A HIGH PRECISION REFLECTOMETER FOR SUBMILLIMETER WAVELENGTHS

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ABSTRACT

A high precision reflectometer has been designed and implemented to measure directly the specular reflectance (R) of materials in the submillimeter (SM) region of the spectrum (300 GHz < < 3000 GHz). Previous laser-based measurement systems were limited to an uncertainty in R of approximately ± 1.0% due to a number of issues such as: lack of an absolute reflection standard, difficulties in the interchange of sample and standard in the laser beam, and instabilities in the laser system. A SM reflection standard was realized by ellipsometrically characterizing the complex index of refraction of high purity, single-crystal silicon to a precision such that its SM reflectivity could be *calculated* to better than ± 0.03%. To deal with alignment issues, a precision sample holder was designed and built to accommodate both sample and silicon reflection standard on an air-bearing rotary stage. The entire measurement system operated under computer control and included ratioing of the reflected signal to a reference laser signal, measured simultaneously, to help eliminate short-term laser instabilities. Many such measurements taken rapidly in succession helped eliminate the effects of both source and detector drift. A liquid helium-cooled bolometer was modified with a large area detecting element to help compensate for the slight residual misalignment between sample and reflection standard as they were positioned into and out of the laser beam. These modifications enabled the final measurement precision for R to be reduced to less than 0.1%. The major contribution to this uncertainty was the difficulty in precisely exchanging the positions of sample and standard into and out of the laser beam and not due to laser or detector noise or instabilities. In other words, further averaging would not help to reduce this uncertainty. This order of magnitude improvement makes possible, for the first time, high precision reflectance measurements of common metals such as copper, gold, aluminum and chromium whose predicted reflectivities exceed 99% in the SM. Furthermore, precise measurement of the high frequency losses in high temperature superconducting materials is now also possible. Measurements reported here of metals at a laser wavelength of = 513.01 μm (584 GHz) indicate a slight discrepancy between experimental and theoretically predicted values, with measured results falling between 0.1% - 0.3% below predicted values.

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INTRODUCTION

As requirements of systems operating at millimeter (MM) and submillimeter frequencies become more demanding, accurate materials characterization at these frequencies becomes critical. Highly conductive materials such as metals and superconductors find important technological applications in communications, radar, and astronomy. From a scientific point of view, precise data are required for elucidating the fundamental nature of these materials.

The high frequency properties of metals are of great technical importance. Discrepancies have been found to exist between measured and theoretical values of absorption (defined by equation (1) below) from the infrared (IR) into the microwave spectral regions. Since demanding communication, radar, and now astrophysical systems operate at these wavelengths, these discrepancies have become a problem. Differences were first observed at microwave (1 - 20 GHz) frequencies and later at millimeter (20 - 300 GHz) frequencies. They are relatively low at 10 GHz but can become appreciable in the millimeter-wave region above 30 GHz. Increases in absorption of 50 to 100 percent have been observed at 35 and 70 GHz.[1] For a dielectric material which exhibits loss (e.g. a metal), conservation of energy requires that the sum of reflected, transmitted and absorbed electromagnetic energy equal unity, i.e., R + T + A = 1. Unless the specimen is sufficiently thin, there will be no transmission, and the absorption can be determined by measurement of the reflected intensity,

$$A = 1 - R \, . \tag{1}$$

Many times the quantity of interest is the real part of the complex surface impedance, or the surface resistance, R_s. This quantity is proportional to the absorption, A, and for good conductors at low frequencies,

$$R_s = \frac{Z}{4} \, (1 - R) \, . \tag{2}$$

Z is the impedance of the medium in which the measurement is made and is 376.99 Ω (MKS units) for free space. A measurement of the reflectance can therefore be used to determine the surface resistance. The difficulty is that R_S of a good conductor is typically very small in the SM (< 1 ohm/square) where the reflectance is near unity. From equation (2), we see that for a material with a reflectance of $\approx$ 99%, an uncertainty in the reflectance of 1% leads to a $\approx$ 100% uncertainty in R_S. Therefore, the reflectance of a highly conductive material should be measured to *at least* 0.1% to be useful. This fact is illustrated in Figure 1 where the theoretical reflectivity is plotted for several common metals using the Hagen-Rubens (HR) relation[2] (equation (3)). Table I lists the resistivities and conductivities of these metals.

$$ R_{HR} = 1 - \sqrt{\frac{2\omega}{\pi\sigma_{DC}}} \quad . \tag{3} $$

The HR relation predicts a reflectivity dependent only on the angular frequency, ω, of the incident radiation and the DC conductivity of the metal, σ_{DC}. It does not take any frequency dependence of the conductivity into account. In reality, as the frequency is increased (from microwave to SM frequencies), the AC conductivity of a metal becomes complex and the HR relation begins to break down. A more exact theory, outlined in the Discussion section, accounts for the conductivity's complex behavior and predicts a reflectivity *lower* than predicted by equation (3). In other words, the HR relation *underestimates* the high frequency loss in a metal.

This research was motivated by the fact that the SM frequency regime lacks a precise, straightforward means of measuring the reflectance of materials. The reflectometer described here was based on a CO_2 optically-pumped SM laser system. The SM region of the spectrum (100 µm < λ < 1000 µm) is about the longest wavelength region that straightforward, free space optical techniques can be used. Components routinely employed at shorter wavelengths such as front surface mirrors, lenses, beamsplitters, polarizers and waveplates can be used equally as well in the

SM. At wavelengths longer than   1 - 2 mm, diffraction prevents one from performing extremely precise optical measurements in a simple, free space manner. On the other hand, SM frequencies are low enough to gain insight on the low frequency (i.e. microwave, MM-wave) behavior of materials. Previous laser-based measurement systems were limited to an uncertainty in R of approximately 1.0% due to a number of issues such as: difficulty in the interchange of sample and standard in the laser beam, instabilities in the laser system, and lack of an absolute reflection standard. This research addresses all of these problems.

The measurement of the reflected intensity of a specimen at SM wavelengths is typically a relative measurement. The accuracy achieved ultimately rests on the assumptions concerning the reflective properties of the reference standard and how well it can be exchanged with a test sample in the measurement system. Long wavelength reflectance measurements are commonly made by comparison to a metal typically in the form of a thin film which is assumed to have a reflectivity of unity. Unless the film is of high quality and sufficiently thick to avoid skin depth problems, its reflectance may depart significantly from unity. There is also the problem that the high frequency properties of metals may not be well defined. At some frequency the skin effect will become anomalous and the metal's reflectance will depart from predictions of the simple theory (such as the Drude theory). Therefore, for precise measurements, reference to a metal surface is invalid. The technique used here relies on the fact that the reflectivity of a solid specimen can be *calculated* more accurately than measured if its optical constants are known with sufficient precision. A SM reflection standard was realized by ellipsometrically characterizing the complex index of refraction of high purity, single-crystal silicon to a precision such that the SM reflectivity of a Si etalon could be calculated to better than ± 0.03%. This idea was apparently first used by Birch.[3] To deal with alignment issues, a precision sample holder was designed and built to accommodate both sample and silicon standard on a computer-controlled rotary stage. The precision sample holder enabled mounting of specimens with an angle-of-incidence repeatability of   0.03°. Several ball and cross-roller bearing rotary stages were tested and found to exhibit appreciable wobble. To deal

with this issue, an air-bearing rotary stage was implemented to position both test sample and silicon etalon reflection standard into and out of the laser beam. A liquid helium-cooled bolometer was modified with a large area detecting element to help compensate for the slight residual misalignment between sample and standard as they were positioned into and out of the laser beam.

Severalreal-time computer averaging techniques were necessary to help compensate for both short and long-term instabilities inherent in the SM laser. Raw laser output contained non-random fluctuations in power of   1% and long-term drift (on the order of several minutes) of 10% - 15% due to vibrations, changes in room temperature, humidity, etc. A reference bolometer continually monitored the laser's output power. An electronic ratiometer simultaneously ratioed outputs of signal and reference detectors and therefore helped compensate for short-term drifts. Long-term drifts were dealt with by repeating many measurements rapidly in succession over a   6 minute period and then averaging the complete data set for the final result.

Several metal films were examined using the precision reflectometer. High conductivity metals such as Au, Al, and Cu have theoretical (Hagen-Rubens) reflectivities which range from   99.5% - 99.9% in the SM as shown in Figure 1. The final measurement system was able to measure the difference in reflectance between these metals. This significant achievement allows, for the first time, the study of highly reflecting materials in the SM region of the spectrum. High precision, room temperature reflectance data at 584 GHz are presented on a variety of metals and are compared with theoretical predictions.

The Reflection Standard

The reflectivity of any standard considered had to be calculable to high precision given quantities such as the material's complex index of refraction (n + i k), angle of incidence and the laser wavelength. A well-studied material in the SM with this potential is single-crystal, high purity silicon. Silicon was chosen in preference to germanium because of phonon bands that have been observed in germanium at wavelengths as long as 100 μm. Also, the fact that the band gap in germanium is nearly half that of silicon's leads to a far greater number of carriers thermally excited across the band gap into the conduction band at room temperature. These features cause structure in germanium's SM optical properties which are best avoided in this application.

Single-crystal silicon has several attractive features such as very low dispersion, availability in ultra high purities and the ability to be optically polished to a precise thickness. Birch[3] appears to be the first to apply silicon as a precision reflection standard in the SM. His characterization consisted of spectroscopic measurements from 5 cm^{-1} - 120 cm^{-1} (2 mm - 83 μm) on a 1000   -cm silicon wafer using the technique of dispersive Fourier Transform Spectroscopy (DFTS). His results for the complex index of refraction show the refractive index, n, decreasing and , the power absorption coefficient (= 4 k/), increasing with wavelength. An increase in   with SM wavelength in semiconductors is usually indicative of absorption due to free carriers. As mentioned previously, it is desirable to work with a material which exhibits as little dispersion as possible. One way to limit the number of free carriers and hence dispersion in   is to use a high resistivity (high purity) material.

The SM optical properties of high resistivity silicon have been studied by Grischkowsky,[4] who used time-domain spectroscopy to measure the absorption and dispersion in several dielectrics and semiconductors. His data on >10,000   -cm silicon displays low dispersion and a power absorption coefficient which *decreases* with wavelength between   = 200 μm - 1000 μm. A small decrease in   with wavelength is theoretically expected due to the residual effects of the wing of

the IR lattice absorption bands and indicates no measurable contribution from free carrier absorption. Therefore, in this work, high resistivity (40,000 - 60,000 Ω-cm) 1" dia., p-type silicon was employed as the material for the precision reflection standard.

The basic design of the reflection standard was in the form of an etalon. It is advantageous to work with an etalon with a relatively low optical thickness (the product of the physical thickness, t, and the real part of the complex index of refraction, n), because uncertainty in the thickness will then have a smaller effect on calculated values of R. The optical thickness is controlled by adjusting t. It is also obvious that the thickness should be adjusted such that a reflectivity maximum occurs at the laser wavelength of interest. Assuming that the etalon's thickness was designed in this way, one needs to know precisely how the uncertainties in the measured quantities n, k, and t correlate to uncertainty in the calculated reflectivity. One can show that the maximum allowable uncertainties in n, k, and t (for an etalon $\approx$ 400 μm thick) are approximately $\pm$ 0.002, $\pm$ 0.0001, and $\pm$ 1.0 μm respectively, if one wishes to calculate the reflectivity to better than 0.1%.[5] Even near normal incidence, the polarization and angle of incidence, i, affect the reflectivity. The p-polarized reflectivity begins to decrease with incident angle (this is the polarization in which Brewster's angle is observed) and the s-polarized reflectivity begins to increase, eventually reaching unity along with the p-polarization at i = 90°. The angle of incidence should be known to within $\approx$ 0.5° (for i < 5°) if one wishes to calculate the reflectivity to better than 0.1%. The equations used to determine the above tolerances are the familiar Fresnel equations as given in reference 6.

Using ellipsometric techniques,[7] the complex index of refraction of the silicon was measured at λ = 513.01 μm and is given in Table II. The uncertainty in n and k using this technique are within the maximum allowable uncertainties previously mentioned. Polishing of the 1" dia. silicon into a 413.0 μm etalon with an uncertainty in thickness of $\pm$ 0.25 μm was performed by Kappler Crystal Optics, Holliston, MA. One can again use the Fresnel equations to

predict the reflectivity of a Si etalon at this wavelength. The predicted Fresnel reflectivity for s-polarized radiation at an angle of incidence of 1.9° and at λ = 513.01 μm was 0.7093. When precise reflectance measurements are made at non-normal angles of incidence, it is necessary to consider the spherical-Gaussian nature of the SM laser beam to account for the incomplete overlap of multiple internal reflections which occur within the etalon.[8] It turns out, however, that at an incident angle of 1.9°, the difference in reflectivity when one accounts for the spherical-Gaussian nature and when one does not account for this effect is < 0.01% [5] and therefore can safely be ignored here.

EXPERIMENT

Measurement System

Reflectance measurements of several metals were made using the high precision reflectometer described here. The metals that were studied included Cu, Ag, Au, Al, Ni, and Cr. These were positioned in a custom-built sample holder which was mounted on an air-bearing rotary stage. A schematic of the optical system is shown in Figure 2. The sample holder/air-bearing assembly was designed to give a smooth, wobble-free, repeatable exchange of sample and standard into and out of the laser beam. The air-bearing rotary system was chosen over more conventional ball and cross-roller bearing rotary stages because of their superior performance. Air-bearings have minimal wobble and radial runout which are best avoided in this application. The sample holder accommodated the silicon etalon reflection standard and up to five test pieces. Samples and standard were mounted in the holder with small springs to ensure they were pressed flat against the ground flat surface of the holder's top mounting plate. This ensured that all reflecting surfaces were located in the same plane.

The metal films were obtained from a variety of sources. Thin Film Lab, West Hurley, NY provided films of Cu, Ag, Au, Ni, and Cr. All of these except Au had a 250 Å protective layer of SiO_2 to prevent oxidation and scratching. These films were deposited with an e-beam deposition system to thicknesses 4000 Å on 1" dia. BK-7 glass substrates with 80/50 surface quality. Deposition parameters such as base pressure, temperature, and deposition rate were set to optimize the film's performance at optical frequencies. The Al films were deposited by PFG Precision Optics, Ocean Springs, MS to a thickness of 3000 Å with a 250 Å SiO protective overcoating. Typical skin depths of common metals at 584 GHz are 1000 Å. The remaining films were Edmund Scientific enhanced aluminum, protected aluminum and gold mirrors. Edmund's protected aluminum mirrors are overcoated with 550 Å of SiO. The enhanced

aluminum is overcoated with an ultraviolet transmitting dielectric (MgF_2) to extend its use into the ultraviolet. The absorption of these protective coatings was calculated to have a negligible effect ($< 10^{-4}$) in the SM.

The signal and reference detectors were liquid helium-cooled bolometers. The signal bolometer was modified by removing its internal parabolic collection cone and replacing the 2 x 2 mm detecting element with a large-area element (12 mm dia.) to help compensate for any residual misalignment between sample and standard. For the given optical configuration described below, an assumed 10% spatial non-uniformity in the detecting element's responsivity leads to a negligible impact on the overall measurement uncertainty (i.e. it contributes less than 0.02% uncertainty). Two off-axis parabolic reflectors were used to focus the radiation on both sample and detecting element. The sample under test was placed at the focus of the off-axis parabolic mirror. The waist size ($1/e^2$ intensity) of the SM laser beam at the sample surface was calculated to be less than 2 . Two samples not in identical alignment had reflected rays which diverged until they reflected from the off-axis parabola, where they then reflected as parallel rays. The two rays would then remain parallel along their path (35") towards the detector's off-axis parabola. The detecting element was placed near the focus of its off-axis parabola. This arrangement was necessary to obtain high precision results which would not have been possible if lenses were employed.

A problem commonly encountered in systems with coherent sources is the standing waves (feedback) that exist between the various components of the optical system (e.g. the laser resonator and reflecting sample). Coherent feedback effects are easily observed when working at normal incidence by translating the reflecting sample into and out of the direction of the oncoming radiation (see Figure 2) and observing a (10%) modulation at the signal detector. To help minimize this problem, reflectance measurements were made slightly off normal incidence (1.9°). To further reduce coherent feedback effects, attenuation (acrylic plastic) was inserted at the laser's output and in front of the signal and reference detectors. Though this attenuation reduced the demodulated

signals to ~ 10 mV, versus 1 V without attenuation, it eliminated the feedback problem. Feedback diminishes with attenuation faster than signal because signal loss is a single pass loss whereas feedback suffers from a double pass loss. Verification that feedback had been suppressed was made by translating the sample fixture as described above and observing no signal modulation. The attenuated laser signal remained far above the detector noise so that the latter had no effect on the measurements.

The laser system used was a CO_2 optically-pumped SM laser. The 85 watt CO_2 laser (mechanically chopped at 100 Hz) pumped the λ = 513.01 μm transition in a 2.7 m, 4.5" dia. formic acid (HCOOH) gas-filled (100 mTorr) cavity. Radiation was coupled out of the cavity through an 8 mm hole output coupler. This produced a high quality, stable TEM_{00} Gaussian mode. Sidelobes on the main transmitted SM laser beam were typically between 18 dB and 22 dB down from the central maximum, as confirmed by spatial mode scans of the beam's cross section. The overall stability of the CO_2/SM laser system was an important factor in the measurement procedure. The CO_2 laser was actively stabilized using a Fabry-Perot frequency locking system.[9] This device monitored the frequency of the CO_2 laser and actively made minor adjustments to its cavity length to keep the CO_2 frequency constant. There was no active stabilization on the SM laser. Its output power would slowly drift due to changes in the laser cavity length caused by thermal fluctuations in the room (e.g. cycling of the air conditioner). This cyclic behavior resulted in a 10% - 15% variation in laser output power every ~ 20 minutes. Most other sources of noise were short-term in nature and had to be dealt with accordingly. Short–term fluctuations can be a result of a variety of sources such as mechanical vibrations, power supply (electrical) noise, CO_2/SM feedback or changes in environmental conditions (such as humidity) along the various beam paths. Several techniques were implemented to minimize these effects and maximize overall system performance. As shown in Figure 2, a reference detector monitored the SM laser's output power. Two lock-in amplifiers demodulated both signal and reference detector outputs. The output of the signal's lock-in was continually ratioed to the reference's lock-in output by an

electronic ratiometer. The comparison is essentially instantaneous if one assumes that the two lock-ins have identical response characteristics. The technique of ratioing was critical in achieving high precision results. Ratiometer output was input to one of the ADC channels of the signal lock–in to be read by the computer. Several different lock-in time constants were tested and 100 ms was found to be optimal. The entire measurement system was automated under computer control.

Even though the etalon's reflectance could not be measured with high precision, its transmittance (T) could be. This is because the "standard" for a transmittance measurement is air with an assumed complex index of refraction of $1.0 + i\ 0.0$. The theoretically calculated transmissivity of the etalon at $= 513.01\ \mu m$ and $i = 1.9°$ is 0.29044. Though it would have been desirable to measure T at this angle of incidence, feedback prevented this. A transmittance measurement was therefore performed at an angle of incidence of $i = 8.5°$. The theoretically predicted value for the transmissivity at 8.5° is 0.28581. The optical configuration for this measurement was the same as the reflectometer set-up except that the silicon etalon was inserted as indicated in Figure 2. The experimentally measured value of 0.2861 is very close to the above predicted transmissivity. This measurement provided experimental verification that the silicon etalon had indeed been designed properly as a reflection standard. After samples and standard were inserted into the fixture, their relative alignment was checked by back reflecting a He-Ne laser onto a far wall. After several measurements of two nominally identical Au mirrors, it was determined that the relative angular alignment of samples should be 0.03° in order to achieve better than ± 0.1% uncertainty in the reflectance. Therefore, before each measurement sequence, this relative alignment was checked and verified. Also just prior to measurements, two nominally identical Au mirrors were inserted and measurements were made to verify that the system measured a ratio of 1.000 ± 0.001 or better.

Measurement Procedure and Data

A measurement sequence consisted of the following steps:

1. The rotary stage was positioned so that the laser reflected from the metal film under test.

2. Several ratiometer outputs were acquired every 10 ms in a 100 ms interval and averaged.

3. The rotary stage rotated to the Si etalon reflection standard.

4. Several ratiometer outputs were again acquired every 10 ms in a 100 ms interval and averaged.

5. The rotary stage was positioned back to the metal film.

Because residual short-term noise still existed on the ratiometer output, it was necessary to acquire several ratioed outputs within a short (100 ms) time interval and then average the results. The above procedure was performed 50 times over a 6 minute period (i.e. the reflectance was measured 50 times) and the mean and standard deviation of the data set were calculated.

Data for Cu, Ag, Au, Al, Ni, and Cr are summarized in Table III. Values of the reflectivity calculated with the HR relation (equation (3)) and its high frequency correction (given in the Discussion section) are also presented. Data for commercially available front surface mirrors are given in Table IV. The overall uncertainty in the measurements was $\pm 0.1\%$. This error is due almost exclusively to positioning error (i.e., the inability to precisely interchange metal film and Si etalon into and out of the laser beam).

DISCUSSION

The use of a high purity silicon etalon as a precision standard for reflectance measurements in the SM has been demonstrated. Originally, several reflection standards had been designed at a variety of SM wavelengths (which was possible because the silicon had been ellipsometrically characterized at a variety of SM wavelengths). The difficulty in designing an etalon at a shorter laser wavelength (e.g.   = 117.7 μm) is that silicon's absorption begins to significantly contribute to its reflectivity and therefore the overall uncertainty in the etalon's reflectivity. The absorption at   = 117.7 μm is roughly three times of that at   = 513.01 μm. One way around this problem is to design a thinner etalon. A thinner etalon will also help reduce the uncertainty in reflectivity due to uncertainty in refractive index n and thickness. However, attempts to polish the 1" dia. silicon wafers to thicknesses less than   400 μm were unsuccessful.

Discrepancies

Reflectance data in Table III indicate a slight discrepancy between experimental and theoretical values. Measured results fall between 0.1% - 0.3% below predicted values for all metals studied. For metals which are expected to be most free-electron-like (Cu, Ag, Au, Al), the average difference between experimental and theoretical (Hagen-Rubens) values is   0.2%. As mentioned in the Introduction, the Hagen-Rubens relation does not take the complex nature of a metal's conductivity into account. It assumes that $(\ )=\ _{DC}$ which essentially says that   << 1 where   is the mean free collision time of the conduction electrons in the metal. In the general case, without any condition imposed on the value of   , the reflectivity of a metal is given by,[5]

$$R = 1 - \sqrt{\frac{2\ }{\ _{DC}}}\ \left[\left(1 + \ ^2\ ^2\right)^{1/2} + \ \right]^{1/2} \quad .$$
(4)

The extra term in brackets in equation (4) is ≈ 1 for $\omega \to 0$ and therefore predicts a *greater* loss than predicted by the Hagen-Rubens relation alone. However at 584 GHz, for typical values of τ, the difference in reflectivity predicted by equations (3) and (4) is $\approx 0.02\%$ (as seen in Tables III, IV and Figure 1) and therefore cannot explain the discrepancies seen here. This difference does become larger at higher frequencies, as expected. At 2.5 THz ($\lambda \approx 120~\mu m$), differences are as large as 0.15%.

Discrepancies between experimentally measured values and theoretical predictions of high frequency (i.e. microwave-SM) absorption in metals have been a long-standing problem. Batt[10] has carefully investigated the surface resistance of gold at 890 GHz. His technique involved evaporating a thin film of gold on the front surface of a pyroelectric detector element. The modified detector was used in the normal way to produce a pyroelectric voltage proportional to the absorbed power of incident SM radiation. The ratio of actual-to-theoretical surface resistance is approximately 2.2 when equations (2), (3) and the bulk conductivity of gold is used. Similar work on copper and silver between 10 - 200 GHz also displays measured-to-theoretical losses approaching 2.5.[11,12] Tischer[13] has investigated the attenuation of commercially available waveguides and has measured a loss ratio of 1.135 ± 0.02 for copper surfaces which were carefully prepared by electropolishing and annealing in hydrogen.

Many times, some portion of a discrepancy can be attributed to the preparation of the surface under test. Thorpe[14] has shown that the electrical properties of copper are a sensitive function of surface preparation. To maximize the conductivity, he found it was necessary to acid-etch and then anneal the copper surfaces at 780 C in a hydrogen environment. By this method, an improvement in loss ratio from 1.32 to 1.01 at 38 GHz was observed. Hinderks and Maione[15] report near theoretically predicted values of R_s for electrodeposited copper at 43 GHz. Their research shows that significant improvement in copper conductivity can be obtained through an annealing step and have reported a measured-to-theoretical loss ratio of 1.04.

At frequencies above the MM-wave region, bulk samples are no longer necessary and

specimens that are sufficiently thick can be prepared by vacuum deposition. Thin metal films are generally preferred over prepared bulk metal surfaces when studying optical properties, however, they are not without limitations. It has been shown that the SM surface resistance can depend upon deposition parameters such as temperature. For aluminum films deposited on diamond substrates, Schiever, et. al.[16] have shown that a substrate temperature of 100 C - 120 C yields a film with a resistance at 105.55 GHz approaching theoretical values. On the other hand, for films deposited at room temperature, loss ratios as high as   2.5 are found. This implies that a material which normally reflects 99.7% in the SM will only reflect 99.5%, or a reduction of approximately 0.2%. Because of this fact, any future work on metal films with this instrument should concentrate on studying the relationship between film preparation and its reflectance.

Application of SM Reflectometry to the Study of High Temperature Superconductors (HTS)

SM reflectometry has been shown to be a viable technique to precisely measure the reflectance of metals.   There is also current interest in the SM optical properties of superconductors.[17] Since the advent of superconductors with transition temperatures (T_c) above 77 K, these materials have generated much interest among microwave and MM–wave engineers. A current frequency limit in microwave circuits is ohmic loss. In addition to reducing ohmic loss, a circuit made from superconducting materials will have lower dispersion. Superconductors, unfortunately, have zero resistance only to DC currents. The surface resistance varies as ^{2}, predicted by the London equations, in contrast to normal metals whose surface resistance varies as $^{1/2}$, predicted by the classical theory. Therefore, beyond some critical frequency (f_c), a normal metal will have a lower surface resistance than a superconductor. At the present time, bulk superconducting materials have critical frequencies of   10 GHz when compared with copper at 77 K.

However, recent measurements on thin film HTS appear to have f_c values beyond 100 GHz. Klein[18] has measured the MM-wave surface resistance of YBCO thin film superconductors at 86.7 GHz and 77 K. Results indicate that R_s is nearly one order of magnitude less than R_s of copper at 77 K and nearly equal to niobium at 7.7 K. As HTS continue to improve, it will become increasingly difficult to determine their surface resistance at these frequencies. Most published measurements at MM-wave frequencies use cylindrical resonators and determine R_s from the cavity Q. In the measurement of a superconducting material, the normal metal sides of the resonator should have as little resistance as possible and the HTS should make up as much of the resonator's walls as possible. Accuracy in determining R_s will typically be poor if the material under study has a lower R_s than the material comprising the rest of the resonator. This is the case for a high quality thin film superconductor measured using a resonator made of copper. Ideally, the resonator should be constructed from superconducting material itself, which is not practical. One way to approach this problem is to measure R_s at higher frequencies where losses are much greater, and scale the results to the MM-wave region. According to the BCS theory, the surface resistance of a superconductor below the gap frequency increases as the square of the frequency. This has been experimentally verified for HTS.[19,20] A SM reflectometer can be used to determine R_s at SM frequencies using equation (2) and then R_s could be scaled to microwave frequencies using ω^2 scaling. The uncertainty in the SM surface resistance in ohms/square is approximately equal to the uncertainty in the reflectivity, ΔR, in percent. Therefore, an uncertainty $\Delta R \approx 0.1\%$ leads to a ΔR_s of ≈ 0.1 ohms/square. The sensitivity at 10 GHz implied by ω^2 scaling of R_s (measured at 1.5 THz) is therefore $\approx 4 \mu\Omega$/square. The SM technique is therefore more sensitive than common microwave measurements. Uncertainties in parallel plate resonator techniques are typically ± 20 $\mu\Omega$/square at 77 K and $\pm 60 \mu\Omega$/square at 95 K.[21] End-wall replacement techniques suffer even greater uncertainties (a few mΩ) due to the metallic walls of the cavity.

Also of current interest is the ability to spatially scan the surface of a superconducting material. As thin film superconducting samples increase in size, the ability to measure the spatial distribution of R_s becomes of interest. The waist of the SM laser used in experiments described previously was 0.7 mm at = 513.01 μm. At shorter wavelengths, smaller waist sizes would be possible. These near-diffraction limited spot sizes could make high spatial resolution measurements of the surface resistance possible.

CONCLUSION

A high precision reflectometer has been designed and built to operate in the SM region of the spectrum. Problems which typically limit measurement precision such as sample positioning, lack of an absolute reflection standard, and instabilities in the SM laser system have been investigated. Critical in this effort was the optical characterization of a high purity silicon wafer such that an etalon fabricated from this material had a calculable SM reflectivity with an uncertainty of less than $\pm\ 0.03\%$. This was achieved using an ellipsometer specifically designed for materials characterization at SM wavelengths. Sample positioning was achieved through construction of a specially designed sample fixture which was mounted on an air-bearing rotary stage. These efforts reduced the overall uncertainty in reflectance from $\pm\ 1\%$ achieved in previous systems to less than $\pm\ 0.1\%$. This order of magnitude improvement makes possible, for the first time, high precision reflectance measurements of materials in the SM. The differences in reflectance between common metals, which are on the order of several tenths of one percent, can now be discerned. The absolute reflectance of several thin metal films was measured at $\quad = 513.01$ μm using the silicon etalon standard. The data indicate a slight discrepancy between experimental and theoretical values, with measured results falling 0.1% - 0.3% below values predicted by the simple theory. This discrepancy has been observed by other researchers as well in the SM and MM-wave frequency regions. High precision reflectometry can also be used as a sensitive technique to measure the surface resistance of high temperature superconducting materials as well as studying the relationship between metal film preparation and its reflectance.

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Figure 1

Reference detector

Translation table to
test for feedback

Sample
fixture

Off-axis parabola

Si etalon was inserted
here for the transmittance
measurement

Off-axis parabola

beamsplitters

Signal detector

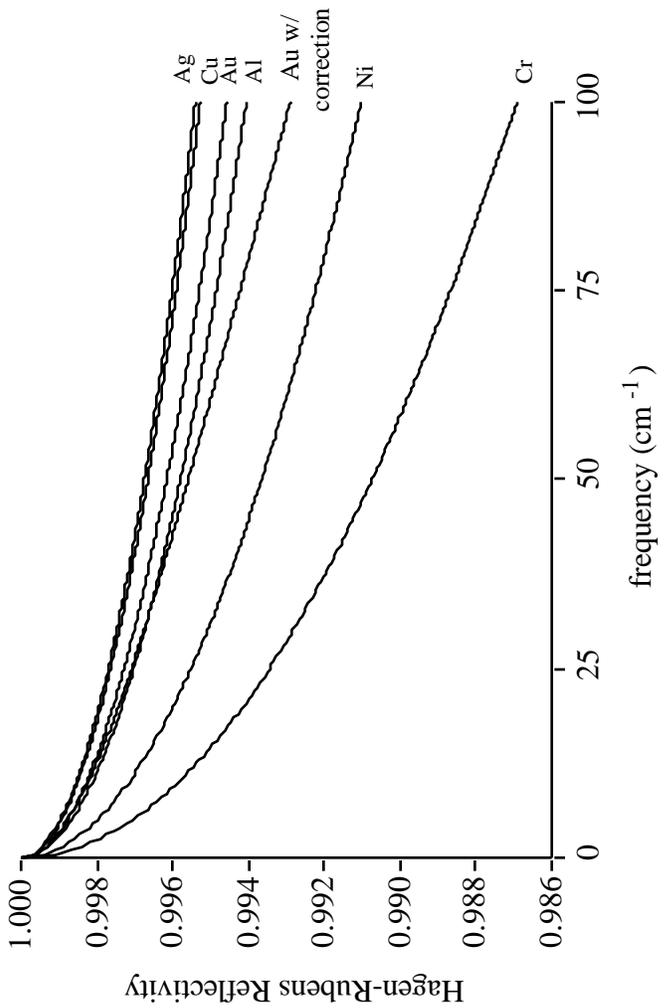anechoic

lens

attenuation

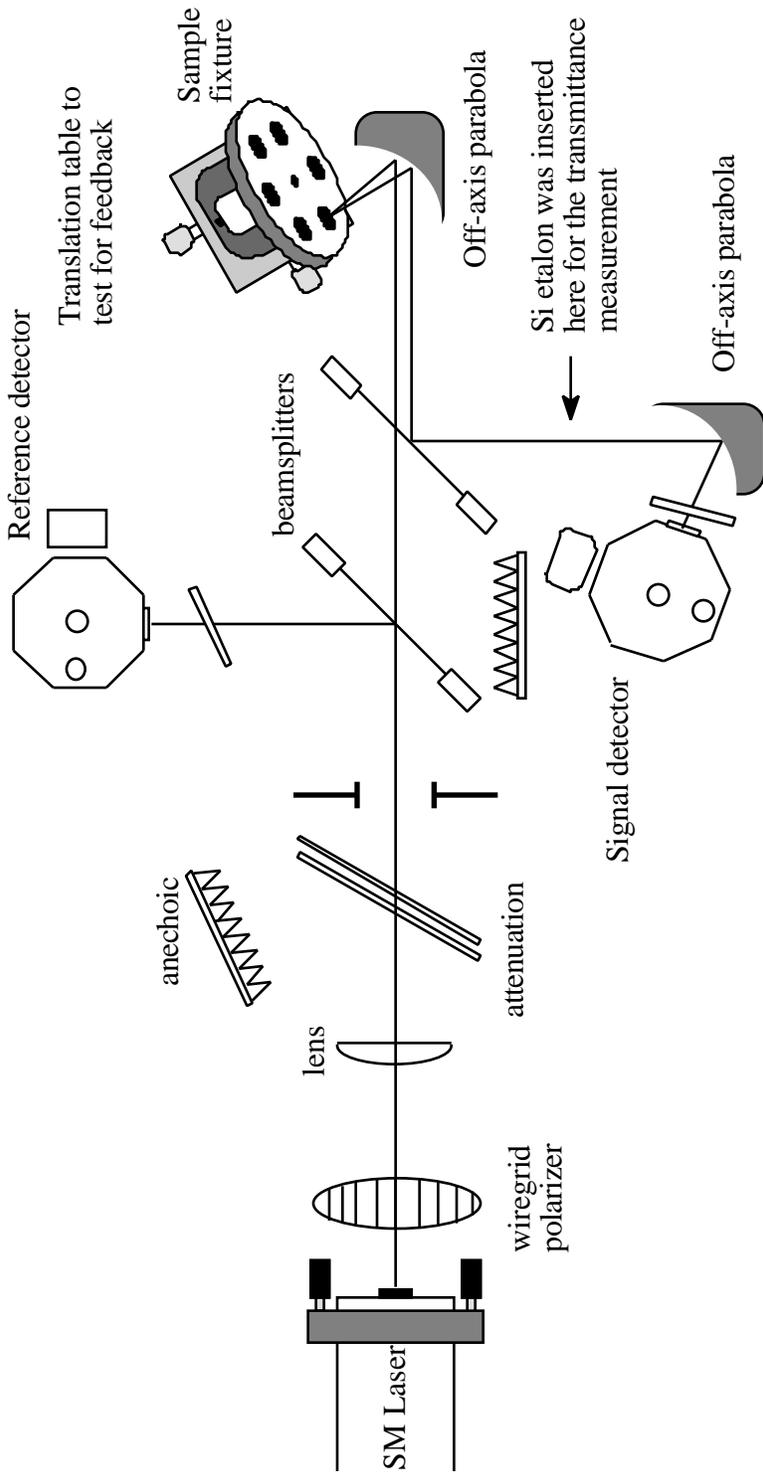wiregrid
polarizer

SM Laser

Figure 2

FIGURE CAPTIONS

1.   Hagen Rubens reflectivity of metals listed in Table I. The high frequency correction described in the Discussion Section is shown for gold.

2.   General optical configuration of the high precision reflectometer showing signal and reference detectors (liquid helium bolometers) and the six position sample holder mounted on an air bearing rotary stage.

Table I   Resistivities and Conductivities of Various Metals

Metal	Resistivity ($\mu\Omega-cm$)	T (K)	Conductivity x 10^{17} sec^{-1}	Drude relaxation time x 10^{-14} sec
Silver (Ag)	1.587	293	5.671	4.0
Copper (Cu)	1.678	293	5.364	2.7
Gold (Au)	2.214	283	4.065	3.0
Aluminum (Al)	2.650	293	3.396	0.8
Nickel (Ni)	6.03	293	1.49	0.65
Chromium (Cr)	12.9	273	0.70	0.17

Table II

Ellipsometrically Determined SM Complex Index of Refraction of Silicon (ρ_{DC} = 40 - 60 KΩ-cm)

wavelength (µm)	frequency (cm^{-1})	Refractive Index n	Extinction Coefficient k	Absorption Coefficient (cm^{-1})
513.0157	19.4926	3.4164 ± 0.0002	$0.00004\,^{+\,0.00008}_{-\,0.00004}$	$0.01\,^{+\,0.02}_{-\,0.01}$

Table III
Results of Reflectivity Measurements at $\lambda = 513.01$ μm

metal	Reflectivity at 513.01 μm	uncertainty	Hagen-Rubens Eqn. (3)	H-R w/ corr. Eqn. (4)	conductivity 10^{17} sec^{-1}	relaxation time 10^{-14} sec
Copper	0.997	0.001	0.9979	0.9978	5.36	2.70
Silver	0.996	0.001	0.9980	0.9978	5.67	4.00
Gold	0.994	0.001	0.9976	0.9975	4.07	3.00
Aluminum	0.995	0.001	0.9974	0.9973	3.40	0.80
Nickel	0.994	0.001	0.9960	0.9960	1.49	0.32
Chromium	0.993	0.001	0.9942	0.9942	0.70	0.33

Table IV
Reflectivity of Commercially Available Front Surface Mirrors

Metal	Reflectivity at 513.01 μm	uncertainty	Hagen-Rubens Eqn. (3)	H-R w/ corr. Eqn. (4)	conductivity 10^{17} sec-1	relaxation time 10^{-14} sec
Enhanced Al	0.992	0.001	0.9974	0.9973	3.40	0.80
Protected Al	0.985	0.001	0.9974	0.9973	3.40	0.80
"Edmund" Gold	0.995	0.001	0.9976	0.9975	4.07	3.00