THE PENNSYLVANIA STATE UNIVERSITY
SCHREYER HONORS COLLEGE

DEPARTMENT OF PHYSICS

CAPACITIVELY COUPLED CONDUCTIVITY DETECTION INSTRUMENTATION AND DESIGN

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Abstract

Capacitively Coupled Contactless Conductivity Detection (C^4D) is a method for identifying ions and biomolecules in solutions separated by capillary electrophoresis. A C^4D device operates by applying an AC voltage across two electrodes set up near to a capillary tube, allowing it to measure the electrical conductivity and permittivity of the substance inside the tube. This technique does not require direct contact with the fluid under observation, giving it several advantages over conventional conductivity detection devices. In this thesis our aim was to develop and optimize a sensitive C^4D device able to operate at high frequencies of AC excitation voltage, allowing for identification of a wider range of ions and biomolecules. We studied and simulated transmission line characteristics, configuration of the excitation electrodes, and electrical shielding in efforts to increase the sensitivity of our devices.

Acknowledgements

The completion of this thesis and my undergraduate education was made possible by the help, support, and guidance of several groups and individuals. Alex Kozhevnikov was instrumental in guiding me through all of the papers, techniques, and approaches I needed to study for this thesis, as well as giving my project direction. I greatly appreciate his patience and helpfulness throughout this research, and without him this thesis would not have been possible. I also had the pleasure of working on this project with fellow undergraduate David McLaughlin. David fabricated several of the waveguides and worked with me on calculations and MATLAB simulations, and it has been fun working with him. I would also like to thank Rick Robinett for his help and enthusiasm throughout my undergraduate education. He has always been there to answer my questions about planning my coursework and summer research opportunities, and he has played a large part in making my Penn State experience fun and rewarding. This work was also made possible by a Teas scholarship through the Research Experiences for Undergraduates grant.

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C^4D	Capacitively Coupled Contactless Conductivity Detection
CE	Capillary Electrophoresis
CPW	Coplanar Waveguide

1 Introduction

Capacitively coupled contactless conductivity detection (C^4D) has been the focus of increasing attention over the last fifteen years. It is used in identifying chemical species separated by capillary electrophoresis (CE) and can detect samples which cannot be identified through optical means. Conductivity detection applies a voltage to a fluid to measure the electrical conductivity σ and permittivity ε of a fluid, which is useful for determining the presence of ionic molecules in a solution.

It is also of interest to measure these electrical parameters over a wide range of applied voltage frequencies. Large biomolecules have dipole moments which will align themselves along an external electric field, which will contribute to the permittivity and conductance of a fluid. If the voltage frequency is too high, the molecules will not have time to orient themselves along the electric field, causing a drop in permittivity. These drops in permittivity can be used to infer the size of biomolecules in the solution.

Contactless detection shows several advantages over conventional techniques which require direct galvanic contact with the fluid under test. Because no direct contact with the fluid is required, construction and alignment of devices is simpler. Contactless devices can also be scaled down more effectively than conventional methods, saving researchers time and money in identifying ionic specimens in samples. The contactless method eliminates the problem of reactions and corrosion at the electrodes, and the devices are generally more durable and rugged.

For these reasons there is motivation to design a C^4D device which is sensitive and can operate over a wide frequency range.

2 Previous Research

Capacitively Coupled Contactless Conductivity Detection was originally developed in the 1950's for use in measuring titrations, but it was later shown to have potential for use with CE. Gas et al. were the first to report how C^4D could be used to identify samples separated by CE[1]. They developed a detector featuring four electrodes arranged radially around a capillary tube through which the sample was run. As ionic specimens in the sample passed under the electrodes, conductivity between the electrodes increased, and different molecules could be identified depending on the change in conductance. A flurry of interest in this procedure was aroused when a new C^4D detector was unveiled independently by Zemann et al. and da Silva and do Lago[2,3]. They described a detector featuring two electrodes wrapped around the capillary tube in an axial arrangement, as shown in Fig. 2.1.

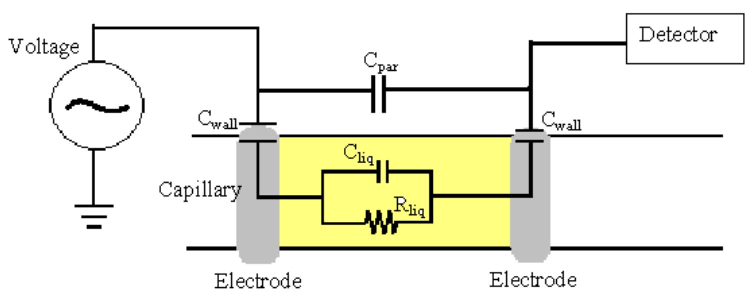

Figure 2.1: Schematic of a C^4D device

An AC source was connected to one electrode, and the other electrode was connected to an AC receiver. This setup could detect the electrical conductance of the fluid in the capillary tube, allowing for detection of ionic specimens within the sample. This setup greatly simplified construction of C^4D

devices, and many groups were able to use this technique to build their own devices and conduct new research.

2.1. *Improvements in Sensitivity*

C^4D operates by measuring the conductance of the test solution between the electrodes, but there is also conductance along other paths outside of the solution. Conduction along these paths introduces a background signal which is not useful with respect to detection. The existence of these baseline signals reduces sensitivity and range of detection in C^4D setups. Several approaches have been investigated for reducing or eliminating the effect of these paths and improving the performance of new devices.

Optimizing the dimensions and orientation of the pickup electrodes can have a helpful effect on device sensitivity. Computer simulations of conductance in various setups show that shape and orientation of electrodes can affect the sensitivity, but the distance between the electrodes is reported to have little effect[4]. Groups have investigated several different electrode designs and geometries and how they affect detection sensitivity[5].

Another approach is to introduce a grounded shield plane in between the two electrodes. This splits the conductance through the air into two paths: between the excitation electrode and the ground plane, and between the ground plane and the receiver electrode. These paths will not have an effect on the signal, and with proper shielding sensitivity can be greatly improved[4,6]. Though the effect of shielding is greatly beneficial for sensitivity, simulations have suggested that there may be adverse effects as well. If the shielding plane is too close to the analyte in the capillary, there will be capacitive coupling between the analyte and the grounding plane, which has an unfavorable effect on device calibration for solutions with low conductivity[4].

A recent study has shown that stray conductance can be even further reduced by introducing an inductor unit in series with the circuit[6]. It is known that it is possible to cancel the impedance of a capacitor at a given signal frequency by introducing a well selected inductor in series with it. This will establish resonance, and the result will decrease the impedance of the circuit. In this way, an inductor unit can be used to cancel the capacitive effects of the space in between the two electrodes and the capillary tube wall. This configuration has been demonstrated to greatly increase sensitivity and detection range in a C^4D device. This method, however, will only work at a given signal frequency; for other frequencies the inductance will need to be chosen differently.

Some groups have aimed to increase the signal to noise ratio in their devices by applying higher excitation voltages to the electrodes[7,8,9]. Tanyanyiwa et al. used peak to peak voltages of 250 V across the electrodes and used a pickup amplifier close to the electrodes[8]. They tested this technique on inorganic cations and showed that high excitation voltages can be used to increase the target signal and improve the signal to noise ratio. Groups have also shown that these high voltage techniques can be integrated into a fully portable C^4D instrument[9].

2.2. *Implementations in Microchips*

Over the last decade interest has been increasing in developing 'lab on a chip' devices, which are small microfabricated chips with channels built into them for fluid injection and measurement. These microfluidic chips require less fluid to make measurements, and they improve measurement speed. They can be small, mass produced, and disposable, and they will increase the portability of detection devices.

An increasing number of detection techniques are being tested for use in these microchips, including conductivity detection. C^4D has been adapted for small scale use and tested in 'lab on a chip' devices[10,11,12,13]. Pumera et al. reported a C^4D configuration that compared favorably with other conductivity detectors[10]. They observed that contactless methods could accommodate greater separation voltages on the ions in the fluid, decreasing the time to make each measurement. C^4D sensitivity in microfluidic applications can be further improved by optimizing the electrode design and shape[12]. A separate group has demonstrated a microfluidic device sensitive enough to detect DNA by conductivity measurement for the first time[13].

2.3. Combination with other Detection Techniques

A useful and recently developing characteristic of C^4D is that it can be combined with other detection techniques to be effective on a larger range of chemical species. Several groups have successfully built detectors that integrated C^4D with optical, photometric, and fluorometric detection techniques[14,15,16].

3 Transmission Line Design

The transmission lines which carry the AC signal from the generators to the electrodes must be carefully selected when designing C^4D devices. Transmission lines contain resistive, capacitive, and inductive elements that will affect the performance of the line. The characteristic impedance of the line, Z_0, describes all three of these components.

If a transmission line of impedance Z_0 is terminated with a load of impedance Z, some of the incident signal will be reflected at the interface between them as described by

$$\frac{V_{ref}}{V_{inc}} = \Gamma = \frac{Z_0 - Z}{Z_0 + Z} \quad (1)$$

where Γ is the reflection coefficient. Unless the impedances of the two materials are matched, some of the incident signal will be reflected back to the source, shown in Fig. 3.1.

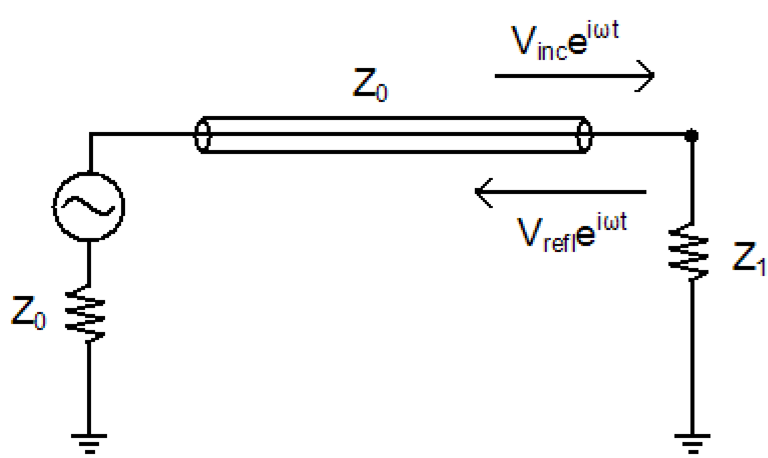

Fig. 3.1. Signal reflection due to unmatched load and transmission line

For the purpose of a conductivity detector, we want as much of our generated signal to reach the capillary tube as possible. Any reflection of the signal at the interface between the source and our transmission lines will reduce our device's sensitivity. To maximize the signal transmitted to the capillary tube, we designed the transmission lines to have the same impedance as the signal generator and detector.

3.1. Microstrip

A microstrip is a transmission line that is useful for microwave frequency applications. An electrical signal is carried by a thin conducting strip separated from a ground plane by a dielectric, shown in Fig 3.1.1.

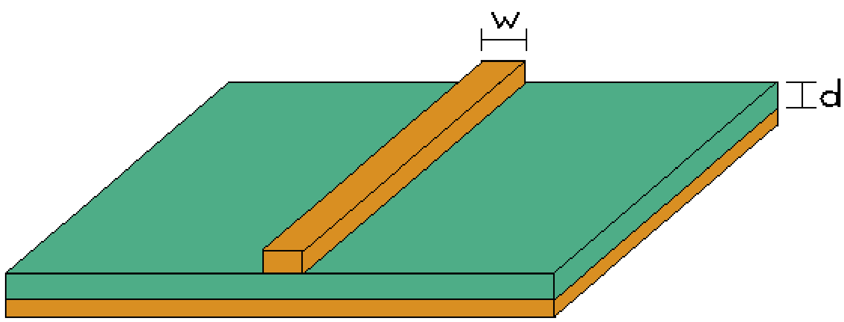

Figure 3.1.1 Microstrip Design

The characteristic impedance of this transmission line is given by [17]

$$Z_0 = \frac{60}{\sqrt{\varepsilon_e}} \ln\left(\frac{8d}{w} + \frac{w}{4d}\right) \qquad (w/d \leq 1) \qquad (2)$$

$$Z_0 = \frac{120\pi}{\sqrt{\varepsilon_e}\left[\frac{w}{d} + 1.393 + 0.667\ln\left(\frac{w}{d} + 1.444\right)\right]} \qquad (w/d \geq 1) \qquad (3)$$

This dependence on the width to depth ratio is plotted below in Figure 3.1.2.

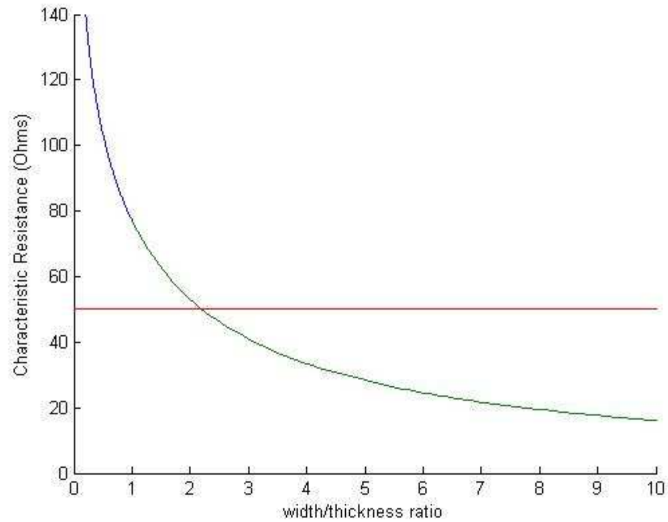

Figure 3.1.2: Characteristic Impedance as a function of width/depth

We used this information to design microstrips with the same 50Ω impedance as our coaxial lines, spectrum analyzer, and signal generator.

To test the impedance of these microstrips, we applied a signal to them and measured the power reflected. To isolate the signal reflected from the microstrips, we connected the strips to a directional coupler, represented by Fig. 3.1.3. The initial signal is sent into the 'COUPLED' port of the directional coupler. From here, the signal flows to the 'IN' port, which is connected to a load. The signal will be partially reflected by the load as governed by Eq. 1 and flow to the 'OUT' port.

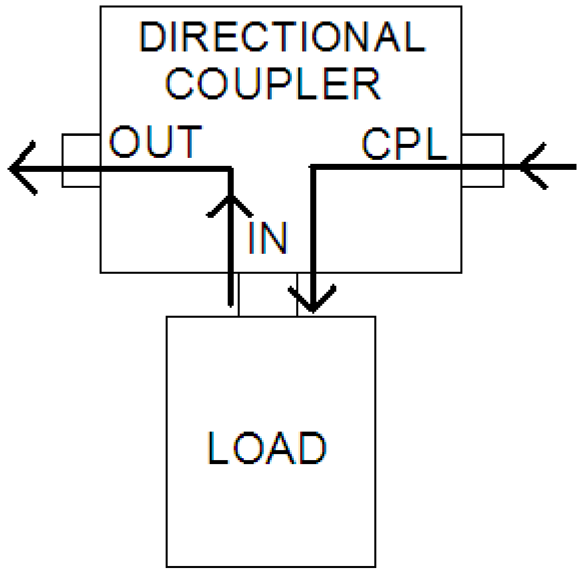

Fig. 3.1.3. Using a directional coupler to measure reflection by a load

The results from our first microstrip test are shown below in Table 3.1.1.

Table 3.1.1. Reflected signal from open, 50Ω, and microstrip terminated with 50Ω loads.

Frequency (MHz)	No Microstrip, open circuit (dB)	No Microstrip, 50Ω termination (dB)	With Microstrip, 50Ω termination (dB)
100	-23	-59	-52
250	-20	-60	-51
500	-21	-54	-52
750	-22	-49	-48
1000	-22	-47	-48

Without a microstrip and no 50Ω terminator, all of the incoming signal is reflected. When the 50Ω terminator is attached, all the power should be absorbed by the load and the reflected signal should be a minimum. When the microstrip is connected and terminated with 50Ω, signal reflection will be dependent on the mismatch between microstrip impedance and line impedance. The magnitude of the power reflected is the difference between the runs with and without the microstrip and 50Ω terminator. The calculated reflection coefficients and impedances of our microstrip are shown below in Table 3.1.2.

Table 3.1.2 Reflection coefficient and impedance at different voltage frequencies

Frequency (MHz)	Magnitude of Reflection Coefficient $\|\Gamma\|$	Magnitude of Impedance Difference $\| Z_{strip} - Z_0 \|$ (Ohms)
100	.035	3.6
250	.040	4.2
500	.035	3.6
750	.056	5.9
1000	.056	5.9

3.2. Coplanar Waveguide

The Coplanar Waveguide (CPW) is similar to a microstrip in many respects and is also useful in high frequency applications. It features a central conducting strip as the microstrip does, but has a grounded strip on either side of the central conductor. These are all separated from a ground plane in the base of the strip by a dielectric slab of thickness h. A diagram is shown below in Fig. 3.2.1.

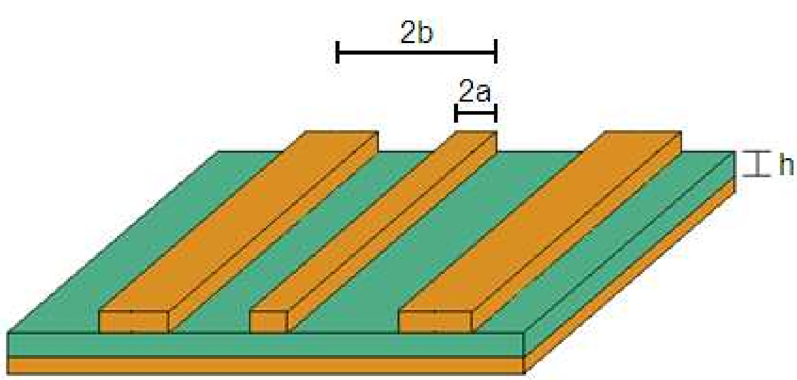

Fig 3.2.1. Coplanar Waveguide Design

The effective permittivity and characteristic impedance of a CPW have been determined[18] to be

$$\varepsilon_{eff} = \frac{1+\varepsilon_r \frac{K(k')\,K(k_3)}{K(k)K(k_3')}}{1+\frac{K(k')\,K(k_3)}{K(k)K(k_3')}} \tag{4}$$

$$Z_0 = \frac{60\pi}{\sqrt{\varepsilon_{eff}}} \frac{1}{\frac{K(k)}{K(k')}+\frac{K(k_3)}{K(k_3')}} \tag{5}$$

where

$$k = a/b$$

$$k_3 = \tanh(\pi a/2h) \,/\, \tanh(\pi b/2h)$$

$$k' = \sqrt{1-k^2}$$

$$k_3' = \sqrt{1-{k_3}^2}$$

and K(k) represents the complete elliptic integral of the first kind.

We simulated these equations in MATLAB to determine the dependence of characteristic impedance on the width of the central conductor. The results of this simulation are shown below in Fig. 3.2.2.

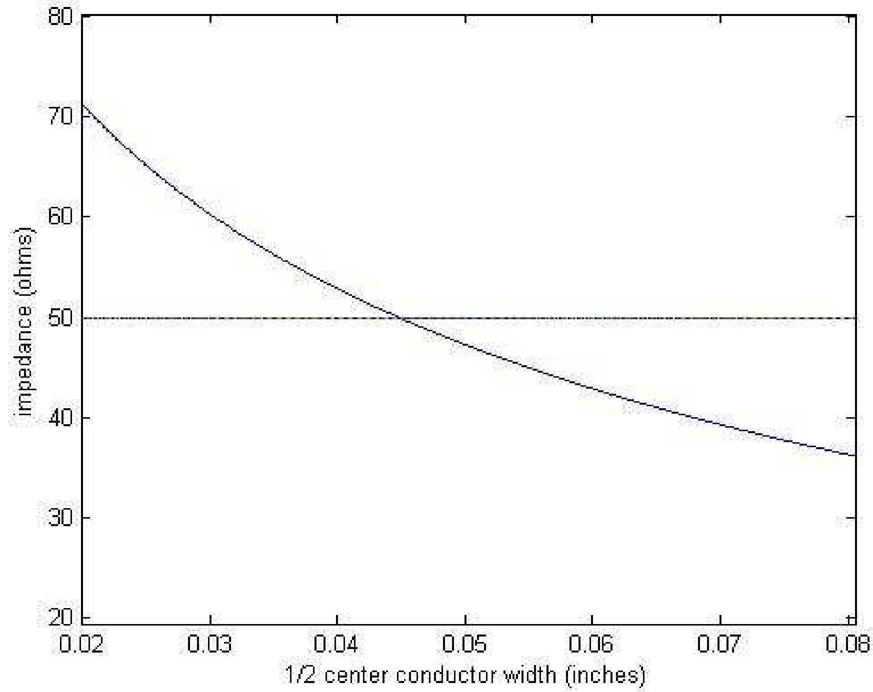

Fig. 3.2.2. CPW impedance versus width of the central conductor. This simulation used a dielectric 55 mils thick, permittivity constant of 3.66, and gap between central conductor and ground strip of 20 mils.

We used these equations to design a CPW with an impedance closely matched to our detector and signal generator. In order to test the impedance of the CPW, we measured the reflectance in the same manner used for our microstrips described in 3.1. Fig. 3.2.3 shows the power reflected for configurations with and without our CPW in the load.

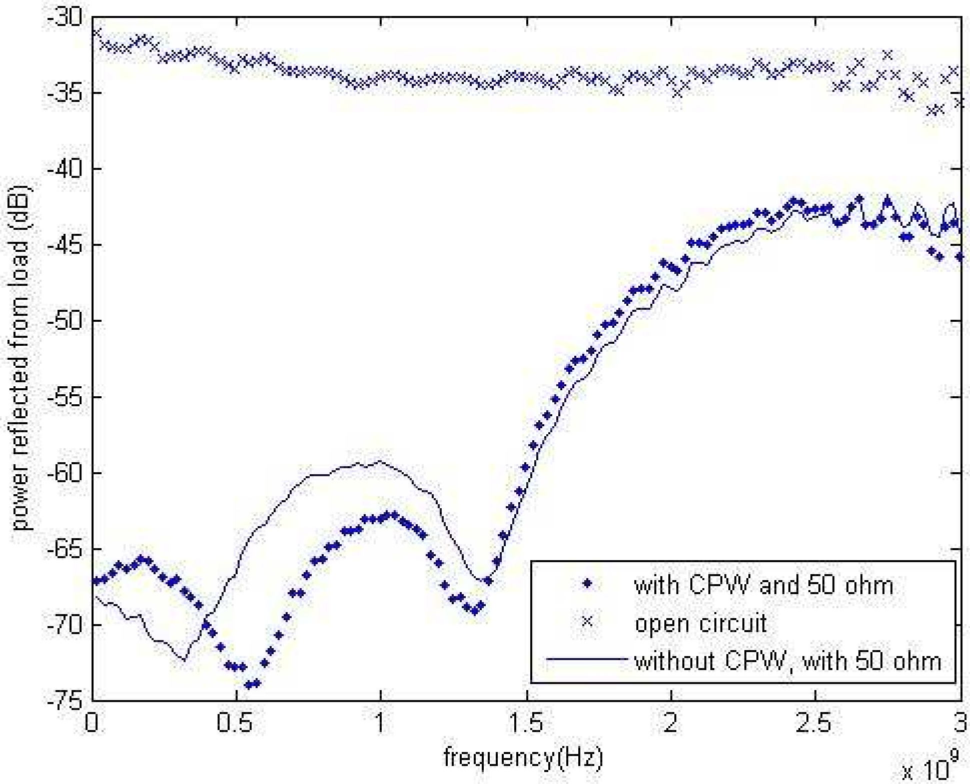

Fig.3.2.3. Reflection test for our coplanar waveguide. The solid line represents the received power when the load was a CPW terminated with 50Ω. The dotted line represents the received power when the load was only the 50Ω termination. The x-ed line represents power reflected when the load was an open circuit.

4 Electrode Design and Geometry

A C^4D device can be represented by the following circuit in Figure 4.1.

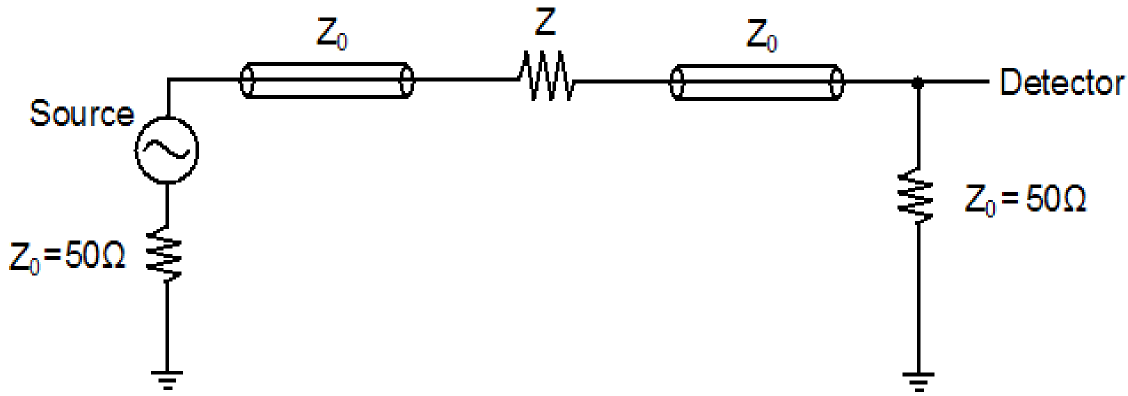

Fig. 4.1 Schematic of a C^4D device.

Here Z represents the impedance between the electrodes of the device. This will depend on the capacitance of the fluid filled capillary C_0, the conductivity of the fluid in the capillary σ_0, and the parasitic capacitance of the air outside of the capillary C_{par}.

Only a portion of the original signal will be measured at the detector, given by

$$\frac{Detector\ Signal}{Source\ Signal} = |T| = \frac{2Z_0}{2Z_0 + Z} \quad (6)$$

where, for a nonconducting fluid,

$$Z = \frac{1}{j\omega(C_{par} + C_0)} \quad (7)$$

By measuring the transmission coefficient T, one can solve Eq. 6 and determine the impedance between the excitation electrodes.

A problem here is that the capacitance through the air between the electrodes, C_{par}, is independent of the fluid that fills the capillary. This results in a baseline level in the total impedance Z. The larger C_{par} is, the less the transmission coefficient T depends on the solution inside the capillary tube, and the less sensitive the C^4D device will be.

The relative sizes of C_{par} and C_0 are largely determined by the setup of the excitation electrodes. To maximize the sensitivity of the device, we sought to design electrodes that minimized the parasitic capacitance.

4.1. Cylindrical Electrodes

Most C^4D devices currently being studied use tubular, cylindrical electrodes wrapped around the capillary tube as shown in Fig. 4.1.1.

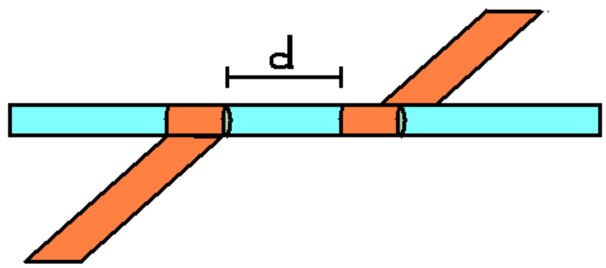

Fig. 4.1.1. Cylindrical electrode configuration

This configuration is simple to construct and has the added benefit of stabilizing the capillary tube.

We built our cylindrical electrodes in one of two ways. Our first approach was to scrape the adhesive off of a small strip of copper tape and wrap it tightly around the capillary tube. Our other approach was to paint the contacts onto the capillary with silver paint.

4.2. Opposing Strip Electrodes

We also tested the effectiveness of using thin strip electrodes on the capillary. Our hope was that more of the signal would flow through the fluid between the electrodes rather than through the air around the capillary. Our setups were similar to the one shown in Fig. 4.2.1.

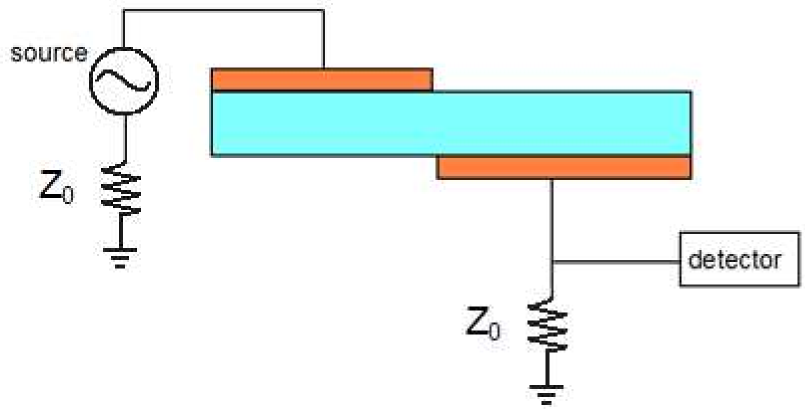

Fig. 4.2.1. Opposing strip electrode configuration

To make the electrodes in this configuration we used either copper tape or silver paint. When using the copper tape, we cut the tape into strips and used scotch tape to secure the strips snugly to the capillary tube to reduce parasitic capacitance through the air.

4.3. Shielding

The use of shielding between the electrodes can substantially improve the sensitivity of a C^4D instrument. Placing a ground plane in between the electrodes will reduce the capacitance through the air between them, which will make the behavior of our transmitted signal more dependent on the properties of the fluid inside the capillary.

We chose to investigate several different approaches to implement shielding between our electrodes. The first was to use a coplanar waveguide with one of the ground strips acting as a shield, as shown in Fig. 4.3.1.

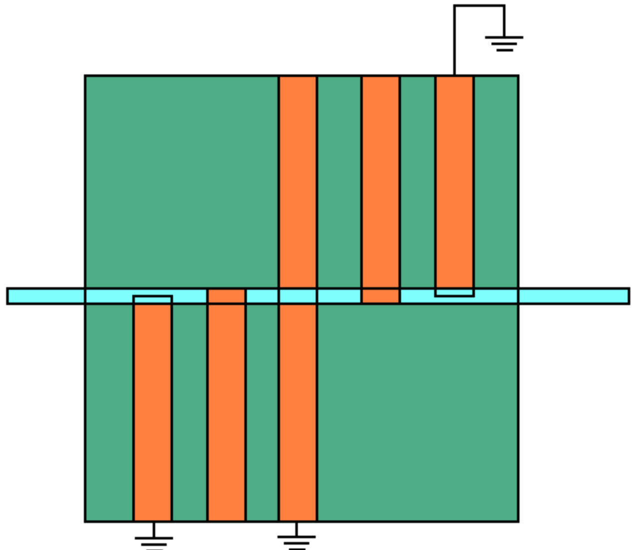

Fig. 4.3.1. Using a CPW for shielding. The middle ground strip is shared between two coplanar waveguides coming from either side of the board, and it will act as a shield between the two signal carrying electrodes.

We also tested a design in which the two signal electrodes were on different planes separated by a ground plane, as shown in Fig. 4.3.2. For this 'sandwich' design we used microstrips to carry the signal towards the capillary tube. We soldered a wire and a conducting ring to the end of the microstrip, where it went into the dielectric slab and wrapped around the capillary.

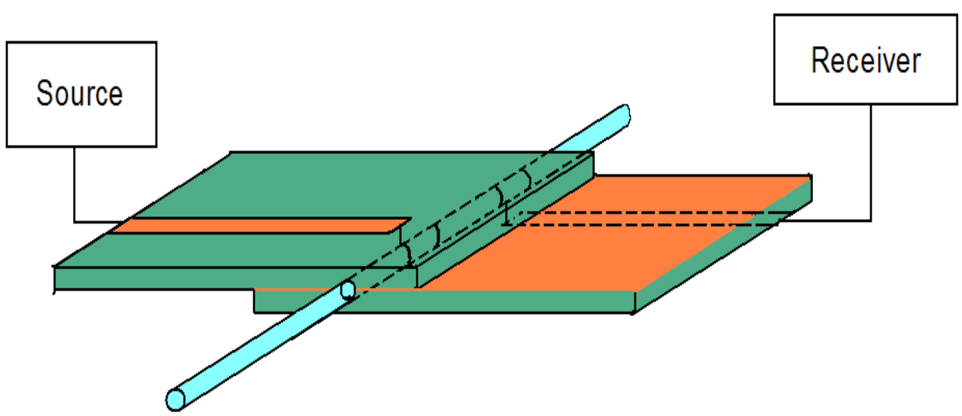

Fig. 4.3.2. The 'sandwich' shielding design. Two microstrips are adjoined with their ground plates touching and microstrips on opposite sides. A wire and conducting loop is connected to the end of the microstrip runs through the dielectric. The loop wraps around the capillary to form an electrode.

The last shielding approach we investigated was using coaxial cables as transmission lines. The ends of the coaxial lines were stripped, and a small conducting collar between the two conducting lines was soldered to the ground of each cable, as shown in Fig. 4.3.3.

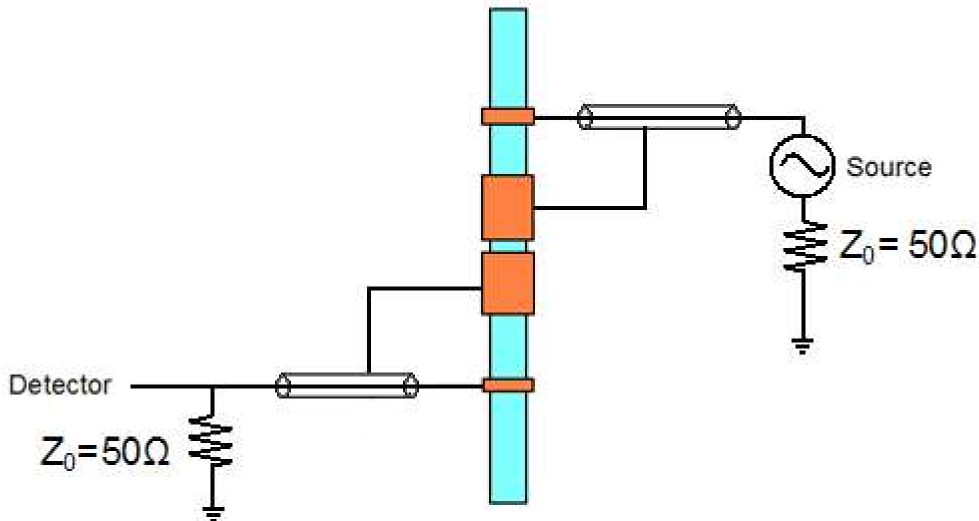

Fig. 4.3.3. Shielding configuration using coaxial cables. Two metal collars are soldered to the inner signal carrying conductors of the coaxial cable and are positioned the furthest from each other. Two other metal collars are soldered to the grounded outer conductor of the coaxial cable and serve as shielding between the two signal carrying electrodes.

5 Phase matching and Interference

Another strategy we used to improve the sensitivity of our device was to use interference to reduce the effects of parasitic capacitance. The interference circuit we used is shown below in Fig. 5.1. A signal is sent into a power splitter, dividing it between two arms of the circuit. In the test arm, the signal flows through our C^4D device to a power splitter on the other side of the circuit. In the reference arm, the signal flows through a variable attenuator, coupler, and line stretcher. The coupler and line stretcher work together as a phase shifter, which can be controlled by the user. At the splitter the two signals recombine and propagate to the output. By controlling the phase shift in the reference arm, we were able to cause destructive interference between the two arms of the circuit at the output.

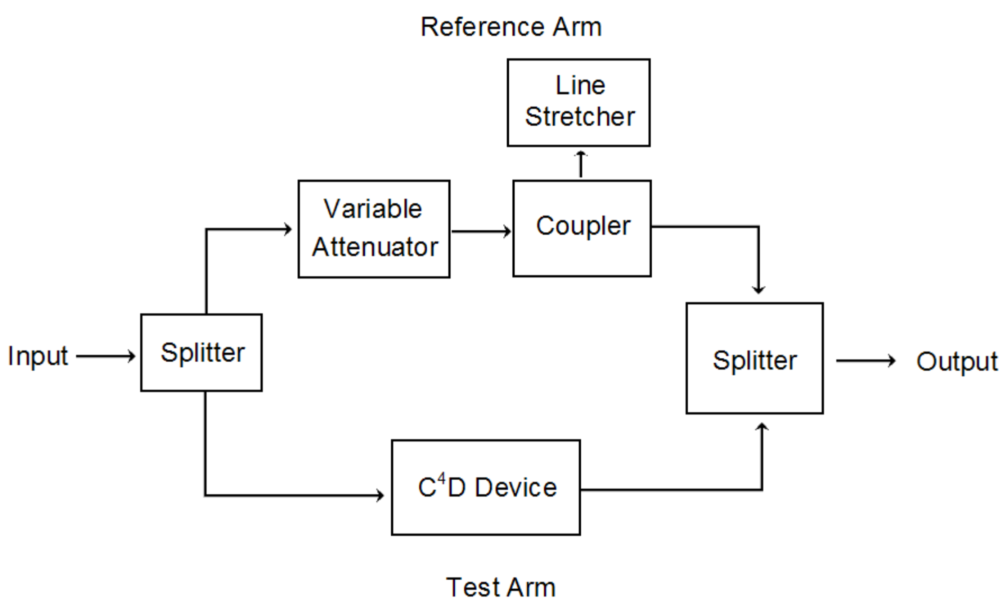

Fig. 5.1. Phase Shifting and Interference Circuit.

Using this interference, we set the magnitude of the output signal to be just above the noise floor of detection. We then filled the capillary with a test fluid and observed the change in the power transmitted to the output. This power increase will be entirely the result of the capacitance of the fluid inside the tube.

6 Testing Device Sensitivity

After constructing a C^4D device, we needed to test its sensitivity. To do this, we measured the power transmission through the device when the capillary was filled with either air or water. The transmission coefficient through our device can be found by combining Eq. 6 and Eq. 7 to get

$$|T| = \frac{2Z_0}{2Z_0 + \frac{1}{j\omega(C_{par} + C_0)}} \tag{8}$$

and because T is small, this relation can be approximated by

$$|T| = 2Z_0 j\omega(C_{par} + \varepsilon C_0) \tag{9}$$

where ε is the permittivity coefficient of the fluid in the capillary.

For C^4D, the device sensitivity is determined by how much the transmitted power changes due to a change in the test fluid. We can use this to determine the sensitivity of our device by the relation

$$S = \frac{1}{|T|} \cdot \left|\frac{dT}{d\varepsilon}\right| \tag{10}$$

Which, applied to Eq. 9, evaluates to

$$S = \frac{C_0}{C_{par} + \varepsilon C_0} \tag{11}$$

From Eq. 11 it is evident that to maximize device sensitivity, we must maximize $\frac{C_0}{C_{par}}$ in our devices.

To measure the sensitivity in our devices, we measured the transmission coefficient when the capillary was filled with either air or water. By comparing the difference between the power transmissions in the two trials, we were able to gauge the sensitivity of the device under test.

7 Results

We started our testing on the cylindrical electrode setup without shielding. We varied the separation between the cylindrical electrodes and measured the power transferred across the electrodes when the capillary was filled with either water or air. The average power difference between the two trials is shown in Table 7.1.

Table 7.1: Power transmission difference in unshielded cylindrical setup

Separation (mils)	Power Difference (dB)
9	3.2
15	3.5
44	3.2
55	3.0
80	2.6

The ideal spacing between the cylindrical electrodes was at 15 mils, corresponding to a power difference of 3.5 dB between the trials.

The opposing strip electrode configuration had similar performance when the electrodes were made with strips of copper tape. The data taken from our best setup with copper tape is shown below in Fig. 7.1. Interestingly, a decay in the power difference can be seen as the frequency increases past 1 GHz.

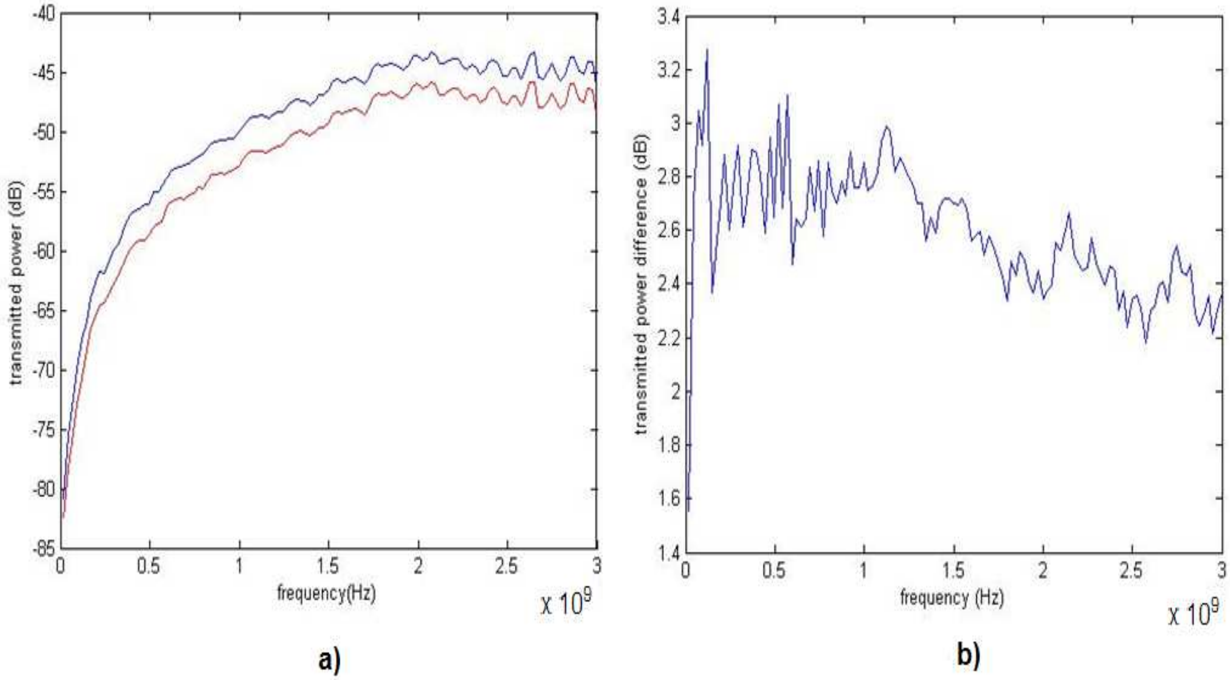

Fig. 7.1. Power transmission for opposing strip copper tape electrode setup. a) shows the power transmission for the air trial in red and the water trial in blue. b) shows the power difference between the two trials.

We also tested the opposing strip design with silver paint as the electrodes instead of the copper tape. This increased the performance slightly over the copper tape design, as shown in Fig. 7.2.

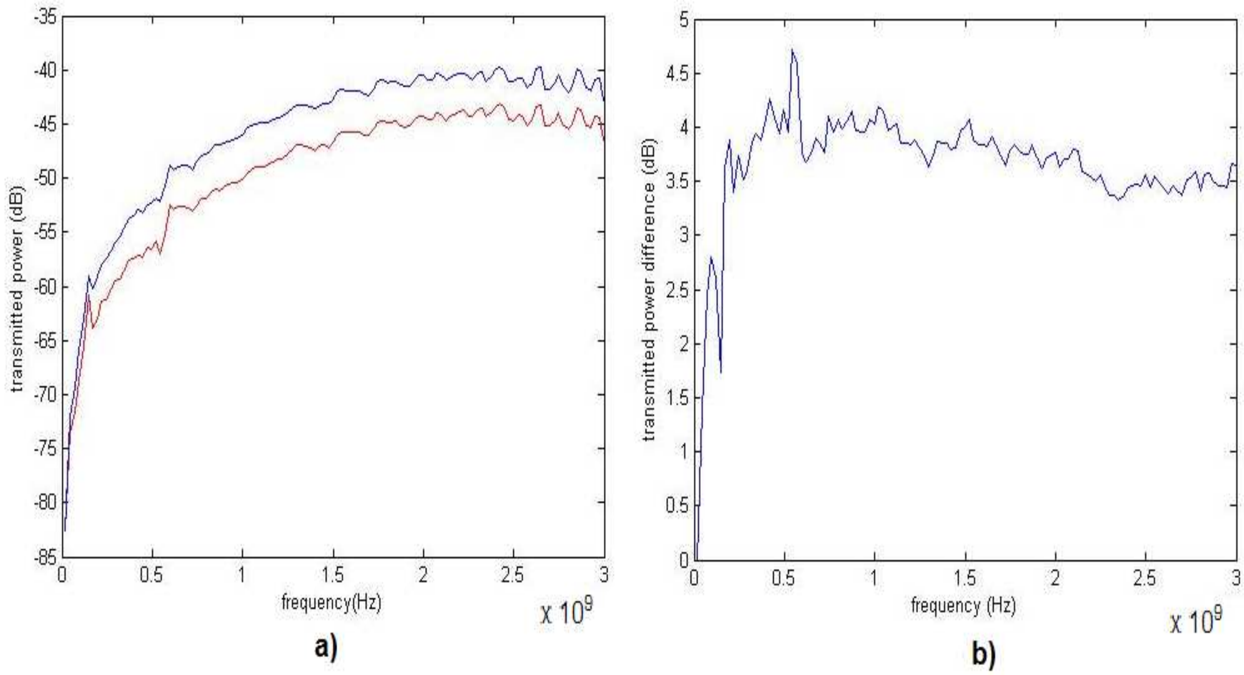

Fig. 7.2 Power transmission for opposing strip silver paint electrode setup. a) shows the power transmission for the air trial in red and the water trial in blue. b) shows the power difference between the two trials.

The performance of this setup showed a power difference of roughly 3.8 dB, and less decay of the sensitivity at high frequency than previous setups.

For the shielded designs, we used a smaller diameter capillary, which decreased the relative sensitivity of our measurements.

When we tested the CPW shielding design, the power difference between the air and water trials remained below 1 dB. Also, the device exhibited odd behavior as shown in Fig. 7.3.

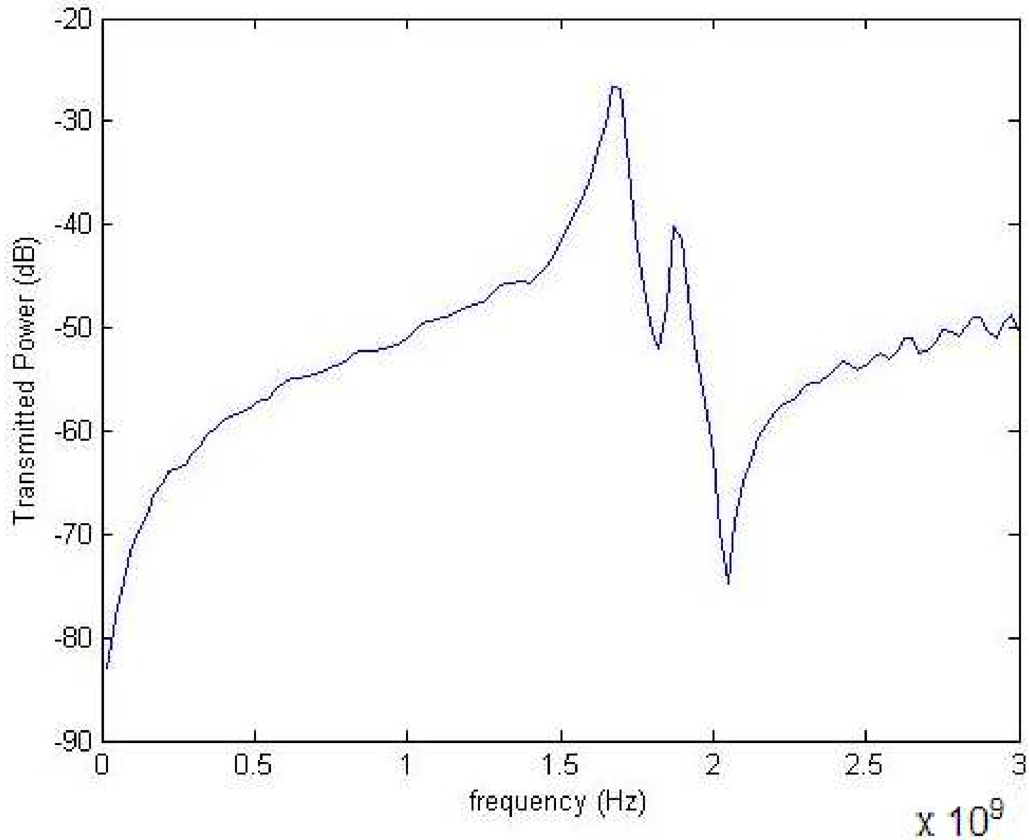

Fig. 7.3. Power transmitted across CPW shielding device with no capillary.

It is possible that at 1.6 GHz the device exhibited antenna-like behavior, with the input conductor acting as the transmitter and the output conductor acting as the receiving antenna.

The ‘sandwich’ design proved to be more effective at low frequency, with a power difference of 12 dB below 500 MHz, but performance quickly dropped off as frequency increased, as shown in Fig. 7.4.

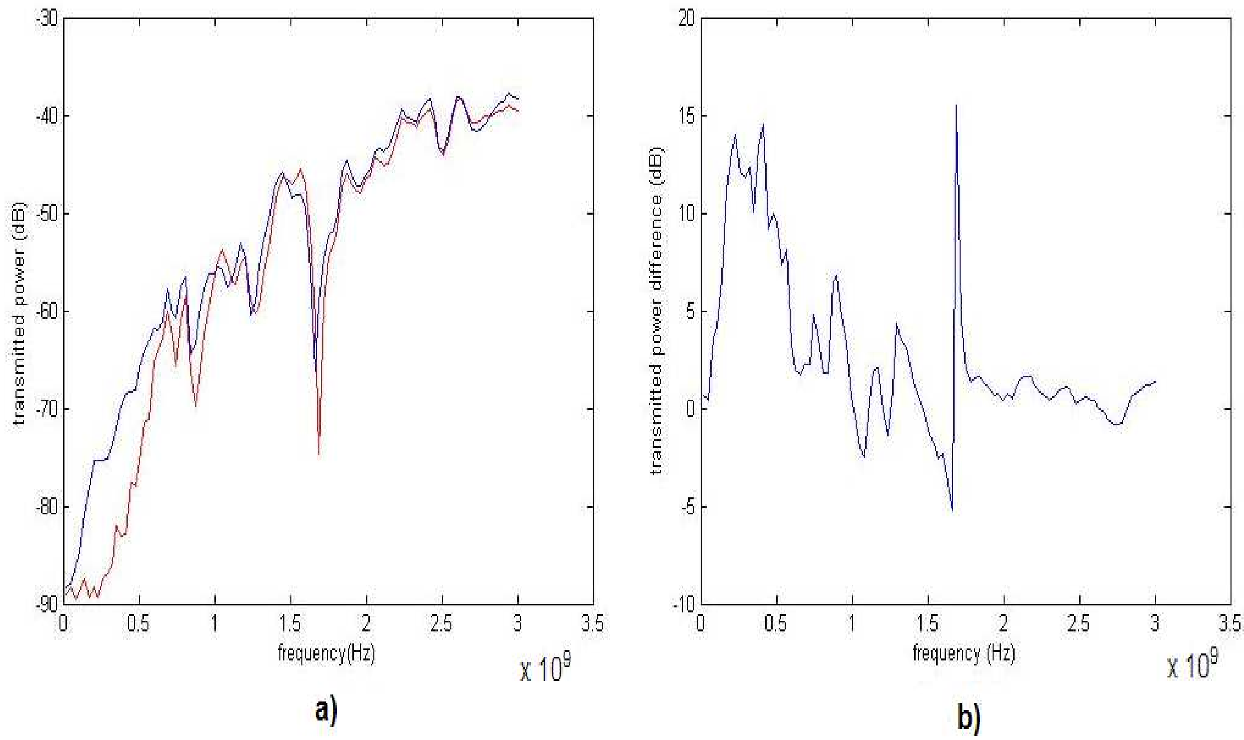

Fig. 7.4. Power transmission data for sandwich shielding design. There is a sharp decline in transmission at about 1.6 GHz for both trials.

We also tested the design using the coaxial cable approach to shielding. We only tested the device from 20 MHz to 1.2 GHz, and the device performed better than the sandwich design. We measured sustained higher sensitivity up to 1.2 GHz, as shown in Fig. 7.5.

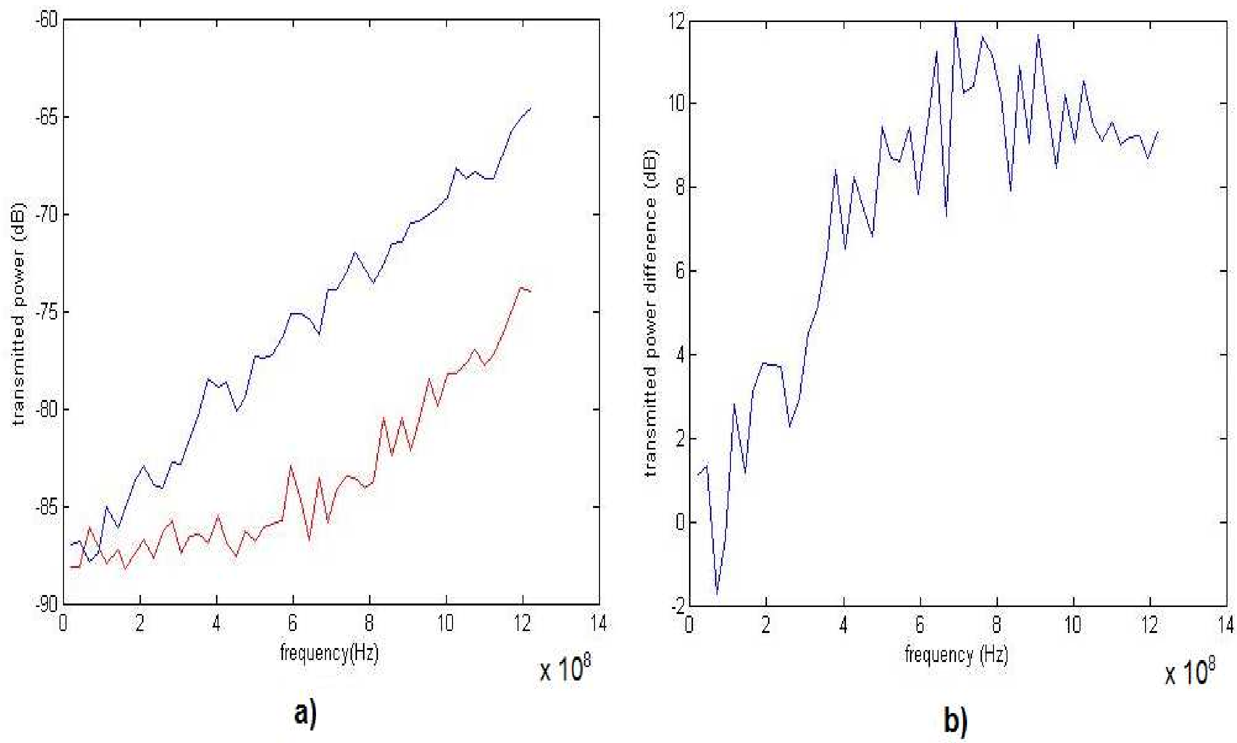

Fig. 7.5 Power transmission for coaxial shielding design. a) shows the power transmission for the air trial in red and the water trial in blue. b) shows the power difference between the air and water trials.

To test our interference circuit we used the ‘sandwich’ shielding setup for our C^4D device. With the capillary filled with air, we configured the circuit to output a small signal just above the noise floor of detection. We then filled the capillary with water and observed the change in the power transmission, shown below in Table 7.2.

Table 7.2. Power transmission in the interference circuit

Frequency (MHz)	Transmitted power with air filled capillary (dB)	Transmitted power with water filled capillary (dB)	Difference in transmitted power (dB)
200	-85	-78	+7
300	-80	-72.5	+7.5
350	-81	-71	+10
400	-80	-72.5	+7.5
450	-80	-70.5	+9.5

Our frequency range was limited by the phase shifter we used, which was functional only from 180-450 MHz.

8 Discussion and Conclusions

In this thesis we have investigated several different configurations and approaches for building a C^4D device. Though all of the aspects of design we investigated can affect device performance, some have more influence than others.

Transmission line design is very important to the propagation of the signal to the device. If the transmission line impedance is poorly matched to the source and detector, a large component of the signal will be reflected before reaching the fluid filled capillary, thereby useless for conductivity detection.

Also, it appears that coaxial cables show better behavior than microstrips and coplanar waveguides in the frequency range we tested. Using microstrips or coplanar waveguides can introduce extra parasitic capacitance, and at some frequencies the conductors may exhibit antenna-like behavior and cause unwanted modes of signal transmission.

Optimizing electrode design and geometry also proved to affect the device sensitivity. In our trials of power transmission with air or water filled capillaries, the best designed electrodes had about a 3 dB improvement in separation between air and water power transmission.

The most important aspect of C^4D design, however, is the electrical shielding. Introducing well designed shielding markedly improved the performance of our devices. Our shielded designs showed 6 dB more separation in air versus water power transmission than unshielded designs, corresponding to a factor of 4 times in power transmission. Shielding seems to have the largest effect in reducing parasitic capacitance and increasing device sensitivity.

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