transmittance results that were measured with the new apparatus. The upper graph shows data taken at 1.2 K, 10 K, and 20 K using the 120 cm<sup>-1</sup> filter. The lower graph shows data taken at 10 K, 20 K, and 100 K using the 60 cm<sup>-1</sup> filter. Notice that the 120 cm<sup>-1</sup> data extends down to 10 cm<sup>-1</sup> and the 60 cm<sup>-1</sup> data extends to 5 cm<sup>-1</sup>. These low frequency limits could probably be improved with more averaging.

In figure 5.8 the 20 K data taken with the new apparatus using the 120 cm<sup>-1</sup> filter is compared with the true transmittance at the same temperature. The data is in good agreement except that the transmittance measured with the new apparatus is about 15% too high. This, of course, is expected since, as discussed previously, improper filtering caused the bolometer sensitivity to be higher when the sample was in place than it was when the reference was in place.

More averaging would probably give smoother spectra, but the fringes which can also be seen in the true transmittance are a real effect caused by interference between multiple reflections off the parallel faces of the CuGeO<sub>3</sub> crystal. Figure 5.9 shows spectra with a cutoff of  $125 \text{ cm}^{-1}$  and a resolution of 1 cm<sup>-1</sup>. This high resolution is undesirable with this sample because the fringes, when fully resolved, tend to dominate the sample spectrum.



Figure 5.7 The top graph shows transmittance of  $CuGeO_3$  measured with a 120 cm<sup>-1</sup> filter at 1.2 K, 10 K, and 20 K. The bottom graph shows results with a 60 cm<sup>-1</sup> filter at 10 K, 20 K, and 100 K. The resolution is 5 cm<sup>-1</sup>.



Figure 5.8 A comparison of the  $20 \text{ K} 120 \text{ cm}^{-1}$  filter results with the true transmittance shows measurements to be about 15% too high. This is consistent with measurements of bolometer DC voltage which showed inadequate filtering.



Figure 5.9 At 1 cm<sup>-1</sup> resolution the sample spectrum is dominated by fringes. Since the fringes do not show up in the reference spectrum, the fringes must be caused by multiple reflections between the surfaces of the sample.

Figure 5.10 compares the 100 K 60 cm<sup>-1</sup> filter results with the true spectrum. As expected, the spectra are now in agreement since the bolometer sensitivity was the same for the sample and the reference. In the results at 10 K and 20 K, the phonon at 50 cm<sup>-1</sup> isn't quite resolved, but this is because it is near the filter cutoff where the signal is small. More averaging is necessary. In practice, the 60 cm<sup>-1</sup> filter spectra would be combined with the 120 cm<sup>-1</sup> filter spectra to produce transmittance spectra that extend over the entire range of the spectrometer.



Figure 5.10 There is good agreement between the 60 cm<sup>-1</sup> filter data and the true transmittance. Although this 100 K data only goes to 60 cm<sup>-1</sup>, it extends as low as  $5 \text{ cm}^{-1}$  or lower.

## 6. FUTURE IMPROVEMENTS

Although the new apparatus has been successfully used to measure transmittance, there are still several improvements which should be made. At least three of these are related to problems discovered during the initial testing. Two that have already been mentioned are the leakage of light around the sample wheel and the need to provide better high frequency filtering in conjunction with the 120 cm<sup>-1</sup> filter. Also, attempts to condense He<sup>3</sup> without pumping on the entire He<sup>4</sup> bath have been unsuccessful. It appears, however, that all of these can be solved with further investigation.

Besides this fine tuning, there are other improvements which must be made in order to achieve the original design goals for the instrument. Two are most important. One of these is the construction of a reflectance insert which, like the transmittance insert, will fit inside the sample chamber. This insert has already been designed by D.A. Bonn at the University of British Columbia and is currently being constructed. The other improvement to be made is the addition of a magnet to apply high fields to the samples.

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6.1 Reflectance

Initial testing was done using transmission experiments, but the main goal for the new cryostat is to perform reflectance experiments. In this configuration light is reflected off samples instead of transmitted through them. Since many samples, particularly conductors, are not transparent, reflectance measurements are desirable. Also, it is possible to obtain the complex optical constants for a material if the reflectance is known at all frequencies (Kittel, 1986).

When light reflects from a sample, both the magnitude and the phase of its electric field may be changed, and these changes will be frequency dependent. The ratio of the electric field after reflection to that before reflection is called the reflectivity coefficient,  $r(\omega)$ , and is given by

$$r(\omega) = \frac{E_r(\omega)}{E_i(\omega)} = \sqrt{R(\omega)}e^{i\theta(\omega)}$$
(6.1)

 $\theta(\omega)$  is the phase of  $r(\omega)$ , and  $R(\omega)$  is its squared amplitude. Knowing these allows the calculation of the various optical constants of the material. These include the index of refraction, the dielectric function, and the optical conductivity.
The complex index of refraction  $N(\omega) = n(\omega) + i\kappa(\omega)$  is given by

$$n(\omega) = \frac{1 - R(\omega)}{1 + R(\omega) - 2\sqrt{R(\omega)}\cos\theta(\omega)}$$
(6.2 a)

$$\kappa(\omega) = \frac{2\sqrt{R(\omega)}\sin\theta(\omega)}{1+R(\omega)-2\sqrt{R(\omega)}\cos\theta(\omega)}$$
(6.2 b)

The complex dielectric function  $\varepsilon(\omega) = \varepsilon_1(\omega) + i\varepsilon_2(\omega)$  is then given by

$$\varepsilon_1(\omega) = n(\omega)^2 - \kappa(\omega)^2$$
 (6.3 a)

$$\varepsilon_2(\omega) = 2n(\omega)\kappa(\omega)$$
 (6.3 b)

The optical conductivity is

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$$\sigma_1(\omega) = \frac{\omega \varepsilon_2(\omega)}{4\pi} \tag{6.4}$$

By comparing this to model dependent expressions for the optical conductivity one can get values for some fundamental properties of the material. For example, the Drude model which is used for free carriers in a metal, gives

$$\sigma_1(\omega) = \frac{ne^2\tau}{m^*(1+\omega^2\tau^2)} \tag{6.5}$$

where  $n, e, \tau$ , and  $m^*$  are the carrier density, electron charge, relaxation time, and effective mass respectively.

All of these calculation become possible once  $R(\omega)$  and  $\theta(\omega)$  are known. Reflectance measurements only give  $R(\omega)$ , but if  $R(\omega)$  is known over all frequencies,  $\theta(\omega)$  can be calculated using the Kramers-Kronig relation,

$$\theta(\omega) = \frac{1}{2\pi} \int_{0}^{\infty} \frac{\ln[R(s)] - \ln[R(\omega)]}{\omega^2 - s^2} ds$$
(6.6)

Of course, it is not possible for one device to measure reflectance at all frequencies. Fortunately, temperature and magnetic fields have little effect at high frequencies, so room temperature, zero field results are often sufficient. At very high frequencies, measured data is extrapolated to infinity using simple models. Just as an open hole is used as a reference to represent 100% transmittance, a reference to represent 100% reflectance can be created by coating the sample with a thin layer of a material like gold or aluminum which has nearly 100% reflectance. By dividing the reflectance spectrum from the sample by that from the coated sample, the effects of sample surface irregularities are removed. As with the transmittance measurements, it is important to have sample and reference measurements taken within a short time of each other so that long term drifts in the apparatus do not affect the results. Of course, it is impossible to quickly switch back and forth between sample and coated sample since they do not exist simultaneously. The solution is to use an intermediate reference, a stainless steel mirror (Cao, 1995).

Take the light reflected from the sample to be a product of three functions,

$$R_{sample} = R(t_1) R_{surface} R_{material}$$
(6.7)

where  $R(t_1)$  includes the effects of the apparatus at the time of a measurement on the sample,  $R_{surface}$  depends on the sample surface, and  $R_{material}$  is the reflectance of the sample material which we are after. If a stainless steel reference mirror is rotated into place immediately after the sample is measured, then

$$R_{reference} = R(t_1)R_{steel} \tag{6.8}$$

Dividing these two gives

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$$\frac{R_{surface}R_{material}}{R_{steel}}$$
(6.9)

The time dependent part has been removed so the sample need not be coated immediately. The process can be repeated at various times for all temperatures and magnetic fields of interest. Then, the sample is coated and everything repeated giving for each temperature and field

$$\frac{R(t)R_{surface}R_{coating}}{R(t)R_{steel}} = \frac{R_{surface}R_{coating}}{R_{steel}}$$
(6.10)

If  $R_{coating}$  is 100% then dividing equation 6.9 by equation 6.10 gives  $R_{material}$  as desired.

6.2 Measurements in Magnetic Field

In order to apply a magnetic field to the sample, a superconducting magnet has been purchased from Cryogenic Limited. The magnet has been tested up to a current of 100 A which produces a field of 7 T. The magnet has a two inch core so that it can fit around the sample can. A mounting bracket has been built to suspend the magnet at the correct height. The setup is shown in figure 6.1. The wires that carry the current to the magnet are copper but still have a nonzero resistance. This means that, unless they have a large diameter, they will dissipate a lot of heat which will boil off helium from the bath. On the other hand, a large diameter would cause a large thermal conductance from the room temperature end of the wire to the helium also causing boiloff. The magnet has a perpetual current mode in which the current is maintained in the magnet even if the current source is disconnected. This means smaller diameter wires can be tolerated since they will only carry current for short times while the field is changed.

To achieve higher fields, the cryostat can be taken to the University of Toronto where T.E. Mason has a high field superconducting magnet that reaches fields as high as 18 T. The cryostat has been designed to fit into the large dewar which contains the magnet. The sample chamber can be inserted into the magnet core which has a diameter of two inches.

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Figure 6.1 A 6 T magnet fits inside the dewar and can be suspended with the sample chamber inside the magnet core. To achieve 18 T fields, the cryostat can be placed in the high field magnet dewar at the University of Toronto.

## APPENDIX A

### PROCEDURE FOR GENERATING SPECTRA

- 1. Record an interferogram from  $x = -L_1$  to  $x = L_2$  with  $L_1$  extending beyond the main features and  $L_2 > L_1$  determining the final resolution. If there is uncertainty as to the exact location of x = 0, then extra data points can be recorded at  $x < -L_1$  and  $x > L_2$  to insure that enough points on both sides of x = 0 are available.
- Subtract the average value from the data points. This removes the zero frequency component of the interferogram corresponding to the baseline which should have been minimized to reduce noise.
- 3. Find the data point with the largest absolute value. Fit a parabola to this point and the points on either side of it. The location of the peak of the parabola is taken as the location of x = 0.
- 4. Select the portion of the interferogram that includes the desired number of data points to the left of x = 0 and the same number of points to the right of x = 0. Take the inverse complex Fourier transform as described in step 6.

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- 195 pt.

- 5. Select the portion of the interferogram that includes the desired number of data points to the left of x = 0 and the number of data points to the right of x = 0 that will give the desired resolution. Multiply this by a linear ramp that goes from zero at the left most point to one an equal distance on the other side of x = 0. Points to the right of this are unaffected. Take the inverse complex Fourier transform as described in step 6.
- 6. Multiply the interferograms by apodization functions which are symmetric about x = 0 in order to bring the ends smoothly to zero. Place both interferograms in arrays with 2m locations where  $m = 2^n$  is larger than the number of points to the right of x = 0 in the larger interferogram. The first location in each array should contain the first point to the right of x = 0 while the last location contains the first point to the left of x = 0. The empty locations in the center of the arrays are filled with zeros. Use a fast Fourier transform routine to calculate the inverse complex Fourier transforms.
- 7. The result of the inverse complex Fourier transform on the interferogram from step 4 is  $P(\omega)e^{i\phi(\omega)} = P_1(\omega) + iP_2(\omega)$  giving  $e^{-i\phi(\omega)} = (P_1-iP_2)/(P_1^2+P_2^2)^{1/2}$ . Take the real part of the product of this and the inverse complex Fourier transform of the interferogram from step 5 to get the phase corrected spectrum.

## **APPENDIX B**

# THERMOMETER RESISTANCE TO TEMPERATURE CONVERSION

 Table B.1 Resistance to Temperature Conversion.

	Resistance (Ω)				
T(K)	X03087	X03261	X02480	X03105	X03265
	He <sup>3</sup> Can	He <sup>4</sup> Can	Sample	Sample	<u>Charcoal</u>
0.30	8369	177837			
0.35	7841	160813			
0.40	7326	138477			
0.45	6831	112419			
0.50	6370	94048			
0.55	5939	80451			
0.60	5529	68940			
0.65	5139	58711			
0.7	4768	49789			
0.8	4092	36092			
0.9	3516	26998			
1.0	3042	20875			
1.1	2660	16538			
1.2	2354	13340			
1.3	2111	10946	20000		
1.4	1917	9158	15890		
1.5	1760	7835	12560		
1.6	1631	6861	10140	709000	
1.7	1526	6140	9000	633000	
1.8	1437	5597	7885	568000	
1.9	1362	5172	7295	502000	
2.0	1297	4823	6765	425500	
2.1	1241	4520	6270	370500	
2.2	1191	4247	5800	325000	
2.3	1146	3994	5340	286800	
2.4	1105	3761	4965	255000	
2.5	1068	3550	4650	225500	
2.6	1033	3362	4355	202800	
2.7	1001	3196	4090	181700	
2.8	971	3044	3850	166200	
2.9	942	2867	3630	152800	

T(K)	He <sup>3</sup> Can	He <sup>4</sup> Can	Sample	Sample	Charcoal
3.0	915	2733	3445	139400	
3.5.	798	2216	2730	91000	
4.0	705	1872	2265	62000	1750
4.5	631	1629	1940	44700	
5.0	573	1449	1700	32900	
5.5	526	1308	1520	25000	
6.0	489	1194	1380	21200	
6.5	459	1100	1265	18100	
7.0	434	1020	1170	15400	
8.0	394	893	1025	11900	
9.0	364	796	915	9600	
10	339	720	830 、	8020	
11	318	659	760	6860	
12	300	609	705	5860	
13	286	567	660	5210	
14	273	531	620	4570	
15	261	501	585	4190	
16	251	474	550	3780	
17	252	451	525	3460	
18	233	430	500	3120	
19	226	411	480	3020	
20	219	394	460	2710	
21	212	379	440	2600	
22	206	365	425	2450	
23	201	352	410	2240	
24	196	339	395	2150	
25	191	328	380	2100	
26	186	318	370	1980	
27	182	308	360	1800	
28	178	298	345	1710	
29	174	290	335	1640	
30	171	281	325	1550	
35	155	247	290	1280	
40	142	220	255	1050	
45	132	199	230	910	
50	123	182	210	790	
55	116	167	190	710	
60	109	155	180	620	
65	103	145	170	550	
70	98	136	160	500	130

#### APPENDIX C

#### **PROCEDURE FOR COOLING THE CRYOSTAT**

- With the bolometer and sample chambers sealed with indium and the transfer tube soldered on, place the cryostat into the dewar and bolt the top plate into position.
- 2. There are three separate chambers in the cryostat that need to be evacuated each having a separate valve at the top of the cryostat. Use the portable pumping station to pump them out one at a time, first roughing them out with the mechanical pump and then opening the diffusion pump. The three chambers are the sample chamber, the bolometer chamber, and the small He<sup>4</sup> chamber inside the bolometer chamber. To pump out the He<sup>4</sup> chamber, it is necessary to close the needle valve by turning the appropriate knob. The ion gauge should read less than 10<sup>-4</sup> torr for all three chambers.
- 3. The transfer tube is soldered to a pipe which extends up through the top plate. Fill the dewar with liquid nitrogen through this pipe and leave it over night. Add about 10 cubic centimeters of helium gas to the sample and bolometer chambers for better heat exchange.

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- 4. Pump the exchange gas out of the bolometer chamber and connect the pipe that leads to the transfer tube to a dewar that will collect the nitrogen. Connect the large pumping port on the dewar to a source of pressurized nitrogen gas. Force the liquid nitrogen up through the transfer tube by applying the pressurized gas to the liquid bath.
- 5. Connect the pumping port on the dewar to the helium gas collection system, and transfer liquid helium into the dewar through the transfer tube. With the pressure in the collection system at about 780 mmHg, it takes about 45 min. for the dewar to fill. The pressure usually increases when the dewar is full.
- 6. Connect the pumping port on the dewar to the large pump, and slowly decrease the pressure of the helium bath. It takes about one hour to completely pump the bath down and bring its temperature to 1.2 K.
- 7. When the temperature of the liquid He<sup>3</sup> chamber has stabilized at 1.2 K, apply about 10 V to the heater on the charcoal pump and monitor its thermometer. When the resistance gets down to 200  $\Omega$ , shut off the power to the heater. The He<sup>3</sup> which was absorbed on the charcoal will be released so that it can condense into the liquid He<sup>3</sup> chamber. When the He<sup>4</sup> bath recools the charcoal, it will pump on the liquid He<sup>3</sup> decreasing its temperature to 0.3 K. It takes about two hours for the charcoal to warm up and recool.

## APPENDIX D

### SYSTEM WIRING DETAILS

There are four connectors at the top of the cryostat, each with ten pins in the configuration shown in figure D.1. The connector for the bolometer and JFET is contained inside a blue metal box. Its wiring is given in table D.1. The wiring for the connector for the two thermometers in the bolometer chamber is given in table D.2. Table D.3 describes the connections for the sample chamber connector, and table D.4 is for the charcoal pump.

Figure D.1 Configuration of Connectors



 Table D.1 Wiring for the Bolometer and JFET

Pin	Group	Function	Wire Colour in Box
Α			
В			
C			
D			
E			
F			
H		9 V Bias	vellow
J	Bolometer	JFET Signal V <sub>S</sub>	green
K	and JFET	Bolometer Bias V <sub>BIAS</sub>	red
L		Bolometer Signal V <sub>B</sub>	white

Pin	Group	Function	Wire Colour on Cable
A			
В			
С		V-	white
D	He⁴ Can	V+	red
E	Thermometer	I-	green
F		I+	black
H		V-	white
J	He <sup>3</sup> Can	V+	red
K	Thermometer	I+	black
L		I-	green

 Table D.2
 Wiring for the Bolometer Chamber Thermometers

Table D.3 Wiring for the Sample Chamber

Pin	Group	Function	Wire Colour on Cable
A	Sample	V+/-	purple
В	Heater	V+/-	purple
С		V	white
D	Thermometer	V+	red
E	X03105	I-	green
F		I+	black
H		V-	white
J	Thermometer X02480	V+	red
K		I+	black
L		<u>I-</u>	green

 Table D.4
 Wiring for the Charcoal Pump

Pin	Group	Function	Wire Colour on Cable
A	Charcoal	V+/-	red
В	Heater	V+/-	green
С		V-	white
D	Charcoal	V+	red
E	Thermometer	I+	black
F		I-	green
H	Charcoal	V+/	white
J	Heater	V+/	black
K			
L		•	

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