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In this work, we study the electrical properties of transparent conducting oxides using physical and optical techniques. The objective is to characterize the conductivity of a series of cadmium oxide zinc-oxide thin films with varying cadmium concentrations using three methods. The Hall Effect estimates carrier concentration, sheet conductivity, and carrier mobility. Optical methods, such as FTIR spectroscopy, can provide estimates of the plasma frequency, which describes a metal's transition from being transparent to opaque to light. THz spectroscopy extracts the complex conductivity of materials in the far infrared spectrum and provides insights on the optical transport properties of conductors. Our findings show that DC/AC measurements of the conductivity of Cd/ZnO thin films exhibit frequency independent conductivity, indicating that the films are homogeneous Drude metals.

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# Investigation on the Electrical Properties of CdO/ZnO thin films using THz Spectroscopy

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In this work, we study the electrical properties of transparent conducting oxides using physical and optical techniques. The objective is to characterize the conductivity of a series of cadmium oxide zinc-oxide thin films with varying cadmium concentrations using three methods. The Hall Effect estimates carrier concentration, sheet conductivity, and carrier mobility. Optical methods, such as FTIR spectroscopy, can provide estimates of the plasma frequency, which describes a metal's transition from being transparent to opaque to light. THz spectroscopy extracts the complex conductivity of materials in the far infrared spectrum and provides insights on the optical transport properties of conductors. Our findings show that DC/AC measurements of the conductivity of Cd/ZnO thin films exhibit frequency independent conductivity, indicating that the films are homogeneous Drude metals.

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*Professor James Heyman patiently advised me on this project.

# Contents

# 1 Introduction

In the endeavor to reduce our reliance on fossil fuel generated energy, new energy portfolios have incorporated renewables, such as solar and wind energy, to begin the shift towards a more sustainable future. One main deterrent of renewable energy sources is their intermittence and how peak power usage does not perfectly align with power production from renewables [1]. Although much is being done to encourage a reduction in peak demand, one area researchers have continuously addressed is how to increase the efficiency of existing solar panel technology. The premise is simple - from a top-down approach, how can we increase the efficiency of solar cells?

Among the answers to that question stand a developing set of materials called transparent conducting oxides. Transparent conducting oxides (TCOs) are thin metal films designed to be optically transparent and electrically conductive. Broadly speaking, TCOs see their best application as surface contact layers for solar cells; solar cells benefit from exposure to a wider range of light beyond the visible to make efficient use of variable energy carried by photons. Ideally, light that passes through a TCO surface contact layer will (1) not be reflected, (2) not be absorbed by the material, and (3) experience absorption at the photovoltaic cell. Effectively, TCOs act as transparent electrodes to collect photo-generated current.

At the forefront of this class of materials is tin-doped indium oxide (often referred to as indium tin oxide). Indium tin oxide (ITO) is a highly transparent material because it possesses a large band-gap (4 eV) - this means that visible light photons pass through the material without being absorbed. ITO also boasts a high conductivity $\sigma = 10^6\,\mathrm{S\,m^{-1}}$, which minimizes resistive power loss.

It may be tempting to assume that ITO will find itself on all future solar panel arrays, but this is far from the truth. The reality is that the known global supply of the precious metal, indium, cannot supply enough material for the scale of operations anticipated. From a scientific perspective, it is the perfect problem to have - we are interested in the development and characterization of alternative composite TCOs that possess particular optical and electronic properties.

The TCOs examined in our research were composite cadmium oxide zinc oxide (CdO/ZnO) thin films. The samples were provided to us by Professor Kin Man Yu from the City University of Hong Kong (originally from Lawrence Berkeley Laboratory). In order to probe the discussed properties of this material, we performed a series of physical and optical measurements using (1) Hall Effect, (2) Fourier Transform Infrared Spectroscopy, and (3) Terahertz Spectroscopy. Although these measurements provide very similar information about conductive materials, each is independent and can provide valuable insight on properties of the material when they do not agree with each other. Namely, if the conductivity of a material measured at DC (as per physical contact methods) differs from the conductivity of the same material measured at AC (via optical methods), then we have encountered something strange and worth further investigation.

Presently, we have described the key qualities of TCOs and how tailoring their optical and electrical properties set them up for applications in optoelectronic circuits. Our primary research interests surround the classification of a set of CdO/ZnO samples, with which we perform three different measurements to determine real and complex components of conductivity as a function of frequency. What can we say about the behavior of this material across DC/AC measurements, and is it good?

## 2 Background

Professor Kin Man Yu from the City University of Hong Kong sent us nine $Cd_xZn_{1-x}O$ samples that fall in the composition range of $0.34 < x < 0.92$. These samples were fabricated with plasma deposition and set onto either glass or sapphire square substrates approximately $5\,\mathrm{mm} \times 5\,\mathrm{mm}$. The samples appear physically different - increasing the CdO concentration changes the color of the film from a light yellow to a dark orange, due to the decrease in the band-gap energy. Another property of these films that changes with CdO concentration is the crystalline structure of the material, shifting from wurtzite to rocksalt at $x \approx 0.69$ [2] - this produces changes in the electron concentration and carrier mobility of the material. Whenever the samples were not being handled, they remained in an environment purged of oxygen in order to prevent degradation of the material.

Our experimental goal was to measure the real and complex parts of conductivity $\sigma$ as a function of frequency. The motivation behind measuring $\sigma$ with the Hall Effect ($\omega = 0$) and THz spectroscopy ($\omega \sim 1 \times 10^{12}\,\mathrm{s}^{-1}$) is that they should produce the same results if the films are homogenous and conventionally resistive. Fourier transform infrared spectroscopy (FTIR) measurements ($\omega \sim 1 \times 10^{14}\,\mathrm{s}^{-1}$) can also provide insights on several electronic properties, most notably the plasma frequency. The plasma frequency $\omega_p$ is a characteristic property of metals that describes when the complex dielectric function $\tilde{\epsilon_r}$ changes from negative to positive, and the material transitions from being opaque to transparent. Specifically, light at a frequency greater than $\omega_p$ will pass through the metal, and light below the plasma frequency will not penetrate completely, or be reflected at the surface. For TCOs, the most ideal samples will have a plasma frequency below the frequencies of visible light contained in the solar spectrum.

In this work, conductivity hitherto has been described as a relatively simple property of a material. The conductivity can be defined in terms of more fundamental properties

$$\sigma = Nq\mu \tag{1}$$

where $N$ is the carrier concentration and $q$ is fundamental charge. The carrier mobility $\mu$ is a measure of how effectively charges move through the medium and can be defined as

$$\mu = \frac{q\tau}{m^*}$$

where $\tau$ is the scattering time and $m^*$ is the effective mass of the charge carriers. The scattering time $\tau$ can be understood as the time between charge carrier-collision events. It is evident that increasing the scattering time or decreasing the effective mass increases the charge mobility, and thus the conductivity, which can be rewritten as

$$\sigma = \frac{Nq^2\tau}{m^*}$$

In the fabrication of thin film TCOs, the quickest way to increase the conductivity is to increase the density of charge carriers $N$. However, we have yet to define the plasma frequency in terms of more fundamental properties, which can be expressed as

$$\omega_p = \sqrt{\frac{Nq^2}{m^*\epsilon_0}} \tag{2}$$

Notice the plight of TCO fabricators? Increasing the carrier concentration $N$ creates a good conductor, but may yield TCOs that are too opaque for visible light to pass through. Fabrication of TCOs is its own research area outside the scope of this work, but these formulas clearly define the relationships between fundamental electrical properties and transparency of thin films.

# 3 Methodology

Prior to measuring $N$, $\mu$, $\sigma$, and $\omega_p$, for each of the nine samples, we carefully considered how each sample would have to be manipulated. For CdO/ZnO samples which were too resistive for initial Hall Effect measurements, we performed cold-welding of indium contact points at the corners to provide Ohmic contacts to the films.

In order to perform optical conductivity measurements on TCOs deposited onto substrates, we require a reference sample. In this case, the reference sample was either a blank glass or sapphire substrate. By taking the ratio between the power of transmission of the sample on glass/sapphire and the power of transmission of a blank piece of substrate, we can find the power of transmission through the material of interest. For THz measurements, it is important that the reference and sample substrate have the same thickness. This is typically taken care of by reserving a separate blank piece of substrate, however, the samples provided to us did not include a reference sample. To produce reference samples, we used a diluted acid solution to chemically etch approximately half of the TCO. The internal set ups of the optical equipment used in this work allow control of the beam path relative to the sample - we can simply move between the half of the sample with TCO and the other half without to extract the transmission power through the film.

## 3.1 Hall Effect

The idea behind the Hall Effect is that it is a four-wire conductivity measurement in the presence of a magnetic field. It's easy to measure the conductivity of a material, but the Hall Effect can also independently measure the carrier mobility $\mu$ and the carrier concentration $N$. The perpendicular magnetic field on the applied current produces a resultant “Hall resistance”, from which we can calculate $\mu$,

$$\mu = \frac{R_H \sigma d}{B}$$

where $R_H$ is the Hall resistance, $d$ is the thickness of the sample, and $B$ is the applied magnetic field.

The Hall Effect is a useful technique in this work because it provides estimates for the (1) density of charge carriers per unit area, (2) sheet conductivity, and (3) carrier mobility. If the thickness of the sample is known, then it is possible to produce the meaningful estimates of the carrier concentration per unit volume $N$, the actual conductivity of the sample $\sigma$, and the carrier mobility $\mu$. An important thing to note about conductivity measurements via the Hall Effect is that it is a DC measurement. Thus it only accurately probes the electrical properties of the underlying material when the sample is free of cracks or structural flaws. Samples that contain large defects, such as a crack that bisects the sample, invalidate the measurement.

Here, we used a Cascade Microtech manual probe station in conjunction with a Keithley 2400 Source Measure apparatus to make electrical contact to the nine Cd/ZnO samples and read voltage vs. current. Additionally, the magnetic field was adjusted incrementally from -0.4 T to 0.4 T using an electromagnet and a Kepco programmable power supply. The experiment was controlled by a LabView program.

## 3.2 Fourier Transform Infrared Spectroscopy

The purpose behind FTIR is to measure light intensity as a function of wavelength. It uses a Michelson Interferometer and a broad-band light source to simultaneously detect the intensity of all wavelengths - this is ideal in infrared spectroscopy because relatively weak infrared sources can be measured quickly with little noise. The raw data that the detectors read then undergoes a Fourier transform to construct the intensity as a function of frequency. Plots of light intensity vs. frequency are then transformed into plots of transmission as a function of frequency by taking the ratio against a reference spectrum,

$$T(f) = \frac{I_{sample}(f)}{I_{ref.}(f)}$$

In our experiment, we use a Thermo-Nicollet IS50 Fourier Transform Infrared Spectrometer to take measurements across the near and mid infrared spectrum, spanning wavelengths between 1 µm to 5 µm.

To estimate the plasma frequency $\omega_p$ from FTIR spectroscopy, we developed a model to simulate the complex dielectric constant $\tilde{\epsilon_r}$ of our films. The complex dielectric constant $\tilde{\epsilon_r}$ can be thought of as a measure of how effectively electromagnetic waves penetrate materials. By modeling the complex dielectric function, it is possible to perform a curve fit to the transmission power vs. frequency plots to numerically estimate $\omega_p$, and by extension, the optical carrier mobility $\mu$ and carrier concentration $N$. This model is an extension of the Drude Model [3], in which the complex dielectric constant of a metal in an AC field is described by

$$\tilde{\epsilon_r} = \epsilon_\infty - \frac{\omega_p^2}{\omega^2 + i\omega\Gamma(\omega)} \tag{3}$$

where $\epsilon_\infty$ is the high-frequency dielectric constant. To better account for the unique optical transport properties of the thin films [4], we introduced a frequency dependent scattering time, parameterized by a complicated function $\Gamma(\omega)$,

$$\Gamma(\omega) = f(\omega)\Gamma_L + (1 - f(\omega))\Gamma_H \left(\frac{\omega}{\Gamma_X}\right)^{-3/2}$$

where,

$$f(\omega) = \frac{1}{1 + \exp\left(\frac{\omega - \Gamma_X}{\Gamma_W}\right)}$$

and $\Gamma_L$ is the low frequency damping constant, $\Gamma_H$ is the high frequency damping constant, $\Gamma_X$ is the cross-over frequency, and $\Gamma_W$ is the cross-over width. In short, these four parameters serve to model the behavior of optically induced charge carrier scattering. Extended Drude theory seems to block the forest for the trees, but the important thing to take away from it is that it provides a model for the complex dielectric constant $\tilde{\epsilon_r}$ of our materials.

Modeling the complex dielectric constant $\tilde{\epsilon_r}$ this way allows us to calculate the real and complex components of the indices of refraction $\tilde{n}$, where the real part is the index of refraction $n = \mathrm{Re}[\sqrt{\epsilon_r}]$, and the imaginary part gives the absorption coefficient $\alpha = 2(\omega/c)\mathrm{Im}[\sqrt{\epsilon_r}]$.

The motivation to calculate the indices of refraction and the absorption coefficients is that they provide a means to calculate the power of transmission through the sample. Logistically, the infrared beam of light moves through three different interfaces: (1) air to sample, (2) sample to substrate, and (3) substrate back out into the air. The transmission coefficient, $t$, at each interface, is defined as

$$t_{i,j} = \frac{2n_i}{n_i + n_j}$$

where $i$ and $j$ are simply indices that correspond to the particular medium. In example, $t_{12}$ corresponds to the infrared beam passing from air into the substrate.

Each of the aforementioned media has a different index of refraction, and the sample itself is not perfectly transparent. Consequently, reflection can occur at each interface as well. The reflection coefficient is

$$r_{i,j} = \frac{n_i - n_j}{n_i + n_j}$$

This produces multi-beam interference, and we can model the effective transmission coefficient through the sample with the following expression

$$t = \frac{t_{12} t_{23} e^{i\delta - \alpha d/2}}{t_{31}(1 - r_{12} r_{21} e^{i\delta - \alpha d})} \tag{4}$$

where $\delta = 4\pi d n \omega$, a parameter which describes decay of electromagnetic waves as it penetrates a sample of thickness $d$. Finally, the transmission coefficient for power, $T$, is given by $T = |t|^2$.

The extended Drude Model seems complicated, but just so that it is clear: we have developed a model which simulates the complex dielectric constant of the material and use that to produce power of transmission vs. frequency curves. By adjusting parameters such as the damping coefficients, the index of refraction of the substrate, the thickness of the thin film $d$, and of special interest, the plasma frequency $\omega_p$, we can perform a best fit curve to the data from our FTIR measurement. It is also possible to optically derive estimates of the carrier mobility $\mu$. Finding $\omega_p$ determines the carrier concentration $N$.

## 3.3 Terahertz Spectroscopy

THz spectroscopy measures light intensity as a function of THz frequency. Although its objective function is similar to FTIR spectroscopy, it is arguably the most interesting measurement technique employed in this research. In this work, long wavelength infrared radiation between 0.1 THz - 3.0 THz is used to probe the behavior of electrons in our thin film conductors, and we measure the transmission at THz frequencies to extract real and complex components of conductivity.

In this work, we use time-domain THz spectroscopy based on a mode-locked Ti-sapphire laser system. This laser system produced ultra-short and intense laser pulses on the upper bound of 100 fs long. Much can be said about the theory of non-linear optics - what follows is a basic description of how THz spectroscopy works.

The FEMTO-COMPACT laser system produces 10 fs pulses with center wavelength of 800 nm. In our system, a femtosecond laser beam is directed towards a beam splitter. The transmitted beam passes through an emitter material, effectively producing THz pulses. The reflected beam serves as a probe pulse, traveling through a delay stage to a detector crystal. Just like in FTIR spectroscopy, when the THz beam strikes the sample, only some of the electromagnetic wave transmits. The THz beam that passes through the sample and the probe pulse are recombined and then sent through an electro-optic crystal. In the electro-optic crystal, the THz electric field changes the polarization of the probe pulse. This is ultimately how the THz pulse is detected. We can sweep the delay stage to measure the electric field as a function of time in the THz pulse.

The raw data provided by the system is the amplitude of THz transmission as a function of THz frequency. However, the relationship between THz transmission and complex conductivity,

$$\tilde{t} = \frac{1}{1 + \alpha\tilde{\sigma}} \tag{5}$$

where,

$$\alpha = \frac{\mu_o c}{1 + n_{substrate}}$$

provides a means to calculate the real and complex conductivity of the sample. Notice the inverse proportionality between transmission and conductivity - namely, conductive materials should reflect most THz radiation.

This is a novel technique that has a lot of potential to probe nano-scale phenomena. Notably, the difference between THz derived conductivity and the Hall Effect is that the optical estimate does not require homogenous films. Often times, the production of TCOs via sputtering can yield large batches of the composite material, but there may be cracks or other defects. Zero frequency, or DC conductivity measurements, can be inconsistent with even small film discontinuities. This speaks to why THz spectroscopy is a compelling practice - if THz and DC conductivity measurements agree, then the films must be relatively homogeneous and free of structural defects.

# 4 Results and Discussion

The series of $Cd_xZn_{1-x}O$ samples were analyzed with the Hall Effect, FTIR and THz spectroscopy. Data on important properties of the samples, such at the CdO concentration, film thicknesses, and crystalline structure are listed in Table 1. The data from our measurements are shown in Table 2.

| Sample | Content. | Film Thickness $d$ (nm) | Crystalline Phase | Substrate Type |
|---|---|---|---|---|
| CZO98 | 0.34 | 124 | Wurtzite | Glass |
| CZO145 | 0.53 | 137 | Mixed | Sapphire |
| CZO50 | 0.54 | 115 | Mixed | Glass |
| CZO115 | 0.64 | 170 | Mixed | Glass |
| CZO125 | 0.64 | 122 | Mixed | Sapphire |
| CZO142 | 0.73 | 130 | Rocksalt | Sapphire |
| CZO130 | 0.86 | 107 | Rocksalt | Sapphire |
| CZO144 | 0.90 | 130 | Rocksalt | Sapphire |
| CZO103 | 0.92 | 135 | Rocksalt | Glass |

Table 1: The samples studied in this work are across the composition range $Cd_xZn_{1-x}O$, where $0.34 < x < 0.92$. Note that increasing the CdO concentration makes the crystalline structure transition from wurtzite to rocksalt.

| Sample | Hall $\sigma$ ($\mathrm{S\,cm^{-1}}$) | THz $\sigma$ ($\mathrm{S\,cm^{-1}}$) | Hall $\mu$ ($\mathrm{cm^2\,V^{-1}\,s^{-1}}$) | Hall $N$ ($\mathrm{cm^{-3}}$) | FTIR $N$ ($\mathrm{cm^{-3}}$) |
|---|---|---|---|---|---|
| CZO98 | 99.2 | 121 | 9.4 | $6.59 \times 10^{19}$ | |
| CZO145 | 483 | 511 | 21.2 | $1.42 \times 10^{20}$ | |
| CZO50 | 557 | 609 | 21.4 | $1.63 \times 10^{20}$ | |
| CZO115 | 878 | 882 | 29.8 | $1.84 \times 10^{20}$ | $1.73 \times 10^{20}$ |
| CZO125 | 69.5 | 82 | 18 | $2.43 \times 10^{19}$ | |
| CZO142 | 1470 | 1540 | 32.7 | $2.82 \times 10^{20}$ | $2.5 \times 10^{20}$ |
| CZO130 | 167 | 140 | 48.7 | $2.19 \times 10^{19}$ | |
| CZO144 | 3720 | 3080 | 85.6 | $2.71 \times 10^{20}$ | $2.3 \times 10^{20}$ |
| CZO103 | 4050 | 4440 | 104 | $2.43 \times 10^{20}$ | |

Table 2: Conductivity measurements performed on samples with varying CdO concentrations. Note that FTIR carrier density estimates are omitted wherever the plasma frequency was too low to measure.

## 4.1 Hall Effect and THz Spectroscopy Measurements

The Hall Effect and THz spectroscopy estimates for conductivity are generally in good agreement, with a 5 - 20 % difference across all samples. This agreement suggests that the CdO/ZnO TCOs are homogeneous and free of cracks or structural defects. From the manufacturer's perspective, it suggests that the films are of good quality and maintain stability across a very broad range of the electromagnetic spectrum.

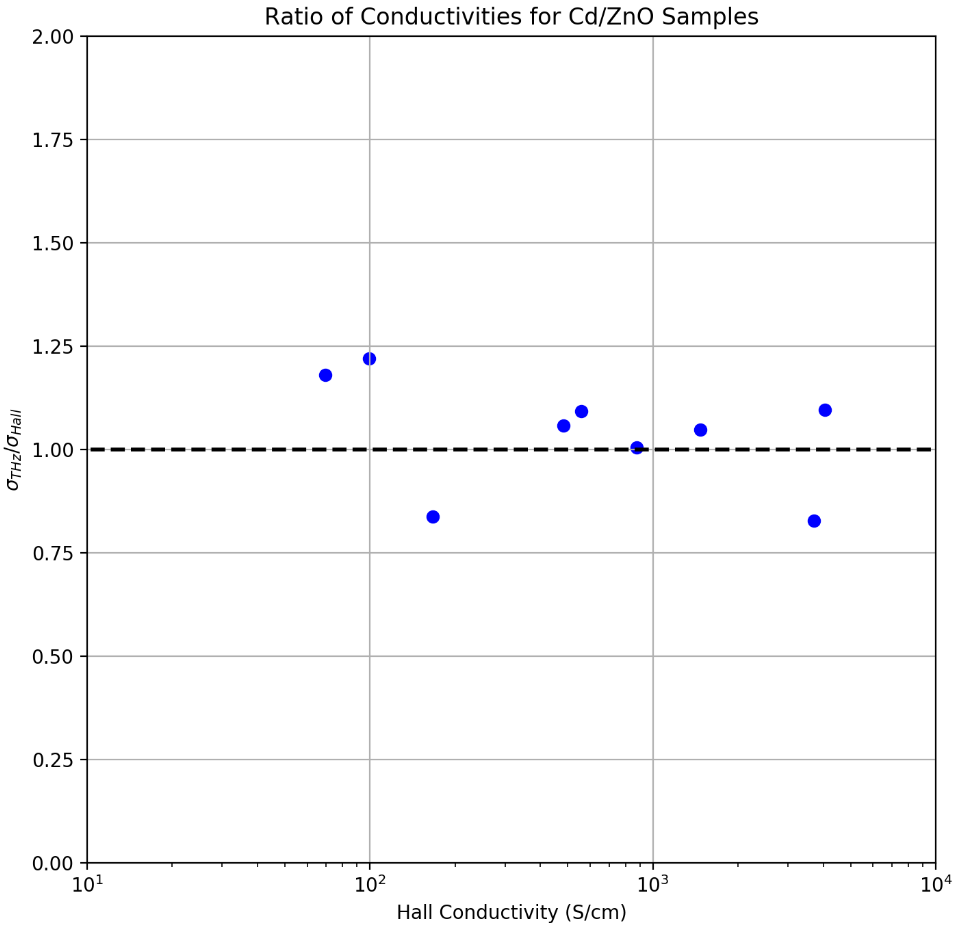
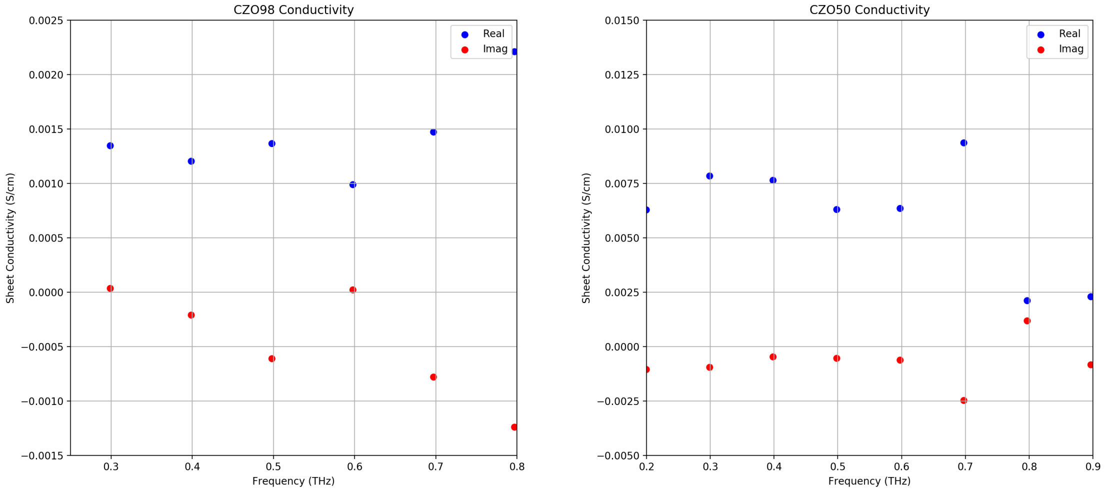
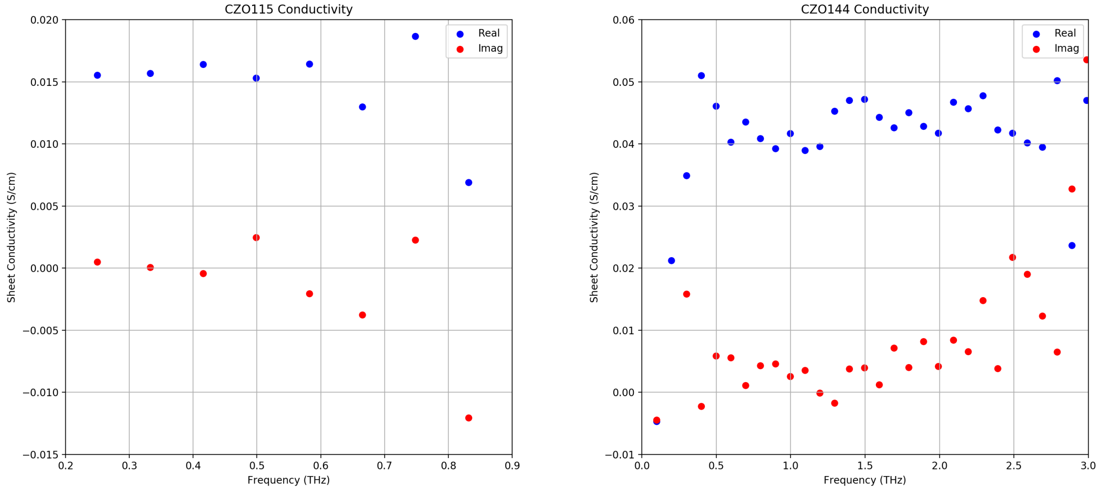


Figure 1: Visual representation of agreement between Hall conductivity and THz conductivity measurements - the dashed line indicates perfect agreement.

More importantly, THz conductivity measurements largely showed that most of the samples exhibit frequency independent conductivity that matched DC measurements, indicating that the are simple Drude metals.

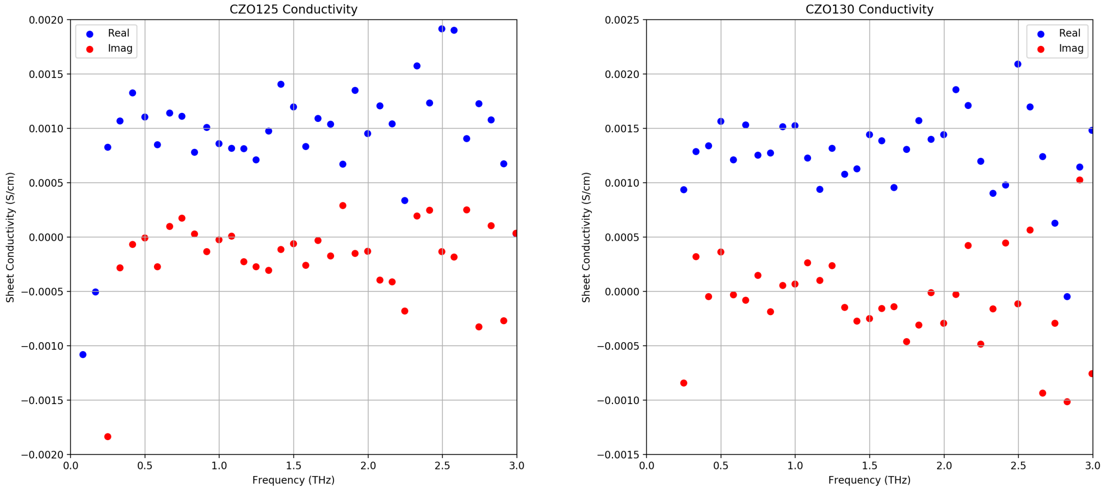


(a) CZO98 (b) CZO50



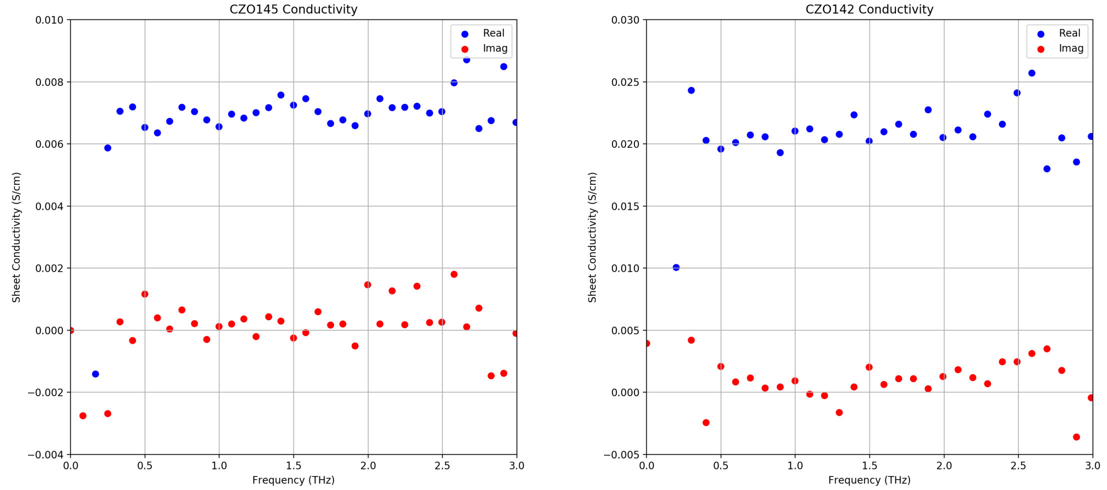
(c) CZO115 (d) CZO144

Figure 2: Sheet conductivity of CdO/ZnO samples as a function of THz frequency. Most samples show frequency independent Re[$\sigma$] and zero Im[$\sigma$].



(a) CZO125

(b) CZO130



(c) CZO145

(d) CZO142

Figure 3: Sheet conductivity of CdO/ZnO samples as a function of THz frequency. Notice that in general, Re[$\sigma$] is constant and Im[$\sigma$] is effectively zero.

However, one exception which arose in our analysis was sample CZO103. Sample CZO103 exhibits a decrease in the real part of the conductivity as we increased THz frequency. Even more peculiar is that sample CZO103 has the highest CdO concentration of all the samples in this research. We expected samples transitioning between wurtzite and rocksalt crystalline structures to behave unlike simple metals, however we did not expect CdO rich samples to behave this way. It's unclear what exactly is driving this behavior and warrants further investigation.

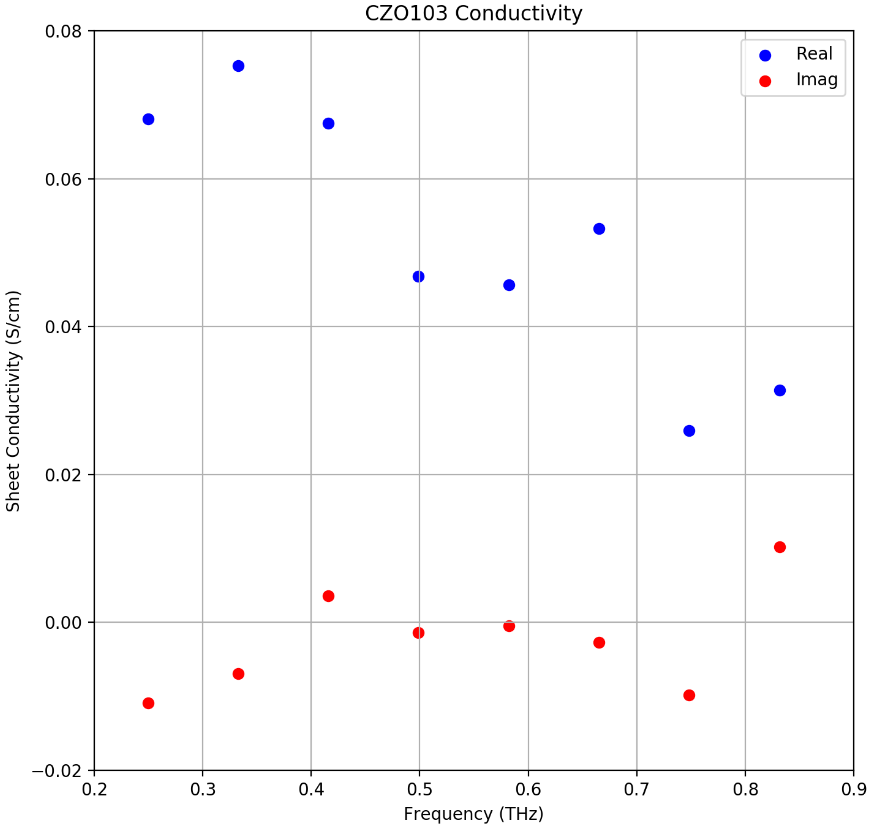


Figure 4: Sheet conductivity as a function of THz frequency for sample CZO103. This sample is the only one in the series that demonstrated a frequency dependent conductivity.

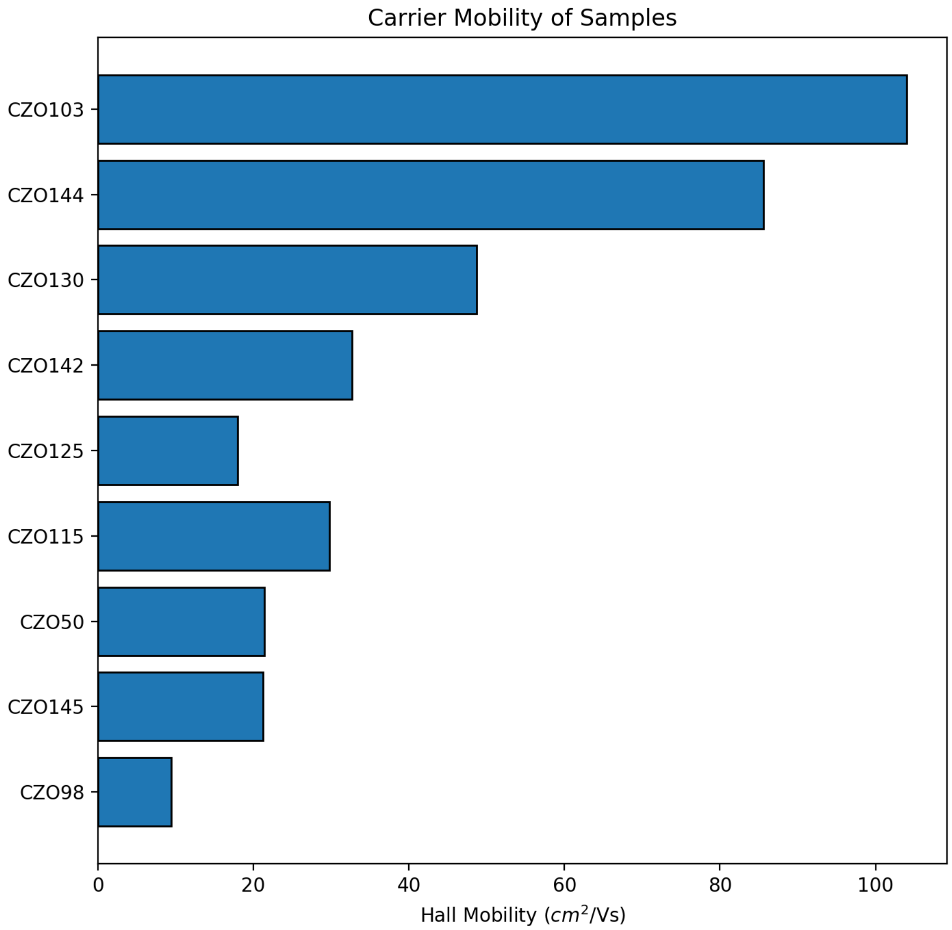


Figure 5: Hall Effect estimates for carrier mobility across the CdO/ZnO composition. Sample CZO98 has the lowest CdO concentration, and the samples above CZO98 have greater CdO concentrations, where CZO103 has the highest CdO concentration. Notice that samples in a rocksalt phase, such as CZO103 and CZO144, exhibit exceptionally large mobilities.

## 4.2 FTIR Measurements

FTIR spectroscopy proved to be less effective at probing the samples than anticipated, and only three of nine samples could be analyzed to extract estimates for optically derived carrier densities. The reason for this is that the plasma frequency for most samples was at lower frequencies than our measurements permitted; at these lower frequencies, the glass or sapphire substrate of the samples becomes opaque and prevents measurement. Nonetheless, optically derived estimates for the carrier concentration $N$ of three samples were obtained by fitting the infrared transmission to determine the plasma frequency $\omega_p$. The results are in good agreement with Hall Effect measurements.

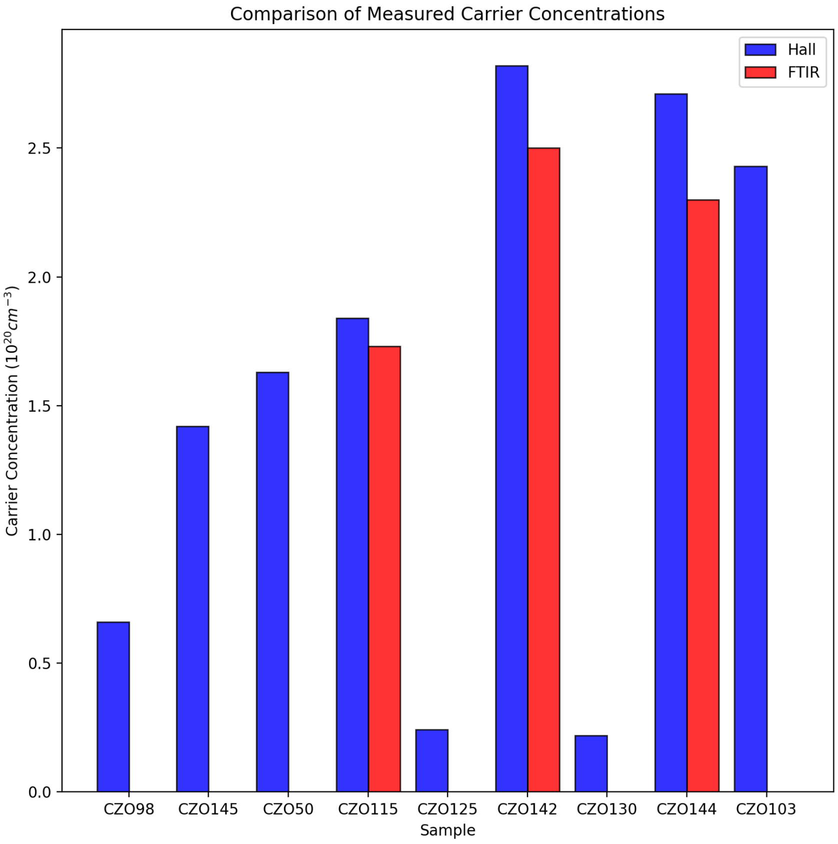


Figure 6: Comparison of Hall Effect and FTIR estimates for the carrier mobility of samples CZO115, CZO142, and CZO144.

Notice that not only are the Hall Effect and FTIR measurements of the carrier concentration comparable, but for all three samples in which a comparison was possible, carrier concentrations derived from FTIR measurements are less than their Hall Effect counterparts. This is interesting, because in principle, optically derived estimates of $N$ could be greater than DC measurements. This is because infrared radiation could excite localized electrons that do not normally contribute to the conductivity at DC. That the results agree in the way we found also presents other questions about these materials that could be explored further.

## 5 Summary and Conclusions

With the development of more complicated microcircuits and a need for better anti - reflective coatings for photovoltaics, testing exotic composite materials' conductivity at higher frequencies is necessary. With the opportunity to examine novel materials such as these CdO/ZnO thin films, we made comparisons across three different experiments to test for changes in conductivity as a function of frequency. The overall agreement in physical and optical conductivity shows that these films contained few flaws and behave like classical resistive metal films. This is promising for fabricators of TCOs, but from a scientific perspective, we were left with more to be desired. In the end, Hall Effect measurements proved to be an effective way to characterize the electrical properties of these films, and it makes our THz conductivity measurements seem superfluous. Fortunately, the search for a competitive alternative to ITO thin films is still on, and there is no shortage of motivation to research the properties of transparent conducting oxides.

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